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Color Masking White Fluorotic Spots by Resin Infiltration and Its Quantitation by Computerized Photographic Analysis: A 12-month Follow-up Study

SA Garg • SM Chavda

Clinical Relevance

Mild to moderate fluorosis spots can be satisfactorily masked by combining a bleaching and resin infiltration technique, quantitation of which can be done by a simple technique of color analysis of photographs using Adobe Photoshop software.

SUMMARY

Objective: To manage three cases of mild to moderate fluorosis by resin infiltration technique and to quantify the tooth color changes by measuring CIE L*a*b* values of digital photographs and calculating ΔE_{00} based on the CIEDE2000 formula using Adobe Photoshop software.

Methods and Materials: Three cases of mild to moderate fluorosis were treated with a combination of bleaching and a resin infiltration technique. CIE L*a*b* values of 18 fluorosed spots were measured from digital photographs of these cases at four different stages—preoperative, postbleaching, postinfiltration and at 12-month follow-up—using Adobe Photoshop software, and ΔE_{00} was calculated based on the CIEDE2000 formula. The ΔE_{00} values of all 18 points obtained at different stages were submitted to statistical analysis (α=0.05)

Results: In all the cases reported, clinically as well as by the photographic color analysis, it was found that the technique masked the lesions, improving the patients' esthetics, which was maintained even at 12-month recall. Statistically significant difference in ΔE_{00} values was present between comparison of all stages ($p<0.001$) except between postinfiltration and the 12-month follow-up stage ($p=0.642$).

Conclusion: A resin infiltration technique helped in the satisfactory management of white spot lesions of fluorosis, which were

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stable even at 12-month follow up. Quantitation of the changes was achieved using Adobe Photoshop software.

INTRODUCTION

Patients often present to dentists with a demand to improve the esthetic appearance of their teeth. Most of them want their teeth to be lightened, but a few also want to darken the white spots present on their teeth.

White spots on teeth can be due either to preeruptive causes, such as fluorosis, traumatic hypomineralization, or molar incisive hypoplasia, or to posteruptive causes, such as demineralization caused by caries and the accumulation of plaque or cementation of orthodontic brackets. Among these, white spot lesions (WSLs) of fluorosis are the most common.[1,2] These are developmental, hypoplastic, and hypomineralized subsurface areas in enamel formed during periods of excessive fluoride exposure during the maturation stage of amelogenesis wherein the matrix protein amelogenin is not completely egressed, impeding complete growth of hydroxyapatite crystals. When fluoride reverts to normal levels, enamel formation also reverts to normal structure. However, the surface enamel is always hypermineralized, as there is a continuous exchange of ions, such as calcium, phosphate, and fluoride, with the oral environment.[3]

The refractive index (RI) of hydroxyapatite is 1.62, that of water/organic content/ethanol is 1.33, and that of air is 1.[4] Healthy enamel is made up of 96% inorganic (hydroxyapatite) and 4% organic content and is almost homogeneous. As most of it is hydroxyapatite; it acts as a single medium, allowing most light to be transmitted through it to the underlying dentin, which absorbs a major portion of it, imparting color to the tooth. However, in fluorotic teeth, the areas of hypomineralized enamel (WSL) are interspersed in normal mineralized enamel. As WSLs have both mineral and organic content, the light is deflected multiple times at their interface, resulting in a scattering of light in multiple directions. Very little to no light reaches the dentin, thus the dentin imparts no color to the area and the lesions appear white clinically. The scattering coefficient of light increases by a power of three[5] with an increase in demineralization, and thus all light is scattered in the well-defined WSLs; however, in diffuse WSLs with no definite borders and less demineralization, some wavelengths of light reach the dentin, giving some color.[4,6]

Various invasive procedures, such as microabrasion, resin composite restorations, veneers, and even full crowns, have been suggested for the management of these WSLs,[1] of which microabrasion was considered to be the minimally invasive technique. It gave satisfactory results for shallow lesions, but for deeper lesions, there may be substantial removal of enamel (360 ± 130 μm when rubbed 20 times with an 18% HCl and pumice mixture)[7] resulting in irregularities or surface defects that may reinvite staining.

In 2009, the concept of resin infiltration was developed in Charite Berlin by two developers—Dr H. Meyer-Luckel and Dr Sebastian Paris—for the treatment of incipient, hypomineralized, porous, opaque white, noncavitated carious lesions.[8] The product is marketed as Icon (DMG America, Englewood, NJ, USA), containing a low-viscosity resin that is infiltrated into porous enamel, replacing air/water, occluding diffusion pathways for acids and dissolved materials, increasing the hardness of enamel, and preventing further progress of caries. The additional advantage is the blending of the white color of the carious lesion with sound adjacent enamel (SAE), as the difference between RIs of air/water and enamel is nearly eliminated by infiltrating resin (RI=1.52). By the same theory, it has been hypothesized that the WSLs of fluorosis, which are also hypomineralized areas, can be managed by the infiltration technique in the most conservative way. Numerous *in vitro* studies[4,6,8] and case reports have been published showing the effectiveness of the technique in the management of hypocalcified lesions of initial caries[9,10] and orthodontic bracket cementation–induced white spots[11,12] as well as those of fluorosis.[13-15] Quantitation of color changes of orthodontic white spots managed by this technique has been reported,[10,11] but none has been reported in WSLs of fluorosis. Moreover, there is limited information about the long-term stability of color masking achieved in these lesions.

Posttherapeutic color difference can be assessed in many ways, such as by visual assessment (photographs or shade tabs), instruments (colorimeter or spectrophotometer), and digital photographic analysis. The results judged by visual assessment of photographs and shade guides are highly subjective and variable. But color assessment done by colorimeter, spectrophotometer as well as the digital photographic analysis done by Adobe Photoshop software provide numeric values, making the method objective, quantitative, reproducible, and analyzable statistically. They all use the color system developed by Commission Internationale de l'Eclair-

age (CIE) involving three parameters to define color: L* (lightness), a* (red/green chromaticity), and b* (yellow/blue chromaticity). It is currently the most popular color system used for dental purposes. However, the use of instruments to measure tooth color *in vivo* is difficult (they are designed to measure flat surfaces), cumbersome, expensive, and not readily available everywhere, whereas digital photographic analysis by image editing software like Adobe Photoshop as described by Bengel[16] is relatively simple, easily available, and cheap. It has been used for the evaluation of color changes of bleached teeth.[16,17]

The purpose of this study was to describe the management of three cases of mild to moderate fluorosis by the resin infiltration technique and to evaluate the color change by visual assessment of photographs and by digital photographic analysis with the help of Adobe Photoshop CS5 software (Adobe Systems Inc, San Jose, CA, USA) using CIE L*a*b* color space by calculating the color difference delta E (ΔE) between healthy and abnormal enamel with the CIEDE2000 formula and comparing values obtained at various stages. ΔE is defined as the Euclidean distance in three-dimensional color space (L*, a*, b*) between two different points.

METHODS AND MATERIALS

Three patients, ages 18, 23, and 24 years, reported to the Government Dental College and Hospital with the esthetic concern of remarkably perceptible white spots and a few light brown spots in maxillary anterior teeth.

The patients' histories revealed them to be born in areas known to be fluoridated, and on clinical examination, the white to light brown stains with intact smooth enamel surfaces were diagnosed as moderate fluorosis, so the patients were included in the study. WSLs in the first two cases were well defined and in the third case were diffuse. As the dark spots (DS) were also present, bleaching followed by resin infiltration with Icon for all maxillary anterior teeth was planned after approval from the institute's ethical committee.

Clinical Intervention

After prophylactic scaling, in-office bleaching with chemically activated Pola Office Plus (SDI, Victoria, Australia) was done according to the manufacturer's instructions in a single visit. Three applications of gel, each for eight minutes without rinsing (only suctioning in between), were done. As nascent oxygen is released during the bleaching procedure, interfering with the polymerization of resin, a 10-day waiting period was observed before resin infiltration.[18]

The infiltration procedure was carried out following the manufacturer's instructions. Each kit for each patient was comprised of three syringes: 15% hydrochloric acid gel (Icon etch, 0.45 mL), ethanol (Icon dry, 0.45 mL), and resin infiltrant 99% triethylene glycol dimethacrylate (TEGDMA[19] [Icon, infiltrant, 0.45 mL]). Following prophylactic polishing with rubber cups (Kerr Corp, Orange, CA, USA), rubber dam (Dental Dam, Coltene Whaledent, Langenau, Germany) isolation was done. To access the subsurface hypomineralized area, Icon etch (15% hydrochloric acid [HCl]) was applied for 120 seconds, rinsed with distilled water for 30 seconds, and air-dried. Then Icon dry (ethanol) was applied for 30 seconds, which changes the RI of enamel. The whiteness of the lesions should have diminished or disappeared; if it did not, it implied the inaccessibility of the lesion to ethanol and subsequently to the resin. Therefore, the sequence of etching and ethanol application was repeated until the whiteness of the spots disappeared (maximum of three times).[14,20]

Next, resin infiltrant was applied with an applicator tip, allowed to penetrate for three minutes, and polymerized for 40 seconds (500 mW/cm^2, Bluephase C5 light, Ivoclar Vivadent, Schaan, Liechtenstein), followed by a second infiltration for one minute, which was done to compensate for the polymerization shrinkage of the first application. Finishing and polishing were done with fine-graded abrasive flexible discs, finishing strips, and rubber cups (Swiss Flex, Coltene Whaledent).

Digital front-view photographs (Coolpix S7000 camera, Nikon, Shinagawa, Japan; macro-lens F/3.4, with camera settings of focal length of 4 mm, maximum aperture of 3.5, no flash, and auto white balance) were taken at four stages—preoperative, postbleaching, postresin infiltration, and at 12-month follow-up—and assessed visually (Figures 1 through 3) as well as with the image editing Adobe Photoshop software (Figure 4). At every stage, the patient position and the distance of the patient from the lens were maintained. The patients were positioned such that the maxillary central incisors were in the plane of focus. Although it is difficult to completely standardize the ambient light, efforts were made by excluding daylight and keeping 16 light tubes constant throughout the procedures in the room where the examination took place.[21] The teeth were kept wet with saliva and water to prevent

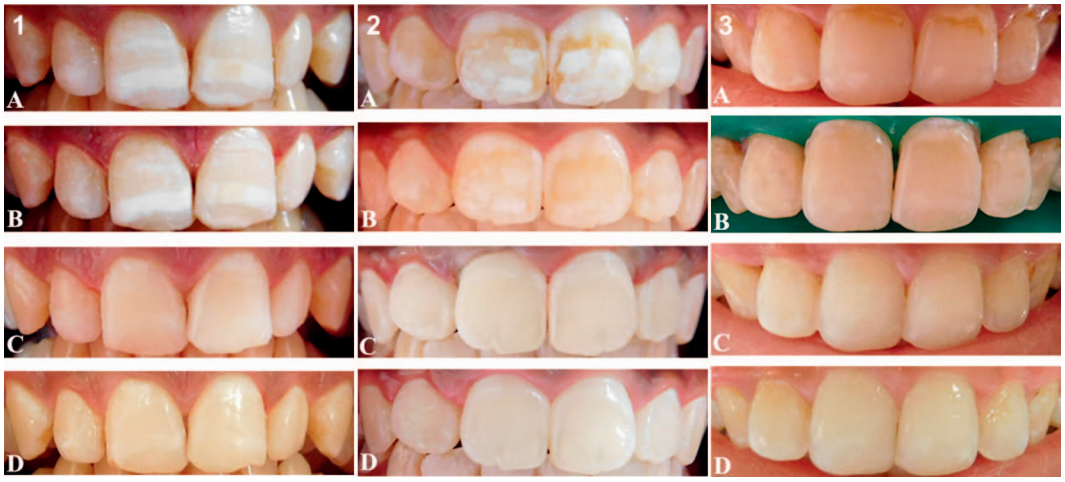


Figure 1. *Case 1. (A): Preoperative. (B): Postbleaching. (C): Immediate postinfiltrative. (D): 12-month follow-up.*

Figure 2. *Case 2. (A): Preoperative. (B): Postbleaching. (C): Immediate postinfiltrative. (D): 12-month follow-up.*

Figure 3. *Case 3. (A): Preoperative. (B): Postbleaching. (C): Immediate postinfiltrative. (D): 12-month follow-up.*

the color alterations caused by the dehydration of teeth. All the clinical procedures, photography, and quantitation of color changes were done by the same operator.

Photographic Analysis

Analysis of images was done by modifying the method described by Bengel.[16] Only central incisors of each case were evaluated. First, the photographs of the case captured at all four stages were opened in Adobe Photoshop CS5 ("Ctrl+O"). "View>show> grid" was used to superimpose a grid on the photographs. "Edit>preferences>guides, grids, and slices>grid line every" was chosen, and values of 10 mm and 5 were input into the "gridline every" and "subdivision," respectively, to change the size of the grid to 2 × 2 mm. Thereafter, the layer panel was made visible (F7 shortcut), and the layers were unlocked by double-clicking the lock symbol to right of the "background." Ctrl + T was selected to open the "resize" dialog box, and keeping the Shift key pressed, the corner of the image was dragged to change the size, and then the right check mark on the right upper corner was clicked to finalize the size. Following the above steps, all the photographs of the same case were resized to the same size taking reference of the superimposed grid. Errors of ambient light standardization and its influence on color can be minimized but cannot be eliminated. Thus, photographs of different stages may vary in lighting conditions, which is eliminated by the use of gray card in various studies[16,17,21] (as described by Bengel[16]). However, as it is not readily available in general dental clinics, it was not used here; instead, the method was modified, and the areas of SAE were compared with WSL/DS in the same photograph where the lighting conditions would have been the same. One point of SAE and three points of fluorosed spots (WSL and DS) were selected on each central incisor, and thus a total of 3 (fluorosed spots) × 6 (central incisors) = 18 abnormal points were evaluated. From the "windows" menu, the "info" tab was selected (F8 shortcut), and the pointer was moved to these points to obtain the x and y coordinates and CIE L*a*b* values. However, variables such as tooth texture, contours, and varying thickness of enamel make CIE L*a*b* values of even SAE different at various points in the same photograph. Hence, to compare ΔE values (color difference between SAE and each WSL/DS) at different stages of treatment, the points selected in the preoperative photographs

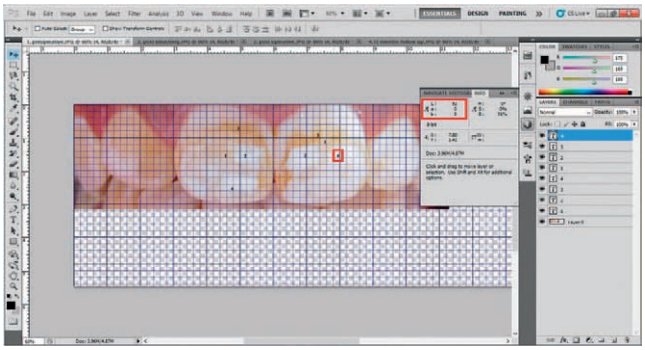


Figure 4. *Screenshot of Adobe Photoshop software showing different points marked in central incisors of case 2. Inset box shows the measured L*, a*, and b* values of point 4 of tooth 9.*

Table 1: *L*, a*, and b* Values Obtained for Central Incisors at Different Stages of Treatment*

Stages	Case 1								Case 2								Case 3							
	Tooth 8				Tooth 9				Tooth 8				Tooth 9				Tooth 8				Tooth 9			
	1 SAE	2 W	3 W	4 W	1 SAE	2 W	3 D	4 W	1 SAE	2 W	3 D	4 W	1 SAE	2 W	3 D	4 W	1 SAE	2 D	3 W	4 D	1 SAE	2 D	3 W	4 D
L*																								
A	74	85	86	83	80	87	83	89	76	86	67	87	82	89	75	92	62	54	71	56	64	63	65	57
B	79	88	91	87	86	89	90	90	82	86	80	89	81	81	79	84	68	65	74	67	70	69	74	67
C	79	78	77	77	82	81	84	81	84	83	82	84	83	84	83	84	74	74	74	74	73	71	74	71
D	84	84	84	85	85	84	87	85	82	82	81	82	87	86	86	89	77	75	75	74	79	80	79	79
a*																								
A	5	0	0	0	1	0	2	0	5	0	8	0	2	0	6	0	18	25	16	24	17	17	16	21
B	5	0	0	1	1	1	2	2	8	5	9	5	9	10	12	7	15	17	11	15	14	12	10	12
C	10	11	10	9	6	7	6	6	3	4	4	3	3	4	3	2	9	9	7	10	9	12	7	10
D	5	4	3	3	3	3	2	2	2	3	3	2	2	2	2	1	3	5	2	7	2	1	1	2
b*																								
A	9	0	0	0	8	0	13	0	14	0	29	0	22	0	40	0	27	43	16	41	24	30	19	37
B	10	0	0	1	9	2	6	4	22	12	23	6	28	24	31	19	21	29	18	27	21	26	17	29
C	18	19	18	19	15	14	16	12	12	12	13	12	11	11	13	11	21	23	19	25	19	22	16	22
D	21	22	20	19	21	20	18	19	11	10	10	10	8	10	9	7	21	24	18	26	19	20	16	22

Abbreviations: SAE, sound adjacent enamel; W, white spot lesion; D, dark spots; A, preoperative; B, postbleaching; C, postinfiltrative; D, 12-month follow-up.

were precisely relocated in the subsequent stage photographs with the reference of x and y coordinates.

ΔE was calculated by the CIEDE2000 formula using an online delta E calculator (http://www.colormine.org/delta-e-calculator/Cie2000).

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C}\right)^2 \left(\frac{\Delta H'}{K_H S_H}\right)^2}$$

Statistical Analysis

ΔE_{00} values at various evaluation periods were compared by a paired t-test at a 95% confidence interval and a 5% significance level using SPSS version 23 software.

RESULTS

Subjective Color Evaluation

From the clinical perspective and visual assessment of photographs, masking of WSLs as well as DS immediately following resin infiltration was achieved in all the cases, which remained stable even at 12-month recall (Figures 1 through 3).

Objective Color Evaluation

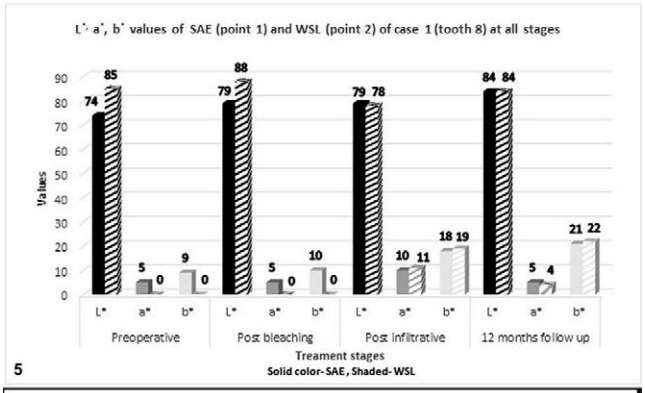
The preoperative, postbleaching, postinfiltrative, and 12-month recall values of all three components of color of the selected spots obtained by the color analysis of images are shown in Table 1. The graphical presentation of comparison of values of one of the WSL with SAE is shown in Figure 5. Table 2 shows the ΔE values of all the spots, and Figure 6 shows the graphical presentation of the changes in ΔE values at various stages.

Table 3 shows the statistical comparison of ΔE values between each stage. A highly statistically significant difference is seen between preoperative and each of the interventional stages (ie, bleaching and postinfiltration stage [$p<0.001$]), indicating an improvement in the appearance of teeth with the treatment, except between the postinfiltration and 12-month follow-up stage ($p=0.642$), which is suggestive of the stability of the result.

DISCUSSION

Various acids, such as 37% phosphoric acid (readily available in a dental office) and 5% HCl, have been tried for removal of the hypermineralized surface layer of enamel (~30 to 50 μm thick), but they were found to etch to a depth of only 7 to 10 μm.[7] Therefore, a stronger acid, such as 15% HCl, is recommended, which, when rubbed for 120 seconds, etches enamel to a depth of about 34.02 μm, and when its application time is increased to four, six, and eight minutes, it etches to a mean depth of 49, 66 and 79 μm, respectively.[22]

For infiltrating the WSLs, various resin adhesives having RIs similar to enamel have been tried, but they have shown limited depth of penetration[23] and



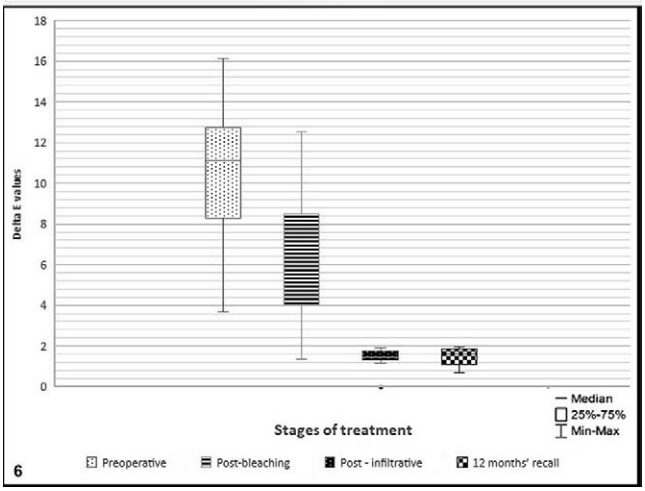


Figure 5. *Graphical representation of comparison of changes in L*, a*, and b* values of SAE (point 1) and WSL (point 2) of case 1(tooth 8) at different stages.*

Figure 6. *Median, interquartile range, and minimum and maximum values of ΔE at various stages of treatment. The high preoperative ΔE values decrease in subsequent stages of treatment.*

have been found to be ineffective.[24] According to Washburn's[25] equation, the penetration coefficient (PC), which describes the penetration of liquids into porous solids, the higher the PC (of light-curing resins) and the longer the application time (within limits), the deeper the penetration in a given porous bed (enamel). The PC of 90% bisphenol A glycidyl methacrylate (BISGMA), 90% diurethane dimethacrylate (UDMA), 100% 2-hydroxyethyl methacrylate (HEMA), and 100% TEGDMA were found to be 0.2, 3.6, 326.8 and 204.1 cm/s, respectively.[26] Although higher PC was observed in 100% HEMA as compared to TEGDMA, the higher content of HEMA resulted in imperfect hardening on curing and therefore was not chosen. Presently, considering all the above factors, TEGDMA seems to be the most optimum resin for infiltration.

A study[19] on the effect of application time has shown that the optimum benefit of penetration could be obtained in three minutes to a depth of 395 to 640 μm. Application time of more than three minutes did not increase the penetration depth, but penetration depth was less when the time was less than three minutes. Therefore, it can also be inferred that only WSLs of approximate depth less than 640 μm can be infiltrated with resins, explaining its inability to mask the deeper ones.

The aim of the treatment is to make the color and value of white spots similar to those of SAE and make ΔE values close to zero. ΔE was calculated using the CIEDE2000 (ΔE_{00}) formula, which provides a higher degree of fit and is an update of the previous formula, adjusting both for the nonuniformity of the CIELAB space and for differences in illuminating conditions.

There is no consensus for the perceptibility and acceptability thresholds of ΔE values. Values as high as 3.7 have been proposed as the acceptance limit in most studies.[21,23,27] But to increase the sensitivity and decrease the probability of missing clinically visible differences, we used the lower values given in a recent study,[28] that is, ΔE_{00} less than 0.8 (clinically perceptible), between 0.8 and 1.8 (clinically perceptible but acceptable), and beyond 1.8 (not acceptable).

Healthy enamel (SAE) has some values for all three components of color: L, a*, and b*. In preoperative photographs, L* values of WSLs in all the cases were more than SAE, the difference being more pronounced in well-defined WSLs (cases 1 and 2) than in the diffuse lesions (case 3); a* and b* values were less in diffuse ones but were zero in well-defined ones (Table 1). ΔE_{00} values of all the spots were in the unacceptable range of 3.70 to 15.49.

After bleaching, there was a generalized lightening of the teeth with an increase in L* values ($p<0.001$). The difference between WSL and SAE also decreased as the striking whiteness of WSL with its surrounding enamel decreased. For the DS, the b* values decreased (decrease in pigment saturation of the spots) but not close to SAE. ΔE_{00} for both decreased but not to an acceptable range. Moreover, the WSLs did not disappear, as their masking requires exactly the opposite effect, namely, a decrease in L* and an increase in b*,[29] which was observed only post resin infiltration (Table 1).

Immediately after infiltration, due to changes in the RI and the transmission of light to the underlying dentin, the whiteness of the WSL disappeared (decrease in L*), and the real tooth color, which was hidden beneath, reappeared (increase in b*). There

Table 2: *ΔE Values (Color Difference Between WSL/DS and SAE)*[a]

	Case 1						Case 2						Case 3					
	Tooth 8			Tooth 9			Tooth 8			Tooth 9			Tooth 8			Tooth 9		
	2 W	3 W	4 W	2 D	3 W	4 W	2 D	3 W	4 W	2 D	3 W	4 W	2 D	3 W	4 D	2 D	3 W	4 D
Preoperative	12.01	12.44	11.22	8.31	4.09	9.09	13.54	8.99	13.88	15.49	8.30	16.14	11.04	11.50	9.83	3.70	4.42	11.87
Post bleaching	11.45	12.54	9.79	5.98	3.79	4.99	6.44	1.36	11.03	2.67	4.1	5.79	6.40	5.51	2.24	4.47	4.87	8.06
Post - infiltrative	1.13	1.41	1.92	1.53	1.50	1.73	1.43	1.90	**0**	1.40	1.33	1.44	1.50	1.55	1.29	1.20	1.90	1.90
12 months' recall	1.48	1.74	1.45	**0.79**	1.85	1.84	1.60	1.74	**0.69**	1.56	0.97	1.12	1.88	1.95	1.06	1.52	1.97	1.61

Abbreviations: W, white spot lesion; D, dark spots.
[a]: White box: $\Delta E_{00} \leq 0.8$, not perceptible clinically; gray box: $\Delta E_{00} \geq 0.8$ and < 1.8, clinically perceptible but acceptable; graphite gray box; black box, $\Delta E_{00} \geq 1.8$, not acceptable. Note all the unacceptable values (black box) turning acceptable (gray box) at the postinfiltrative stage and three unacceptable points (graphite gray box) turning acceptable (gray box) at the 12-month recall.

was an increase in a* values also, but it has little influence on color change.[27,30] The DS also (which are hypomineralized as well) probably got infiltrated with resin and thus merged with the surrounding tooth structure.

Out of the 18 points (11 WSLs and seven DSs) evaluated, ΔE_{00} values for 14 were found in the acceptable range, and the result was found to be highly significant statistically as compared to the preoperative and postbleaching stages ($p<0.001$). Only four points (three WSLs and one DS) had marginally higher values (1.9 [Table 2]), showing a 77% rate of success. TEGDMA in ICON is an unfilled resin having poor abrasion resistance and a higher water sorption rate and was found to have increased surface roughness and subsequent staining after thermocycling and water storage in an *in vitro* study.[31] Despite these unfavorable factors for color stability, the camouflaging effect of infiltration was stable for 13 points at 12-month recall, but one point turned into the unacceptable range with a marginal difference. Additionally, three (two WSLs and one DS) of the four unacceptable points also came down to the acceptable range, showing a success rate of 88% at 12 months and indicating improvement of color over time, which is in agreement with other studies.[1,12,14,15] Moreover, the changes in ΔE_{00} values were nonsignificant ($p=0.642$) as compared to the postinfiltration stage, validating the long-term stability of the result. As the patients were satisfied with the results achieved and felt an increase in their self-esteem, the treatment can be considered to be successful.

CONCLUSION

The resin infiltration technique has opened a new scope for the management of white spots of fluorosis in a minimally invasive manner. Quantitative color assessment by image analysis using Adobe Photoshop is a simple, easy, and promising method of color assessment. Further trials of the method with extended periods of observation and more sample size will reveal more nuances of the method.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the institutional ethics committee of the Government Dental College and Hospital. The approval code for this study is IEC GDCH/CO.3/2017.

Table 3: *Individual Intergroup Comparison of Mean ΔE Values*[a]

Comparisons	Mean	N	Standard Deviation	Standard Error of the Mean	Mean Difference	95% Confidence Interval		*p*-Value
						Lower	Upper	
Pair 1								
Pretreatment	10.33	18	3.64	0.86	4.132	2.212	6.053	<0.001*
Postbleaching	6.19	18	3.22	0.76				
Pair 2								
Pretreatment	10.33	18	3.64	0.86	8.878	7.000	10.756	<0.001*
Postinfiltrative	1.45	18	0.44	0.10				
Pair 3								
Pretreatment	10.33	18	3.64	0.86	8.836	6.966	10.705	<0.001*
12-month follow-up	1.49	18	0.40	0.09				
Pair 4								
Postbleaching	6.19	18	3.22	0.76	4.746	3.057	6.434	<0.001*
Postinfiltrative	1.45	18	0.44	0.10				
Pair 5								
Postbleaching	6.19	18	3.22	0.76	4.703	3.065	6.342	<0.001*
12-month follow-up	1.49	18	0.40	0.09				
Pair 6								
Postinfiltrative	1.45	18	0.44	0.10	−0.042	−0.231	0.146	0.642 NS
12-month follow-up	1.49	18	0.40	0.09				

[a] * highly significant ($p<0.05$); NS, not significant ($p>0.05$).

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Sherwood IA (2010) Fluorosis varied treatment options *Journal of Conservative Dentistry* **13(1)** 47-53.
2. Akpata ES (2014) Therapeutic management of dental fluorosis: A critical review of literature *Saudi Journal of Oral Science* **1(1)** 3-13.
3. Bronckers A, Lyaruu D, & Denbesten P (2009) The impact of fluoride on ameloblasts and the mechanisms of enamel fluorosis *Journal of Dental Research* **88(10)** 877-893.
4. Rocha Gomes Torres C, Borges AB, Torres LM, Gomes IS, & de Oliveira RS (2011) Effect of caries infiltration technique and fluoride therapy on the color masking of white spot lesions *Journal of Dentistry* **39(3)** 202-207.
5. Ko CC, Tantbirojn D, Wang T, & Douglas WH (2000) Optical scattering power for characterization of mineral loss *Journal of Dental Research* **79** 1584-1589.
6. Paris S, Schwendicke F, Keltsch J, Dorfer C, & Meyer H (2013) Masking of white spot lesions by resin infiltration in vitro *Journal of Dentistry* **41(5)** e28-e34.
7. Tong LS, Pang MK, Mok NY, King NM, & Wei SH (1993) The effects of etching, microabrasion, and bleaching on surface enamel *Journal of Dental Research* **72(1)** 67-71.
8. Kugel G, Arsenault P, & Papas A (2009) Treatment modalities for caries management, including a new resin infiltration system. *Compendium of Continuing Education in Dentistry* **30(3)** 1-10.
9. Paris S & Meyer-Lueckel H (2009) Masking of labial enamel white spot lesions by resin infiltration—A clinical report *Quintessence International* **40(9)** 713-718.
10. Martignon S, Ekstrand KR, Gomez J, Lara JS, & Cortes A (2012) Infiltrating/sealing proximal caries lesions: A 3-year randomized clinical trial. *Journal of Dental Research* **91(3)** 288-292.
11. Hammad SM, El Banna M, El Zayat I, & Mohsen MA (2012) Effect of resin infiltration on white spot lesions after debonding orthodontic brackets *American Journal of Dentistry* **25(1)** 3-8.
12. Eckstein A, Helms HJ, & Knösel M (2015) Camouflage effects following resin infiltration of post orthodontic white spot lesions in vivo: One year follow up *Angle Orthodontist* **85(3)** 374-380.
13. Muñoz MA, Arana-Gordillo LA, Gomes GM, Gomes OM, Bombarda NH, Reis A, & Loquerico AD (2013) Alternative esthetic management of fluorosis and hypoplasia stains: Blending effect obtained with resin infiltration techniques *Journal of Esthetic and Restorative Dentistry* **25(1)** 32-39.
14. Tirlet G, Chabouis HF, & Attal JP (2013) Infiltration, a new therapy for masking enamel white spots: A 19-month follow-up case series *European Journal of Esthetic Dentistry* **8(2)** 180-190.

15. Cocco AR, Lund RG, Torre EN, & Martos J (2016) Treatment of fluorosis spots using a resin infiltration technique: 14-month follow-up. *Operative Dentistry* **41(4)** 357-362.
16. Bengel WM (2003) Digital photography and the assessment of therapeutic results after bleaching procedures *Journal of Esthetic and Restorative Dentistry* **15(Supplement 1)** 21-32.
17. Ziebolz D, Helms K, Hanning C, & Attin T (2007) Efficacy and oral side effects of two highly concentrated tray-based bleaching systems *Clinical Oral Investigations* **11(3)** 267-275.
18. Grossman LI (2010) Bleaching of discolored teeth. In: Sureshchandra B, Gopikrishna B (eds) *Grossman's Endodontic Practice* **12th edition** Wolters Kluwer Health, New Delhi 342-360.
19. Meyer-Lueckel H, Chatzidakis A, Naumann M, Dorfer CE, & Paris S (2011) Influence of application time on penetration of an infiltrant into natural enamel caries *Journal of Dentistry* **39(7)** 465-469.
20. Greenwall L (2013) White lesion eradication using resin infiltration *International Dentistry—African Edition* **3(4)** 54-62.
21. Sluzker A, Knosel M, & Athanasiou AE (2011) Sensitivity of digital dental photo CIE L*a*b* analysis compared to spectrophotometer clinical assessments over 6 months. *American Journal of Dentistry* **24** 300-304.
22. Arnold WH, Haddad B, Schaper K, Hagemann K, Lippold C, & Danesh G (2015) Enamel surface alterations after repeated conditioning with HCl *Head and Face Medicine* **11** 32.
23. Oliveira GC, Boteon AP, Ionta FQ, Moretto MJ, Honório HM, Wang L, & Rios D (2015) In vitro effects of resin infiltration on enamel erosion inhibition. *Operative Dentistry* **40(5)** 492-502.
24. De Lacerda AJ, da Silva Ávila DM, Borges AB, & Pucci CR (2016) Adhesive systems as an alternative material for color masking of white spot lesions: Do they work? *Journal of Adhesive Dentistry* **18(1)** 43-50.
25. Meyer-Lueckel H & Paris S (2008) Progression of artificial enamel caries lesions after infiltration with experimental light curing resins *Caries Research* **42** 117-124.
26. Paris S, Meyer-Lueckel H, Cölfen H, & Kielbassa AM (2007) Penetration coefficients of commercially available and experimental composites intended to infiltrate enamel carious lesions *Dental Materials* **23(6)** 742-748.
27. Barry TN, Bailey CW, Ashcraft-Olmscheid D, & Vandewalle KS (2017) Effect of a new bleaching gel on tooth whitening *Operative Dentistry* **42(5)** 559-556.
28. Paravina RD, Ghinea R, Herrera LJ, Bona AD, Igiel C, Linninger M, Sakai M, Takahashi H, Tashkandi E, & Perez Mdel M (2015) Color difference thresholds in dentistry *Journal of Esthetic and Restorative Dentistry* **27(Supplement 1)** S1-S9.
29. Kim Y, Son H-H, Yi K, Kim HY, Ahn J, & Chanq J (2013) The color change in artificial white spot lesions measured using a spectroradiometer. *Clinical Oral Investigations* **17(1)**139-146.
30. Wiegand A, Drebenstedt S, Roos M, Magalhães AC, & Attin T (2008) 12-month color stability of enamel, dentine, and enamel–dentine samples after bleaching *Clinical Oral Investigations* **12(4)** 303-310.
31. Zhao X & Ren YF (2016) Surface properties and color stability of resin-infiltrated enamel lesions *Operative Dentistry* **41(6)** 617-626.

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Underlying Resin Infiltration and Direct Composite Veneers for the Treatment of Severe White Color Alterations of the Enamel: Case Report and 13-Month Follow-Up

C Sekundo • C Frese

Clinical Relevance

The combination of underlying resin infiltration and direct composite veneers presents a minimally invasive alternative for the correction of tooth color and shape in cases of developmental enamel defects, such as severe dental fluorosis in adolescent patients.

SUMMARY

Pronounced white color alterations due to structural anomalies of the enamel are often insufficiently masked by bleaching techniques or resin infiltration procedures alone. This frequently leads to the choice of more invasive prosthetic restorations in order to correct tooth color and form. This article describes a minimally invasive treatment option for esthetic and functional rehabilitation in the case of a 13-year-old female patient with suspected severe fluorosis and misalignment of the anterior teeth. The restorations were performed using underlying resin infiltration to homogenize the tooth shade. In a second step, direct composite veneers were applied on top to attain a natural tooth color and adjust tooth alignment and form. By joining the two minimally and noninvasive techniques, this treatment option combines the directive for preservation of hard tooth structure while treating adolescents with the benefits of easy adaptation and repair when the occlusion is still in adjustment.

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INTRODUCTION

Developmental anomalies of the enamel can have an important effect on dental esthetic appearances. Often, these manifest as white color defects with high opacity, presenting a significant challenge for the clinician attempting to mask these alterations. The most frequent developmental whitish enamel defects include traumatic hypomineralization, mo-

lar-incisor hypomineralization, and fluorosis.[1] Whereas the first two affect only single teeth or groups of teeth, severe cases of dental fluorosis can affect the whole dentition, with discolorations ranging up to an overall parchment-like affect of the tooth surface. Although the underlying mechanisms of dental fluorosis are not yet completely understood, it is recognized that influences on ameloblastic metabolism affect the pre-eruptive mineralization of the enamel, leading to an increase in porosity.[2] The result is a difference in refraction indices, causing a deviation of incoming light rays and thus appearing as white lesions to the observer.[4,5] Posteruptively, further discoloration can take place by the penetration of extrinsic pigments.[6] In severe cases, the extent of the hypomineralized zone leaves only a very thin layer of intact surface enamel, prone to pitting by means of posteruptive trauma. Due to the growing establishment of preventive care in modern dentistry, including the widespread use of fluoride-containing oral hygiene products, an increase in these dental fluorosis manifestations may be expected. There are great variations in the reported prevalence, depending on the time and place of observation. For instance, a European multicenter survey among eight-year-old children ranged the prevalence of smaller diffuse lesions between 28% and 61%, depending on the study site. However, only 0% to 4% of examined children showed severe manifestations.[7]

Consequently, dentists are typically confronted with mild cases of fluorosis, usually manifesting in the form of white spots, lines, or diffuse opacities. Several treatment options have been proposed to mask these discolorations. One approach is bleaching of the affected teeth, aimed at removing any incorporated extrinsic stains from the porosities and adapting the tooth color to minimize the differences and therefore the visibility of the opacities.[8,9] However, bleaching therapy cannot reach full color masking, as the lesions themselves are not altered. Microabrasion, a technique described since 1984,[10,11] using a combination of acidic and abrasive agents to remove the altered enamel, is a slightly more invasive alternative. Yet again, this technique is limited by the depth of the lesion[12] and can result in a substantial reduction of enamel thickness, depending on the number of applications and the materials used.[13,14]

In recent years, resin infiltration of early-stage carious lesions[15-17] has gained popularity as a new microinvasive therapeutic option. The uppermost intact enamel layer must equally be removed by means of hydrochloric acid to gain access to the porosities. However, the altered enamel remains and is infiltrated by a low-viscosity resin, adjusting the refractive index. Furthermore, it has been used in cases of developmentally hypomineralized enamel, improving appearances of tooth color, but not managing to mask extensive lesions.[18,19]

Thus, in cases with more pronounced white color defects, when confronted with an insufficient esthetic outcome after application of the previously mentioned techniques, many practitioners fall back on more invasive treatments. These include veneering or crowning of affected teeth.[8,20-23]

This article proposes a minimally invasive alternative for the esthetic management of severe white color alterations in young patients using a combination of resin infiltration and direct composite veneers.

PATIENT PRESENTATION AND TREATMENT PLAN

A 13-year-old female patient without general diseases presented at the Department of Conservative Dentistry, Heidelberg University Hospital, on referral by her orthodontist, where she was currently being treated for a class II malocclusion with removable braces (class II activator). She was seeking a diagnosis and esthetic rehabilitation due to generalized structural anomalies of the enamel. She also wished for esthetic and functional corrections of the slight deviations in tooth position of the maxillary and mandibular incisors, as her orthodontist did not want to bond fixed braces to the severely altered enamel. In her family history, there had been no similar case. Her medical history further revealed that she had simultaneously received dietary fluoride supplements, prescribed by her pediatrician, as well as fluoridated toothpaste (500 ppm) and fluoridated salt (250 ppm) during early childhood. Moreover, according to the anamnesis, her height and weight during these first years were significantly lower than the average in her age group. No other medication use with risks of altered tooth development could be determined.

Clinical examination showed generalized white and yellowish opacities, encompassing the complete permanent dentition, extending over the whole tooth surface. Pitting of the enamel surface was ubiquitously present but differed in degree. Her maxillary central incisors and first molars were particularly affected. The interplay of anamnesis, distribution, form, and coloration of the structural defects suggested the diagnosis of a TF5 fluorosis (Thylstr-

up-Fejerskov Index[24]). The irregular tooth surface also provided niches for plaque retention, leading to an accompanying generalized gingivitis. The previously mentioned orthodontic diagnoses may also be noted (Figure 1).

The following treatment goals were decided on:

1. Establishment of a harmonious tooth shade and color within the visible area
2. Esthetic correction of the tooth position and form of the anterior teeth
3. Prevention of further loss of hard tooth structures
4. Establishment of sufficient oral hygiene on highly plaque-affected sites

The treatment plan foresaw the following measures:

1. Underlying microinvasive resin infiltration of the facial surfaces of all anterior teeth and premolars in order to achieve a homogenization and removal of the white opaque color
2. Direct composite veneers for the correction of tooth form, position, and final establishment of a natural tooth color
3. Professional teeth cleaning and oral hygiene instructions with close follow-up intervals

TWO-STEP CLINICAL TREATMENT CONCEPT

The correction of form and color was planned as a two-step clinical treatment concept. Due to the extended defects of this patient, the treatment took place in five consecutive sessions. First, an underlying resin infiltration for homogenization of the tooth shade was performed, followed by direct composite veneers for the correction of tooth form, position, and color. Patient counseling included the creation of a diagnostic wax-up to visualize the potential outcome (for illustration, see Figures A.1 and A.2 in the Appendix). Because of the large extent of the measures envisaged, the anterior maxillary teeth were treated first, followed by the anterior mandibular teeth and, finally, all premolars. The two stages are described next.

First Step: Underlying Resin Infiltration

After cleaning of the buccal surfaces, relative isolation of the working field was achieved by use of a lip and cheek retractor (Optragate, Ivoclar Vivadent, Schaan, Liechtenstein), a resinous gingival barrier (OpalDam, Ultradent, Cologne, Germany), and cotton rolls. Afterward, the buccal surfaces were etched with hydrochloric acid for two minutes (Icon Etch, HCl 15%-20%, DMG, Hamburg,

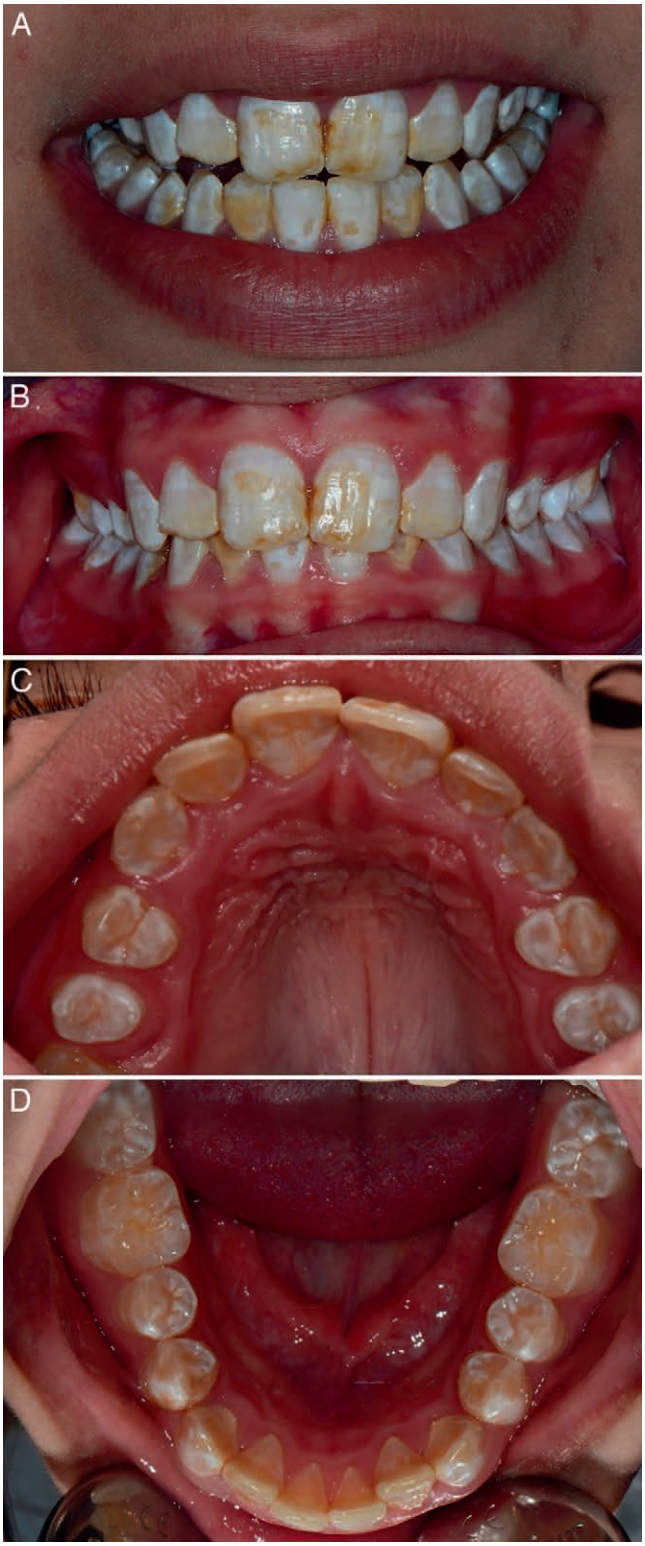

Figure 1. *Preoperative view of the 13-year-old female with a suspected TF5 fluorosis. The dual occlusion due to the current class II malocclusion treatment as well as the anterior misalignment of teeth may also be noted. (A): Facial view during smiling. (B): Facial view in occlusion. (C): Occlusal view of upper jaw. (D): Occlusal view of lower jaw.*

Germany). Rewetting was performed to assess the possible outcome. If the white discoloration did not vanish within a few seconds, etching was repeated until it did. On average, the etching procedure was repeated three or four times. Teeth were then dried with ethanol (Icon Dry, C2H6O<100%, DMG). Thereafter, the resin infiltrant (Icon Infiltrant, DMG) was gently applied for approximately six minutes and excess dispersed with air and then light cured (800 to 1000 mW/cm^2). Another infiltration for approximately one minute followed with subsequent light curing. Finally, surfaces and proximal areas were cleaned. The process is shown for the anterior teeth in Figure 2. In the first step of the treatment concept, a satisfactory homogenization of the tooth shade could be achieved, and the prominent white opaque lesions were removed. Figure 3 illustrates this change in tooth shade for the anterior teeth after full rehydration.

Second Step: Direct Composite Veneers

In the second step, the tooth color, form, and position were corrected by direct composite veneers. The desired tooth shade was chosen in consultation with the patient. For the benefit of minimal invasiveness, no preparation of the teeth was performed. A dry work environment was achieved by usage of the lip and cheek retractor, cotton rolls and intrasulcular retraction cords (Ultrapak, Ultradent). The teeth to be treated were etched, air-dried, and bonded according to standard protocol of total etch technique (Email Preparator, Ivoclar Vivadent; OptiBond FL, Kerr, Biberach, Germany). The composite veneers were created by direct modeling in a multilayer technique. A nanohybrid composite in the colors dentin A2 and enamel A2 (Tetric Evo Ceram, Ivoclar Vivadent) was used. No translucent effect colors were applied in the incisal edge, as a thin basal layer of opaque dentin color was needed to cover the slight remaining inhomogeneities and the color of the resin infiltrant.

The proximal contacts were created using the individual matrix technique according to Hugo[25] and Klaiber.[26] In this technique, a transparent matrix is inserted into the proximal sulcus vertically. A light-curing provisional composite is then injected between the matrix and the adjacent tooth. The matrix is curved toward the adjacent tooth according to the desired proximal contour with a small spatula, simultaneously shaping the provisional composite in the interdental space. Next, the provisional composite is light cured while firmly pressing the matrix against the adjacent tooth in the desired

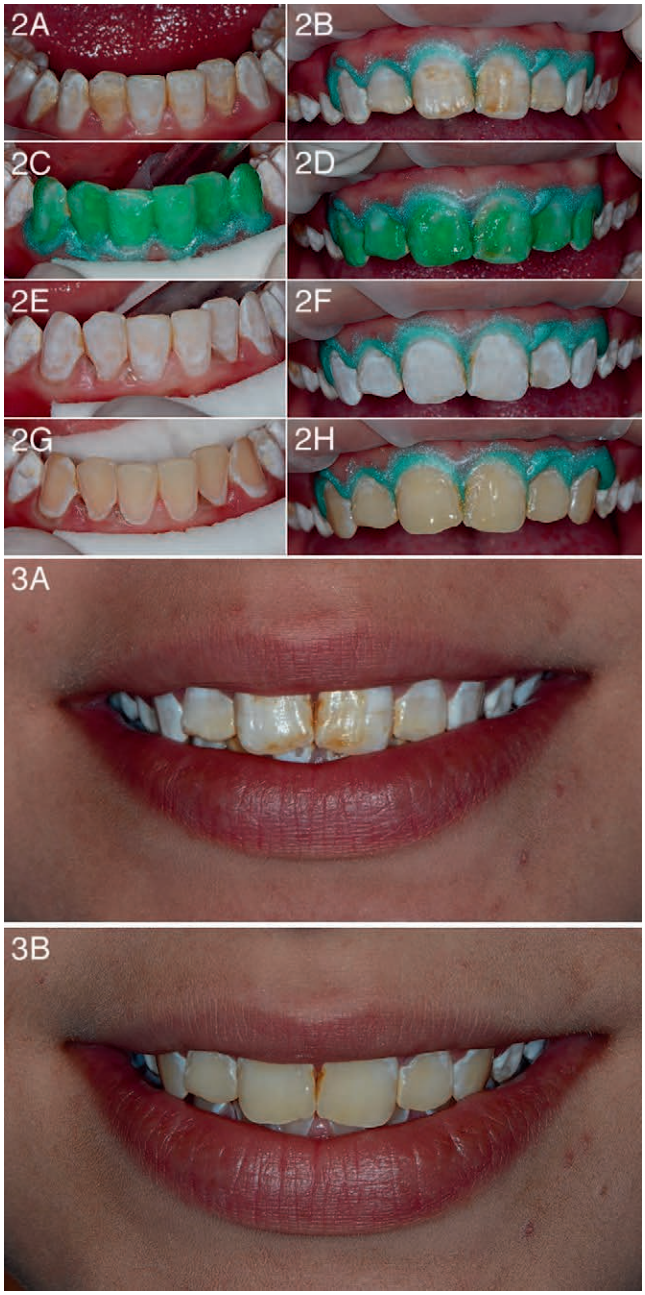

Figure 2. *Resin infiltration of the anterior teeth in the mandible (left side) and maxilla (right side). (A): Preoperative view. (B): Preoperative view after application of the liquid rubber dam. (C, D): Application of the hydrochloric acid. (E, F): Drying of the teeth with ethanol. (G, H): Application of the infiltrant. The liquid rubber dam could not fully withstand the repeated etching with hydrochloric acid. Small chemical burns could be observed at the marginal gingiva. However, these did not cause the patient any pain and healed within a few days. No postoperative sensibilities occurred.*

Figure 3. *Homogenization of tooth color after resin infiltration. (A): Preoperative view. (B): Four-day postoperative result after resin infiltration of the anterior teeth of mandible and maxilla.*

position. This holds the matrix in place, subsequently enabling easy proximal composite placement. For detailed step-by step illustrations, see Klaiber.[26] No proximal contacts were created in the posterior tooth region, as consultation with the attending orthodontist revealed that further extrusion of the teeth was planned as part of the class II malocclusion therapy. Finishing and polishing were performed using a flame-shaped finishing diamond, a sickle-shaped scalpel No. 12 (B. Braun, Melsungen, Germany), Sof-Lex disks (3M ESPE, Neuss, Germany), and flame-shaped polishing rubbers (AstroPol, Ivoclar Vivadent). The preliminary treatment outcome is shown in Figure 4.

During all phases of the treatment and follow-up, professional teeth-cleaning sessions and structured oral hygiene instructions took place repeatedly. Hereby, the regular use of accurately fitting interdental brushes was demonstrated to the patient. After the two-step treatment, a highly esthetic and remarkably less plaque-retentive situation was created, resulting in a considerable improvement in plaque levels and gingival condition (see Appendix Figure A.3). In view of the patient's deficient enamel, there may be an increased risk of excessive attrition. However, as the patient is still undergoing orthodontic treatment, the removable braces serve as protection so that no additional protective means, such as an occlusal splint, were implemented.

Follow-Up Examinations

Due to difficulties scheduling convenient appointments for the patient, the creation of a more pronounced macromorphological relief (ie, the three-dimensional shape of the tooth surface) as well as high-gloss polishing were realized in the course of the four-month follow-up. The shape of the labial surfaces was improved by accentuating the marginal ridges, thus giving the tooth a more natural appearance. The procedure and final result are presented in Figure 5. For further images on composite shaping and polishing, see Appendix Figure A.4.

At 13-month follow-up (Figure 6), all restorations presented intact, and no further abrasive changes to the enamel could be diagnosed. Transitions from tooth to restoration were smooth. The gingival situation presented without any inflammatory signs, and no bleeding on probing occurred. The patient was highly satisfied with the outcome and did not report any complaints. The class II malocclusion therapy is still in place, and further extrusion of the posterior teeth is anticipated. It should also be noted that due to improved oral hygiene and accompanying reduction of the gingival swelling, a small black triangle has appeared between the maxillary central incisors. However, the patient does not feel esthetically impaired by this occurrence.

DISCUSSION

The present case report demonstrated a novel two-step approach of minimally invasive procedures for the esthetic rehabilitation of a 13-year-old patient with suspected severe fluorosis. Here, the use of an underlying resin infiltration for the homogenization of tooth shade was performed before direct composite veneers were applied on top. To the best of our knowledge, such an elaborate procedure for severely affected teeth has not yet been described in literature. It is important to stress that high compliance is necessary when choosing a treatment option with all aspects performed directly on the patient. However, in our case, it showed that the apparent esthetic improvements during the treatment procedures can lead to high intrinsic motivation and equally high patient satisfaction.

Until now, resin infiltration on developmental defects has always been evaluated depending on whether full masking of the structural anomaly was possible. At a certain extent of the enamel hypomineralization, however, full recovery of the dental esthetics cannot be expected. An incomplete disguise is often discussed as a limitation or disadvantage of the infiltration procedure.[1,19,27-29] This case aims to change this judgment by viewing the infiltration process as a pretreatment. Instead of aiming at instant full color masking, the ability of resin infiltration to homogenize the tooth shade can be used as a preparation for further restorations in a new two-step approach.

Every practitioner is aware that discolorations, especially white opacities, can shine through partially translucent restorations, such as ceramic veneers, ceramic crowns, or classical composite shades. The application of an opaque interlayer has been suggested;[30] however, the natural tooth color always comprises a certain translucency.[31] While opaque restorations can achieve a relatively natural outcome in elderly patients, the latter treatment option cannot be recommended for adolescent patients who possess a higher translucency in their tooth shade.[32]

Other proposed solutions usually encompass an augmented layer thickness of the restoration or removal of the discoloration, both resulting in a

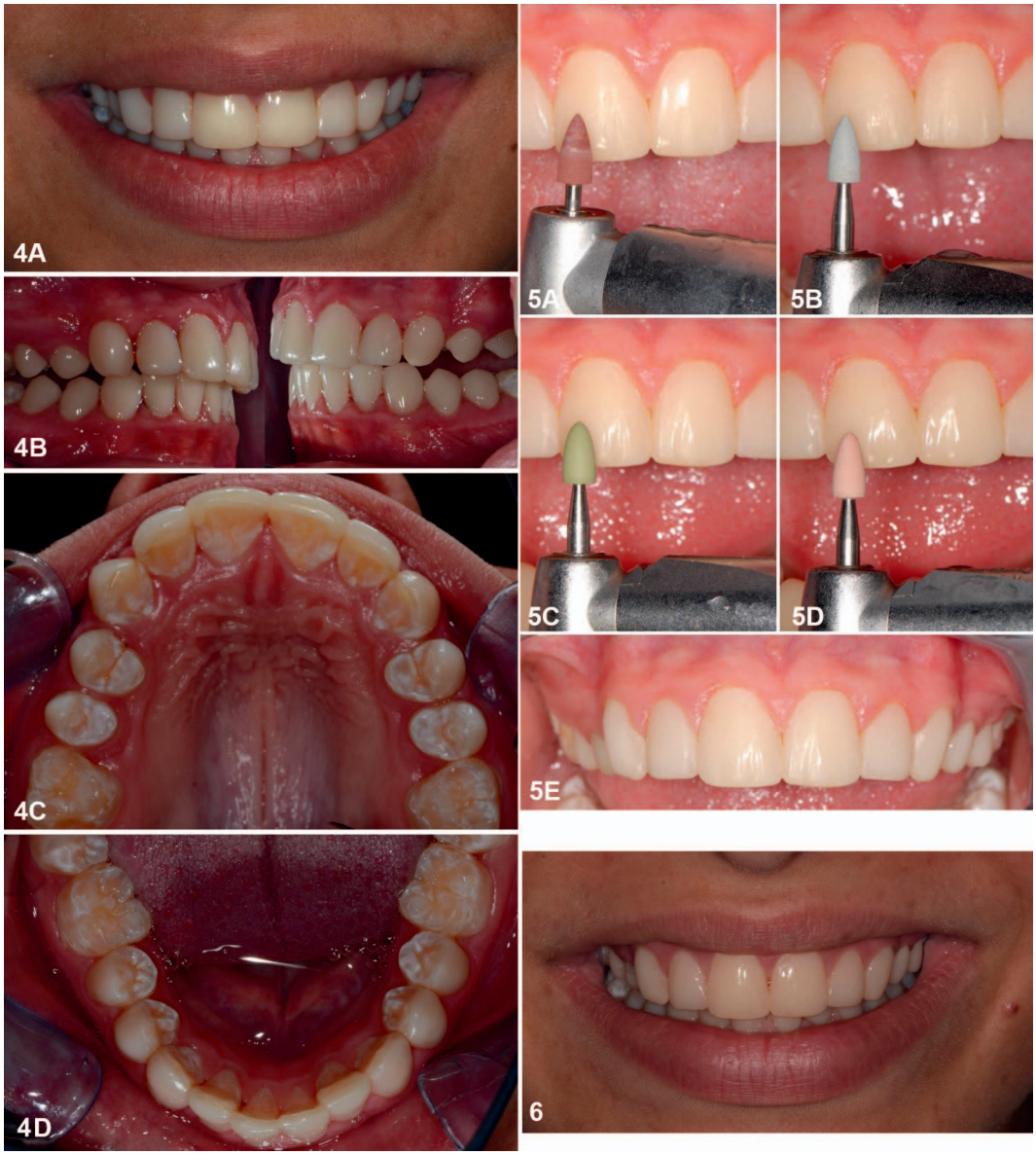

Figure 4. *Preliminary result of the two-step treatment concept with underlying resin infiltration and direct composite veneers at the top. (A): Facial view during smiling. (B): Right and left side. (C): Occlusal view of upper jaw. (D): Occlusal view of lower jaw. Macromorphological shaping of the relief has not yet been realized.*

Figure 5. *Realization of a more pronounced macromorphological relief with flame-shaped polishing rubbers. (A): Macromorphological impressions were first shaped with a brownie. (B-D): A special three-step polishing system (AstroPol, Ivoclar Vivadent) was used for high-gloss polishing. (E): Result after macromorphological shaping and polishing.*

Figure 6. *13-month follow-up.*

greater removal of hard tooth structures. This is particularly critical when treating young patients with extensive pulp chambers and a high risk of preparation trauma. It also leads to an early onset of the "vicious circle" of dental treatment, where the sacrifice of hard tooth structure always results in the necessity of an even more invasive following restoration.[33] Although some enamel is also removed during the (often multiple) etching before resin infiltration, it is nonetheless much less invasive than preparation or even microabrasion, as only the upmost layer above the porous enamel must be removed, whereas the lesion itself can remain.

Moreover, in this patient, indirect restorations in the posterior tooth region were contraindicated, as full tooth eruption had not yet taken place and

placement of indirect restorations would have resulted in their frequent replacement. In contrast, composite presents easy extension and repair possibilities.[34] In addition, direct veneers using adhesive resin composite technology demonstrate good clinical performance rates with a five-year survival rate of 84.6% to 89%.[35-38] Functional survival rates are even higher due to the previously mentioned reparability, as most failure events do not necessitate the restoration's removal but present as chipping fractures or color deterioration and can be restored by polishing or adhesive resin application. Survival rates of porcelain veneers may be even better,[39] but this must be balanced against the advantages of reduced cost and reduced destruction of hard tooth structure of direct composite veneers.

The influence of the infiltrant on adhesive composite restorations has been examined *in vitro* in several studies. The results indicate either no influence or even a beneficial effect on the bond strength.[40-42] Therefore, to our knowledge, there is currently no contraindication for an underlying resin infiltration before reshaping teeth by direct composite veneers or buildups. Successful case reports with less severe baseline conditions have already been published.[43,44] Nonetheless, it must be stressed that no reliable long-term prognosis can be made due to lacking evidence. Long-term studies are necessary to validate this treatment option. Prognosis is also dependent on the patient's oral hygiene and continuing compliance. However, the current restorations do not impede an indirect treatment at a later point in the patient's life if necessary and may then be accomplished with lower risk of iatrogenic pulp trauma and a more stable occlusion.

CONCLUSION

Based on this case report, pronounced white color alterations in adolescent patients can be successfully managed minimally invasively by means of a two-step treatment concept using underlying resin infiltration and direct composite restorations.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Clinic for Oral, Dental, and Maxillofacial Diseases, University Hospital Heidelberg.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Torres CR & Borges AB (2015) Color masking of developmental enamel defects: A case series *Operative Dentistry* **40(1)** 25-33.
2. Aoba T & Fejerskov O (2002) Dental fluorosis: Chemistry and biology *Critical Reviews in Oral Biology and Medicine* **13(2)** 155-170.
3. Fejerskov O, Larsen MJ, Richards A, & Baelum V (1994) Dental tissue effects of fluoride *Advances in Dental Research* **8(1)** 15-31. doi:10.1177/08959374940080010601
4. Limeback H (1994) Enamel formation and the effects of fluoride *Community Dentistry and Oral Epidemiology* **22(3)** 144-147.
5. Tredwin CJ, Scully C, & Bagan-Sebastian JV (2005) Drug-induced disorders of teeth *Journal of Dental Research* **84(7)** 596-602. doi:10.1177/154405910508400703
6. DenBesten PK & Thariani H (1992) Biological mechanisms of fluorosis and level and timing of systemic exposure to fluoride with respect to fluorosis *Journal of Dental Research* **71(5)** 1238-1243. doi:10.1177/00220345920710051701
7. Cochran JA, Ketley CE, Árnadóttir IB, Fernandes B, Koletsi-Kounari H, Oila A-M, Van Loveren C, Whelton HP, & O'Mullane DM (2004) A comparison of the prevalence of fluorosis in 8-year-old children from seven European study sites using a standardized methodology *Community Dentistry and Oral Epidemiology* **32(Supplement 1)** 28-33. doi:10.1111/j.1600-0528.2004.00136.x
8. Akpata ES (2001) Occurrence and management of dental fluorosis *International Dental Journal* **51(5)** 325-333.
9. Bussadori SK, do Rego MA, da Silva PE, Pinto MM, & Pinto AC (2004) Esthetic alternative for fluorosis blemishes with the usage of a dual bleaching system based on hydrogen peroxide at 35% *Journal of Clinical Pediatric Dentistry* **28(2)** 143-146.
10. Croll TP & Cavanaugh RR (1986) Enamel color modification by controlled hydrochloric acid-pumice abrasion. I. Technique and examples *Quintessence International* **17(2)** 81-87.
11. McCloskey RJ (1984) A technique for removal of fluorosis stains *Journal of the American Dental Association* **109(1)** 63-64.
12. Celik EU, Yildiz G, & Yazkan B (2013) Clinical evaluation of enamel microabrasion for the aesthetic management of mild-to-severe dental fluorosis *Journal of Esthetic and Restorative Dentistry* **25(6)** 422-430. doi:10.1111/jerd.12052
13. Pini NI, Sundfeld-Neto D, Aguiar FH, Sundfeld RH, Martins LR, Lovadino JR, & Lima DA (2015) Enamel microabrasion: An overview of clinical and scientific considerations *World Journal of Clinical Cases* **3(1)** 34-41. doi:10.12998/wjcc.v3.i1.34
14. Dalzell DP, Howes RI, & Hubler PM (1995) Microabrasion: Effect of time, number of applications, and pressure on enamel loss *Pediatric Dentistry* **17(3)** 207-211.

15. Knosel M, Eckstein A, & Helms HJ (2013) Durability of esthetic improvement following Icon resin infiltration of multibracket-induced white spot lesions compared with no therapy over 6 months: A single-center, split-mouth, randomized clinical trial *American Journal of Orthodontics and Dentofacial Orthopedics* **144(1)** 86-96. doi:10.1016/j.ajodo. 2013.02.029

16. Feng CH & Chu XY (2013) [Efficacy of one year treatment of icon infiltration resin on post-orthodontic white spots] *Beijing Da Xue Xue Bao Yi Xue Ban* **45(1)** 40-43.

17. Senestraro SV, Crowe JJ, Wang M, Vo A, Huang G, Ferracane J, & Covell DA Jr (2013) Minimally invasive resin infiltration of arrested white-spot lesions: A randomized clinical trial *Journal of the American Dental Association* **144(9)** 997-1005.

18. Marouane O, Douki N, & Chtioui F (2018) A combined approach for the aesthetic management of stained enamel opacities: External bleaching followed by resin infiltration *Case Reports in Dentistry* **2018** 4. doi:10.1155/2018/1605842

19. Crombie F, Manton D, Palamara J, & Reynolds E (2014) Resin infiltration of developmentally hypomineralised enamel *International Journal of Paediatric Dentistry* **24(1)** 51-55.

20. Farid H & Khan FR (2012) Clinical management of severe fluorosis in an adult *BMJ Case Reports* **2012(Dec 10)**. doi:10.1136/bcr-2012-007138

21. El Mourad AM (2018) Aesthetic rehabilitation of a severe dental fluorosis case with ceramic veneers: A step-by-step guide *Case Reports in Dentistry* **2018** 4063165.

22. Jain AR & Hemakumar V (2016) Evaluation of esthetic rehabilitation of teeth with severe fluorosis using direct and indirect laminate veneer: A case report. *Biology and Medicine*. doi:10.4172/0974-8369.1000358

23. Rodd HD & Davidson LE (1997) The aesthetic management of severe dental fluorosis in the young patient *Dental Update* **24(10)** 408-411.

24. Thylstrup A & Fejerskov O (1978) Clinical appearance of dental fluorosis in permanent teeth in relation to histologic changes *Community Dentistry and Oral Epidemiology* **6(6)** 315-328.

25. Hugo B (2001) Optimale Approximalkontakte—Neue approximale Matrizen- und Aufbautechnik bei Frontzahnfüllungen *Ästhetische Zahnmedizin* **3** 241-250.

26. Klaiber B (2006) Alles noninvasiv-Zahnformveränderung, Lückenschluss, Reduktion schwarzer Dreiecke *zm Online* **96(10)** 52-59. https://www.zm-online.de/archiv/2006/10/titel/alles-noninvasiv-zahnformveraenderung-lueckenschluss-reduktion-schwarzer-dreiecke/

27. Mazur M, Westland S, Guerra F, Corridore D, Vichi M, Maruotti A, Nardi GM, & Ottolenghi L (2018) Objective and subjective aesthetic performance of icon treatment for enamel hypomineralization lesions in young adolescents: A retrospective single center study *Journal of Dentistry* **68** 104-108.

28. Borges AB, Caneppele TM, Masterson D, & Maia LC (2017) Is resin infiltration an effective esthetic treatment for enamel development defects and white spot lesions? A systematic review *Journal of Dentistry* **56** 11-18. doi:10.1016/j.jdent. 2016.10.010

29. Kim S, Kim EY, Jeong TS, & Kim JW (2011) The evaluation of resin infiltration for masking labial enamel white spot lesions *International Journal of Paediatric Dentistry* **21(4)** 241-248. doi:10.1111/j.1365-263X.2011.01126.x

30. Sapir S & Shapira J (2007) Clinical solutions for developmental defects of enamel and dentin in children *Pediatric Dentistry* **29(4)** 330-336.

31. Joiner A (2004) Tooth colour: A review of the literature *Journal of Dentistry* **32** 3-12. doi.org/10.1016/j.jdent.2003.10.013

32. Hasegawa A, Ikeda I, & Kawaguchi S (2000) Color and translucency of in vivo natural central incisors *Journal of Prosthetic Dentistry* **83(4)** 418-423. doi.org/10.1016/S0022-3913(00)70036-9

33. Brantley CF, Bader JD, Shugars DA, & Nesbit SP (1995) Does the cycle of rerestoration lead to larger restorations? *Journal of the American Dental Association* **126(10)** 1407-1413.

34. Maneenut C, Sakoolnamarka R, & Tyas MJ (2011) The repair potential of resin composite materials *Dental Materials* **27(2)** e20-e27. doi.org/10.1016/j.dental.2010.09.006

35. Frese C, Schiller P, Staehle HJ, & Wolff D (2013) Recontouring teeth and closing diastemas with direct composite buildups: A 5-year follow-up *Journal of Dentistry* **41(11)** 979-985. doi:10.1016/j.jdent.2013.08.009

36. Peumans M, Van Meerbeek B, Lambrechts P, & Vanherle G (1997) The 5-year clinical performance of direct composite additions to correct tooth form and position. I. Esthetic qualities *Clinical Oral Investigations* **1(1)** 12-18.

37. Peumans M, Van Meerbeek B, Lambrechts P, & Vanherle G (1997) The 5-year clinical performance of direct composite additions to correct tooth form and position. II. Marginal qualities *Clinical Oral Investigations* **1(1)** 19-26.

38. Gresnigt MM, Kalk W, & Ozcan M (2012) Randomized controlled split-mouth clinical trial of direct laminate veneers with two micro-hybrid resin composites *Journal of Dentistry* **40(9)** 766-775. 10.1016/j.jdent.2012.05.010

39. Morimoto S, Albanesi RB, Sesma N, Agra CM, & Braga MM (2016) Main clinical outcomes of feldspathic porcelain and glass-ceramic laminate veneers: A systematic review and meta-analysis of survival and complication rates *International Journal of Prosthodontics* **29(1)** 38-49. 10.11607/ijp.4315

40. Yetkiner E, Wegehaupt FJ, Attin R, & Attin T (2013) Caries infiltrant combined with conventional adhesives for sealing sound enamel in vitro *Angle Orthodontist* **83(5)** 858-863.

41. Wiegand A, Stawarczyk B, Kolakovic M, Hammerle CH, Attin T, & Schmidlin PR (2011) Adhesive performance of a caries infiltrant on sound and demineralised enamel *Journal of Dentistry* **39(2)** 117-121.

42. Chay PL, Manton DJ, & Palamara JE (2014) The effect of resin infiltration and oxidative pre-treatment on micro-

shear bond strength of resin composite to hypomineralised enamel *International Journal of Paediatric Dentistry* **24(4)** 252-267.

43. Perdigao J, Lam VQ, Burseth BG, & Real C (2017) Masking of enamel fluorosis discolorations and tooth misalignment with a combination of at-home whitening, resin infiltration, and direct composite restorations *Operative Dentistry* **42(4)** 347-356.

44. Giannetti L, Murri Dello Diago A, Corciolani E, & Spinas E (2018) Deep infiltration for the treatment of hypomineralized enamel lesions in a patient with molar incisor hypomineralization: A clinical case *Journal of Biological Regulators and Homeostatic Agents* **32(3)** 751-754.

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Randomized Prospective Clinical Trial of Class II Restorations Using Low-shrinkage Flowable Resin Composite

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Clinical Relevance

Class II restorations using low-shrinkage resin composites showed satisfactory clinical performance after one year.

SUMMARY

Purpose: The aim of this prospective, randomized, split-mouth clinical trial was to evaluate postoperative sensitivity, clinical performance, and interproximal contacts after using different restorative systems.

Methods and Materials: Fifty-three subjects each received three class II restorations according to the restorative systems: conventional resin composite (PA: Peak Universal+Amelogen Plus, Ultradent), low-shrinkage flowable and nanoparticulate resin composites (ABF: Adper Single Bond 2+Filtek Bulk Fill Flow+ Filtek Z350XT, 3M ESPE), and low-shrinkage flowable and microhybrid resin composites (XST: XP Bond+SDR+TPH3, Dentsply). Postoperative sensitivity was assessed at 24 hours, seven days, 90 days, and six months. The clinical performance and interproximal contacts were evaluated at baseline, six months, and one year. Friedman, Wilcoxon, Kruskal-Wallis, and Mann-Whitney tests were used to evaluate postoperative sensitivity and interproximal contacts. The equality test of two proportions and logistic regression analysis were used to assess the clinical performance.

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Results: No statistically significant differences were observed among groups for postoperative sensitivity. The highest spontaneous sensitivity was reported at 24 hours. ABF was the only group that did not present a reduction in cold sensitivity. Color, marginal discoloration, and superficial staining showed differences among the groups. XST did not show superficial staining after one year. No differences were observed among groups in relation to interproximal contacts. XST resulted in the loss of interproximal contact after one year.

Conclusions: Different types of restorative systems do not influence postoperative sensitivity; however, ABF maintained cold sensitivity over time. Marginal discoloration occurred for all groups but occurred earliest for PA. XST presented a reduction of interproximal contact after one year of evaluation.

INTRODUCTION

Resin composites have increased in popularity in the clinical practice of dental professionals. The replacement of amalgam restorations with resin composite has been frequent, especially for esthetic reasons.[1] Despite the development of adhesive and restorative materials, posterior resin composite restorations still require extreme technical accuracy for their best performance. The use of incremental techniques, combined with photoactivation protocols, makes the restorative procedure relatively time consuming. Nevertheless, this care has been determined as essential for achieving restorative success.[2]

Posterior restorations, especially those involving proximal surfaces, still represent a challenge for the dental professional when re-establishing the contour and interproximal contact to resemble those of natural teeth[3] since the most common restorative failures are observed in this region.[4] Polymerization shrinkage stress is one of the causes of these failures, generating tension at the adhesive interface and compromising the bonding integrity over time.[5] In addition, polymerization stresses make cavity margins more susceptible to leakage and the development of carious lesions.[6]

Therefore, there is a growing tendency to use materials that generate lower stress at the restorative interface[7] while allowing restorations to be performed in a considerably shorter time with the advantage of reducing the technical sensitivity of the operative technique. Low-shrinkage flowable resin composites present a main advantage of minimal shrinkage stress during polymerization, with a high level of flow in the cavity, to facilitate handling of the material.[7] These materials are presented as a restorative alternative for class II cavities because they can quickly replace lost dental tissue by inserting single increments of up to 4 mm thick.[8-10]

Clinical studies with conventional resin composites indicate that up to 30% of patients report some discomfort or pain after receiving a restoration in posterior teeth, which is more common in class II restorations.[11,12] This sensitivity has been attributed to the stresses caused by the contraction of the resinous restorative material, leading to possible failures of marginal sealing,[13,14] which may favor the movement of fluids in the dentin-pulp complex or cause pulp injury.[15]

Currently, there are a few randomized clinical trials in the literature that have evaluated low-shrinkage resin composites with regard to the occurrence of postoperative sensitivity,[16,17] clinical performance of the material,[17-19] and maintenance of interproximal contact.

Thus, the objective of this study was to evaluate postoperative sensitivity, clinical performance, and interproximal contact after the placement of direct resin composite restorations in class II cavities, using three different restorative strategies. The null hypotheses tested were that there would be no difference among the three restorative systems at each evaluation period for the clinical parameters described above and that there would be no differences for the same restorative strategy at the different evaluation periods.

METHODS AND MATERIALS

Study Design

This clinical trial was a prospective, randomized, double-blind (volunteers and examiners), and split-mouth model. It was carried out after gaining approval from the local ethics committee (approval code: 1,235,100), and it was registered (#RBR-3gg3mg) and conducted according to CONSORT guidelines (Figure 1). Three restorative systems were used: conventional resin composite - considered the control group (PA: Peak Universal+Amelogen Plus, Ultradent, South Jordan, UT, USA), low-shrinkage flowable and nanoparticulate resin composites (ABF: Adper Single Bond 2+Filtek Bulk Fill Flow+Filtek Z350XT, 3M ESPE, St Paul, MN, USA), and low-shrinkage flowable and microhybrid resin composites, (XST: XP Bond+SDR+TPH3, Dentsply,

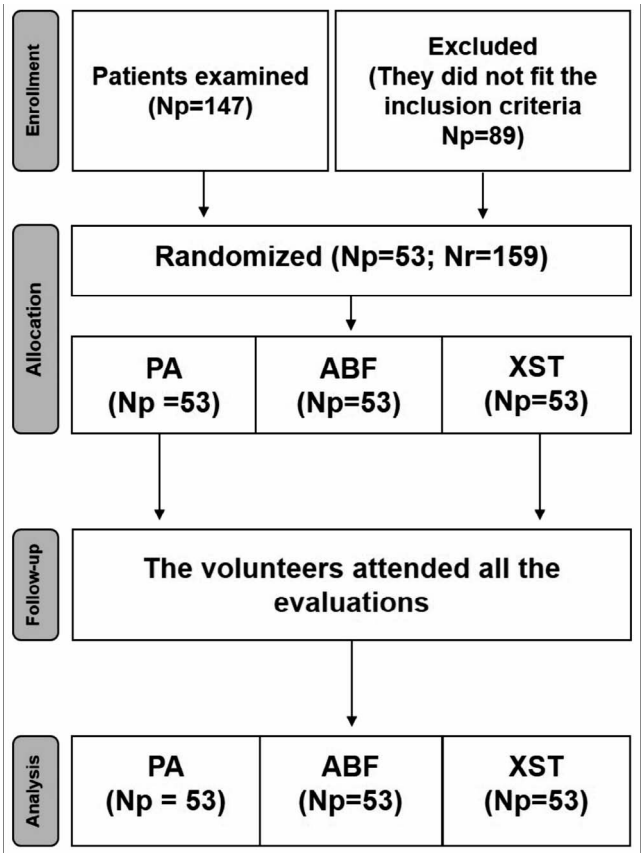

Figure 1. *Flowchart of patients. Np, number of patients; Nr, number of restorations.*

Milford, DE, USA). The two last restorative systems were considered the test groups.

Patient Selection

The sample power for two proportions, when considering 95% success achieved for the control group and 80% for the test group, indicated that an experimental sample with 159 restorations had a high power of 98.3%.

The following inclusion criteria were used: patients presenting at least three unsatisfactory class II restorations that were at least 3 mm deep in a vital permanent premolar or molar of the maxilla or mandible with an adjacent tooth, patients who were at least 18 years old, patients with good periodontal health, and patients with no clinical history of allergies to dental products. The exclusion criteria were the following: pregnant or lactating women, patients receiving orthodontic treatment, a tooth without an antagonist, active and untreated periodontal disease, endodontically treated teeth, dental mobility, history of previous tooth sensitivity, or subjects undergoing any kind of treatment using analgesic or anti-inflammatory drugs.

Fifty-three subjects were selected from the local undergraduate clinic, and patients were submitted to clinical and radiographic examination after signing the informed consent form.

Calibration and Randomization

Two calibrated operators (residents), with clinical experiences of 19 years and one year, were trained by a faculty member specializing in restorative dentistry to perform the restorative procedures. For calibration, each operator performed two restorations for each group from patients who were not selected for the research. The operators were identified on the procedure sheets. Visible plaque index (VPI), gingival bleeding index (GBI), and the decayed, missing, and filled teeth (DMFT) index were assessed at baseline. The subjects then received oral hygiene instructions, and initial photographs were taken.

All subjects received local anesthesia prior to restorative procedures. The randomization was performed by putting numbers in a sealed envelope and drawing which restorative procedure would be performed on each of the selected teeth. Each subject received three restorations, one from each group.

Restorative Procedures

The cavity preparations were performed using spherical diamond burs (#1015-1017, KG Sorensen, Barueri, Brazil) that were replaced after every three procedures. When there was carious tissue, smooth spherical carbide burs (#1/2-4, Dentsply Maillefer, Ballaigues, Switzerland) were also used in a slow speed hand piece.

Prophylaxis with pumice and water was carried out, and rubber dam isolation was performed in all restorations. In order to evaluate the depth of the preparation in mm, a periodontal probe was used (#6 Satin Steel Handle, Hu-Friedy, Chicago, IL, USA) to measure the greatest depth of the preparation. This was done to limit the use of the low-skrinkage resin composites to the maximum thickness of 4-mm depth as well as to preserve 2 mm of space for inserting the covering resin on the occlusal surface. This measurement was performed in all preparations, even in those that would receive the restorations using the incremental technique.

Then 35% phosphoric acid gel (Ultra-Etch, Ultradent) was used for 30 seconds on enamel and 15 seconds on dentin. Thereafter, the preparation was

Table 1: *Products (Material, Manufacturer, Composition, and Mode of Application) Used in This Study*

Group	Material	Manufacturer	Composition	Application
Control PA	Peak Universal	Ultradent (South Jordan, UT, USA)	Ethyl alcohol and 2-hydroxyethyl methacrylate	Dentin was dried, and the adhesive was applied with a microbrush by rubbing on the cavity for 10 s. Adhesive was air-dried for 10 s and photoactivated for 10 s.
	Amelogen Plus		Organic matrix: Bis-GMA, TEGDMA. Filler: silica dioxide and silicate particles (76% wt)	Oblique 2-mm increments were inserted and photoactivated for 20 s. The last increment was photoactivated for 40 s.
Test ABF	Adper Single Bond 2	3M ESPE (St Paul, MN, USA)	Water, ethanol, Bis-GMA, HEMA, UDMA, bisphenol A glycerolate, silica nanofillers treated with acid copolymer, dimethacrylate	Dentin was left slightly moist. The adhesive was applied with a microbrush and air-dried for 5 s. A second layer of the adhesive was applied and air-dried for 5 s. Photoactivation was performed for 20 s.
	Filtek Bulk Fill Flow		Organic matrix: Bis-GMA, Bis-EMA, UDMA, procrylat. Filler: ytterbium trifluoride filler with a range of particle sizes from 0.1 to 5.0 microns and zirconia/silica with a particle size range of 0.01 to 3.5 μm (64.5% wt)	A single increment was inserted in the cavity without submerging the tip of the syringe into the material already dispensed and photoactivated for 40 s. Material was kept 2 mm below the occlusal margin.
	Filtek Z350XT		Organic matrix: Bis-GMA, Bis-EMA, UDMA, and TEGDMA. Filler: agglomerated silica nanofillers and nanoagglomerated zirconia/ silica (78.5% wt)	Oblique increments of up to 2 mm were inserted, finishing the restorations. Each increment was photoactivated for 20 s and the last increment for 40 s.
Test XST	XP Bond2	Dentsply Caulk (Milford, DE, USA)	PENTA, UDMA, dimethacrylate modified by carboxilic acid (TCB resin), triethileneglycol dimethacrylate, hydroxyethylmethacrylate, canphoroquinone, ethyldimethylaminebenzoato, tert-butylhydroquinon, silica, tert-butanol (T-butanol)	Dentin was left slightly moist. One drop of XP Bond was applied with a microbrush, allowed to sit for 20 s, air-dried for 5 s, and photoactivated for 20 s .
	SureFil SDR		Organic matrix: SDR-UDMA, EBPADMA, TEGDMA, CQ, butyl hydroxy toluene; stabilizers UV, titanium dioxide, iron oxide pigments. Filler: barium glass fluoride aluminum silicate, strontium glass (68% wt)	A single increment was inserted using a constant and slow pressure in the deepest part of the cavity, keeping the tip inside the material until an increment of not more than 4 mm was obtained. The material was kept 2 mm below the cavosurface angle for posterior insertion of the universal resin and photoactivated for 40 s.
	TPH3		Organic matrix: Bis-GMA, silica dimethacrylate, EDAB, and others. Filler: silanized barium glass aluminum borosilicate, silanized barium glass, fluoride, aluminum borosilicate (75% wt)	Resin was placed using the incremental technique, and each increment was photoactivated for 20 s. The last increment was photoactivated for 40 s.

*Abbreviations: Bis-GMA, bisphenol A glycydil methacrylate; TEGDMA, triethylene-glycol dimethacrylate; HEMA, hydroxyethyl methacrylate; UDMA, urethane dimethacrylate; Bis-EMA, bisphenol A ethoxylate methacrylate; PENTA, dipentaerythritol*penta *acrylate monophosphate; EBPADMA, bisphenol A ethoxylated dimethacrylate; CQ, camphorquinone; EDAB, ethyl-4-dimethylamino benzoate.*

rinsed with an air/water spray for 10 seconds. Subsequently, adhesive systems and restorative materials were applied, following the recommendations of the respective manufacturers. Table 1 presents the specifications for each group.

To restore the shape of the proximal walls, wooden wedges, preformed metal matrices, and rings (Unimatrix sectional matrix system, TDV Dental Ltda, Pomerode, Brazil) were used. Subsequently, the preparations were restored. In cases where the depth in the proximal box was greater than 6 mm, the low-shrinkage flowable resin composites were inserted in two increments, with the first increment being 4 mm thick. Otherwise, low-shrinkage flowable resin composites were inserted, reserving 2 mm of the restorative depth for the final occlusal resin composite. In order to compare the interproximal contacts of the restorations over time, care was taken to perform restorations so that the point of contact with the adjacent tooth was reconstructed with the bulk-fill resin. Adhesive and resin composites were light cured with a Valo curing light (Ultradent) in the standard application mode and an output of 1000 mW/cm^2.

Table 2: *Modified US Public Health Service Criteria Rating System for Clinical Evaluation of the Restorations*

Retention
Alpha (A): presence of the restoration
Bravo (B): partial retention
Charlie (C): total absence of the restoration
Marginal integrity
Alpha (A): There is no visual evidence of marginal fracture, and the tip of the dental probe is not trapped in the tooth/restoration interface.
Bravo (B): There is visible and tactile evidence of a cleft, but the dentin and/or base is not exposed, nor does the restoration present mobility.
Charlie (C): The dental probe penetrates the tooth/restoration interface, presenting exposed dentin and/or base, but the restoration is not mobile, fractured, or lost.
Marginal discoloration
Alpha (A): There is no visual evidence of marginal discoloration at the tooth/restoration interface.
Bravo (B): There is visual evidence of marginal discoloration at the tooth/restoration interface that can be removed with polishing.
Charlie (C): There is visual evidence of deep marginal discoloration at the tooth/restoration interface that cannot be removed with polishing.
Surface texture
Alpha (A): smooth and shiny, similar to enamel
Bravo (B): slightly rough
Charlie (C): high roughness, not reflective
Wear
Alpha (A): no wear, continuous interface
Bravo (B): discontinuous interface, no exposed dentin
Charlie (C): discontinuous interface, exposed dentin
Secondary caries
Alpha (A): There is no visual evidence of tooth decay at the tooth/restoration interface.
Charlie (C): There is visual evidence of tooth decay at the tooth/restoration interface.
Anatomical form
Alpha (A): The restoration presents continuity with the anatomical form of the existing tooth.
Bravo (B): The restoration has a slight overcontour or undercontour.
Charlie (C): There is loss of restorative material leading to exposure of dentin and/or base.
Surface staining
Alpha (A): absent
Bravo (B): present
Color
Alpha (A): nonapparent interface with the tooth
Bravo (B): subtle visualization between tooth and restoration
Charlie (C): clear visualization between tooth and restoration
Gingival tissue
Alpha (A): no inflammation
Bravo (B): mild inflammation
Charlie (C): severe inflammation

After finishing the restorative procedures, the rubber dam was removed, and occlusal adjustments were made using fine and ultrafine diamond burs (#1190F, 3118F, 1190FF, and 3118FF, KG Sorensen). All restorations were finished using polishing points (Jiffy, Ultradent).

Evaluation

Two independent and calibrated examiners, neither of whom placed the restorations, were responsible for the clinical evaluations. The examiners were kept blind at all assessments.

At 24 hours, seven and 90 days, and six months after finishing the restorative procedures, the subjects were evaluated for postoperative sensitivity. The analysis included spontaneous pain as well as that caused by hot and cold thermal stimuli.

Isolation of the operative field during the evaluations was performed using cotton rolls. The cold test was performed with an anesthetic tube filled with frozen water and by placing the ice in direct contact with the restoration for 15 seconds. The hot thermal test was performed using gutta-percha (Dentsply Maillefer) directly heated by a lamp and placed in direct contact with the restoration for 15 seconds (temperature $<60°C$). Patients received a scale that was used to identify the pain intensity reported according to the modified visual analog scale[20] using scores ranging from 1 to 6: 1, no pain; 2, slight pain; 3, moderate pain; 4, a little worse pain; 5, very bad pain; and 6, the worst pain.

The clinical performance of the restorations was performed through visual and tactile inspection, using a flat dental mirror (SS White, Rio de Janeiro, Brazil) and a periodontal probe (#6 Satin Steel Handle, Hu-Friedy).

After 24 hours, six months, and one year, the restorations were evaluated using the modified US Public Health Service (USPHS) criteria,[21] as described in Table 2. The tightness of the proximal contact was determined based on the resistance to dental floss (Sanifill, São Paulo, Brazil) between the restored surface and the adjacent tooth. The following scores were used: 0, no contact; 1, minimum contact; 2, ideal contact; 3, tight contact; and 4, very tight contact.[22] In cases where more than one proximal surface was involved, the worst score of the two contacts was recorded.

Statistical Methods

The kappa index was used to measure the degree of agreement between the two evaluators. The Fried-

Table 3: *Characteristics of the Cavities and Restorative Procedures*

Variables	Characteristics	n	Groups		
			PA	ABF	XST
Operator	1 (experience of 19 y)	81	27	27	27
	2 (experience of 1 y)	78	26	26	26
Teeth	Maxillary premolar	67	22	23	22
	Maxillary molar	34	11	13	10
	Mandibular premolar	27	7	9	11
	Mandibular molar	31	13	8	10
Restored faces	2	87	30	30	27
	3	67	20	23	24
	4	5	3	0	2
Previous condition	Unsatisfactory amalgam	106	39	35	32
	Unsatisfactory resin composite	52	14	18	20
	Primary caries lesions	1	0	0	1
Deep	3 mm	29	12	9	8
	≥4 mm	61	17	19	25
	≥5 mm	69	24	25	20
Previous dentin	Normal	34	10	15	9
	Sclerotic	125	43	38	44
Anesthesia	Yes	156	52	52	52
	No	3	1	1	1
Restorative time	≤10 min	133	43	45	45
	≤20 min	26	10	8	8
Operator perception	Easy	113	39	38	36
	Medium	38	13	12	13
	Dificult	8	1	3	4

Abbreviations: PA, Peak Universal + Amelogen Plus; ABF, Adper Single Bond 2 + Filtek Bulk Fill Flow + Filtek Z350XT; XST, XP Bond + SDR + TPH3.

man and Wilcoxon tests were used to evaluate postoperative sensitivity and interproximal contacts within each group, and the Kruskal-Wallis and Mann-Whitney tests were used within the same evaluation period. The equality test of two proportions was used to evaluate clinical performance.

Logistic regression analysis was performed to predict the probability of success of the clinical performance results at one year, using the characteristics cited in Table 3. Afterward, the Hosmer-Lemeshow test was performed to evaluate the efficacy of the logistic regression model. "A" and "B" scores were considered because there were only two observations for the "C" score (color and gingival tissue). All tests were performed at a significance level of 0.05%.

RESULTS

The mean age of the 53 subjects was 48.3 years (±10.0), and all participants attended all of the evaluations. The oral health characteristics were (in percentage) VPI, 22.92 ± 20.3; GBI, 14.52 ± 18.8; and DMFT, 22.71 ± 3.91. A total of 65 molars and 94 premolars were restored. The characteristics of the preparations and the restorative procedures are described in Table 3.

There was a statistically significant agreement among the evaluators at the periods analyzed ($p<0.001$), showing that there was an excellent concordance of kappa (baseline=0.79, six months =0.91, one year=0.89).

Data regarding postoperative sensitivity are shown in Table 4. No statistically significant difference was observed at any of the evaluated periods among all groups ($p>0.05$). There was a statistically significant reduction in comparison to spontaneous sensitivity between the periods for all study groups when comparing the initial evaluation to the subsequent periods ($p<0.05$). Regarding cold stimulus, ABF was the only group that did not present a reduction in cold sensitivity over time ($p>0.05$). No statistically significant differences between periods were found for the hot stimulus ($p>0.05$).

Table 4: *Means (Standard Deviation) of the Postoperative Sensitivities Experienced by Patients for All Groups and Evaluation Periods Using a Modified Visual Analog Scale*[a]

	Spontaneous			Cold			Hot		
	PA	ABF	XST	PA	ABF	XST	PA	ABF	XST
24 h	1.62(1.10) Aa	1.49(0.87) Aa	1.51(1.03) Aa	2.57(1.55) Aa	2.51(1.37) Aa	2.39(1.59) Aa	1.55(0.91) Aa	1.62(1.08) Aa	1.51(0.87) Aa
7 d	1.23(0.61) Ab	1.13(0.44) Abc	1.21(0.57) Abc	2.38(1.19) Aab	2.23(1.15) Aa	1.98(1.20) Aab	1.55(0.82) Aa	1.47(0.80) Aa	1.47(0.84) Aa
90 d	1.15(0.4) Ab	1.13(0.44) Abc	1.13(0.44) Abc	2.06(1.35) Abc	2.17(1.39) Aa	1.81(1.11) Ab	1.30(0.54) Aa	1.24(0.51) Aa	1.31(0.61) Aa
6 mo	1.17(0.60) Ab	1.11(0.37) Ac	1.06(0.23) Ac	1.94(1.18) Ac	2.09(1.21) Aa	1.96(1.17) Ab	1.45(0.75) Aa	1.28(0.53) Aa	1.32(0.64) Aa

Abbreviations: PA, Peak Universal + Amelogen Plus; ABF, Adper Single Bond 2 + Filtek Bulk Fill Flow + Filtek Z350XT; XST, XP Bond + SDR + TPH3.
[a] *Uppercase letters compare groups within a same evaluation period (columns), and lowercase letters compare the periods of each group individually (lines), both for each type of postoperative sensitivity separately.*

The data from the USPHS criteria are presented in Table 5. When the analysis among groups was carried out, a statistically significant difference was observed for the following criteria: color, marginal discoloration, and superficial staining. A statistically significant color alteration was observed for PA when compared to ABF and XST at six months and one year. After one year, a low percentage of color alteration was observed for XST, which was statistically different from the other groups. Regarding marginal discoloration, the only difference observed was for ABF at six months, which presented a greater number of restorations with the bravo score when compared to the other groups.

When comparing the evaluation periods for each of the groups, a statistically significant difference was observed for marginal discoloration, marginal integrity, and superficial staining. With regard to marginal discoloration, a statistical difference occurred in both PA and XST after one year when compared to the other periods. ABF presented statistically significant differences in marginal discoloration between the baseline and the subsequent evaluations. Initial degradation of marginal integrity was observed for all groups; PA presented continuous and increased degradation over time, while ABF and XST presented differences only at the one-year follow-up. With regard to superficial staining, PA and ABF presented alterations over time; ABF staining was observed starting at the six-month evaluation period, while PA presented alterations only after one year.

All XST restorations presented an A score for superficial staining at six months, which was statistically different from the other groups. For the one-year evaluation, the highest number of bravo scores for superficial staining was observed for ABF, which was statistically different from the other groups.

The interproximal contacts data are shown in Table 6. When the performance of each group was compared over time, a significant reduction in the intensity of contacts for XST was observed after one year. There was no significant difference among the groups.

The probability of success after one year was observed in the following variables: number of surfaces for the retention, operator for the marginal integrity, sclerotic dentin for marginal discoloration, and mandibular molar for surface texture ($p<0.05$).

DISCUSSION

The present study represents a prospective, randomized, double-blind, split-mouth model, which provides the best evidence for a clinical trial.[23] With the split-mouth design, it is possible to analyze the test and control groups under the same conditions, where the three analyzed groups are present in the same subject, increasing the statistical efficiency and decreasing the amount of patients required for the study.[24] The method used for the analysis of postoperative sensitivity was the visual analog scale, which has been the most frequently used scale for this type of clinical trial.[25] According to the results found in our study, no differences were found among groups for postoperative sensitivity. These results are in accordance with another clinical study in which the sensitivity risk was not affected by the type of adhesive or the restorative strategy used.[16,26]

The highest mean of sensitivity was found during the first 24 hours after the restorative procedure, which leads us to reject the first null hypothesis. The higher intensity of spontaneous sensitivity found in this period may be associated not only with the restorative materials used but also with the trauma generated during the cavity preparation and restorative procedures.[11,16,27]

Table 5: *Clinical Evaluation of Resin Composite Restorations (US Public Health Service). Percentage Values of "A" Score and Numbers of "A," "B," and "C" Scores in Parentheses, Respectively*[a]

Category	Groups	Baseline	6 Mo	1 Y
Retention	PA	100% (53-A/0-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa	94.3% (50-A/2-B/1-C) Aa
	ABF	100% (53-A/0-B/0-C) Aa	100% (53-A/0-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa
	XST	100% (53-A/0-B/0-C) Aa	100% (53-A/0-B/0-C) Aa	96.2% (51-A/2-B/0-C) Aa
Marginal integrity	PA	100% (53-A/0-B/0-C) Aa	92.5% (49-A/4-B/0-C) Ab	71.7% (38-A/15-B/0-C) Ac
	ABF	100% (53-A/0-B/0-C) Aa	94.3% (50-A/3-B/0-C) Aa	73.6% (39-A/14-B/0-C) Ab
	XST	100% (53-A/0-B/0-C) Aa	94.3% (50-A/3-B/0-C) Aab	83.0% (44-A/9-B/0-C) Ab
Marginal discoloration	PA	98.1% (52-A/1-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa	73.6% (39-A/14-B/0-C) Ab
	ABF	98.1% (52-A/1-B/0-C) Aa	83.0% (44-A/9-B/0-C) Bb	73.6% (39-A/14-B/0-C) Ab
	XST	100% (53-A/0-B/0-C)Aa	96.2% (51-A/2-B/0-C) Aa	77.4% (41-A/12-B/0-C) Ab
Surface texture	PA	100% (53-A/0-B/0-C) Aa	100% (53-A/0-B/0-C) Aa	96.2% (51-A/2-B/0-C) Aa
	ABF	100% (53-A/0-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa	94.3% (50-A/3-B/0-C) Aa
	XST	100% (53-A/0-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa	92.5% (49-A/4-B/0-C) Aa
Wear	PA	100% (53-A/0-B/0-C) Aa	100% (53-A/0-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa
	ABF	100% (53-A/0-B/0-C) Aa	100% (53-A/0-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa
	XST	100% (53-A/0-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa
Secondary caries	PA	100% (53-A/0-C) Aa	100% (53-A/0-C) Aa	100% (53-A/0-C) Aa
	ABF	100% (53-A/0-C) Aa	100% (53-A/0-C) Aa	98.1% (52-A/1-C) Aa
	XST	100% (53-A/0-C) Aa	100% (53-A/0-C) Aa	98.1% (52-A/1-C) Aa
Anatomical form	PA	98.1% (52-A/1-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa
	ABF	100% (53-A/0-B/0-C) Aa	100% (53-A/0-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa
	XST	100% (53-A/0-B/0-C) Aa	100% (53-A/0-B/0-C) Aa	100% (53-A/0-B/0-C) Aa
Surface staining	PA	100% (53-A/0-B) Aa	96.2% (51-A/2-B) Bab	84.9% (45-A/8-B) Ab
	ABF	100% (53-A/0-B) Aa	86.8% (46-A/7-B) Bb	66.0% (35-A/18-B) Bc
	XST	100% (53-A/0-B) Aa	100% (53-A/0-B) Aa	94.3% (50-A/3-B) Aa
Color	PA	71.7% (38-A/13-B/2-C) Ba	75.5% (40-A/12-B/1-C) Ba	84.9% (45-A/8-B/0-C) Ba
	ABF	92.5% (49-A/3-B/1-C) Aa	90.6% (48-A/4-B/1-C) Aa	92.5% (49-A/4-B/0-C) Bba
	XST	92.5% (49-A/4-B/0-C) Aa	94.3% (50-A/3-B/0-C) Aa	96.2% (51-A/2-B/0-C) Aa
Gingival tissue	PA	98.1% (52-A/0-B/1-C) Aa	98.1% (52-A/0-B/1-C) Aa	96.2% (51-A/1-B/1-C) Aa
	ABF	96.2% (51-A/2-B/0-C) Aa	98.1% (52-A/1-B/0-C) Aa	96.2% (51-A/2-B/0-C) Aa
	XST	100% (53-A/0-B/0-C) Aa	98.1% (52-A/0-B/1-C) Aa	98.1% (52-A/1-B/0-C) Aa

Abbreviations: PA, Peak Universal + Amelogen Plus; ABF, Adper Single Bond 2 + Filtek Bulk Fill Flow + Filtek Z350XT; XST, XP Bond + SDR + TPH3.
[a] *Uppercase letters compare groups within the same evaluation period (columns),and lowercase letters compare the periods of each group individually (lines). Numbers in parentheses: number of scores present in Table 2.*

Table 6: *Means (Standard Deviation) of the Interproximal Contacts for Groups and Evaluation Periods*[a]

Groups	Evaluation Periods		
	24 H	6 Mo	1 Y
PA	1.92(0.51) Aa	1.87(0.48) Aa	1.79(0.49) Aa
ABF	1.85(0.45) Aa	1.79(0.41) Aa	1.79(0.41) Aa
XST	1.94(0.41) Aa	1.83(0.38) Aa	1.73(0.44) Ab

Abbreviations: PA, Peak Universal + Amelogen Plus; ABF, Adper Single Bond 2 + Filtek Bulk Fill Flow + Filtek Z350XT; XST, XP Bond + SDR + TPH3.
[a] *Uppercase letters compare groups within a same evaluation period (columns), and lowercase letters compare the periods of each group individually (lines).*

When the data obtained for cold sensitivity testing were analyzed, only one restorative system with a low-shrinkage flowable resin composite (ABF) maintained the same intensity of discomfort at all evaluation times. On the other hand, the other restorative system that used a low-shrinkage flowable resin composite (XST) resulted in a reduction of this type of sensitivity after 90 days. These data corroborate the findings of another clinical study that observed low postoperative sensitivity after 30 days in restorations performed with the same low-shrinkage resin used in the present study.[17]

The distinct performance of the two restorative systems containing low-shrinkage flowable resin composites is possibly a result of the different

monomers present in the composition of these resin composites. Although the materials had similar percentages of filler loading (Filtek Bulk Fill Flow 64.5%, SDR 68% by weight), the monomers of Filtek Bulk Fill Flow present a similar structure to conventional resins,[28] while Surefil SDR presents a patented monomer (SDR-UDMA), according to the manufacturer. Furthermore, Surefil SDR showed a significantly higher degree of conversion when compared to Filtek Bulk Fill Flow.[29]

No differences in sensitivity were observed when the hot stimulus was applied since reversible or irreversible pulpal inflammation is required for a response to this test.[30] Other studies in the literature that assessed postoperative sensitivity did not evaluate restorations with the hot stimulus test;[16,17] therefore, it is not possible to compare the results found in this study with other studies.

With regard to the clinical analysis of the restorations, the modified USPHS criteria have been used to evaluate the survival of restorations in several clinical studies,[18,19,31] although there are other criteria for the clinical evaluation of restorations, such as those used by the World Dental Federation.[16] It is worth mentioning that the kappa test revealed excellent agreement among the evaluators. This was possible due to the calibration carried out prior to the evaluations using projected images of the different criteria that would be analyzed.

After the one-year follow-up, the restorations in all groups showed similar clinical performance. In three- and five-year follow-ups, 27.6% and 35.9%, respectively, of class II restorations with minimal marginal discoloration were found when the low-shrinkage Surefil SDR resin composite was used.[18,19] Similar results were obtained in the present study, in which 22.6% of the restorations presented bravo scores for this criterion after one year. These data also show that, up to the one-year evaluation period, the restorative system is not responsible for the degree of marginal discoloration of the restorations.

When considering superficial staining, only the XST restorations remained without a statistical difference after the one-year follow-up, rejecting the second null hypothesis. On the other hand, the beginning of superficial staining of the ABF restorations began at the six-month evaluation, with a greater number of B scores at the last evaluation. The good performance of XST over time may be related to the absence of triethylene-glycol dimethacrylate in the coating resin, leaving this restorative option less susceptible to liquid absorption when compared to the other options evaluated.[32] Additionally, the decreased behavior of ABF when compared to PA may be related to the greater sorption capacity of the nanoparticulated resin in ABF when compared to the microhybrid resin in PA.[33]

When the criterion of marginal integrity was analyzed over time, the teeth that received restorations with the incremental technique presented alterations starting at the six-month evaluation and became worse at the one-year evaluation. On the other hand, the groups that received the low-shrinkage flowable resin composites presented marginal degradation only after one year. In a recent study comparing the same low-shrinkage resin composites used in this study, Surefil SDR and Filtek Bulk Fill Flow showed similar polymerization shrinkage when evaluated using microtomography in class II cavities.[34] These materials differ from conventional resin composites because they have increased polymerization depth, which can be attributed to an increase in translucency and specific monomers. However, the literature is inconsistent in determining the depth of polymerization, although it is reported that these materials are more suitable for narrow preparations with depths greater than 4 mm since they have a greater potential for adaptation.[35] This fact can probably be explained by the low modulus of elasticity of these materials, reducing the stresses generated by the polymerization contraction and, thereby, maintaining the marginal integrity.[36] This information may justify the similarity found among the groups evaluated after one year of clinical evaluation.

In order to evaluate the interproximal contact of the restorations over time using the new low-shrinkage materials, the restorations restored the area of contact with the adjacent tooth. The literature is scarce in clinical work evaluating the intensity of the interproximal contacts of resin composite restorations. Teich and others[22] attempted to standardize the evaluation of the thickness of the contact areas using different brands of dental floss. They carried out a study assigning scores to measure the intensity of the interproximal contacts in natural and restored teeth. The same scores were used in this study to evaluate the interproximal contacts re-established by the restorations using the low-shrinkage resin composites in comparison to the incremental technique.

The current study found no difference among the groups tested, which can be justified by the stan-

dardization of the matrix used since the use of different types of matrices can influence the intensity of the interproximal contact as opposed to the technique or material used.[3] A decrease in the intensity of contact was observed only for XST. However, it should be noted that not all patients had a harmonious and standardized occlusion, and dental movement may have occurred in some cases. Furthermore, a one-year period is not sufficient time to develop variations in most categories. These are potential limitations of this study.

According to the logistic regression analysis, some factors influenced the results of the present study after one year of evaluation. A lower number of surfaces restored increased the retention of restorations, with a smaller interface area being subject to failure. The operator factor influenced the marginal integrity results, where the most experienced operator obtained a higher number of A scores. There are reports in the literature that the most experienced operators influence the success of noncarious class V restorations and restorations using the active release technique.[37,38] Other studies have reported that the clinical success of restorations depends on several factors, including tooth type and location, operator, socioeconomic status, demographics, and behavioral elements.[39] The properties of the material showed a smaller effect on the longevity of the restorations.[39] The presence of sclerotic dentin influenced the present results; it was observed that most of the teeth restored in this study had sclerotic dentin (78.6%), which may have caused a bias in the interpretation of the results.

It is important to emphasize that a new generation of low-shrinkage resin composite has been released in the market, the "full-body" bulk-fill resin composites, which contain a high filler load and are preferable in more extensive restorations.[35] It is believed that clinical trials evaluating this new generation of resin composite are necessary. In addition, a longer evaluation of this study can also elucidate the differences between the restorative systems used.

CONCLUSIONS

The different restorative systems did not influence spontaneous postoperative sensitivity, and after 90 days, there was a reduction of this sensitivity for all study groups. ABF maintained the same degree of cold sensitivity over time. Marginal discoloration was identified earlier for ABF, although marginal discoloration was observed for all study groups after one year. A reduction in interproximal contact was observed for XST after one year of evaluation. Furthermore, other factors should be considered during a clinical evaluation, such as current composite chemistry enhancements and the C-factor.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the ethics committee. The approval code for this study is 1,235,100.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Sunnegårdh-Grönberg K, van Dijken JW, Funegård U, Lindberg A, & Nilsson M (2009) Selection of dental materials and longevity of replaced restorations in Public Dental Health clinics in northern Sweden *Journal of Dentistry* **37(9)** 673-678.
2. Park J, Chang J, Ferracane J, & Lee IB (2008) How should composite be layered to reduce shrinkage stress: Incremental or bulk filling? *Dental Materials* **24(11)** 1501-1505.
3. El-Shamy H, Saber MH, Dörfer CE, El-Badrawy W, & Loomans BAC (2012) Influence of volumetric shrinkage and curing light intensity on proximal contact tightness of class II resin composite restorations: In vitro study *Operative Dentistry* **37(2)** 205-210.
4. Gomes IA, Mariz DC, Borges AH, Tonetto MR, Firoozmand LM, Kuga CM, & Bandeca MC (2015) In vivo evaluation of proximal resin composite restorations performed using three different matrix systems *Journal of Contemporary Dental Practice* **16(8)** 643-647.
5. Moosavi H, Kimyai S, Forghani M, & Khodadadi R (2013) The clinical effectiveness of various adhesive systems: An 18-month evaluation *Operative Dentistry* **38(2)** 134-141.
6. Kwon Y, Ferracane J, & Lee IB (2012) Effect of layering methods, composite type, and flowable liner on the polymerization shrinkage stress of light cured composites *Dental Materials* **28(7)** 801-809.
7. Rosatto CMP, Bicalho AA, Veríssimo C, Bragança GF, Rodrigues MP, Tantbirojn D, & Soares CJ (2015) Mechanical properties, shrinkage stress, cuspal strain and fracture resistance of molars restored with bulk-fill composites and incremental filling technique *Journal of Dentistry* **43(12)** 1519-1528.
8. Czasch P & Ilie N (2013) In vitro comparison of mechanical properties and degree of cure of bulk fill composites *Clinical Oral Investigations* **17(1)** 227-235.
9. El-Damanhoury HM & Platt JA (2014) Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites *Operative Dentistry* **39(4)** 374-382.
10. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, & Leloup G (2015) Physico-mechanical characteristics of

commercially available bulk-fill composites *Journal of Esthetic and Restorative Dentistry* **27(4)** 231-236.

11. Briso ALF, Mestrener SR, Delício G, Sundfeld RH, Bedran-Russo AK, De Alexandre RS, & Ambrosano GM B (2007) Clinical assessment of postoperative sensitivity in posterior composite restorations *Operative Dentistry* **32(5)** 421-426.
12. Hajizadeh H, Ghavamnasiri M, & Majidinia S (2013) Randomized clinical evaluation of the effect of chlorhexidine on postoperative sensitivity of posterior composite resin restorations *Quintessence International* **44(10)** 793-798.
13. Roggendorf MJ, Krämer N, Appelt A, Naumann M, & Frankenberger R (2011) Marginal quality of flowable 4-mm base vs. conventionally layered resin composite *Journal of Dentistry* **39(10)** 643-647.
14. Tantbirojn D, Pfeifer CS, Braga RR, & Versluis A (2011) Do low-shrink composites reduce polymerization shrinkage effects? *Journal of Dental Research* **90(5)** 596-601.
15. Moosavi H, Maleknejad F, Sharifi M, & Ahrari F (2015) A randomized clinical trial of the effect of low-level laser therapy before composite placement on postoperative sensitivity in class V restorations *Lasers in Medical Science* **30(4)** 1245-1249.
16. Costa TRF, Rezende M, Sakamoto A, Bittencourt B, Dalzochio P, Loguercio AD, & Reis A (2017) Influence of adhesive type and placement technique on postoperative sensitivity in posterior composite restorations *Operative Dentistry* **42(2)** 143-154.
17. Hickey D, Sharif O, Janjua F, & Brunton PA (2016) Bulk dentine replacement versus incrementally placed resin composite: A randomised controlled clinical trial *Journal of Dentistry* **46** 18-22.
18. van Dijken JW & Pallesen U (2014) A randomized controlled three year evaluation of "bulk-filled" posterior resin restorations based on stress decreasing resin technology *Dental Materials* **30(9)** e245-e251.
19. van Dijken JW & Pallesen U (2016) Posterior bulk-filled resin composite restorations: A 5-year randomized controlled clinical study *Journal of Dentistry* **51** 29-35.
20. Baker CM & Wong DL (1987) QUEST: A process of pain assessment in children *Orthopaedic Nursing* **6(1)** 11-21.
21. Ryge G (1980) Clinical criteria *International Dental Journal* **30(4)** 347-358.
22. Teich ST, Joseph J, Sartori N, Heima M, & Duarte S (2014) Dental floss selection and its impact on evaluation of interproximal contacts in licensure exams *Journal of Dental Education* **78(6)** 921-926.
23. Pihlstrom BL & Barnett ML (2010) Design, operation, and interpretation of clinical trials *Journal of Dental Research* **89(8)** 759-772.
24. Pandis N (2012) Sample calculation for split-mouth designs *American Journal of Orthodontics and Dentofacial Orthopedics* **141(6)** 818-819.
25. Gillam DG, Aris A, Bulman JS, Newman HN, & Ley F (2002) Dentine hypersensitivity in subjects recruited for clinical trials: Clinical evaluation, prevalence and intra-oral distribution *Journal of Oral Rehabilitation* **29(3)** 226-231.
26. Perdigão J & Swift EJ Jr (2013) Post-op sensitivity with direct composite restorations *Journal of Esthetic and Restorative Dentistry* **25(4)** 284-288.
27. Reis A, Loguercio AD, Schroeder M, Luque-Martinez I, Masterson D, & Maia LC (2015) Does the adhesive strategy influence the post-operative sensitivity in adult patients with posterior resin composite restorations? A systematic review and meta-analysis *Dental Materials* **31(9)** 1052-1067.
28. Alshali RZ, Silikas N, & Satterthwaite JD (2013) Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals *Dental Materials* **29(9)** e213-e217.
29. Lempel E, Czibulya Z, Kovács B, Szalma J, Tóth Á, Kunsági-Máté S, & Böddi K (2016) Degree of conversion and BisGMA, TEGDMA, UDMA elution from flowable bulk fill composites *International Journal of Molecular Sciences* **17(5)** 732.
30. Jafarzadeh H & Abbott PV (2010) Review of pulp sensibility tests. Part I: General information and thermal tests *International Endodontic Journal* **43(9)** 738-762.
31. Pazinatto FB, Gionordoli Neto R, Wang L, Mondelli J, Mondelli RFL, & Navarro MFDL (2012) 56-month clinical performance of class I and II resin composite restorations *Journal of Applied Oral Science* **20(3)** 323-328.
32. Lemos CA, Mauro SJ, Dos Santos PH, Briso AL, & Fagundes TC (2017) Influence of mechanical and chemical degradation in the surface roughness, gloss, and color of microhybrid composites *Journal of Contemporary Dental Practice* **18(4)** 283-288.
33. Rahim TNAT, Mohamad D, Akil HM, & Ab Rahman I (2012) Water sorption characteristics of restorative dental composites immersed in acidic drinks *Dental Materials* **28(6)** e63-e70.
34. Algamaiah H, Sampaio CS, Rigo LC, Janal MN, Giannini M, Bonfante EA, & Hirata R (2017) Microcomputed tomography evaluation of volumetric shrinkage of bulk-fill composites in class II cavities *Journal of Esthetic and Restorative Dentistry* **29(2)** 118-127.
35. van Ende A, De Munck J, Lise DP, & Van Meerbeek B (2017) Bulk-fill composites: A review of the current literature *Journal of Adhesive Dentistry* **19(2)** 95-109.
36. Campos EA, Ardu S, Lefever D, Jassé FF, Bortolotto T, & Krejci I (2014) Marginal adaptation of class II cavities restored with bulk-fill composites *Journal of Dentistry* **42(5)** 575-581.
37. Kemoli A, van Amerongen WE, & Opinya G (2009) Influence of the experience of operator and assistant on the survival rate of proximal ART restorations: Two-year results *European Archives of Paediatric Dentistry* **10(4)** 227-232.
38. Scotti N, Comba A, Gambino A, Manzon E, Breschi L, Paolino D, & Berutti E (2016) Influence of operator experience on non-carious cervical lesion restorations: Clinical evaluation with different adhesive systems *American Journal of Dentistry* **29(1)** 33-38.
39. Demarco FF, Corrêa MB, Cenci MS, Moraes RR, & Opdam N J (2012) Longevity of posterior composite restorations: Not only a matter of materials *Dental Materials* **28(1)** 87-101.

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3D Printing in Dentistry—State of the Art

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Clinical Relevance

3D printing has been found to exhibit properties and performance comparable or superior to those of traditional manufacturing processes. Additive manufacturing has the potential to overcome the disadvantages of the subtractive production method.

SUMMARY

Three-dimensional (3D) printing is a rapidly developing technology that has gained widespread acceptance in dentistry. Compared to conventional (lost-wax technique) and subtractive computer numeric controlled methods, 3D printing offers process engineering advantages. Materials such as plastics, metals, and ceramics can be manufactured using various techniques. 3D printing was introduced over three decades ago. Today, it is experiencing rapid development due to the expiration of many patents and is often described as the key technology of the next industrial revolution. The transition to its clinical application in dentistry is highly dependent on the available materials, which must not only provide the required accuracy but also the necessary biological and physical properties. The aim of this work is to provide an up-to-date overview of the different printing techniques: stereolithography, digital light processing, photopolymer jetting, material jetting, binder jetting, selective laser sintering, selective laser melting, and fused filament fabrication. Additionally, particular attention is paid to the materials used in dentistry and their clinical application.

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INTRODUCTION

The application of computer-aided design (CAD) and computer-aided manufacturing (CAM) in dentistry has progressed strongly over the past few decades. It has led to the development of new classes of materials and to the digitization and automation of various work processes. Until recently, in dentistry, the CAM process was synonymous with the subtractive manufacturing process.

In this process, an object is created out of a blank by milling, grinding, drilling, turning, or polishing using specific tools. From a procedural and ecological point of view, subtractive production has the disadvantage in that the surface resolution is limited by the smallest tool radius. The material loss by computer numeric controlled milling can reach 90%.[1] In addition, the subtractive technique also has a limitation with regard to the number of objects it can produce per machining operation, and it is not

capable of reproducing more complex geometries. Furthermore, the tools used show signs of wear after repeated use, which can lead to cracks in the objects produced.

Alternative ways of producing CAD files are the additive manufacturing processes. All additive manufacturing processes have in common that on the basis of 3D design data, the physical object is built up by the sequential application of thin layers of material. In addition to the term "additive process," the synonyms "generative process," "rapid prototyping," and "3D printing" are often used. With the development of the first CAD programs, the first experiments in the 3D printing sector were carried out from 1980 on. The inventor of the 3D printer, Chuck Hull, took his place in history in 1986 with his patent application for stereolithographic printing. Shortly thereafter, a number of alternative processes were developed.

Legally protected patents on the various additive processes led to high costs and prevented the new technologies from spreading rapidly. With the expiration of important patents a few years ago, commercial and industrial use and further development of the additive processes started at even lower costs.

In contrast to subtractive methods, additive processes can save material and produce more complex geometries. As a result, this manufacturing method is a suitable solution in the dental field. From a process engineering point of view, the additive process has the potential to overcome the disadvantages of the subtractive production method.[2-4]

The basis for 3D printing is a complete description of the surface in a 3D CAD file. The object must be self-contained (watertight) and is usually available in the STL (Stereolithography, Standard Transformation Language, Surface Tessellation Language, or Standard Triangulation Language) file format of the standard interface. The STL format contains the description of the surface of 3D bodies with the help of triangulation (tessellation). Each triangular facet is characterized by its three corner points and the corresponding surface normal of the triangle. Curved surfaces are approximated by polyhedra. Increasing the number of polyhedra minimizes the secant error and describes the object surface with a higher resolution (Figure 1). The STL files can be stored as ASCII files with human-readable source code and much fewer data than binary machine code. Before printing, the CAM software cuts the STL file into

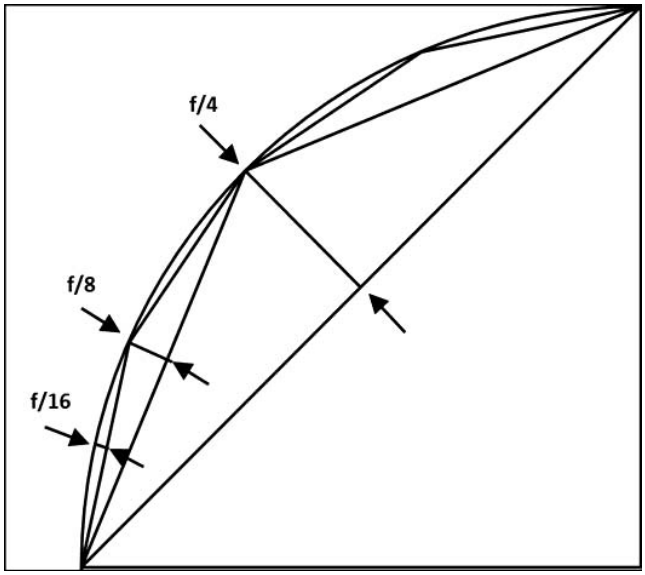

Figure 1. *Secant error when approaching a circle by 4 (f/4), 8 (f/8), or 16 line sections (f/16).*

multiple horizontal layers (xy plane) (slicing). The different slices contain the path information (xy coordinates). The result of slicing is the so-called G code, which contains the machine command for the printer. Thinner film thicknesses are associated with smoother objects but also with a longer printing time. The resolution of the printer is determined by its layer thickness, namely, the z-axis, which represents the vertical accuracy and is one of the essential technical features of any 3D printer. Staircase-shaped gradations of the object are characteristic of additively produced surfaces. They occur most distinctly on planes with low inclination and represent only an approximation of the actual object surface.

The contributions of this technology to general medicine, which began in the 1990s with the production of 3D models, improvements in diagnosis and operation planning, and reduction in surgical risks,[5-9] is now being expanded to many areas of dentistry. In the following, the various additive processes and their use in the dental field are presented.

Stereolithography and Digital Light Processing

Stereolithography (SLA) is the oldest and most commonly used method of 3D printing in dentistry. This technique can be subdivided according to the build platform motion and laser movement. The principle of SLA is based on the layered structure of

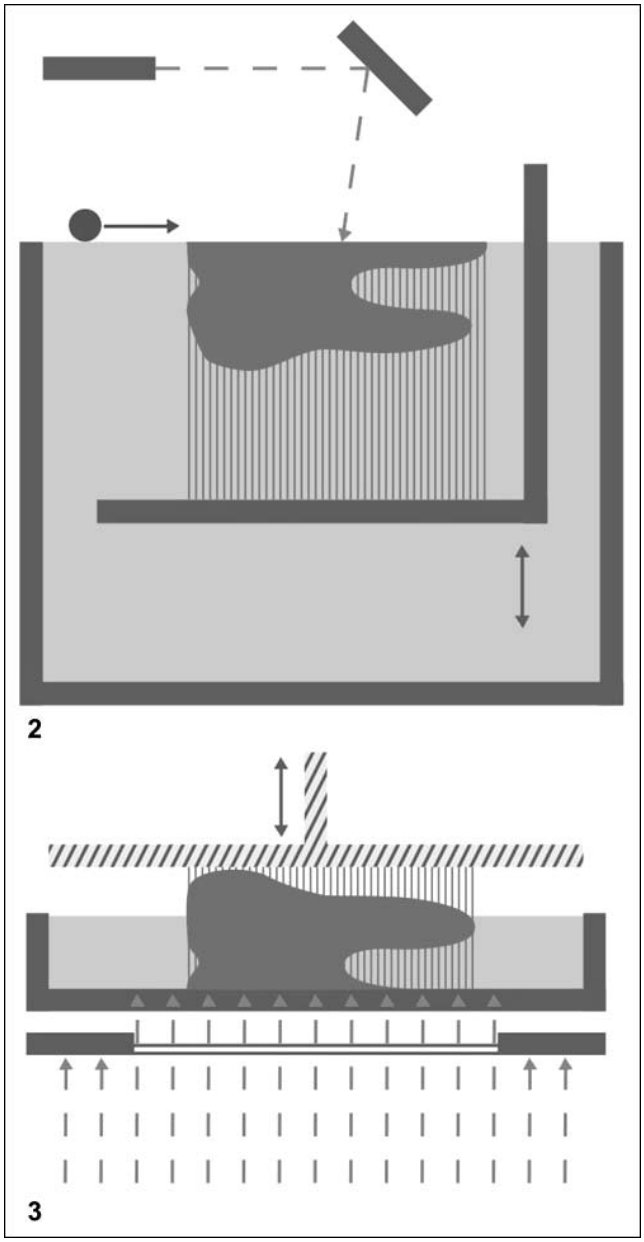

Figure 2. *Stereolithography.*

Figure 3. *Digital light processing.*

an object made of a UV-sensitive liquid monomer, which is polymerized and solidified by a laser.

In the top-down approach, the platform can be lowered vertically and is immersed in a reservoir with liquid monomer near the bottom of the reservoir. A layer of monomer can spread between the platform and the bottom. This monomer layer is exposed to a laser that scans from the bottom of the reservoir. The exposure of the monomer activates the polymerization reaction, which stops locally due to the solidification and limited movement of the free monomers. After each exposure cycle, the build platform is lifted to ensure that the resin flows into the gap between the platform and reservoir. In contrast, the bottom-up approach utilizes a laser that scans from the top of the reservoir. The movable build platform is covered with a thin film of resin and is localized in the resin reservoir (Figure 2). After scanning the first layer with a laser, the build platform moves down, and a roller applies a new layer of uncured resin. The cycle is repeated for each layer until the object is built up. There are many advantages of the top-down approach, so most of the currently available SLA printers operate by this technique. First, the integration of the laser reduces the potential risk to the operator. Second, by curing the resin in the depth of the reservoir, inhibition of the polymerization reaction by oxygen can be prevented. Third, the resin is refilled automatically, and, fourth, the printed layers have a smooth surface due to the contact of the building platform with the bottom of the reservoir.[10]

In addition to the activation of the monomer by a scanning laser in the SLA process, a projection-based SLA technique named digital light processing (DLP) is the second technique that is commonly used (Figure 3). DLP technology contains a microsystem with a rectangular mirror arrangement called a digital micromirror device. The angle of the micromirrors can be individually adjusted, and each one usually has two stable end states. The micromirrors, which act as light switches, project the light from the source as individual pixels onto the projection surface. The resolution of the projected image corresponds to the number of mirrors. The advantage of DLP technology in comparison to the SLA technique is that every layer can be cured with a single shot of laser exposure by producing patterned laser light rather than scanning each area one after the other with the laser. This advantage makes the construction time independent of the respective layer geometry or the number of objects. The resolution can be higher, depending on the system, due to the pixel-based exposure in the DLP method, but neither of the two techniques can be attributed a fundamental superiority over the other. In principle, the printing process in the SLA and DLP techniques can be divided into three discontinuous steps: light exposure, platform moving, and resin refilling, each of which is separate from the other, with no real printing taking place in the last two. A new technology invented by Tumbleston and others,[11] called continuous liquid interface production (CLIP),

addressed this concern, allowing a part to be printed continuously and speeding up the building process. The technology can be combined with SLA or DLP in the bottom-up building approach and takes advantage of the inhibition of radical polymerization by oxygen. An oxygen-permeable membrane is attached to the top layer of the reservoir and is permeable to both UV light and oxygen. Normally, the last printed layer adheres to the surface of the reservoir and causes high peel forces when the building platform is lifted. With CLIP technology, the oxygen-permeable membrane avoids polymerization and adhesion of the bottom layer, which is called the "dead zone." This dead zone is fundamental for continuous printing, as it ensures that a fresh layer of resin is always present below the printed part. The thickness of the dead zone can be controlled by oxygen flux. As a result, the build platform motion can be continuous, and the speed of printing increases. However, CLIP technology also has some disadvantages. First, filled resins can lead to uncontrolled scattering of light at the particles in the dead zone whereby the intensity of the actual laser light reaching the resin may not be enough, potentially compromising print quality. Second, resins differ in their affinity to oxygen, which leads to varying thickness of the dead zones. Thus, the dead zone thickness and oxygen penetration rate are further variables that should be included in the printing settings.[10]

Typically, layer thicknesses between 25 and 100 μm can be converted.[12] A lower layer thickness leads to high-resolution object surfaces but is not conducive to a fast production time. Layer thicknesses of 5 μm along the xy-axis and 10 μm along the z-axis can already be achieved using micro-SLA methods.[13] The layer thickness is influenced by the amount of photoinitiators, the irradiation conditions (wavelength, power, and exposure time), and the temperature of the monomer and absorbent ingredients, such as the pigments. Since most monomers are acrylate based and cannot be activated directly by irradiation, photoinitiators are necessary. Photoinitiators can be classified into two major categories according to the mechanism of free radical generation: α-cleavage and H-abstraction initiators.

α-Cleavage photoinitiators are unimolecular radical generators. During the absorption of UV light, a specific bond within the initiator structure is homolytically cleaved. Both newly produced compounds contain a free radical. Each α-cleavage photo initiator requires UV irradiation within a specific range. Photoinitiators used frequently in dentistry include hydroxyacetophenone, benzoin, benzoin ethers, and phosphine oxides, such as TPO and BAPO.[14-16]

H-abstraction forms the second category of photo initiators. This type of initiator requires a coinitiator, usually an alcohol or amine. Benzophenone is a representative H-abstraction photoinitiator that is commonly used. By absorbing UV irradiation, the photoinitiator enters an excited electron state and abstracts an electron or hydrogen from the coinitiator. The donor molecule then reacts with a monomer to initiate polymerization.[10] Due to the initially short exposure time in 3D printing, 3-5 wt% of photoinitiators are added to the monomers.[12] The initiators should be matched to the light source and have a high molar absorption capacity to achieve high polymerization efficiency and a low curing depth.[12] Depending on the printer, the wavelength of the laser is set at 385 or 405 nm, with new printers tending to use 385 nm. If a 405-nm laser is employed, more initiator is required than that needed for a 385-nm laser due to the absorption spectra of TPO and BAPO. Since the complementary color of the absorbed blue light is yellow, the resins, which are designed for 405 nm, often have an undesirable yellowish tinge. Therefore, for the consumer, it is important to check whether the selected resin can be used with the printer and to ensure that the corresponding printing parameters are stored. By using a pulsed near-infrared laser in combination with two photon initiators, layers up to 300 nm below the light diffraction limit can be generated. However, this approach to the SLA method has thus far been limited to 3D microlabeling.[17-19] By contrast, the iron oxides used as pigments in conventional composites cannot be used in 3D printing resins because of their density, which causes sedimentation to occur. Manufacturers therefore use organic pigments; however, these pigments have the disadvantage of being less stable in the long term than inorganic pigments.

The monomers used for SLA and DLP printers should have a low to moderate viscosity. In SLA-printed structures, brittleness is often observed due to the inhomogeneous cross-linked network resulting from the uneven diffusion of unreacted monomers/oligomers in the vitrification stage or the fast reaction rate in the gelation stage. The most commonly used monomers are methacrylate, epoxy, and functionalized vinyl ether resins. The mechanical properties are limited by the increase in viscosity of the monomers. If fillers are added to increase the mechanical properties (eg, temporary materials or models), then the polarity of the resins,

which is also an important factor, is affected. To achieve a uniform dispersion of fillers, a polarity similar to that of the monomers is advantageous. Therefore, surface modification is often used to adjust the filler-resin polarity.[10] If the viscosity is increased by increasing the content of fillers, gravity will no longer be capable of producing a smooth surface. To ensure a homogeneous dispersion of the fillers, manufacturers advise different mixture methods, such as shaking, stirring, or special roller/tilting stirring devices.

The incorporation of fillers also has considerable effects on the printing accuracy, as the light scattering of the material changes due to the addition of these agents. The accuracy depends significantly on the curing depth, which is described by the Beer-Lambert law:[20]

$$z_p = \delta_p \ln \frac{t_p}{T_c}$$

where z_p is the penetration depth in the z direction, t_p is the time it takes to reach the critical dose for polymerization at depth z_p, T_c is the time it takes to reach the critical dose for curing at depth z_0, and δ_p is the characteristic penetration depth, which is also expressed as $\delta_p = 1/\alpha$:

$$\alpha = \frac{4\pi k}{\lambda}$$

where λ is the wavelength of the laser and k is the extinction coefficient.

The extinction coefficient k depends on the intrinsic properties of the resin, which includes the loading of fillers, the surface of the fillers, and the refractive index of both the fillers and the resin. If there is a mismatch in refractive index between the fillers and the resin, the laser light is scattered significantly, resulting in a reduced polymerization depth. This effect can result in the previous layer not connecting to the next layer and the scattered light curing more resin around the laser beam, resulting in a reduced resolution.[10]

In addition to the layer thickness, the orientation of the objects on the building panel is another variable that can influence the printing result with regard to accuracy and mechanical properties like bending strength.[21] Recent research projects developed slant beam rotation scanning, where a laser can rotate and cure the resin from different angles. In a study of printed total dentures, Alharbi and others[22] found an angle of 135° to the building platform to be the most accurate. Vertically printed temporary materials showed significantly higher compressive strength than horizontally printed materials.[23]

To fix the object to the building platform, support structures are printed with both techniques. The support structures, which are in the form of small columns, also prevent overhanging structures from sinking, which would occur due to a change in density during polymerization. After printing, postprocessing follows. This step includes the removal of excess resin with isopropanol, postpolymerization, and reduction of the residual monomer content using light polymerization boxes and separation of the support structures. Postprocessing can vary greatly depending on the manufacturer. For example, UV-, LED-, or flash-based polymerization devices can be used.

SLA and DLP technology are still limited in the processing of several materials in one construction process. A realization of property gradients is therefore not yet possible. Furthermore, the layer-by-layer technique seems to prevent SLA/DLP-printed objects from achieving the mechanical properties of their monolithic counterparts. Additionally, the object is determined by the "stair-stepping" effect caused by the printer's capability to fabricate only straight layers.

While there are many companies that purchase and relabel printing resins from original equipment manufacturers, an example of the actual number of manufacturers is limited (Table 1). SLA and DLP technology is the most advanced 3D printing technology in dentistry. As a result, a large number of application areas are available. 3D-printed tooth models are widely implemented. In a study by Patzelt and others,[24] the superiority of 3D-printed models (dental casts) over milled models could be observed. Hazefeld and others[25] compared three printing technologies (DLP, photopolymer jetting, and binder jetting) and conventional production with regard to the accuracy of the models. However, they could not determine any superiority of a particular method. 3D-printed patient models are based on scanned surfaces or radiological volume data sets and can be helpful to the dentist for planning. However, they are also very popular in teaching and further education.[26]

The use of printed drilling templates to transfer virtual implant planning into reality has become firmly integrated as a standard fabrication method within navigated implantology and is superior to other procedures, such as milling.[27-31] In total prosthetics, patient-specific prosthetic teeth have

already been successfully printed using DLP technology.[32,33] Furthermore, there is the possibility of printing long-term temporaries with a release of up to 48 months as well as occlusal splints and orthodontic appliances.

Photopolymer Jetting and Material Jetting

In the photopolymer jetting and material jetting processes, the object is built up in layers by a print head with several linear nozzles (Figure 4). The principle is comparable largely to that of a conventional inkjet printer. Instead of ink drops, a liquid photomonomer is used for photopolymer jetting, and wax is used for material jetting. Subsequently, either the monomer is cured in layers by UV light or the wax solidifies thermally on the building platform. Following the same pattern as the other printing processes, the construction platform lowers by one Z gradation after each layer, and the next layer can be applied. This process allows several print heads to work simultaneously. As a result, objects with different materials, colors, and property gradients are possible.[34] The monomers can contain silica nanofillers, which increase the viscosity and improve the controlled application and the mechanical properties of the finished object.[35]

To print overhangs on objects, support material is required in the same way as for the other procedures. The support either is made of a lower melting wax or, conventionally, consists of columns of the actual building material. If wax is used as a support material, it can be melted out by heat in postprocessing.[36] This is called the "hands-free" method and is particularly suitable for sensitive objects. The surface quality of the objects as well as the print resolution is very high in the photopolymer jetting and material jetting processes and does not require any surface finishing with layer thicknesses of less than 20 μm.[12] Similar to SLA and DLP, the photopolymer is vulnerable to sunlight and heat, and the material can creep over time. For printing, photopolymer jetting and material jetting are the most expansive technologies.

Models with high surface quality can be produced using photopolymer jetting and material jetting processes. Braian and others[37] reported in a comparative study in which four photopolymer jetting printers were compared with respect to two model configurations (inlay and bridge), and the accuracy of the models was <100 μm. Another study confirmed a significantly better fit of interim crowns in the proximal, marginal, and internal areas than ground or directly fabricated PMMA interim crowns

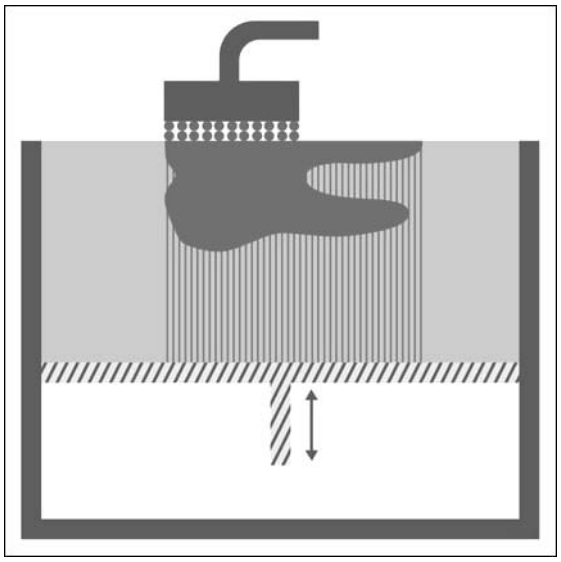

Figure 4. *Photopolymer jetting and material jetting.*

using overimpressions.[38] Metal crowns that were milled conventionally using the lost-wax method and produced using material jetting were also examined. A higher accuracy with regard to the marginal and internal fit of the metal crowns that were made from the printed wax crowns was determined.[39]

Another area of application for the jetting process is the production of prosthetic teeth and implant drilling templates.

Binder Jetting

A variation of the photopolymer jetting process is to apply an adhesive to a powdery substrate using pressure nozzles (Figure 5). After each layer, the building platform descends, and a fresh layer of powder at the level of a Z layer is applied by a blade. Additional support structures are not necessary, as the printed object is completely enclosed by a supporting substrate. If metal and glass powders are used, the object can then be subjected to a sintering process in which the adhesive is burned out. Due to the large adhesive content, the resulting objects exhibit high sinter shrinkage and subsequent porosity and must be subsequently infiltrated. By using several print heads, objects with different colors can be created. Due to the complicated geometries in dentistry, the binder jetting process using powder/adhesive is limited mostly to surgical planning models.

Selective Laser Sintering and Laser Melting

All powdery materials that can be sintered or melted by laser radiation and solidify after cooling can

Table 1: *Overview of Most Common Manufacturers of Stereolithographic and Digital Light Processing Materials*

	Drilling Template	Splint	Orthodontic	Temporary
Deltamed (Friedberg, Germany)	3Delta Guide			3Delta Temp
Detax (Ettlingen, Germany)	Freeprint Ortho Freeprint Splint	Freeprint Ortho Freeprint Splint	Freeprint Ortho	Freeprint Temp
DMG (Hamburg Germany)	Luxaprint Ortho	Luxaprint Ortho Plus	Luxaprint Ortho Plus	
Dreve (Unna, Germany)	FotoDent Guide	FotoDent Splint		
Nextdent (Soesterberg, Netherlands)	NextDent SG	NextDent Ortho Clear, NextDent Ortho Rigid	NextDent Ortho IBT	NextDent MFH, NextDent C&B
Keystone, (Burlington, VT, USA)	KeyGuide	KeySplint Hard, KeySplint Soft		
VOCO (Cuxhaven, Germany)	V Print SG	V Print Ortho		

generally be used for the selective laser sintering or laser melting process. The material spectrum ranges from plastics and metallic materials to ceramic materials. In dentistry, these methods are used mainly for metallic materials.

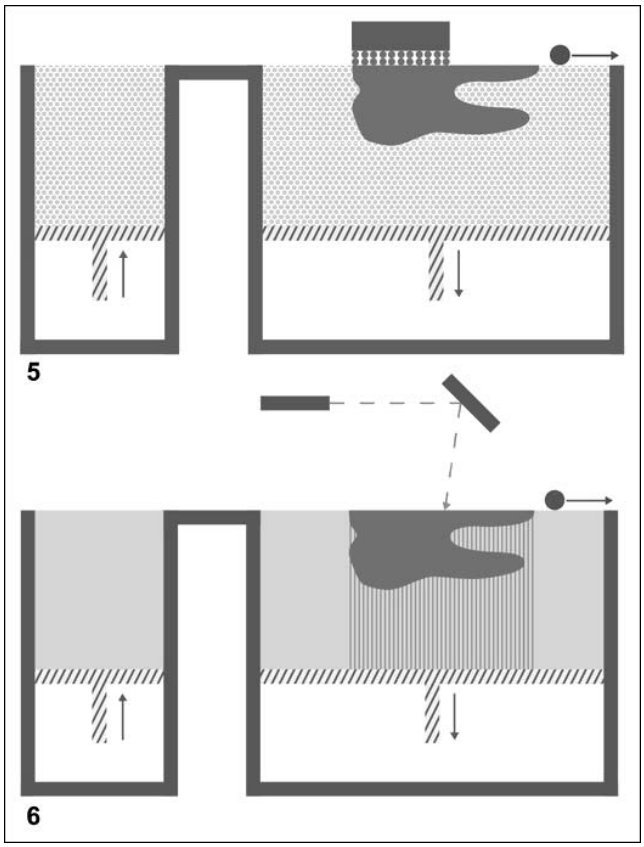

Figure 5. *Binder jetting.*

Figure 6. *Selective laser sintering and laser melting.*

The powder-filled tank is first preheated close to the melting point of the material and above the temperature required for recrystallization during the cooling cycle. Due to the preheating of the powder, the laser requires less energy to fuse or sinter the individual powder particles together, thus avoiding large thermal differences that can otherwise lead to distortion of the objects. High-power CO_2 lasers locally melt or sinter the powder particles two-dimensionally before the installation space is reduced by one-layer thickness after each cycle and a new thin powder layer is applied to the previous layer by a blade (Figure 6). Due to the lack of compression of the particles in the tank, the particle size, shape, and density, as well as the thermal behavior, are decisive factors in the selection of materials. Spherical particles have a lower rolling resistance than irregular particles and can be packed more tightly. Particles that are too small cause processing difficulties due to excessive cohesion or electrostatic repulsion forces.[12] Factors such as the preheating temperature of the powder bed[40] also have an effect on the density of the powder particles.

Because the object is completely enclosed by nonmelted powder, no additional support structure is theoretically required. In practice, however, they have proven their worth, as these structures dissipate heat, reduce internal stress, and decrease distortion of the work piece.[41,42] Since the support structures must be removed in postprocessing, the work piece should be aligned before printing in such a way that the support structures do not lie in the area of the fitting surfaces of the work pieces (eg, partial dentures).[41]

Table 1: *Overview of Most Common Manufacturers of Stereolithographic and Digital Light Processing Materials (ext.)*

Model	Castable	Tray	Denture Base	Gingiva Mask	Others
3Delta Model, 3Delta Model Ortho	3Delta Cast, 3Delta Cast P				
Freeprint model T, Freeprint Model	Freeprint Cast	Freeprint Tray			
Luxaprint Model	Luxaprint Cast	Luxaprint Tray			
FotoDent Model, FotoDent Model2, FotoDent Setup	FotoDent Cast	FotoDent Tray	FotoDent Denture	FotoDent Gingiva	
NextDent Model 2.0, NextDent Model Ortho	NextDent Cast	NextDent Tray	NextDent Denture 3+	NextDent Gingiva Mask	NextDent Try-In
KeyModel, KeyOrthoModel	KeyCast			KeyMask	
V Print Model					V Education

The terms "laser sintering" and "laser melting" are interpreted inconsistently. The two processes are further divided into several subcategories, some of which represent the brand names of certain companies (eg, direct metal laser sintering or laser CUSING). However, the basic printer construction principle is the same. Selective laser sintering is defined as sintering the individual layers of an object, which means that a laser fuses the individual material particles on the surface. Thus, only a partial melting process occurs.

In selective laser melting, however, the material powder is locally melted directly at the processing point. If an electron beam is used instead of a laser, the process is called electron beam melting. It is advantageous to carry out both processes under inert gas. The process is used on a range of metals, alloys, and plastics in dentistry for the fabrication of frameworks, crowns, model casting bases, and models. The most common metals used are Cr-Co and titanium. To achieve a high resolution in the vertical direction with metals, lasers with a power of more than 100 W, a beam diameter of 0.2-0.4 μm, and a resulting layer thickness of 30 μm are used.[42] Printed model casting bases are now comparable with traditionally produced bases.[41] Optimized processes lead to a material density of 99.98% for titanium, but the resulting products have a rough surface because of the size of the powder particles and require finishing.[42]

Metal-free materials include polyamides (Pa6, Pa12, PA10, Pa11, P12, and nylon), polystyrenes, polycarbonates, acrylonitrile-butadiene-styrene, and polyether ether ketone (PEEK), which are increasing in use in dentistry. Since most commercial SLS printers reach a maximum operating temperature of approximately 200°C, they do not allow printing of high-performance polymers such as PEEK, which require temperatures of up to 345°C.[43] Moreover, the current high costs of machines that can print polymers such as PEEK make them inaccessible to the majority of users. Furthermore, the high processing temperatures limit the potential recycling of the non-fused PEEK powder, which increases the production effort and costs. Polyamides are processed with a layer thicknesses of 100 μm and polymer particles of 30-90 μm at a laser power of 20-50 W. The layer thickness along the Z axis thus consists of an average of two to four particles.[12] Initial tests have already been carried out with PMMA printing, but the printing resolution and mechanical properties are still too low for commercial use in dentistry.[44]

Fused Filament Fabrication

The melt layer process was developed over 20 years ago by the founder of Stratasys (Edina, MN, USA) and protected by the trade name "fused deposition modeling." The processes called fused deposition modeling and the nonpatented term "fused filament fabrication" (FFF) work according to the principle of strand extrusion (Figure 7). Thermoplastic materials, such as polylactides, acrylonitrile-butadiene-styrene, and waxes, are supplied as semifinished products in various strand thicknesses to the extruder, where they are melted in the hot end and applied to the building board through a die at the respective xy-coordinate. Heated construction chambers can be used to minimize heat distortion in cases of uneven cooling. After completion of a one-layer plane, the construction panel is lowered onto the z-axis, and the next layer is started. Due to the reduced bonding of

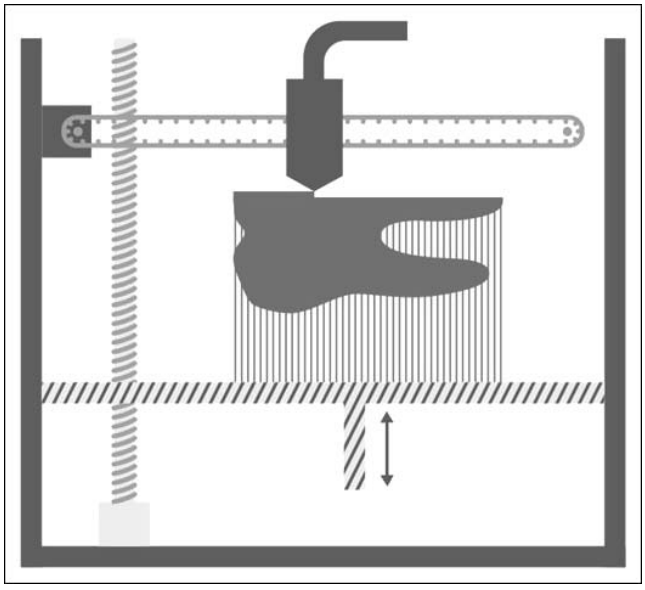

Figure 7. *Fused filament fabrication.*

the individual layers in FFF compared to the SLS and SLA processes, objects with increased anisotropy are created. This results in direction-dependent material properties, thus requiring special attention to be paid to the alignment of the objects before the printing process. If support structures are needed, they can be built up with the same material and then removed or produced from a water-soluble wax using a dual extruder. The surface of the objects is usually more stepped than with other methods due to the layer thicknesses of 200 μm.

The advantage of FFF is its cost efficiency and the lack of restrictions on materials. In general, all materials that can be extruded could be used. In terms of process technology, FFF shows potential superiority over other processes since objects with different material gradients can be produced with several extruders. At present, however, its use in the dental field is very limited. In addition to foam models, individual impression trays have been produced.[45]

Table 2: *Overview of 3D Printing Techniques and Their Most Important Features*

	SLA/DLP	PJ/MJ	BJ	SLS/SLM	FFF
Additive manufacturing process	Photopolymerization	Material jetting	Jetting	Powder bed fusion	Material extrusion
Material	Photopolymer resin	Photopolymer resin	Material in powder consistency (metal, ceramic, plastic)	Material in powder consistency (Co-Cr, titanium, PEEK, polyamides)	Thermoplastic filament (PLA, ABS, TPU, ASA)
Average layer thickness (μm)	25-100	16	50-100	30-100	178 or 254
Average xy resolution (μm)	30-150	42	60-100	200	200-400
Acquisition costs	$-$$	$$$	$$	$$$$	$
Application in dentistry	Model, castable, surgical guide, splint, tray, temporary restauration, gingiva mask, denture	Model, castable, surgical guide	Model	Crowns, implants, partial dentures	Model
Multicolor	No	Yes	Yes	no	Yes
Support structure needed	Yes	Yes	No	no	Yes
Strength	Smooth surface, fine details, most materials	Smoothest surface, fine details, multicolor	Low cost, multicolor, large build volumes, fast build, no support structure	High detail, objects with high density and mechanical properties, no support structure	Low cost, multicolor
Weakness	Only photopolymers, relative brittle materials, vulnerable to sunlight and heat	Only photopolymers, relatively brittle materials, high costs for photopolymer printing, vulnerable to sunlight and heat	Low mechanical properties, low details	Highest cost, special CAD software required, rough surface	Brittle materials, rough surface and low details, anisotropic mechanical properties

Abbreviations: SLA, stereolithography; DLP, digital light processing; PJ, photopolymer jetting; MJ, material jetting; BJ, binder jetting; SLS, selective laser sintering; SLM, selective laser melting; FFF, fused filament fabrication; PLA, polylactides; ABS, acrylonitrile-butadiene-styrene; TPU, Thermoplastic Polyurethane; ASA, Acrylonitrile Styrene Acrylate; CAD, computer-aided design.

CONCLUSIONS

This article intended to provide a practical and scientific overview of the nature, application, advantages, and disadvantages of the different additive procedures in dentistry (Table 2). Various additive processes are on a par with or superior to established manufacturing processes and already offer considerable advantages. Due to the elimination of production restrictions, it is possible to produce dental work on an industrial level, economically and with increased complexity on-site. As an integral part of Industrialization 4.0, we are currently experiencing the beginnings of the additive age. At present, promising processes are developing in parallel with each other; which of these processes will ultimately prevail is still unknown.

Future developments in dentistry must aim at optimizing surface quality and increasing process reliability and property gradients within the materials at lower costs and with shorter production times.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Strub JR, Rekow ED, & Witkowski S (2006) Computer-aided design and fabrication of dental restorations: Current systems and future possibilities *Journal of the American Dental Association* **137(9)** 1289-1296.
2. Wang J & Shaw LL (2006) Fabrication of functionally graded materials via inkjet color printing *Journal of the American Ceramic Society* **89(10)** 3285-3289.
3. Tay B, Evans J, & Edirisinghe M (2003) Solid freeform fabrication of ceramics *International Materials Reviews* **48(6)** 341-370.
4. Noguera R, Lejeune M, & Chartier T (2005) 3D fine scale ceramic components formed by ink-jet prototyping process *Journal of the European Ceramic Society* **25(12)** 2055-2059.
5. Klein HM, Schneider W, Alzen G, Voy ED, & Gunther RW (1992) Pediatric craniofacial surgery: Comparison of milling and stereolithography for 3D model manufacturing *Pediatric Radiology* **22(6)** 458-460.
6. Potamianos P, Amis AA, Forester AJ, McGurk M, & Bircher M (1998) Rapid prototyping for orthopaedic surgery *Proceedings of the Institution of Mechanical Engineers H* **212(5)** 383-393, 10.1243/0954411981534150
7. Petzold R, Zeilhofer HF, & Kalender WA (1999) Rapid protyping technology in medicine—Basics and applications *Computerized Medical Imaging and Graphics* **23(5)** 277-284.
8. Webb PA (2000) A review of rapid prototyping (RP) techniques in the medical and biomedical sector *Journal of Medical Engineering and Technology* **24(4)** 149-153.
9. Swann S (1996) Integration of MRI and stereolithography to build medical models: A case study *Rapid Prototyping Journal* **2(4)** 41-46.
10. Manapat JZ, Chen Q, Ye P, & Advincula RC (2017) 3D printing of polymer nanocomposites via stereolithography *Macromolecular Materials and Engineering* **302(9)** 1600553.
11. Tumbleston JR, Shirvanyants D, Ermoshkin N, Janusziewicz R, Johnson AR, Kelly D, Chen K, Pinschmidt R, Rolland JP, Ermoshkin A, Samulski ET, & DeSimone JM (2015) Additive manufacturing: Continuous liquid interface production of 3D objects *Science* **347(6228)** 1349-1352, 10.1126/science.aaa2397
12. Stansbury JW & Idacavage MJ (2016) 3D printing with polymers: Challenges among expanding options and opportunities *Dental Materials* **32(1)** 54-64, 10.1016/j.dental.2015.09.018
13. Liska R, Schuster M, Inführ R, Turecek C, Fritscher C, Seidl B, Schmidt V, Kuna L, Haase A, & Varga F (2007) Photopolymers for rapid prototyping *Journal of Coatings Technology and Research* **4(4)** 505-510.
14. Lecamp L, Youssef B, Bunel C, & Lebaudy P (1997) Photoinitiated polymerization of a dimethacrylate oligomer: 1. Influence of photoinitiator concentration, temperature and light intensity *Polymer* **38(25)** 6089-6096.
15. Williams RM, Khudyakov IV, Purvis MB, Overton BJ, & Turro NJ (2000) Direct and sensitized photolysis of phosphine oxide polymerization photoinitiators in the presence and absence of a model acrylate monomer: A time resolved EPR, cure monitor, and PhotoDSC study *Journal of Physical Chemistry B* **104(44)** 10437-10443.
16. Schneider LF, Cavalcante LM, Prahl SA, Pfeifer CS, & Ferracane JL (2012) Curing efficiency of dental resin composites formulated with camphorquinone or trimethylbenzoyl-diphenyl-phosphine oxide *Dental Materials* **28(4)** 392-397, 10.1016/j.dental.2011.11.014
17. Infuehr R, Pucher N, Heller C, Lichtenegger H, Liska R, Schmidt V, Kuna L, Haase A, & Stampfl J (2007) Functional polymers by two-photon 3D lithography *Applied Surface Science* **254(4)** 836-840.
18. Sugioka K & Cheng Y (2014) Femtosecond laser three-dimensional micro- and nanofabrication *Applied Physics Reviews* **1(4)** 041303.
19. Belfield KD, Schafer KJ, Liu Y, Liu J, Ren X, & Stryland EWV (2000) Multiphoton-absorbing organic materials for microfabrication, emerging optical applications and non-destructive three-dimensional imaging *Journal of Physical Organic Chemistry* **13(12)** 837-849.
20. Gong H, Beauchamp M, Perry S, Woolley AT, & Nordin GP (2015) Optical approach to resin formulation for 3D printed microfluidics *RSC Advances* **5(129)** 106621-106632.
21. Unkovskiy A, Bui PH, Schille C, Geis-Gerstorfer J, Huettig F, & Spintzyk S (2018) Objects build orientation, positioning, and curing influence dimensional accuracy and flexural properties of stereolithographically printed

resin *Dental Materials* **34(12)** e324-e333, 10.1016/j.dental.2018.09.011

22. Osman RB, Alharbi N, & Wismeijer D (2017) Build angle: Does it influence the accuracy of 3D-printed dental restorations using digital light-processing technology? *International Journal of Prosthodontics* **30(2)** 182-188, 10.11607/ijp.5117
23. Alharbi N, Osman R, & Wismeijer D (2016) Effects of build direction on the mechanical properties of 3D-printed complete coverage interim dental restorations *Journal of Prosthetic Dentistry* **115(6)** 760-767, 10.1016/j.prosdent.2015.12.002.
24. Patzelt SB, Bishti S, Stampf S, & Att W (2014) Accuracy of computer-aided design/computer-aided manufacturing-generated dental casts based on intraoral scanner data *Journal of the American Dental Association* **145(11)** 1133-1140, 10.14219/jada.2014.87
25. Hazeveld A, Huddleston Slater JJ, & Ren Y (2014) Accuracy and reproducibility of dental replica models reconstructed by different rapid prototyping techniques *American Journal of Orthodontics and Dentofacial Orthopedics* **145(1)** 108-115, 10.1016/j.ajodo.2013.05.011
26. Reymus M, Fotiadou C, Hickel R, & Diegritz C (2018) 3D-printed model for hands-on training in dental traumatology *International Endodontic Journal* **51(11)** 1313-1319, 10.1111/iej.12947
27. Sarment DP, Sukovic P, & Clinthorne N (2003) Accuracy of implant placement with a stereolithographic surgical guide *International Journal of Oral and Maxillofacial Implants* **18(4)** 571-577.
28. Chen X, Yuan J, Wang C, Huang Y, & Kang L (2010) Modular preoperative planning software for computer-aided oral implantology and the application of a novel stereolithographic template: A pilot study *Clinical Implant Dentistry and Related Research* **12(3)** 181-193, 10.1111/j.1708-8208.2009.00160.x
29. Jayme SJ, Muglia VA, de Oliveira RR, & Novaes AB (2008) Optimization in multi-implant placement for immediate loading in edentulous arches using a modified surgical template and prototyping: A case report *International Journal of Oral and Maxillofacial Implants* **23(4)** 759-762.
30. Lal K, White GS, Morea DN, & Wright RF (2006) Use of stereolithographic templates for surgical and prosthodontic implant planning and placement. Part I. The concept *Journal of Prosthodontics* **15(1)** 51-58, 10.1111/j.1532-849X.2006.00069.x
31. Di Giacomo GA, Cury PR, de Araujo NS, Sendyk WR, & Sendyk CL (2005) Clinical application of stereolithographic surgical guides for implant placement: Preliminary results *Journal of Periodontology* **76(4)** 503-507, 10.1902/jop.2005.76.4.503
32. Bilgin MS, Erdem A, Aglarci OS, & Dilber E (2015) Fabricating complete dentures with CAD/CAM and RP technologies *Journal of Prosthodontics* **24(7)** 576-579, 10.1111/jopr.12302.
33. Inokoshi M, Kanazawa M, & Minakuchi S (2012) Evaluation of a complete denture trial method applying rapid prototyping *Dental Materials Journal* **31(1)** 40-46.
34. Hofmann M (2014) *3D Printing Gets a Boost and Opportunities With Polymer Materials* ACS Publications, Washington, DC.
35. Sugavaneswaran M & Arumaikkannu G (2015) Analytical and experimental investigation on elastic modulus of reinforced additive manufactured structure *Materials & Design (1980-2015)* **66** 29-36.
36. Fahad M, Dickens P, & Gilbert M (2013) Novel polymeric support materials for jetting based additive manufacturing processes *Rapid Prototyping Journal* **19(4)** 230-239.
37. Braian M, Jimbo R, & Wennerberg A (2016) Production tolerance of additive manufactured polymeric objects for clinical applications *Dental Materials* **32(7)** 853-861, 10.1016/j.dental.2016.03.020
38. Mai HN, Lee KB, & Lee DH (2017) Fit of interim crowns fabricated using photopolymer-jetting 3D printing *Journal of Prosthetic Dentistry* **118(2)** 208-215, 10.1016/j.prosdent.2016.10.030
39. Fathi HM, Al-Masoody AH, El-Ghezawi N, & Johnson A (2016) The accuracy of fit of crowns made from wax patterns produced conventionally (hand formed) and via CAD/CAM technology *European Journal of Prosthodontics and Restorative Dentistry* **24(1)** 10-17.
40. Nelson JC, Xue S, Barlow JW, Beaman JJ, Marcus HL, & Bourell DL (1993) Model of the selective laser sintering of bisphenol-A polycarbonate *Industrial and Engineering Chemistry Research* **32(10)** 2305-2317.
41. Bibb R, Eggbeer D, & Williams R (2006) Rapid manufacture of removable partial denture frameworks *Rapid Prototyping Journal* **12(2)** 95-99.
42. Masri R & Driscoll C (2015) *Clinical Applications of Digital Dental Technology* John Wiley & Sons, New York, NY.
43. Goodridge R, Tuck C, & Hague R (2012) Laser sintering of polyamides and other polymers *Progress in Materials Science* **57(2)** 229-267.
44. Polzin C, Spath S, & Seitz H (2013) Characterization and evaluation of a PMMA-based 3D printing process *Rapid Prototyping Journal* **19(1)** 37-43.
45. Chen H, Yang X, Chen L, Wang Y, & Sun Y (2016) Application of FDM three-dimensional printing technology in the digital manufacture of custom edentulous mandible trays *Science Reports* **6** 19207, 10.1038/srep19207

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Comparison of Flexural Properties of Bulk-fill Restorative/Flowable Composites and Their Conventional Counterparts

AH Eweis • AU Yap • NA Yahya

Clinical Relevance

Bulk-fill restorative resin-based composites (RBCs), though stiffer than their flowable and conventional counterparts, were mostly weaker. Bulk-fill restorative RBCs should thus be used with caution in areas of high flexural stresses and an overlying final layer of conventional composite may be still be prudent.

SUMMARY

The objectives of the study were to compare the flexural modulus and strength of restorative and flowable bulk-fill resin-based composites (RBCs) to their conventional counterparts and to determine the effects of conditioning environment on their flexural properties. The materials evaluated included three conventional RBCs (Filtek Z350, Tetric N Ceram, and Beautifil II), three restorative bulk-fill RBCs (Filtek Bulk-Fill Restorative, Tetric N Ceram Bulk-Fill, and Beautifil Bulk-fill Restorative), as well as three flowable bulk-fill RBCs (Filtek Bulk-Fill Flowable, Tetric N Flow Bulk-Fill, and Beautifil Bulk-Fill Flowable). Specimens were fabricated using customized stainless-steel molds, finished, measured, and randomly divided into four groups. The various RBCs were conditioned in the following mediums (n=10) for seven days at 37°C: air, artificial saliva (SAGF), 0.02 N citric acid, and 50% ethanol-water solution. After conditioning, the specimens were rinsed, blotted dry, measured, and subjected to flexural testing using a universal testing machine. Data were subjected to statistical analysis using analysis of variance and the Tukey test at a significance level of $\alpha = 0.05$. Significant differences in flexural properties were observed between materials and conditioning mediums. Bulk-fill restorative RBCs exhibited higher flexural modulus than their bulk-fill flowable

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and conventional counterparts. With the exception of Filtek Bulk-Fill Flowable, bulk-fill flowable RBCs had significantly higher flexural strength than bulk-fill restorative and conventional RBCs. Flexural properties were highest when RBCs were conditioned in air and generally the lowest after exposure to ethanol.

INTRODUCTION

With advances in materials science and clinical techniques, the indications of resin-based composites (RBCs) have expanded to include large posterior stress-bearing restorations that were traditionally restored using amalgam.[1,2] Nonetheless, the posterior RBC restorations are still technically challenging to perform due to the incremental layering technique and depth-of-cure issues.[3,4] The incremental layering technique is time consuming and might lead to void formation between composite layers. Bulk-fill RBCs were developed to address the previously mentioned problems associated with conventional materials. They can be placed and cured in layers of up to 4 to 5 mm in thickness.[5] The greater depth of cure is achieved by means of novel resins, modified initiator systems, polymerization boosters, unique fillers, and filler control.[4,6,7] Moreover, special modulators and light-sensitive fillers are incorporated into some products to provide expanded working time by acting as a protective shield against operatory and ambient lights. Bulk-fill RBCs come in either restorative (sculptable/packable) or flowable forms.[5] Typically, flowable bulk-fill RBCs, with their lower filler content, are used as liners or bases in large class I/II restorations where they are placed and bulk cured in 4-mm increments and "capped" occlusally with more highly filled RBCs.[2,8] On the other hand, restorative bulk-fill RBCs are more highly filled and can be used in stress-bearing situations without the need for another overlying final layer.[9]

The mechanical properties of bulk-fill RBCs have been the subject of some debate. While some authors have reported lower mechanical properties than conventional highly filled RBCs, others have reported values that are close to conventional materials.[5,10,11] Prior data on curing efficiency had also been ambivalent, with some reporting depths of cure of more than 4 mm and others describing insufficient curing at 4-mm layers.[12-16] The differences in mechanical properties as well as depth of cure may be attributed to variances in resin compositions, material translucency, viscosity, filler type, and content.[8]

Until now, a limited number of studies have investigated the flexural properties of bulk-fill RBCs, and few, if any, have assessed the performance of bulk-fill giomers or prereacted glass (PRG) composites. Giomers are based on PRG technology where acid-reactive fluoride containing glass is prereacted with polyacids in the presence of water, freeze-dried, milled, silanized, ground, and used as fillers.[4] PRG technology has been incorporated into many Shofu products, ranging from restoratives to bonding agents. Besides being biocompatible and having antiplaque formation properties, giomers can also release and recharge fluoride.[17,18]

Physical properties of RBCs are well known to be affected by their surrounding chemical environment. Different chemicals from food substances can cause softening and dissolution of matrices of RBCs as well as filler damage, debonding, and leaching, resulting in decreased restoration durability and longevity.[19,20] The use of dietary solvents permits the accelerated assessment of dental RBCs in short periods of time together with an appraisal of chemical affinity and the elution process.[21] The chemical environment and food substances have been found to affect the viscoelastic properties of bulk-fill RBCs as well.[22]

The objectives of the study were to compare the flexural modulus and strength of restorative and flowable bulk-fill RBCs to their conventional materials and to determine the effects of conditioning environment on the flexural properties of bulk-fill composites. The null hypotheses were as follows: 1) there are no differences in flexural modulus and strength between restorative and flowable bulk-fill RBCs as well as their conventional counterparts, and 2) the flexural properties of bulk-fill restorative and flowable RBCs are not affected by their conditioning environment.

METHODS AND MATERIALS

Materials and Specimen Preparation

Table 1 shows the materials evaluated and their technical profiles. They included three conventional RBCs (Filtek Z350 [FZ], Tetric N Ceram [TN], and Beautifil II [BT]), three restorative bulk-fill RBCs (Filtek Bulk-Fill Restorative [FB], Tetric N Ceram Bulk-Fill [TB], and Beautifil Bulk-fill Restorative [BB]), as well as three flowable bulk-fill RBCs (Filtek Bulk-Fill Flowable [FF], Tetric N Flow Bulk-Fill [TF], and Beautifil Bulk-Fill Flowable [BF]).

The International Organization for Standardization (ISO) recommends the use of specimens that are 25 × 2 × 2 mm for flexural testing.[23] Such specimens are challenging to fabricate without flaws and necessitate the use of multiple overlapping irradiation because of the comparably smaller

Table 1: *Technical Profiles and Manufacturers of the Materials Evaluated*

Manufacturer	Material (Abbreviation)	Type	Resin (Photoinitiator)	Filler	Filler Content % by weight/% by Volume	Lot Number
3M ESPE (St Paul, MN, USA) [A]	Filtek Z350 (FZ)	Conventional nanohybrid composite	Bis-GMA Bis-EMA UDMA TEGDMA (CQ)	Zirconia/silica cluster and silica nanoparticle	78.5/63.3	N771467
	Filtek Bulk-Fill Restorative (FB)	Bulk-fill restorative composite	AUDMA AFM DDDMAA UDMA (CQ)	Zirconia/silica cluster, ytterbium trifluoride	76.5/58.4	N693019
	Filtek Bulk-Fill Flowable (FF)	Flowable bulk-fill composite	Bis-GMA Bis-EMA UDMA (CQ, EDMAB)	Zirconia/silica, ytterbium trifluoride	64.5/42.5	N884479
Ivoclar Vivadent Inc (Amherst, NY, USA) [B]	Tetric N Ceram (TN)	Conventional microhybrid composite	Bis-GMA Bis-EMA UDMA (CQ)	Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide, prepolymers	81.2/57	V35260
	Tetric N Ceram Bulk-Fill (TB)	Bulk-fill restorative composite	Bis-GMA Bis-EMA UDMA (CQ, TPO, Ivocerin)	Barium glass filler, ytterbium fluoride, spherical mixed oxide	79/60	S38368
	Tetric N Flow Bulk-Fill (TF)	Flowable bulk-fill composite	Bis-GMA UDMA TEGDMA (Ivocerin)	Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide	64.9/NA	V49336
Shofu Inc (Kyoto, Japan) [C]	Beautifil II (BT)	Conventional giomer	Bis-GMA TEGDMA (CQ)	S-PRG based on F-Br-Al-Si glass	83.3/68.8	31731
	Beautifil-Bulk Restorative (BB)	Bulk-fill restorative giomer	Bis-GMA UDMA Bis-MPEPP TEGDMA (CQ)	S-PRG based on F-Br-Al-Si glass	87/74.5	51623
	Beautifil-Bulk Flowable (BF)	Flowable bulk-fill giomer	Bis-GMA UDMA Bis-MPEPP TEGDMA (CQ)	S-PRG based on F-Br-Al-Si glass	73/60	101615

Abbreviations: Bis-GMA, bisphenol-A glycidyl methacrylate; Bis-EMA, ethoxylated bisphenol-A-glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; CQ, camphorquinone; AUDMA, aromatic urethane dimethacrylate; AFM, addition-fragmentaion monomers; DDDMA, 1,12-dodecanediol dimethacrylate; EDMAB, ethyl 4-dimethyl aminobenzoate; TPO, 2,4,6-trimethylbenzoyl diphenylphosphine oxide; NA, not available; S-PRG, surface-modified prereacted glass; F-Br-Al-Si, fluoroboroaluminosiliciate; Bis-MPEPP, bisphenol-A polyethoxy-dimethacrylate.

light exit windows of curing tips.[24] Moreover, these ISO specimens are not clinically relevant since the mesio-distal widths of molars are around 11 mm, and the cervico-incisial length of central incisors usually do not exceed 13 mm.[25] The miniflexural test, employing 12-mm specimens, was selected due to their significant correlation to the ISO flexural test, clinical relevance, and better efficiency.[26,27] Forty beam-shaped test specimens (12×2×2 mm) of each of the various RBCs were fabricated using customized stainless-steel molds. The conventional and bulk-fill RBCs were placed in a single increment, while the flowable materials were injected into the molds. Excess material was removed by compressing the molds between two Mylar strips with glass slides. The top surface of the specimens were light polymerized through the glass slide with two overlapping irradiations of 10 seconds each using a calibrated LED curing light (Demi Plus, Kerr, Orange, CA, USA) with an output irradiance of 1330 mW/cm^2 and wavelength range of 450 to 470 nm. The glass slides were removed, and the

specimens were light cured for another 10 seconds. The Mylar strips were subsequently discarded, and the composite beams were removed from their molds. Any minor material excess, or "fins," was gently removed by fine polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA). The composite specimens were visually examined for the presence of voids, and any defective specimens were replaced. The final dimensions of the specimens and the parallelism between their opposite surfaces were verified with a digital caliper (Mitutoyo Corp, Kawasaki, Japan).

Conditioning Mediums and Time

Specimens of the various materials were randomly divided into four groups of 10 (n=10) and conditioned in the following mediums for seven days at 37°C: air (control), artificial saliva (SAGF),[28] 0.02 N citric acid, and 50% ethanol-water solution. Containers used to house the various solutions were sealed to minimize evaporation and stored in air within an incubator (IN-450, Memmert, Schwabach, Germany). The pH of the artificial saliva was verified via a digital pH meter (pH2700, Eutech, Singapore) and adjusted to 6.8 with diluted hydrochloric acid (where needed) to resemble natural saliva pH when it is released from the salivary ducts.[21]

Flexural Testing

After the seven-day conditioning period, the composite specimens were loaded until fracture using a universal testing machine (Shimadzu Corp, Kyoto, Japan) with a load cell of 5 KN and crosshead speed of 0.5 mm/min. Flexural strength, σ, in megapascals (MPa), was calculated using the following equation:

$$\sigma = \frac{3PL}{2BH^2}$$

where P is the maximum load exerted on the specimen in newtons, L is the distance between the supports in millimeters (10 mm), B is the width of the specimen in millimeters, and H is the height of the specimen in millimeters.

Flexural modulus, E′, in MPa, was calculated using the following equation:

$$E' = \left(\frac{F}{D}\right)\left(\frac{L^3}{4BH^3}\right)$$

where F/D is the slope, in newtons per millimeter, measured in the straight-line portion of the load-deflection graph; L, B, and H are defined in the flexural strength equation. Flexural modulus was subsequently converted to gigapascals (GPa).

Statistical Analysis

The SPSS statistical program (version 12.0.1, SPSS Inc, Chicago, IL, USA) was used to analyze the data obtained. Normality testing was done using the Shapiro-Wilk test, and parametric data analysis was permissible, as data were normally distributed. Homogeneity of variance was assessed using the Levene test, and equal variances were assumed. The interactions between the independent variables (materials and conditioning mediums) and each of the dependent variables (flexural modulus and flexural strength) were evaluated using two-way analysis of variance (ANOVA). One-way ANOVA, followed by Tukey *post hoc* tests, was used to determine intermedium and intermaterial differences for flexural modulus and strength for the same material type from different manufacturers as well as for different material types from the same manufacturer. All statistical analyses were carried out at significance level of $\alpha = 0.05$.

RESULTS

Mean flexural modulus and strength for the various RBCs after conditioning in the different mediums are reflected in Table 2. Figures 1 and 2 compare the mean flexural modulus and strength between mediums for each material. Statistical comparisons of mean flexural properties between RBCs when grouped by manufacturers and material type after conditioning in the various mediums are summarized in Tables 3 and 4, respectively. Two-way ANOVA presented significant interactions ($p<0.001$) between materials and mediums for both flexural modulus and strength.

Comparison Between Material Types by Manufacturers

Manufacturer (A) RBCs—With the exception of air, FB had the highest flexural modulus for all mediums. FZ showed the highest flexural modulus in air. For all mediums, FF had significantly lower flexural modulus than FZ and FB. The highest flexural strength was observed with FB for all mediums. FZ had the lowest flexural strength for all mediums aside from air, where FF was the lowest. There was, however, no significant difference between the three different manufacturer (A) RBCs when conditioned in air.

Table 2: *Mean Flexural Modulus (GPa) and Flexural Strength (MPa) of the Various Resin-Based Composites (Standard Deviations in Parentheses)*

Medium/Material (Abbreviation)	Flexural Modulus (GPa)				Flexural Strength (MPa)			
	Air	Artificial Saliva	Citric Acid	Ethanol	Air	Artificial Saliva	Citric Acid	Ethanol
Filtek Z350 (FZ)	8.23 (0.89)	6.58 (0.76)	6.38 (0.78)	6.89 (0.94)	135.20 (17.08)	91.71 (10.10)	89.03 (11.84)	62.50 (7.56)
Filtek Bulk-Fill Restorative (FB)	8.04 (1.11)	7.64 (1.07)	8.00 (1.05)	7.25 (0.99)	144.00 (19.32)	122.39 (16.63)	115.26 (10.51)	120.94 (12.75)
Filtek Bulk-Fill Flowable (FF)	3.59 (0.28)	3.52 (0.39)	3.06 (0.17)	1.91 (0.15)	127.89 (7.19)	101.09 (12.43)	105.13 (11.86)	66.56 (5.33)
Tetric N Ceram (TN)	5.22 (0.32)	4.62 (0.28)	4.01 (0.23)	2.94 (0.18)	109.84 (10.34)	86.08 (12.10)	89.53 (7.05)	60.25 (4.45)
Tetric N Ceram Bulk-Fill (TB)	6.51 (0.81)	5.72 (0.49)	4.86 (0.56)	3.33 (0.43)	106.85 (6.80)	99.17 (8.89)	93.20 (8.23)	55.77 (4.78)
Tetric N Flow Bulk-Fill (TF)	4.56 (0.29)	3.89 (0.29)	4.05 (0.39)	3.10 (0.33)	119.73 (11.72)	106.92 (7.74)	98.06 (10.01)	82.56 (5.04)
Beautifil II (BT)	6.66 (0.93)	6.51 (0.55)	6.43 (0.47)	5.82 (0.34)	110.36 (13.40)	79.50 (8.79)	86.94 (9.71)	68.26 (8.02)
Beautifil-Bulk Restorative (BB)	8.19 (1.12)	7.34 (0.92)	5.93 (0.84)	6.80 (0.72)	117.53 (10.22)	86.60 (3.57)	87.23 (8.06)	85.76 (6.86)
Beautifil-Bulk Flowable (BF)	5.56 (0.48)	5.51 (0.42)	5.21 (0.48)	4.47 (0.38)	113.91 (11.89)	105.56 (12.01)	100.07 (11.54)	99.81 (11.98)

Manufacturer (B) RBCs—TB had the highest flexural modulus for all mediums. TF showed the lowest flexural modulus when conditioned in air and artificial saliva, whereas TN presented the lowest modulus in citric acid and ethanol. The highest flexural strength was observed with TF for all mediums. TB showed the lowest flexural strength when conditioned in air and ethanol, whereas TN was the lowest in artificial saliva and citric acid. Nonetheless, no significant difference in strength was observed between the three different manufacturer (B) RBCs when conditioned in citric acid.

Manufacturer (C) RBCs—Besides citric acid, BB had the highest flexural modulus in all mediums. BT

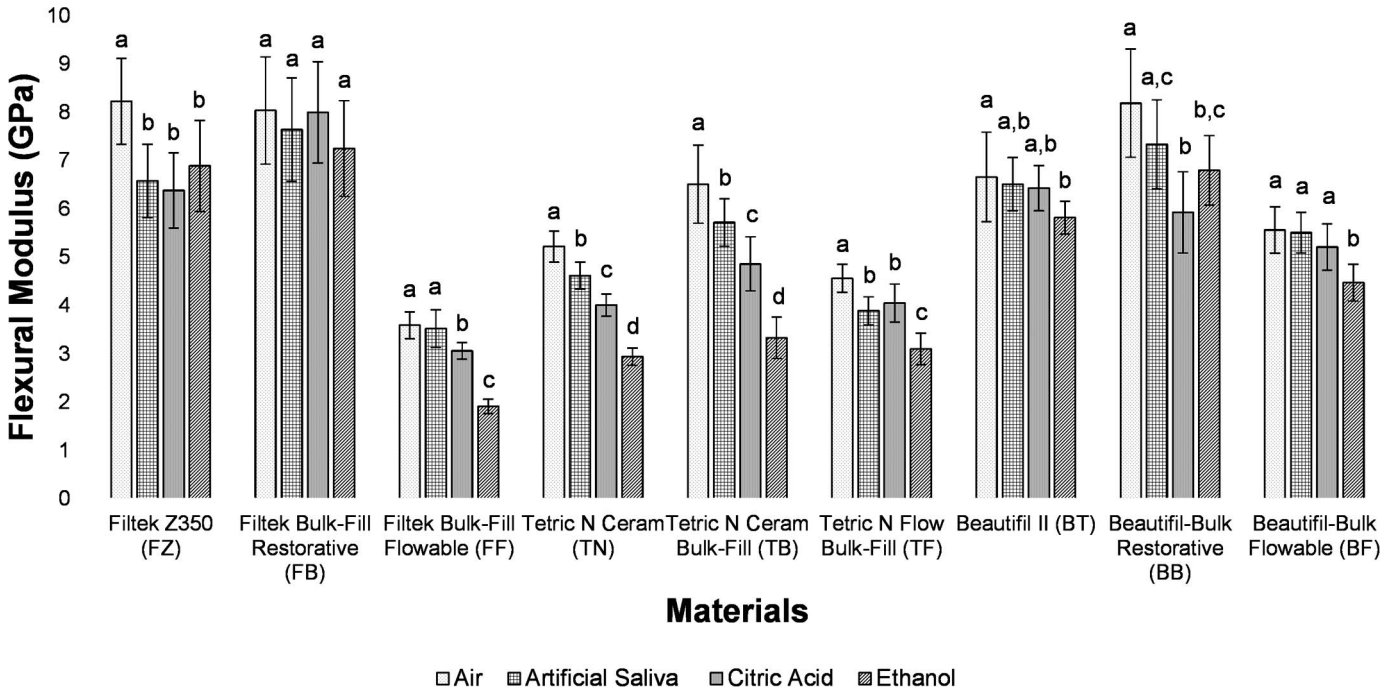

Figure 1. *Mean flexural modulus values (GPa) for the different materials after storage in the different mediums. Results of one-way analysis of variance and* post hoc *Tukey test (*$p<0.05$*). Same letters above the bars indicate no statistical significance between different mediums for each material.*

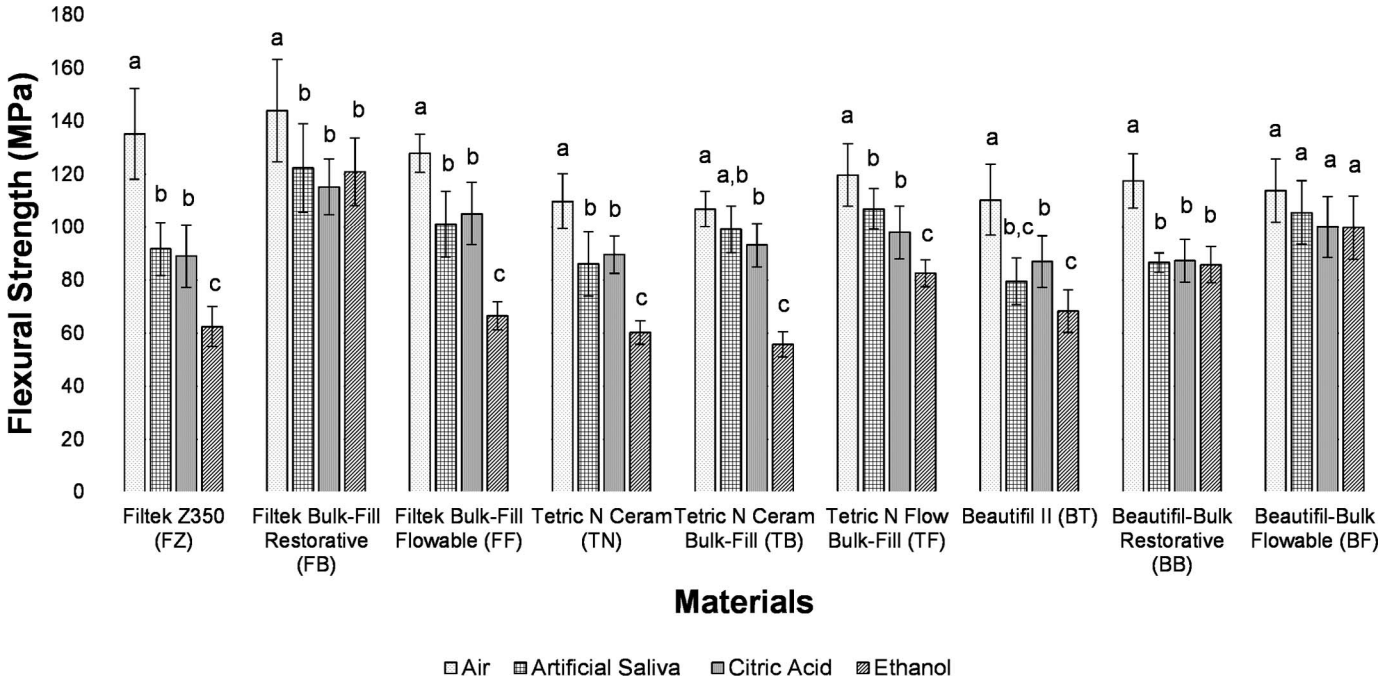

Figure 2. *Mean flexural strength values (MPa) for the different materials after storage in the different mediums. Results of one-way analysis of variance and* post hoc *Tukey test ($p<0.05$). Same letters above the bars indicate no statistical significance between different mediums for each material.*

showed the highest flexural modulus in citric acid. Regardless of mediums, BF had significantly lower modulus than BT and BB. With the exemption of air, BF had significantly higher flexural strength than the other two giomers. BB showed the highest flexural strength in air. However, no significant difference in flexural strength was observed between the three different manufacturer (C) RBCs when conditioned in air. BT had the lowest flexural strength for all mediums.

Comparison Between Different Manufacturers for Each RBC Type

Conventional RBCs—With the exception of citric acid, FZ had the highest flexural modulus for all mediums. BT presented the highest flexural modulus in citric acid. TN had significantly lower flexural modulus than FZ and BT for all mediums. FZ had the highest flexural strength when conditioned in air and artificial saliva, whereas TN and BT displayed the highest flexural strength when conditioned in citric

Table 3: *Comparison of Mean Flexural Modulus and Strength Between Resin-Based Composites Grouped by Manufacturer After Conditioning in the Various Mediums*[a]

Properties	Medium	Differences		
		A	B	C
Flexural modulus	Air	FZ, FB > FF	TB > TN > TF	BB > BT > BF
	Artificial saliva	FB > FZ > FF	TB > TN > TF	BB > BT > BF
	Citric acid	FB > FZ > FF	TB > TF, TN	BT, BB > BF
	Ethanol 50%	FB, FZ > FF	TB > TN	BB > BT > BF
Flexural strength	Air	Nonsignificant	TF > TB	Nonsignificant
	Artificial saliva	FB > FF, FZ	TF, TB > TN	BF > BB, BT
	Citric acid	FB, FF > FZ	Nonsignificant	BF > BB, BT
	Ethanol 50%	FB > FF, FZ	TF > TN, TB	BF > BB > BT

Abbreviations: FZ, Filtek Z350; FB, Filtek Bulk-Fill Restorative; FF, Filtek Bulk-Fill Flowable; TB, Tetric N Ceram Bulk-Fill; TN, Tetric N Ceram; TF, Tetric N Flow Bulk-Fill; BB, Beautifil-Bulk Restorative; BT, Beautifil II; BF, Beautifil-Bulk Flowable.

[a] *Results of one-way analysis of variance and* post hoc *Tukey test ($p<0.05$); > indicates statistical significance.*

Table 4: *Comparison of Mean Flexural Modulus and Strength Between Resin-Based Composites Grouped by Material Type After Conditioning in the Various Mediums*[a]

Properties	Medium	Differences		
		Conventional	Bulk-Fill Restorative	Bulk-Fill Flowable
Flexural modulus	Air	FZ > BT > TN	BB, FB > TB	BF > TF > FF
	Artificial saliva	FZ, BT > TN	FB, BB > TB	BF > TF, FF
	Citric acid	BT, FZ > TN	FB > BB > TB	BF > TF > FF
	Ethanol 50%	FZ > BT > TN	FB, BB > TB	BF > TF > FF
Flexural strength	Air	FZ > BT, TN	FB > BB, TB	FF > BF
	Artificial saliva	FZ > BT	FB > TB > BB	Nonsignificant
	Citric acid	Nonsignificant	FB > TB, BB	Nonsignificant
	Ethanol 50%	BT > TN	FB > BB > TB	BF > TF > FF

Abbreviations: FZ, Filtek Z350; BT, Beautifil II; TN, Tetric N Ceram; BB, Beautifil-Bulk Restorative; FB, Filtek Bulk-Fill Restorative; TB, Tetric N Ceram Bulk-Fill; BF, Beautifil-Bulk Flowable; TF, Tetric N Flow Bulk-Fill; FF, Filtek Bulk-Fill Flowable.

[a] *Results of one-way analysis of variance and* post hoc *Tukey test ($p<0.05$); > indicates statistical significance.*

acid and ethanol, respectively. TN showed the lowest flexural strength when conditioned in air and ethanol, while BT had the lowest strength in artificial saliva and citric acid. No significant difference in strength was observed between the three conventional materials when conditioned in citric acid.

Bulk-Fill Restorative RBCs—With the exception of air, FB had the highest flexural modulus. BB showed the highest flexural modulus in air. TB had significantly lower flexural modulus than FB and BB for all mediums. FB had significantly higher flexural strength than BB and TB for all mediums. TB showed the lowest flexural strength when conditioned in air and ethanol, whereas BB had the lowest strength in artificial saliva and citric acid.

Bulk-Fill Flowable RBCs—BF had significantly higher flexural modulus than TF and FF for all mediums. FF showed the lowest flexural modulus for all mediums. However, FF presented the highest flexural strength when conditioned in air and citric acid, whereas TF and BF had the highest flexural strength when conditioned in artificial saliva and ethanol, respectively. On the other hand, FF had the lowest flexural strength when conditioned in artificial saliva and ethanol, while TF and BF showed the lowest flexural strength when conditioned in citric acid and air, respectively. However, there was no significant difference between the three different bulk-fill flowable materials when conditioned in artificial saliva and citric acid.

Comparison Between Conditioning Mediums

Manufacturer (A) RBCs—Conditioning in air showed the highest flexural modulus and strength for all manufacturer (A) materials. The lowest flexural modulus and strength were generally observed after conditioning in ethanol. Conditioning in citric acid showed the lowest flexural modulus for FZ and the lowest flexural strength for FB. No significant difference in flexural modulus was observed between mediums for FB. For FZ, conditioning in air resulted in significantly higher flexural modulus than all other mediums, whereas no significant difference was observed between ethanol, artificial saliva, and citric acid. Flexural modulus of FF after conditioning in air and artificial saliva was significantly higher than in citric acid, which in turn was significantly higher than in ethanol. For all manufacturer (A) RBCs, no significant difference in flexural strength was noted between conditioning in artificial saliva and citric acid. When conditioned in artificial saliva and citric acid, FZ and FF showed significantly higher flexural strength than in ethanol.

Manufacturer (B) RBCs—Conditioning in air again resulted in the highest flexural modulus and strength. The lowest flexural modulus and strength were obtained after conditioning in ethanol. For TN and TB, significant differences in flexural modulus was noted between the four mediums. For TF, conditioning in air showed significantly higher flexural modulus than in saliva and citric acid, which in turn was greater than in ethanol. There was no significant difference in flexural strength between artificial saliva and citric acid for all manufacturer (B) RBCs. When conditioned in these mediums, all materials showed significantly higher flexural strength than ethanol.

Manufacturer (C) RBCs—Conditioning in air showed the highest flexural modulus and strength for all manufacturer (C) RBCs. The lowest flexural modulus and strength were observed after conditioning in ethanol except for BB, which had the

lowest flexural modulus after conditioning in citric acid. For BB, conditioning in citric acid resulted in significantly lower flexural modulus than in air and artificial saliva. Flexural modulus after conditioning in air was significantly higher than in ethanol for BT, whereas for BF, it was significantly lower than all other mediums when conditioned in ethanol. There was no significant difference in flexural strength between artificial saliva and citric acid for all giomers. For BT, conditioning in ethanol resulted in significantly lower flexural strength than citric acid. No significant difference in flexural strength was observed between all mediums for BF.

DISCUSSION

The current study investigated the differences in the flexural properties between bulk-fill and conventional RBCs and the effect of the conditioning environment on the different materials. As flexural properties were material and conditioning medium dependent, both the null hypotheses were rejected. The different RBCs were immersed continually in the various conditioning mediums for seven days at 37°C before flexural testing was performed. This conditioning period might appear considerably long since restorations come into contact with foods and liquids infrequently and for short durations intraorally. Continuous exposure may, however, take place when chemicals are absorbed by food particles or calculus at the margins or grooves of restorations.[29,30]

Flexural testing is widely used in characterizing RBCs since it determines both flexural modulus and strength.[23] Flexural modulus describes the stiffness of RBCs, whereas flexural strength represents the maximum stress that RBCs can be subjected to prior to failure. Flexural testing yields complex tensions arising from the combination of compression, shear, and tensile stresses.[24] The variation between the flexural properties of various RBCs is useful in the different clinical situations.[31,32] For example, in class I, II, III, and IV cavities, RBCs with high flexural properties are usually selected to minimize fracture or deformation under the high occlusal forces, while in class V cavities, RBCs having low flexural modulus are preferred, as they can flex with the teeth during function and parafunction, which in turn reduces the stresses at the adhesive interface and decreases the chances of debonding.[31,33]

Bulk-fill restorative RBCs were generally stiffer than their bulk-fill flowable and conventional counterparts. This may be attributed to the similar or higher filler content of the bulk-fill restoratives in comparison to the other RBCs. Results corroborated those of El-Safty and others,[34] who reported a significant positive correlation between modulus and filler loading. FZ had a higher filler content than FB, which explains the higher flexural modulus of FZ in air. Weak intraoral acids, such as citric acid, have been reported to degrade the inorganic fillers in RBCs.[30] This might explain why BB, with its relatively higher inorganic and prereacted glass ionomer filler content, was somewhat more susceptible to modulus degradation than BT. The flexural modulus of the bulk-fill flowable RBCs were mostly significantly lower than their bulk-fill restorative and conventional counterparts. This was consistent with the work of Jung and others.[35] These authors suggested that bulk-fill flowable RBCs, with their lower modulus, may not provide an effective buffer to occlusal stress and recommended that they be capped with conventional materials. In high-stress–bearing areas, RBCs of higher stiffness are required to prevent restoration deformation, which could accelerate marginal and restoration failures. In addition to modulus, other physical properties of RBCs, including strength, fracture, and wear resistance, must also be considered for stress-bearing situations. However, with their greater flexibility, bulk-fill flowable RBCs are preferred over bulk-fill restorative and conventional materials in deep class V cavities, as they appear to offer better marginal adaptation.[36]

For materials from manufacturer (A), the bulk-fill restorative had higher flexural strength than the other RBCs. However, for manufacturer (B) and (C) materials, bulk-fill flowable RBCs were generally stronger than their bulk-fill restorative and conventional counterparts despite their relatively lower filler loading. These results contradicted those of Tomaszewska and others[35] and Jung and others,[37] which reported that bulk-fill flowable RBCs have lower mechanical properties when compared to either highly filled nanohybrid or bulk-fill restorative RBCs. The variance in results may be attributed in part to the differences in bulk-fill flowable RBCs evaluated and experimental designs. Commercial flowable composites have a wide range of filler loading from 52% to 68% weight.[38] The higher flexural strength of the bulk-fill flowable RBCs evaluated in the present study when compared to bulk-fill restorative and conventional materials could be attributed to their relatively high filler loading, resiliency, and ability to withstand higher stress prior to fracture (Table 1).

When comparing different products, TN and TB from manufacturer (B) and FF from manufacturer (A) had significantly lower modulus than the other conventional, bulk-fill restorative and bulk-fill flow-

able RBCs regardless of conditioning mediums. The significantly lower modulus of these RBCs may be attributed to their relatively lower filler loading, disparities in fillers, and resin matrices used. As for flexural strength, no obvious trends were observed when conventional and bulk-fill flowable RBCs were compared. Differences between products varied with conditioning mediums, highlighting the importance of conducting flexural testing with different dietary solvents. Variances can again be attributed to differences in filler and resin content/type employed. For bulk-fill restorative materials, FB from manufacturer (A) was significantly stronger than both TN and BB from manufacturers (B) and (C), respectively. FB contains two novel monomers: a high-molecular-weight aromatic dimethacrylate and addition fragmentation monomers that act to decrease the polymerization shrinkage stress.[39] According to the manufacturer, aromatic dimethacrylate decreases the reactive groups in the resin, controlling the volumetric shrinkage and the rigidity of the final polymeric matrix, whereas addition fragmentation monomers contain a third reactive site that cleaves through a fragmentation process. This in turn helps provide a relaxation mechanism of the network being developed, leading to stress relief. The fragments can still react with each other or with other reactive sites ensuring that the physical properties of the material are preserved. This might play a role in developing shorter and stiffer polymeric chains, leading to a more rigid and stronger bulk-fill RBC.[40]

When storage environments were compared, conditioning in air presented the highest flexural modulus and strength for all RBCs regardless of their type or manufacturer. Conditioning in air does not result in the leaching out of silica and filler particles that occurs with storage in aqueous mediums.[29] The lowest flexural modulus was observed when the RBCs were conditioned in ethanol with the exception of FZ and BB, which showed the lowest flexural modulus after storage in citric acid. Other than FB, which was weakest after storage in citric acid, the lowest flexural strength was also observed with conditioning in ethanol for all RBCs. The effect of citric acid on RBCs has already been elaborated on. Ethanol is known to soften the resin matrix of RBCs by removing unreacted monomers and linear polymers from the polymeric structure.[24,29] With resin dissolution, filler exposure and dislodgement may ensure weakening the RBCs. The dietary habits of patients should thus be considered during material selection to enhance the clinical longevity of composite restorations.

The current study can be improved in some areas. To begin with, the conditioning period could be extended to determine the longer-term effects of conditioning environment on flexural properties.[21,41] Static flexural testing that was carried out in the present study cannot provide insights into material structure, as dental RBCs are viscoelastic in nature and exhibit both viscous and elastic characteristics when undergoing deformation. Dynamic testing with dynamic mechanical analysis can be performed to better assess the viscoelastic properties of the RBCs.[42] Dynamic mechanical analysis can be carried out using various frequencies, temperatures, and displacements that resemble the variations of forces and temperatures in the oral cavity. Moreover, unlike static testing, dynamic testing enables retesting of specimens over extended time periods, as it is nondestructive.[22,43] As flowable RBCs are not a homogeneous group of materials, appraisal of their rheological properties and correlating this to their flexural properties may be beneficial.

CONCLUSIONS

Within the limitations of this study, the following can be concluded:

- Flexural properties of bulk-fill restorative, bulk-fill flowable, and conventional RBCs were both material and conditioning medium dependent.
- Bulk-fill restorative RBCs were generally stiffer than their bulk-fill flowable and conventional counterparts.
- With the exception of FF, bulk-fill flowable RBCs were stronger than their bulk-fill restorative and conventional counterparts.
- While no patterns were observed for flexural strength, manufacturer (B) bulk-fill and conventional RBCs were less rigid than comparable products from manufacturers (A) and (C).
- Conditioning in air resulted in the highest flexural properties, while exposure to ethanol generally presented the lowest.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Ferracane JL (2011) Resin composite—State of the art *Dental Materials* **27(1)** 29-38.
2. Fugolin APP & Pfeifer CS (2017) New resins for dental composites *Journal of Dental Research* **96(10)** 1085-1091.
3. Versluis A, Douglas WH, Cross M, & Sakaguchi RL (1996) Does an incremental filling technique reduce polymerization shrinkage stresses? *Journal of Dental Research* **75(3)** 871-878.
4. Yap AU, Pandya M, & Toh WS (2016) Depth of cure of contemporary bulk-fill resin-based composites *Dental Materials Journal* **35(3)** 503-510.
5. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, & Leloup G (2014) Physico-mechanical characteristics of commercially available bulk-fill composites *Journal of Dentistry* **42(8)** 993-1000.
6. Fleming GJP, Awan M, Cooper PR, & Sloan AJ (2008) The potential of a resin-composite to be cured to a 4 mm depth *Dental Materials* **24(4)** 522-529.
7. Lassila LVJ, Nagas E, Vallittu PK, & Garoushi S (2012) Translucency of flowable bulk-filling composites of various thicknesses *Chinese Journal of Dental Research* **15(1)** 31-35.
8. Jang JH, Park SH, & Hwang IN (2015) Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin *Operative Dentistry* **40(2)** 172-180.
9. Hirata R, Kabbach W, De Andrade OS, Bonfante EA, Giannini M, & Coelho PG (2015) Bulk fill composites: An anatomic sculpting technique *Journal of Esthetic and Restorative Dentistry* **27(6)** 335-343.
10. Ilie N, Bucuta S, & Draenert M (2013) Bulk-fill resin-based composites: An in vitro assessment of their mechanical performance *Operative Dentistry* **38(6)** 618-625.
11. El Gezawi M, Kaisarly D, Al-Saleh H, ArRejaie A, Al-Harbi F, & Kunzelmann K (2016) Degradation potential of bulk versus incrementally applied and indirect composites: Color, microhardness, and surface deterioration *Operative Dentistry* **41(6)** 195-208.
12. Bucuta S & Ilie N (2014) Light transmittance and micromechanical properties of bulk fill vs. conventional resin based composites *Clinical Oral Investigations* **18(8)** 1991-2000.
13. Garcia D, Yaman P, Dennison J, & Neiva GF (2014) Polymerization shrinkage and depth of cure of bulk fill flowable composite resins *Operative Dentistry* **39(4)** 441-448.
14. Czasch P & Ilie N (2013) In vitro comparison of mechanical properties and degree of cure of bulk fill composites *Clinical Oral Investigations* **17(1)** 227-235.
15. Alrahlah A, Silikas N, & Watts DC (2014) Post-cure depth of cure of bulk fill dental resin-composites *Dental Materials* **30(2)** 149-154.
16. Ilie N, Kessler A, & Durner J (2013) Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites *Journal of Dentistry* **41(8)** 695-702.
17. Itota T, Carrick TE, Yoshiyama M, & McCabe JF (2004) Fluoride release and recharge in giomer, compomer and resin composite *Dental Materials* **20(9)** 789-795.
18. Saku S, Kotake H, Scougall-Vilchis RJ, Ohashi S, Hotta M, Horiuchi S, Hamada K, Asaoka K, Tanaka E, & Yamamoto K (2010) Antibacterial activity of composite resin with glass-ionomer filler particles *Dental Materials Journal* **29(2)** 193-198.
19. Wu W, Toth EE, Moffa JF, & Ellison JA (1984) Subsurface damage layer of in vivo worn dental composite restorations *Journal of Dental Research* **63(5)** 675-680.
20. Drummond JL (2008) Degradation, fatigue, and failure of resin dental composite materials *Journal of Dental Research* **87(8)** 710-719.
21. Vouvoudi EC & Sideridou ID (2012) Dynamic mechanical properties of dental nanofilled light-cured resin composites: Effect of food-simulating liquids *Journal of the Mechanical Behavior of Biomedical Materials* **10** 87-96.
22. Eweis AH, Yap AU-J, & Yahya NA (2017) Dynamic analysis of bulk-fill composites: Effect of food-simulating liquids *Journal of the Mechanical Behavior of Biomedical Materials* **74C** 183-188.
23. International Organization for Standardization (2009) ISO 4049 Dentistry polymer-based filling, restorative and luting materials *Geneve: International Organization for Standardization* 15-18.
24. dos Santos SG, Moyses MR, Alcantara CEP, Ribeiro JCR, & Ribeiro JGR (2012) Flexural strength of a composite resin light cured with different exposure modes and immersed in ethanol or distilled water media *Journal of Conservative Dentistry* **15(4)** 333-336.
25. Wheeler RC (1965) *A Textbook of Dental Anatomy and Physiology* WB Saunders, Philadelphia PA.
26. Yap AU & Teoh SH (2003) Comparison of flexural properties of composite restoratives using the iso and mini-flexural tests *Journal of Oral Rehabilitation* **30(2)** 171-177.
27. Yap AU, Eweis AH, & Yahya NA (2018) Dynamic and static flexural appraisal of resin-based composites: Comparison of the ISO and mini-flexural tests *Operative Dentistry* **43(5)** e223-e231.
28. Gal JY, Fovet Y, & Adib-Yadzi M (2001) About a synthetic saliva for in vitro studies *Talanta* **53(6)** 1103-1115.
29. Yap AU, Tan DTT, Goh BKC, Kuah HG, & Goh M (2000) Effect of food-simulating liquids on the flexural strength of composite and polyacid-modified composite restoratives *Operative Dentistry* **25(3)** 202-208.
30. Akova T, Ozkomur A, & Uysal H (2006) Effect of food-simulating liquids on the mechanical properties of provisional restorative materials *Dental Materials* **22(12)** 1130-1134.

31. Yap AU, Chandra S, Chung S, & Lim C (2002) Changes in flexural properties of composite restoratives after aging in water *Operative Dentistry* **27(5)** 468-474.

32. Rodrigues SAJ, Zanchi CH, Carvalho RV, & Demarco FF (2007) Flexural strength and modulus of elasticity of different types of resin-based composites *Brazilian Oral Research* **21(1)** 16-21.

33. Pontes LF, Alves EB, Alves BP, Ballester RY, Dias CGBT, & Silva CM (2013) Mechanical properties of nanofilled and microhybrid composites cured by different light polymerization modes *General Dentistry* **61(3)** 30-33.

34. El-Safty S, Akhtar R, Silikas N, & Watts DC (2012) Nanomechanical properties of dental resin-composites *Dental Materials* **28(12)** 1292-1300.

35. Jung J & Park S (2017) Comparison of polymerization shrinkage, physical properties, and marginal adaptation of flowable and restorative bulk fill resin-based composites *Operative Dentistry* **42(4)** 375-386.

36. Szesz A, Parreiras S, Martini E, Reis A, & Loguercio A (2017) Effect of flowable composites on the clinical performance of non-carious cervical lesions: A systematic review and meta-analysis *Journal of Dentistry* **65** 11-21.

37. Tomaszewska IM, Kearns JO, Ilie N, & Fleming GJP (2015) Bulk fill restoratives: To cap or not to cap—That is the question? *Journal of Dentistry* **43(3)** 309-316.

38. Moszner N & Salz U (2001) New developments of polymeric dental composites *Progress in Polymer Science* **26(4)** 535-576.

39. Kalliecharan D, Germscheid W, Price RB, Stansbury J, & Labrie D (2016) Shrinkage stress kinetics of bulk fill resin-based composites at tooth temperature and long time *Dental Materials* **32(11)** 1322-1331.

40. Braga RR, Ballester RY, & Ferracane JL (2005) Factors involved in the development of polymerization shrinkage stress in resin-composites: A systematic review *Dental Materials* **21(10)** 962-970.

41. Mesquita RV, Axmann D, & Geis-Gerstorfer J (2006) Dynamic visco-elastic properties of dental composite resins *Dental Materials* **22(3)** 258-267.

42. Jacobsen PH & Darr AH (1997) Static and dynamic moduli of composite restorative materials *Journal of Oral Rehabilitation* **24(4)** 265-273.

43. Mesquita RV & Geis-Gerstorfer J (2008) Influence of temperature on the visco-elastic properties of direct and indirect dental composite resins *Dental Materials* **24(5)** 623-632.

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Efficacy of Direct Restorative Materials in Proximal Box Elevation on the Margin Quality and Fracture Resistance of Molars Restored With CAD/CAM Onlays

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Clinical Relevance

Specimens with type II glass ionomer/proximal box elevation (PBE) behave similarly in terms of margin quality and fracture resistance to specimens restored with resin-based composite/PBE and without PBE. Dental professionals may elect type II glass ionomer/PBE in appropriate clinical situations.

SUMMARY

Purpose: The purpose of this study was to investigate the effect of four direct restorative materials that can be used in the proximal box elevation (PBE) technique.

Methods and Materials: Seventy-five molar teeth were randomly assigned to one of five groups (n=15): type II glass ionomer (GI), type II resin-modified glass ionomer (RMGI), resin-based composite (RBC), bulk-fill (BF) resin-based composite, and a control with no box elevation procedure. Specimens were prepared for a standard mesio-occlusal-distal, computer-aided design/computer-aided manufactured (CAD-CAM) resin, nanoceramic onlay with mesial cervical margins located 1 mm above the cementoenamel junction (CEJ) and distal cervical margins located 2 mm below the CEJ. PBE was used to elevate the distal margins to 1 mm above the CEJ in all groups except the control group. For the control group the onlay margin was placed directly on the prepared distal tooth structure without PBE. A Lava Ultimate CAD/CAM resin, nanoceramic onlay restorative was manufactured and bonded on all specimens with RelyX Ultimate adhesive resin cement. The quality of the

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tooth-PBE material and PBE material-onlay interface was evaluated with scanning electron microscopy using epoxy resin replicas before and after cyclic loading (100,000 cycles, 1.2 Hz at 65N and 37°C). In addition to margin quality, the fracture resistance of each group was measured using a universal testing machine. Fracture pattern was recorded by visual examination. The Levene test for homogeneity and the Welch analysis of variance were completed for fracture resistance and margin quality. A χ^2 test was completed for break mode.

Results: For dentin margins, a statistically significant difference was detected between the RMGI and control groups at baseline (p=0.0442). All other groups—GI, RBC, and BF—showed no difference from the control at baseline (p>0.05). No statistical significance was observed among groups for post-cyclic fatigue (p=0.8735). For onlay margins, no statistical significance was observed among groups for pre-cyclic fatigue, post-cyclic fatigue, or change (p=0.9713, p=0.528, p=0.4385, respectively). No significant difference was observed for the fracture resistance among groups or for the type of break by material used (p=0.1593, p=0.77, respectively).

Conclusion: Within the parameters of this study, after mechanical fatigue, the materials used for PBE: RMGI, RBC, and BF, did not influence results in terms of margin quality and fracture resistance. Therefore, collective findings suggest that these materials might be suitable for PBE procedures. Nevertheless, clinical caution is recommended with any PBE procedure and further testing of GI materials is needed.

INTRODUCTION

Every dentist faces challenging clinical decisions when planning and restoring severely damaged teeth. Deep proximal surface destruction presents additional restorative complexities. With the lack of enamel for durable adhesive bonding, the presence of root concavities, and gingival tissue interferences, clinicians might elect adjunctive procedures when restoring teeth with deep proximal boxes. Surgical crown lengthening or orthodontic extrusion provide predictable restorative outcomes in teeth with deep surface destruction. Considering all possible restorative options delivers treatment focal to the needs of the patient. To simplify the restoration process, it is typically recommended that teeth with damage below the gingiva undergo surgical crown lengthening.[1] A conservative alternative to the former procedure is the proximal box elevation (PBE) technique. The PBE technique was initially purposed by Dietschi and Spreafico.[2] PBE has been revisited and refined by several authors.[3-13] Placing indirect prosthesis margins on direct restorative materials has been suggested for use in deep anterior Class III and V restorations as well.[14]

In certain clinical situations, the PBE procedure may be added to the list of possible adjunctive procedures for the patient and clinician to choose from. The PBE procedure has the potential to save time, resources, and biologic tissue. Additional benefits of placing indirect restoration margins on an elevated margin using direct materials are noted in the literature.[3-13] Indirect restoration preparation and delivery have inherent complexities, especially for onlays and inlays, which can be further complicated by deep proximal defects.[7,8] When utilizing PBE, a simplified preparation design gives rise to more manageable tooth and restoration intaglio surfaces. PBE facilitates impressions, rubber dam isolation, and clean-up for bonded restoration delivery.[3-13,15] Lastly, some publications report that PBE performs similar, in terms of margin quality and strength, to restorations placed without PBE.[3-13]

Computer-aided design/computer-aided manufactured (CAD/CAM) resin nanoceramics used in conjunction with the PBE technique offer the possibility to conserve tooth structure, improve esthetics, minimize cost, and ease adjustment and reparability; the approach also results in minimal enamel wear rates with CAD/CAM or traditional ceramic-based restorations.[7,8,12]

According to the literature, PBE is typically completed with resin-based composite (RBC) and a bonded occlusal indirect restoration. An alternative box elevation material, one that is water-based, hydrophilic, and historically placed in the subgingival area in conjunction with the open-sandwich technique (OST) is logical to implement when performing PBE.[16-18] Current literature on PBE using RBC makes no mention of the inconspicuous fluid environment or the required matrix adaptation when placing material subgingivally during PBE. [3-13] These are details to consider when justifying the clinical significance of *in vitro* studies.

Clinical advantages and disadvantages of type II glass ionomer (GI) materials to that of RBC are known, but the performance of GIs in place of RBCs

in the PBE function needs to be investigated. When used properly, reported performance of materials in the GI family were comparable to RBC in the OST.[16-18] It is commonly accepted that GIs possess several benefits over current RBC systems. These include, but are not limited to; chemical adhesion to tooth structure, fluoride release, stable microtensile bond strength with moisture contamination, pulpal biocompatibility, comparable elastic modulus to dentin, *Streptococcus mutans* resistance, biocompatibility to periodontal tissues, lower contraction stress, and the self-polymerizing benefit specific to GI.[16,19-21]

Questions remain when placing a restorative material beneath a milled restoration regarding the margin quality durability and strength of direct restorative materials suitable for box elevation procedures. This study investigates the effect of four PBE materials on the fracture resistance and margin quality of molar teeth restored with resin, nanoceramic CAD-CAM onlays following mechanical cyclic fatigue.

METHODS AND MATERIALS

Sample Preparation

Seventy-five, caries-free first or second human mandibular molar teeth were procured and stored in a 5000 ppm chloramine-thymol solution. Selection criteria were caries-free intact mandibular molars.

Using a universal mounting device, all 75 specimens were mounted in clear acrylic resin (Great Lakes Orthodontics, Tonawanda, NY, USA) at a level 3-mm apical to the specimen cementoenamel junction (CEJ). Prior to mesio-occlusal distal (MOD) preparation, all specimens were reduced occlusally with a wheel diamond (863C, Two Striper, Burs-Premier Dental USA, Plymouth Meeting, PA, USA) until the distance from the CEJ to the prepared occlusal surface was 4 mm. Indirect CAD-CAM MOD onlay preparations were completed on all specimens by a single clinician using depth cutting (DC1.0, DC1.5), egg shape (287.4 fine) and tapered diamonds (712.3KR, 703.8KR, Two Striper). The buccal to lingual isthmus preparation dimension was prepared to a width of one-half of the intercuspal distance and a pulpal depth of 1 mm. For both the mesial and distal box preparations, the axial wall depth at the gingival floor was 1.5 mm measured from the cavo-surface margin to the axial wall, and the buccal-lingual extent of the box measured 3 mm at the gingival floor. The mesial gingival floor was located 1 mm occlusal to the CEJ, and the distal gingival floor was located 2 mm apical to the CEJ to test different restorative materials for PBE. All internal angles were rounded and smoothed to optimize optical impressions, machined onlay milling, and seating.

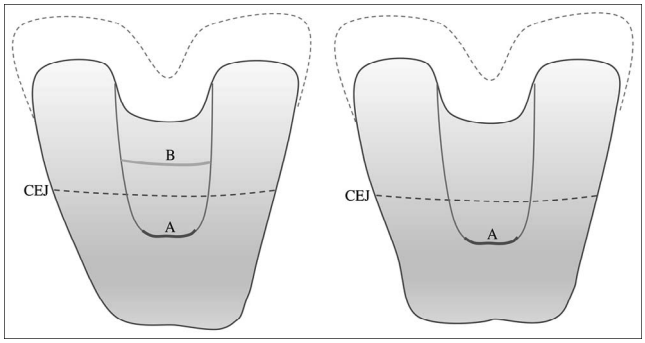

Figure 1. *Schematic of margin locations for experimental (left) and control group (right).*

Bonding Procedure for Proximal Box Elevation

Specimens were randomly assigned to one of five groups (n=15) according to the restorative material used for the distal PBE. The PBE restoration material groups were as follows: GI group (Fuji IX, GC America, Chicago, IL, USA) placed in a single 3-mm increment, resin-modified glass ionomer (RMGI) group (Fuji II LC, GC America) placed in two 1.5-mm increments, resin-based composite (RBC) group (Filtek Supreme Ultra, 3M ESPE, St Paul, MN, USA) placed in two 1.5-mm increments, bulk-fill (BF) group (Filtek Bulk Fill Posterior Restorative, 3M ESPE) placed in a single 3-mm increment, and control group (no PBE).

Specimens in the GI, RMGI, RBC, and BF groups (Figure 1) underwent PBE of the distal box to raise the gingival margin 3 mm, resulting in a material gingival floor location 1 mm occlusal to the CEJ using Tofflemire matrix bands (Henry Schein, Melville, NY, USA). For specimens in the GI and RMGI groups, according to manufacturer instructions, Cavity Conditioner (GC America) was applied and rinsed; materials were then injected into the distal boxes with nominal manipulation to minimize voids. The GF material in GI group specimens was allowed to self-polymerize for 6 minutes, while the RMGI material in the RMGI group specimens received light polymerization for 20 seconds from the occlusal; after removal of the matrix band the material was cured for 20 seconds each from the distal, buccal, and lingual. All polymerization performed in this study was accomplished using a Valo light-curing (LC) unit

(Ultradent, South Jordan, UT, USA) (20 seconds, 18 J/cm^2 at 0 mm). Following the manufacturer instructions, the distal boxes of specimens in the RBC and BF groups were selectively etched with Scotchbond Universal Etchant (3M ESPE) (32% phosphoric acid etchant), rinsed and dried, and coated with Scotchbond Universal Adhesive (3M ESPE). The Scotchbond layer received a 10-second LC unit polymerization time. The RBC and BF resin-based composite materials were placed in the distal boxes and polymerized for 20 seconds from the occlusal per increment. After the matrix band was removed, the material was polymerized for 20 seconds each from the distal, buccal, and lingual.

To aid the CAD-CAM workflow, the gingival floor of the PBE material was reduced and flattened to a level 1 mm occlusal to the CEJ using a flat-end cylinder diamond bur (515.7 fine) (Two Striper Burs, Premier Dental, USA). To facilitate visualization of the dentin to box-up material interface when using scanning electron microscopy (SEM), the distal-proximal surface of the PBE material was polished with a series of Sof-Lex Extra-Thin Contouring and Polishing Discs (coarse, medium, and fine) (3M ESPE). To ensure that all specimens had minimal margin discontinuity, all margins were evaluated for defects using 3.5× loupe magnification and tactile exploration with a sharp explorer. If any specimen had a detectable margin visually or with an explorer, the PBE elevation procedure was repeated until the margin was continuous. Specimens were stored in artificial saliva during the onlay fabrication process as described in the sections that follow.

Digital Impression, Design, Processing, and Bonding of Onlay

All onlay preparations were optically impressed and digitally designed using the Cerec Omnicam acquisition unit (CEREC AC, software package 4.4.3, Dentsply/Sirona, York, PA, USA). Onlay design mode was set to biogeneric copy. Occlusal schemes were pulled from the 4.4.3 software package library. Each onlay design had an occlusal thickness of 1.5 mm at the central fossa. Lava Ultimate onlays (LAVU) (n=75) (3M ESPE) were fabricated for each specimen. Size 14, A2 LAVU blocks were manufactured with the Cerec MC XL unit (Dentsply/Sirona). Once manufactured, sprues were removed with a coarse Sof-Lex Extra-Thin Contouring and Polishing Disc. The onlay occlusal surfaces were polished with a soft Abbott-Robinson bristle brush (Brasseler, Savannah, GA, USA) and Enamelize polishing paste (Cosmedent, Chicago, IL, USA). Each onlay was dried, then microetched with 50-μm Al_3O_2 at 30 psi (3M ESPE) until the intaglio surface appeared matte. All onlays were steam cleaned and dried; then 70% EtOH was applied and allowed to evaporate. The intaglio surface of each onlay was treated with Scotchbond Universal Adhesive, air thinned and received no light polymerization.

Following manufacturer's directions, all specimens received selective etching with a Scotchbond Universal Etchant (32% phosphoric acid), then rinsed and dried. Scotchbond Universal Adhesive was applied to all specimens, dried, and then polymerized for 10 seconds using a Valo LC unit. Onlays were bonded with RelyX Ultimate (3M ESPE) resin cement which was injected on the tooth and the onlay intaglio surface. Onlays were seated with finger pressure, then tack polymerized for 2 seconds. The excess cement was removed with a sickle scaler; then, the onlays were polymerized for 20 seconds on each of the five surfaces: occlusal, lingual, buccal, and both proximal surfaces.

Replica Fabrication and Margin Analysis

All specimens were stored in artificial saliva at 37°C for 24 hours prior to pre-fatigue replica fabrication. After 24 hours the distal proximal surfaces of all 75 specimens were cleaned with EtOH, dried, and impressed with Exaflex Puddy (GC America). Extrude light body (Kerr, Orange, CA, USA) was placed over the distal surfaces of each specimen then replacement of the Exaflex Putty matrix was completed with finger pressure. The Extrude light body material was allowed to polymerize and then was removed from the specimen following manufacturer's recommendations.

The impressions were then allowed to fully polymerize over 12 hours. After that period each impression was poured with Epoxicure epoxy resin (Buehler, Lake Bluff, IL, USA). After removing replicas from impressions, they were trimmed, cleaned with EtOH and placed on SEM stubs (Ted Pella, Redding, CA, USA). Following gold-sputtering with a Gold Sputter K550 (Emitech Ltd, Ashford, England) the replicas were evaluated under SEM (200×) with a S-4800 electron microscope (Hitachi High-Technology Corporation, Tokyo, Japan). Specimens were observed for initial margin quality. Margin qualities were classified to have a continuous margin, gap/irregularity, or a not judgeable artifact according to a protocol described by Frankenberger and others.[22] The percentage of continuous margin (in length, complete margin continuity) in relation to the entire judgeable margin was calculated as a

Table 1: *Percentage of Continuous Margin Quality in Dentin by Groups*[a]

Material	N	Dentin Pre %Continuous Mean (SD)	Dentin Post %Continuous Mean (SD)	Post-Pre %Change in Margin Quality Mean (SD)
GI	15	91.3 (11.5)AB	88.2 (16.7)A	−3.1 (12.3)A
RMGI	15	85.7 (22.6)A	93.5 (8.9)A	+7.9 (15.3)B
RBC	15	94.9 (9.7)AB	92.9 (11.3)A	−2.0 (3.7)AB
BF	15	96.2 (7.5)AB	93.1 (9.0)A	−3.2 (5.6)A
Control	15	98.8 (2.4)B	92.1 (8.2)A	−6.7 (7.9)A

Abbreviations: BF, bulk-fill; GI, glass ionomer; RBC, resin-based composite; RMGI, resin-modified glass ionomer.
[a] *Same letter in columns denotes no statistical difference.*

percentage. For specimens in the four intervention groups, two different interfaces were evaluated on the distal surfaces: B) between the tooth and the direct restorative material and, A) between the direct restorative material and the onlay restoration for the experimental groups (Figure 1). For specimens in the control group, one interface was evaluated on the distal surface: A) between the tooth and the onlay restoration (Figure 1).

Cyclic Fatigue

To simulate the clinical environment, the specimens were randomized to one of four stations within a wear instrument (Modified University of Alabama wear simulator) and submitted to mechanical loading under a 65 N, 1.2 Hz cyclic load for 100,000 cycles in a water bath at a constant 37°C. The load that was designated is associated with higher than normal chewing forces. The load was applied at the onlay central fossa with a 4-mm steel sphere. Afterward, to evaluate the post-fatigue margin quality, replicas were fabricated and evaluated with SEM, as described previously.

Fracture Resistance Testing

To evaluate levels of failure, all specimens were loaded until failure with a Universal 10kN Zwick instrument (Zwick/Roell, Ulm, Germany). Force was applied at the onlay central fossa with an antagonist identical to the sphere used for mechanical fatigue (4-mm steel sphere, 0.5 mm/min crosshead speed, at 0° to the long axis of the tooth). The fracture resistance and mode were recorded. Fracture modes were either catastrophic failure (fracture of specimen surface at or below clear acrylic resin or within root surfaces), combined fracture of coronal tooth structure and restoration, or fracture of the restoration.

Statistical Analysis

Statistical analysis was performed for margin quality among groups initially and after mechanical loading, for fracture resistance and type of break. The Levene test for homogeneity one-way analysis of variance (ANOVA) of squared deviations from group means was used to test for normal distribution of fracture resistance and onlay and dentin margin quality variance. Least squares means were completed for effect of material. The type of break was analyzed by material using the χ^2 test. The Welch ANOVA was performed for comparisons at the 95% level for margin quality.

RESULTS

Regarding dentin margins (Table 1), a statistically significant difference was detected between the RMGI group and the control group for pre-cyclic fatigue dentin margins ($p=0.0442$). This finding indicated that the margin quality was significantly lower for the RMGI group than the control group at baseline. All other groups (GI, RBC, and BF) showed no difference in dentin margin quality compared with the control group at baseline ($p>0.05$). The RMGI group was not statistically significantly different than the GI, RBC, and BF groups at baseline ($p>0.05$). No statistical significance was observed among groups for post-cyclic fatigue ($p=0.8735$). In terms of change in dentin margins (post-pre), all materials and the control group had comparable decreases in continuous margins except for the RMGI group ($p=0.0443$). RMGI showed a statistically significantly positive mean value for continuous margins percent change, indicating improved margins after fatigue cycling, whereas all other groups showed a decline or a negative mean value for continuous margins ($p<0.05$). However, RMGI and RBC were not statistically different from one another.

The results for onlay margin quality can be viewed in Table 2. Concerning onlay margins (margin located between onlay and PBE material), no statistically significant difference was observed among groups for pre-cyclic fatigue, post-cyclic

Table 2: *Percentage of Continuous Margin Quality for Onlay by Groups*[a]

Material	N	Onlay Pre %Continuous Mean (SD)	Onlay Post %Continuous Mean (SD)	Post-Pre %Change in Margin Quality Mean (SD)
GI	15	98.5 (4.89)A	97.8 (6.7)A	−0.7 (1.9)A
RMGI	15	99.0 (3.2)A	99.3 (1.8)A	0.3 (1.7)A
RBC	15	98.6 (3.8)A	98.0 (3.9)A	−0.5 (1.0)A
BF	15	98.4 (4.1)A	98.1 (4.1)A	−0.3 (0.6)A

Abbreviations: BF, bulk-fill; GI, glass ionomer; RBC, resin-based composite; RMGI, resin-modified glass ionomer; SD, standard deviation.
[a] *Same letter in columns denotes no statistical difference.*

fatigue, or the change (post-pre percentage) (p=0.9713, p=0.528, p= 0.4385, respectively). However, the RMGI group did have a small amount of improved margins after cycling compared with the other groups, which all had declines in continuous margins.

The results for fracture resistance are shown in Table 3. No statistically significant difference was observed for fracture resistance among groups or fracture mode by material used (p=0.1593 and p=0.77, respectively). In terms of fracture mode, only two specimens had a break type within restoration/tooth structure. These specimens were combined with the break type of restoration to allow for χ^2 test evaluation.

DISCUSSION

Our study investigated the effects of four different materials, two RBCs and two restorative GI- based materials, when used in the PBE technique. Margin quality and fracture resistance were the outcome measures to evaluate each material's performance following mechanical cyclic fatigue. Overall, the material used for proximal box elevation had no effect on margin quality, fracture resistance, or fracture mode.

In terms of dentin margin quality, the RMGI group showed an unexpected positive value for change, 7.9% (Table 1). Percent change was calculated by subtracting the post-fatigue continuous margin percent from that of baseline for each group. This finding indicates that pre-fatigued RMGI margins were less continuous than after specimens underwent 100,000 cycles of fatigue. It was not uncommon for RMGI specimens to appear distended following fatigue. This result may be explained by RMGI's hygroscopic expansion when placed in water.[23] It can be theorized that the RMGI specimens underwent a greater degree of hygroscopic expansion than the other materials, resulting in reduction of some marginal defects (Figure 2a,b).[24,25] The reasons of this material specific outcome for RMGI may be due to its hydroxyethyl methacrylate (HEMA) content. The HEMA monomer is known to be unstable and could have contributed to some expansion.[26] Current literature shows that RBCs, GIs, and compomers initially shrink, then undergo some measure of hygroscopic expansion with subsequent reduction in marginal defects. All other groups showed an expected overall negative value for change in terms of margin quality following cyclic fatigue (Figure 3a.b). This finding is consistent with published studies on PBE (Table 1).[3-13]

The RMGI group had the only positive percentage value for change and fewest margin post-fatigue defects at dentin margins. At first glance, one might conclude that RMGI is the material to use in PBE; however, we must consider other factors and materials. Following the RMGI group was the RBC group, which showed a –1.95 % change in margin

Table 3: *Results for Fracture Resistance and Mode of Failure*[a]

Material	N	Fracture Resistance Mean (SD)	CAT Frequency/%	REST Frequency/%
GI	15	1968.5 (505.6)A	10/66.7	5/33.3
RMGI	15	1700.6 (308.4)A	7/46.7	8/53.3
RBC	15	1968.3 (458.2)A	7/46.7	8/53.3
BF	15	2029.9 (478.5)A	9/60.0	6/40.0
Control	15	1843.8 (440.7)A	8/53.3	7/46.7

Abbreviations: BF, bulk-fill; CAT, catastrophic failure; GI, type II glass ionomer; RBC, resin-based composite; REST, restoration failure; RMGI, resin-modified glass ionomer; SD, standard deviation.
[a] *Same letter in columns denotes no statistical difference.*

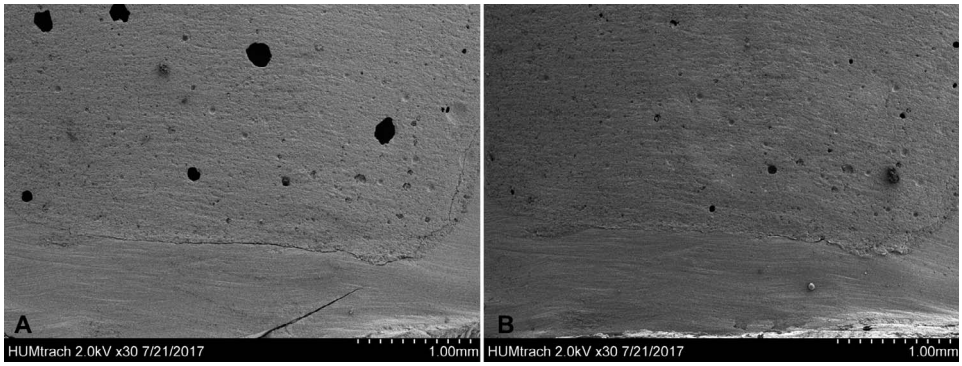

Figure 2. *(a): Resin-modified glass ionomer baseline. (b): Resin-modified glass ionomer post fatigue.*

quality (Table 1). Interestingly, neither post-fatigue nor change calculations for the RBC group were significantly different from any other group ($p>0.05$). In an optimal clinical situation, these results may give reasonable rationale to use RBC over other materials in PBE.

In comparing dentin margin continuity from this study to other studies on PBE using Lava Ultimate (CAD/CAM resin, nanoceramic restorative) results from Ilgenstein and others were similar in magnitude to our findings.[4] They restored teeth with and without resin composite PBE then either a Vitablock Mark II or Lava Ultimate onlay. Our study and that of Ilgenstein present a –6 to –11% range change in dentin margins. In contrast to our control group showing the lowest margin quality, as calculated with change (Table 1), Ilgenstein's Lava Ultimate group without box elevation had the highest margin quality. Ilgenstein attributes this result to the shock-absorbing capability of Lava Ultimate. We suggest that our luting agent was more susceptible to degradation than the other restorative materials used at the dentin margin, resulting in excessive leaching of cement in control specimens from the dentin margins and consequential dentin margin breakdown. This observation, however, was not found to be statistically significant ($p>0.05$).

At the onset of our study we selected the exclusive use of Lava Ultimate, rather than ceramic or a combination of onlay materials, for clearer and streamlined analyses, resulting in sound results. The decision to use Lava Ultimate exclusively was also made after a thorough evaluation of published literature evaluating box elevation. Interestingly, most box elevation studies showed indirect resin restorations to be comparable or outperform ceramic restorations in terms of dentin margins following fatigue.[3-6,9] A prime example of this observation brings us back to Ilgenstein and others whose specimens restored with or without box elevation and a ceramic onlay performed worse than Lava Ultimate in terms of dentin margin quality.[4] They noted that composite resin has the ability to absorb and transfer energy more effectively than ceramic, resulting in less stress at tooth-restoration margins.

Regarding onlay to restorative material margin quality, no statistically significant difference was observed among groups ($p>0.05$). Here again, the RMGI group showed a positive value for change 0.3% (Table 2). This finding is consistent with RMGI margins in dentin previously discussed. All other

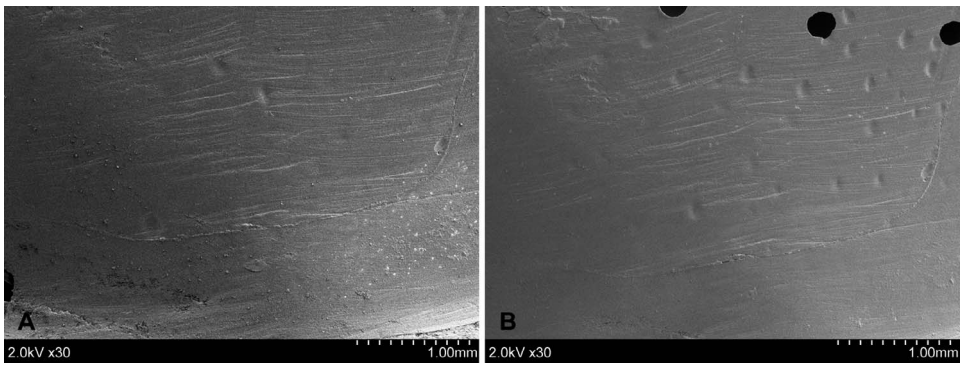

Figure 3. *(a): Resin-based composite baseline. (b): Resin-based composite post fatigue.*

groups showed a change in margin continuity between –0.7 and –0.3% (Table 2). These values were consistent with results of other studies using Lava Ultimate.[4,12]

PBE with the BF composite resulted in the second-highest margin quality post-fatigue at dentin, and it also showed the greatest fracture resistance; however, these findings were not statistically significant in comparison to the values from other groups (Tables 1 through 3). No studies to date have evaluated BF composite in conjunction with the PBE technique. This outcome may be attributed to the material's simplicity of placement, optimized consistency, and minimal instrument pullback. For these reasons, assuming ideal clinical conditions, BF composite may be a clinician's material of choice when performing PBE.

Only one other study utilized fracture resistance as an outcome measure for PBE, Ilgenstein and others.[4] For comparison purposes, they reported the fracture resistance of nanoceramic indirect onlays with no PBE to be higher than those with PBE; however, no statistically significant difference was found. This finding was attributed to the different stress patterns induced by the long distal wing extension in specimens without PBE. All specimens in our study fractured within the same range 1700.6–2029.9 N as in the study above, including GI and RMGI proximal elevation specimens (Table 3). Fracture resistance, regardless of PBE material used, was similar to the control group; therefore, PBE within this study's parameters may resist a maximum bite force of 600–1200 N and withstand forces during normal mastication.[27]

Mode of fracture results are presented in Table 3. No statistically significant difference in mode of fracture was detected in this study ($p>0.05$). Overall, most specimens fractured catastrophically. The GI group had the most catastrophic failures and the least failures within the restoration. This may be attributed to the chemical bond of GI to dentin. These findings parallel the results of Ilgenstein in terms of mode of fracture, where overall most specimens fractured catastrophically.

One limitation of this study was the type of fatigue cycling used. We used a 65 N, 1.2 Hz cyclic load for 100,000 cycles in a water bath at constant 37°C. According to the literature, this simulates only about 3 months of chewing.[28] Current literature evaluating PBE reports 100,000–1,200,000 cycles of fatigue. Even though no set international thermocycling parameter has been agreed upon, a handful of studies implemented additional or simultaneous thermocycling of specimens from 5°C to 55°C.[3-5,29] We did not subject our specimens to thermocycling. Adding a thermocycling element to our methodology would have made our results more generalizable when comparing with other studies.

In the same vein, we were unable to fully simulate the oral environment and clinical realities of restoration placement. The main reason for implementing the PBE technique in daily practice is to eliminate the inherent difficulty of capturing a deep margin with an impression, optically or otherwise. The patient variable, necessity of gingival tissue control, material placement and restricted access were aspects our methodology did not assess. Ideal conditions (ie, contamination free and uninterrupted access) were used during specimen preparation. Depending on the clinical situation, materials like GI or RMGI may offer better clinical success due to their moisture forgiveness and chemical adhesion to dentin.[16,18,21] However, caution is recommended in extrapolating our findings to clinical situations.

Future investigation is recommended before specific protocols of proximal box elevation can be universally recommended in patients. Laboratory results encourage the success of PBE, but a clinical trial would bring reliable box elevation outcomes to the forefront. Disadvantages of PBE shown in the literature are most recently noted in an *in vivo* 12-month study showing increased bleeding on probing associated with the procedure.[15] Ferrari and others used flowable RBC to elevate margins from below the CEJ, followed by indirect cuspal restorations.[15] Teeth with PBE were compared with control teeth, which underwent no PBE, and indirect restoration margins were placed below the CEJ. No GI type materials were utilized for box elevation in the Ferrari and others study, possibly giving reason for further investigation of type II GIs in this role.

CONCLUSION

Within the parameters of this study, following mechanical fatigue, the materials used for PBE: RMGI and GI, RBC and BF composite, did not influence results in terms of margin quality and fracture resistance. Therefore, collective findings suggest that any of these materials might be suitable for PBE procedures. Nevertheless, clinical caution is recommended with any PBE procedure and further testing of GI materials is needed.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Iowa. The approval code for this study is 201709826.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Schmidt JC, Sahrmann P, Weiger R, Schmidlin PR, & Walter C (2013) Biologic width dimensions—a systematic review *Journal of Clinical Periodontology* **40(5)** 493-504.
2. Dietschi D & Spreafico R (1998) Current clinical concepts for adhesive cementation of tooth-colored posterior restorations *Practical Periodontics and Aesthetic Dentistry* **10(1)** 47-54; quiz 56.
3. Frankenberger R, Hehn J, Hajto J, Kramer N, Naumann M, Koch A, & Roggendorf MJ (2013) Effect of proximal box elevation with resin composite on marginal quality of ceramic inlays in vitro *Clinical Oral Investigations* **17(1)** 177-183.
4. Ilgenstein I, Zitzmann NU, Buhler J, Wegehaupt FJ, Attin T, Weiger R, Attin T, Weiger R, & Krastl G (2015) Influence of proximal box elevation on the marginal quality and fracture behavior of root-filled molars restored with CAD/CAM ceramic or composite onlays *Clinical Oral Investigations* **19(5)** 1021-1028.
5. Roggendorf MJ, Kramer N, Dippold C, Vosen VE, Naumann M, Jablonski-Momeni A, & Frankenberger R (2012) Effect of proximal box elevation with resin composite on marginal quality of resin composite inlays in vitro *Journal of Dentistry* **40(12)** 1068-1073.
6. Zaruba M, Gohring TN, Wegehaupt FJ, & Attin T. Influence of a proximal margin elevation technique on marginal adaptation of ceramic inlays *Acta Odontologica Scandinavica* **71(2)** 317-324.
7. Dietschi D & Spreafico R (2015) Evidence-based concepts and procedures for bonded inlays and onlays. Part I. Historical perspectives and clinical rationale for a biosubstitutive approach. *International Journal of Esthetic Dentistry* **10(2)** 210-227.
8. Rocca GT, Rizcalla N, Krejci I, & Dietschi D (2015) Evidence-based concepts and procedures for bonded inlays and onlays. Part II. Guidelines for cavity preparation and restoration fabrication *International Journal of Esthetic Dentistry* **10(3)** 392-413.
9. Zaruba M, Kasper R, Kazama R, Wegehaupt FJ, Ender A, Attin T, & Mehl A (2014) Marginal adaptation of ceramic and composite inlays in minimally invasive mod cavities *Clinical Oral Investigations* **18(2)** 579-587.
10. Da Silva Goncalves D, Cura M, Ceballos L, & Fuentes MV (2017) Influence of proximal box elevation on bond strength of composite inlays *Clinical Oral Investigations* **21(1)** 247-254.
11. Muller V, Friedl KH, Friedl K, Hahnel S, Handel G, & Lang R (2017) Influence of proximal box elevation technique on marginal integrity of adhesively luted Cerec inlays *Clinical Oral Investigations* **21(2)** 607-612.
12. Spreafico R, Marchesi G, Turco G, Frassetto A, Di Lenarda R, Mazzoni A, Cadenaro M, & Breschi L (2016) Evaluation of the in vitro effects of cervical marginal relocation using composite resins on the marginal quality of CAD/CAM crowns *Journal of Adhesive Dentistry* **18(4)** 355-362.
13. Rocca GT, Gregor L, Sandoval MJ, Krejci I, & Dietschi D (2012) In vitro evaluation of marginal and internal adaptation after occlusal stressing of indirect class II composite restorations with different resinous bases and interface treatments. "Post-fatigue adaptation of indirect composite restorations" *Clinical Oral Investigations* **16(5)** 1385-1393.
14. Metz M, Durski M, Chou J, Crim G, Harris B, & Lin WS (2016) Microleakage of lithium disilicate crown margins finished on direct restorative materials *Operative Dentistry* **41(5)** 552-562.
15. Ferrari M, Koken S, Grandini S, Cagidiaco EF, Joda T, & Discepoli N (2018) Influence of cervical margin relocation (CMR) on periodontal health: 12-month results of a controlled trial *Journal of Dentistry* **69(2)** 70-76.
16. Andersson-Wenckert IE, van Dijken JW, & Horstedt P (2002) Modified Class II open sandwich restorations: evaluation of interfacial adaptation and influence of different restorative techniques *European Journal of Oral Sciences* **110(3)** 270-275.
17. Dietrich T, Kraemer M, Losche GM, Wernecke KD, & Roulet JF (2000) Influence of dentin conditioning and contamination on the marginal integrity of sandwich Class II restorations *Operative Dentistry* **25(5)** 401-410.
18. van Dijken JW, Kieri C, & Carlen M (1999) Longevity of extensive class II open-sandwich restorations with a resin-modified glass-ionomer cement *Journal of Dental Research* **78(7)** 1319-1325.

19. Ronald L. Sakaguchi D, PhD & Powers JM (2012) *Craig's Restorative Dental Materials* **13th edition** Elsevier, Philadelphia PA.
20. Fritz UB, Finger WJ, & Uno S (1996) Resin-modified glass ionomer cements: bonding to enamel and dentin *Dental Materials* **12(3)** 161-166.
21. Shimazu K, Karibe H, & Ogata K (2014) Effect of artificial saliva contamination on adhesion of dental restorative materials *Dental Materials Journal* **33(4)** 545-550.
22. Frankenberger R, Strobel WO, Lohbauer U, Kramer N, & Petschelt A (2004) The effect of six years of water storage on resin composite bonding to human dentin *Journal of Biomedical Materials Research Part B Applied Biomaterials* **69(1)** 25-32.
23. Suiter EA, Watson LE, Tantbirojn D, Lou JS, & Versluis A (2016) Effective expansion: balance between shrinkage and hygroscopic expansion *Journal of Dental Research* **95(5)** 543-549.
24. Al Sunbul H, Silikas N, & Watts DC (2015) Resin-based composites show similar kinetic profiles for dimensional change and recovery with solvent storage *Dental Materials* **31(10)** e201-e217.
25. Rosales-Leal JI, Castillo-Salmeron RD, Molino-Serrano MA, Gonzalez-Moreira H, & Cabrerizo-Vilchez MA (2013) Effect of hygroscopic expansion of resin filling on interfacial gap and sealing: a confocal microscopy study *Journal of Adhesive Dentistry* **15(5)** 423-430.
26. Dursun E, Nguyen JF, Tang ML, Attal JP, & Sadoun M (2016) HEMA release and degree of conversion from a resin-modified glass ionomer cement after various delays of light activation *Dental Materials* **32(5)** 640-645.
27. Shinogaya T, Bakke M, Thomsen CE, Vilmann A, & Matsumoto M (2000) Bite force and occlusal load in healthy young subjects—A methodological study *European Journal of Prosthodontics and Restorative Dentistry* **8(1)** 11-15.
28. Barkmeier WW, Latta MA, Erickson RL, & Lambrechts P (2004) Comparison of laboratory and clinical wear rates of resin composites *Quintessence International* **35(4)** 269-274.
29. Morresi AL, D'Amario M, Capogreco M, Gatto R, Marzo G, D'Arcangelo C, & et al (2014) Thermal cycling for restorative materials: Does a standardized protocol exist in laboratory testing? A literature review *Journal of the Mechanical Behavior of Biomedical Materials* **29(Jan)** 295-308.

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Fluorescence-aided Composite Removal in Directly Restored Permanent Posterior Teeth

C Dettwiler • F Eggmann • L Matthisson • C Meller • R Weiger • T Connert

Clinical Relevance

The fluorescence-aided identification technique can be a useful and time-saving aid for the repair and replacement of direct composite restorations with the potential to preserve tooth substance and reduce the risk of treatment-related complications.

SUMMARY

Aim: The aim of this study was to quantitatively compare conventional composite removal and composite removal supported by the fluorescence-aided identification technique (FIT) regarding the completeness, selectivity, and duration of the procedure in directly restored permanent posterior teeth.

Methods and Materials: Two operators removed standardized direct class II composite restorations (n=32 per operator) in human tooth models under simulated clinical conditions. According to a randomized allocation scheme, removal was performed with either the conventional technique (contra-angle handpiece) or supported by FIT. The duration of each removal procedure was recorded. The completeness and selectivity were volumetrically assessed through superimposition of three-dimensional surface scans. Statistical significance was tested by examining the overlap of 95% confidence intervals (CI). Multiple comparison was performed with Tukey tests for each variable.

Results: Compared with the conventional technique, composite removal with FIT was faster (329 seconds [95% confidence interval (CI): 268-390 seconds] vs 179 seconds [95% CI: 150-208 seconds]), generated less tooth substance loss (4.53 mm^3 [95% CI: 3.77-5.30 mm^3] vs 2.77 mm^3 [95% CI: 2.11-3.43 mm^3]), and left behind less

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composite residue (1.58 mm^3 [95% CI: 1.23-1.94 mm^3] vs 0.53 mm^3 [95% CI: 0.39-0.67 mm^3]).

Conclusion: Within the limitations of this *in vitro* study, FIT facilitated the selective and expeditious removal of tooth-colored composites in directly restored posterior teeth.

INTRODUCTION

Composites are now the material of choice for direct posterior restorations because of their good clinical performance and esthetic properties.[1,2] However, restorations have a limited lifespan, with secondary caries and fractures being the predominant reasons for the failure of posterior composite restorations.[2,3] The replacement of failing and defective restorations is therefore a commonplace procedure: replacement restorations account for more than half of restorations placed by dentists.[1,4] Refurbishment and repair are restorative treatment approaches that, underpinned by a considerable body of evidence, can extend the lifespan of direct composite restorations.[5-8] Nevertheless, partially defective composite restorations are frequently treated with the replacement of the entire restoration.[2,9]

Removal of composite with an exact color match can pose a formidable challenge in restorative dentistry; inadvertent removal of adjacent dental hard tissue often is all but inevitable.[10] As a consequence, a cavity tends to increase in size with each invasive intervention, which may, in turn, negatively affect the long-term prognosis of a tooth.[4,11-13]

Different avenues have been explored to facilitate the selective removal of tooth-colored composites. For instance, carbon dioxide lasers guided by spectral feedback allow a higher level of ablation selectivity compared with the traditional composite removal with a high-speed handpiece.[14] Another approach, termed fluorescence-aided identification technique (FIT), uses the fluorescence properties of composites to make them more easily detectable and thus facilitate their selective removal.[10,15] Within the visible light spectrum, many composite materials have distinct fluorescence properties from dental hard tissues at wavelengths in the range of 405 $\pm$ 10 nm.[16] Consequently, the use of an illumination source emitting blue light in this range facilitates visually distinguishing between composite and tooth substance.[15] Recent studies have reported the successful application of FIT for orthodontic debonding procedures and the removal of composite bonded trauma splints.[17-19]

When providing replacement restorations and repairing partially defective restorations, it is crucial to avoid inadvertent removal of sound dental hard tissue. The aim of this *in vitro* study was therefore to quantitatively compare conventional composite removal and composite removal aided by FIT regarding the completeness, selectivity, and duration of the composite removal procedure in directly restored permanent posterior teeth.

METHODS AND MATERIALS

Model Preparation

From a pool of irreversibly anonymized human teeth, 32 permanent teeth were selected to produce a maxillary and mandibular model with a complete set of teeth. The teeth, stored in a 0.2% thymol solution, were free of caries, restorations, and significant signs of tooth wear. The setup of the dental arches was done in wax. An impression of the setup was taken with C-silicone putty material (Coltoflax, Coltène/Whaledent AG, Altstätten, Switzerland). The root ends were cut off, and a dowel pin (BI-PIN, Renfert GmbH, Singen, Germany) was fixed to each root with DuraLay (Reliance Dental Manufacturing LLC, Alsip, IL, USA). The model was produced according to the laboratory procedures required for a full arch master model with removable dies. The base of the model was cast with a self-curing denture base material (ProBase Cold, Ivoclar Vivadent AG, Schaan, Liechtenstein). The dowel pins attached to the root ends permitted either anchorage to the base or separate removal of each tooth from the base. A pink-colored gingiva mask was fabricated with an addition curing silicone (Gingiform 05410, BISCO Dental Products, Schaumburg, IL, USA).

Imaging

Optical surface scans were made with a five-axis dental laboratory scanner (inEOS X5, Dentsply Sirona Inc, York, PA, USA) at baseline and before and after each restorative procedure. The experimental design of the study is outlined in Figure 1. To ensure unobstructed scanning of the class II cavities, the neighboring teeth were removed from the base. Therefore, two scans per jaw model were made in each imaging session: maxillary/mandibular model with first premolars and first and third molars in place; maxillary/mandibular model with second premolars and second molars in place and canines removed.

Restorative Procedure

Before the cavity preparation, an impression of each posterior tooth was taken with a clear two-compo-

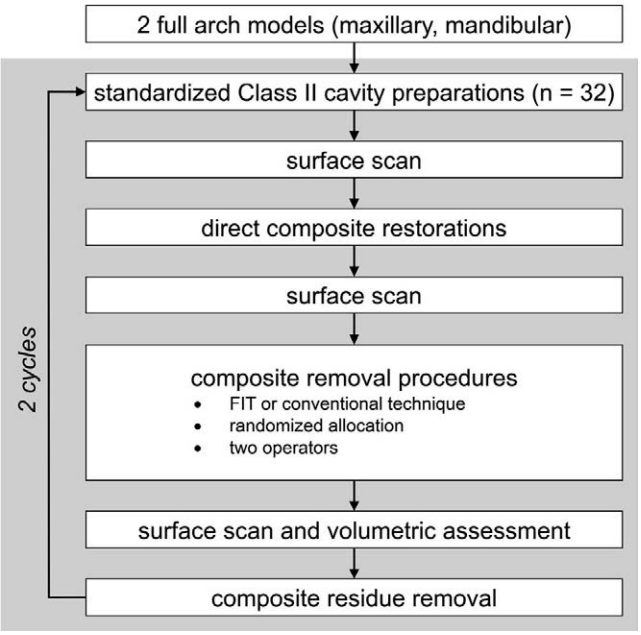

Figure 1. *Study flow diagram.*

nent silicone (SHERACRYSTAL, SHERA Werkstoff-Technologie GmbH & Co KG, Lemförde, Germany). The silicone impressions were trimmed to obtain keys with a uniform proximal thickness of about 3 mm. These silicone keys were used to provide direct composite restorations that replicated the original form of the proximal surface and marginal ridge.[20] To determine the base shade of the teeth, measurements were carried out on the central part of the proximal tooth surfaces with an intraoral spectrophotometer (VITA Easyshade V, VITA Zahnfabrik H. Rauter GmbH & Co KG, Bad Säckingen, Germany). The matching composite was selected for each class II cavity according to the corresponding shade measurement. Standardized class II cavities were prepared on the mesial and distal surfaces of all posterior teeth apart from the third molars for a total of 32 cavities. The cavities were prepared under constant water cooling with an ultrasonic preparation device (SIROPREP M2/D2 Standard, Dentsply Sirona Inc) in the region of the bucco-lingual position of the contact to the adjacent tooth. The approximate dimensions of the box-shaped cavities were as follows: the cavities were 2.7 mm wide in the bucco-lingual dimension, 1.6 mm mesio-distally, and 4 mm deep, with the gingival depth extending below the contact area. The gingival floor of the proximal boxes was located above the cementum-enamel junction. Cavity margins were not beveled.[21] The cavities were conditioned with a phosphoric acid etchant (Ultra-Etch, Ultradent

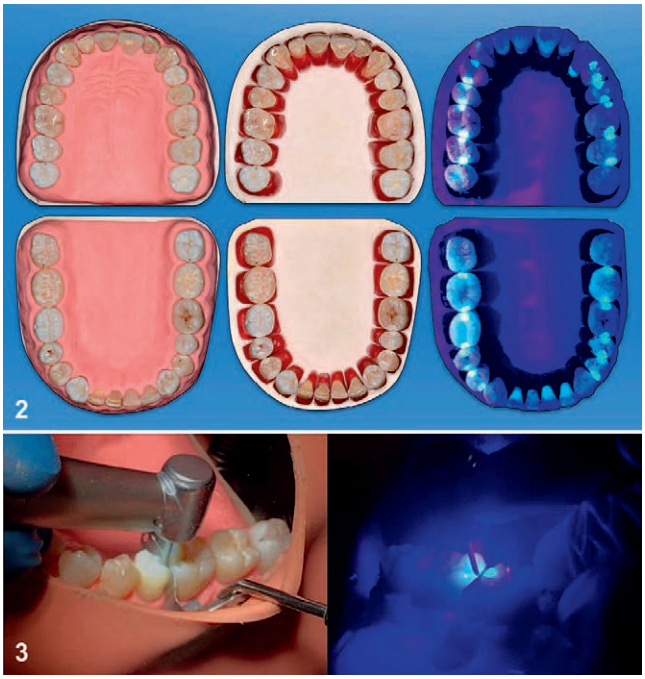

Figure 2. *Maxillary and mandibular full arch models with gingiva mask in place (left), with gingiva mask removed to show the separately removable teeth (middle), and under FIT illumination (right).*

Figure 3. *Conventional composite removal (left) and composite removal supported by FIT (right) under simulated clinical conditions.*

Products Inc, South Jordan, UT, USA), and an etch-and-rinse adhesive was applied according to the manufacturer's instructions (OptiBond FL, Kerr Italia Srl, Scafati, Italy). Light curing was performed for 20 seconds at 1200 mW/cm^2 (Bluephase, Ivoclar Vivadent AG). The pretreated class II cavities were filled in one increment with a nanohybrid direct composite material (IPS Empress Direct, Ivoclar Vivadent AG). Composite was applied in slight excess in the silicone key, which was then seated onto the tooth under constant finger pressure. Light curing was performed through the clear silicone key for 40 seconds at 1200 mW/cm^2 and after removing the silicone key for another 40 seconds at same output intensity (Bluephase, Ivoclar Vivadent AG). Excess material was removed with surgical scalpel blades (No. 12D, Gebrüder Martin GmbH & Co KG, Tuttlingen, Germany) under an operating microscope (OPMI PROergo, Carl Zeiss AG, Oberkochen, Germany). Figure 2 shows the model with and without the gingiva mask and under FIT illumination.

Operators 1 and 2 (FE and LM, respectively), two general dentists with normal vision, were tasked to completely remove the class II restorations without extending the cavities. Both operators had no color

vision deficiency, which was assessed beforehand with Ishihara plates. Operators 1 and 2 had four and two years of professional experience, respectively.

The 32 restorations were evenhandedly assigned to the operators: each operator was responsible for 16 restorations, with an equal distribution of tooth types (premolars, molars), maxillary and mandibular teeth, right and left teeth, and mesial and distal restorations. Each operator performed the removal procedures in two sessions. Between the sessions, the volumetric assessment was undertaken, and the removed restorations were replaced (details see below). Both operators removed each restoration conventionally and with FIT (ie, 2 × 16 removal procedures per operator; Figure 3). A randomized allocation scheme, generated with online freeware (www.randomizer.org), determined for each restoration which technique to use first.

To simulate a clinical situation, the models were mounted in a dental manikin (P-6, Frasaco GmbH, Tettnang, Germany) whose head position was vertically and laterally adjustable. A single-ended shepherd's hook explorer, a dental mirror, and a triple function syringe were placed at the disposal of the operators. For conventional composite removal, the operators used a high-speed contra-angle handpiece (1:5, KaVo Master Series, KaVo Dental GmbH, Biberach, Germany) with a set of piriform, cylindrical and flame-shaped diamond burs (ISO 314 235 524 010, ISO 314 157 524 011, ISO 314 158 504 013, and ISO 314 248 514 011, Intensiv SA, Montagnola, Switzerland). For composite removal with FIT, a modified micromotor that was equipped with a light source emitting blue light at a wavelength of 405 nm was used (MX2, Bien-Air Dental SA, Bienne, Switzerland; Power LED LZ1-00UB00-00U7, LED Engin Inc, San Jose, CA, USA), and the operators had the same contra-angle handpiece and set of burs at their disposal (Figure 3). To protect adjacent teeth from iatrogenic damage, preventive stainless-steel aids with a thickness of 0.2 mm were used during composite removal (InterGuard, Ultradent Products Inc). Clear protective glasses were worn during the removal procedures. The operators used neither optical magnification devices nor filter lenses. The time for each removal procedure was recorded.

After the first session, an investigator (CD) examined the cavities with a FIT illumination source under an operating microscope and removed composite remnants in cavities where the removal procedure did not achieve complete composite removal with the same ultrasonic preparation device as used for the initial preparations. The cavities were finished with the ultrasonic preparation device to obtain class II cavities of substantially the same dimensions as in the first session and to have finished tooth substance as adhesion substrate for the following composite restoration. The direct composite restorations were made following the very same procedure as described above.

Volumetric Assessment

Obtained data from the optical scans were uploaded as surface tessellation language (STL) files to the OraCheck Software (Version 2.13.8676, Cyfex AG, Zurich, Switzerland). Technique allocation was concealed to the investigators (CD, TC) who carried out the quantitative assessments with OraCheck software. The best-fit method was used to superimpose scans.[22] Each tooth surface of interest was separately selected and overlapped independently from other surfaces to obtain a more accurate superimposition. A software tool with a color-coded scale clearly visualized volumetric changes between the scans: green marked unchanged areas; blue and violet colors indicated substance loss; and yellow, red, and pink indicated excess material. The cavities were analyzed using software tools that performed linear and volumetric measurements of selected areas (Figure 4). Areas of interest (ie, the class II cavities) were selected and analyzed with the "volume analysis" tool. Volumes were measured in cubic millimeters. The "cursor-distance" tool was used to quantify hard tissue defects, defined as any loss of dentin and/or enamel, and composite remnants. The highest and lowest points were recorded followed by a volumetric measurement.

Statistical Analysis

For each operator and removal technique, a descriptive analysis regarding volume of tooth substance defects, volume of composite residue, and treatment duration was performed. Statistically significant differences were expressed by nonoverlapping 95% confidence intervals (CIs). Multiple comparison was performed with Tukey tests for each variable with the level of significance set at 5%. Statistical analyses were conducted with JMP 11 (SAS Institute Inc, Cary, NC, USA). The relevant datasets are available on request.

RESULTS

The mean preoperative volume of the composite restoration was 16.65 mm^3 (SD: 2.23; 95% CI: 15.85-17.45) for the conventional technique and 16.91 mm^3 (SD: 2.05; 95% CI: 16.17-17.63) for the FIT group.

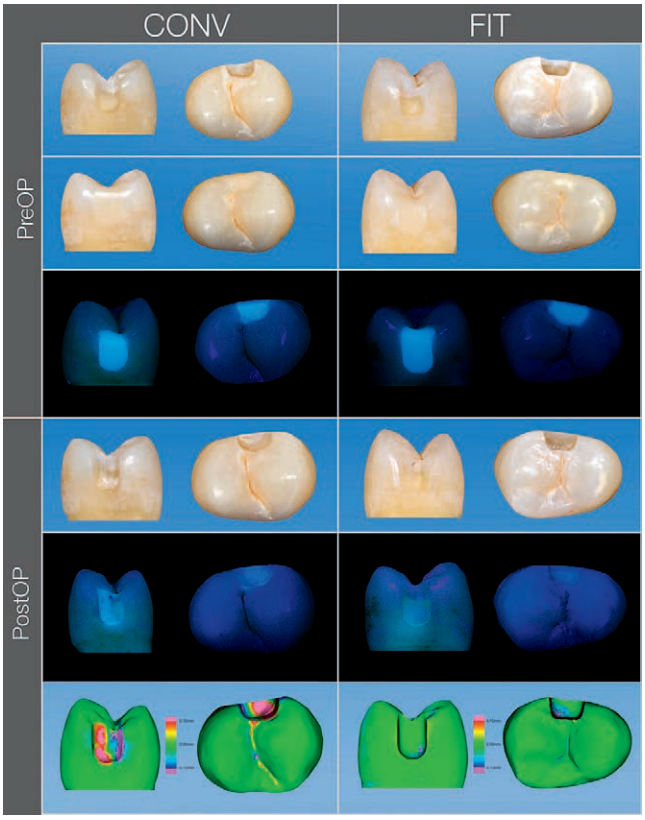

Figure 4. *Preoperative (PreOP) and postoperative (PostOP) images of the volumetric assessment of a premolar with the conventional technique (CONV, left) and with FIT (right).*

The mean volume of composite residue was 1.58 mm^3 (SD 0.99 mm^3; 95% CI: 1.23-1.94 mm^3) for the conventional technique and 0.53 mm^3 (SD: 0.39 mm^3; 95% CI: 0.39-0.67 mm^3) for FIT (Figure 5). The mean volume of dental hard tissue defects was 4.53 mm^3 (SD: 2.12 mm^3; 95% CI: 3.77-5.30 mm^3) for the conventional technique and 2.77 mm^3 (SD: 1.83 mm^3; 2.11-3.43 mm^3) for FIT (Figure 6). The mean duration of the removal procedure per restoration for was 329 seconds (SD: 169 seconds; 95% CI: 268-390 seconds) for the conventional technique and 179 seconds (SD: 80 seconds; 95% CI: 150-208 seconds) for FIT (Figure 7). Table 1 lists detailed results (overall and per operator) regarding volume of composite residue, maximum height of composite residue, percentage of composite removal, volume of defect, maximum depth of defect, and time.

DISCUSSION

The present *in vitro* study compared conventional composite removal with a fluorescence-aided approach. The results showed that FIT improved the selectivity of composite removal and resulted in fewer defects in the adjacent dental hard tissue.

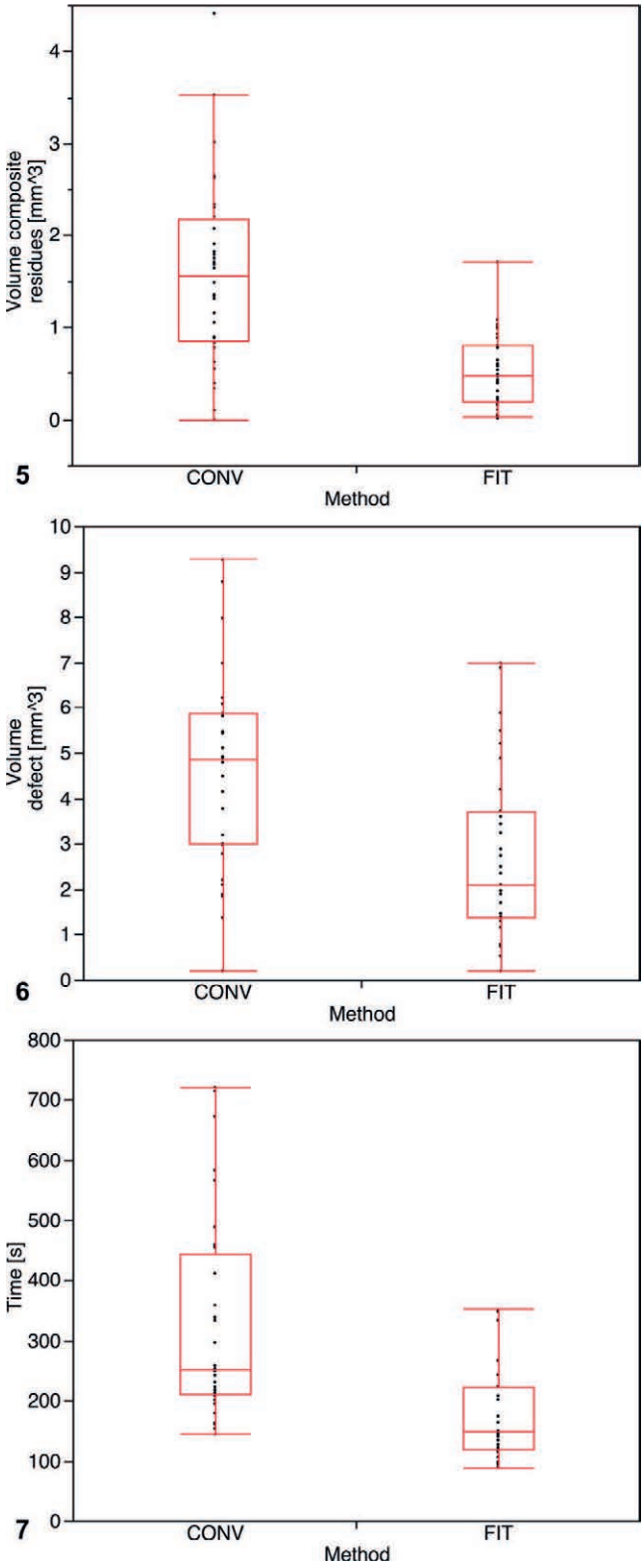

Figure 5. *Box plots of the volume of the composite residues (mm^3) for the conventional technique (CONV) and FIT.*
Figure 6. *Box plots of the volume of dental hard tissue defects (mm^3) for the conventional technique (CONV) and FIT.*
Figure 7. *Box plots of the treatment duration (seconds) per restoration for the conventional technique (CONV) and FIT.*

Table 1: *Mean Results (SD, 95% CI) Regarding Composite Residues (Volume, Height, and Percentage of Removal), Defect Volume and Depth, and Duration*[a]

Operator and Technique	Volume Composite Residue (mm^3)	Maximum Height Composite Residue (mm)	Percentage of Composite Removal (%)
Operator 1: conventional technique	1.18 (0.74; 0.79-1.58) B,C	0.72 (0.46; 0.47-0.96) A	92.99 (3.90; 90.92-95.07) B,C
Operator 1: FIT	0.37 (0.32; 0.20-0.54) D	0.23 (0.14; 0.16-0.30) B	97.82 (1.79; 96.87-98.78) A
Operator 2: conventional technique	1.99 (1.07; 1.41-2.56) A	1.05 (0.58; 0.74-1.36) A	88.12 (6.47; 84.67-91.56) D
Operator 2: FIT	0.68 (0.40; 0.47-0.90) C,D	0.24 (0.14; 0.17-0.31) B	95.93 (2.76; 94.46-97.40) A,B
Overall: conventional technique	1.58 (0.99; 1.23-1.94) A,B	0.88 (0.54; 0.69-1.08) A	90.55 (5.81; 88.46-92.65) C,D
Overall: FIT	0.53 (0.39; 0.39-0.67) D	0.24 (0.13; 0.19-0.28) B	96.88 (2.48; 95.83-97.77) A

[a] Tukey test: significant differences within each column are indicated by different superscript letters.

Furthermore, composite removal with FIT was less time-consuming than conventional composite removal procedures.

FIT makes use of illuminant metamerism, which describes the perceived color match of two materials with different spectral power distributions under certain illumination conditions but not under others.[10,15,23] The fluorescent behavior of the majority of composites is distinct from that of dental hard tissues.[16,24] Thus, the use of illumination sources with wavelengths in the range of 405 ± 10 nm makes it easy to visually distinguish between tooth substance and color-matched metameric composites.[15] FIT allows to quickly and accurately detect tooth-colored composite restorations.[15] Moreover, FIT facilitates the removal of trauma splints and orthodontic brackets, minimizing the risk of iatrogenic damage to the enamel.[17,19] Other approaches for the selective removal of composite have been described. For example, carbon dioxide lasers guided by spectral feedback render a high level of ablation selectivity possible for composites.[14] However, compared with laser-based ablation techniques, FIT may be more readily implementable in clinical practice as it is suited for intraoral examinations and restorative procedures alike, and it only requires an illumination source with a light spectrum in the of range of 405 ± 10 nm.[15,24] When using FIT, it is important to follow the safety instructions of the LED manufacturer and to use adequate eye protection such as safety glasses with filter lenses to avoid potentially detrimental health effects of blue-violet and ultraviolet light.

The direct restorations in the present study were placed with a nanohybrid direct composite material that has strong fluorescent properties.[16] Clinical studies report only minor differences in the clinical behavior of direct restorations placed with different composite materials.[2] Although some filler characteristics may have an impact on late failings of composite restorations,[25] the physical properties of available composites are considered to be of subordinate significance for restoration longevity.[2,26] When placing direct restorations in posterior teeth, it may therefore be advantageous to choose metameric composites that have a documented good performance in clinical trials plus strong fluorescent properties.[16,24] The latter may facilitate repair or replacement in the restorative cycle provided that FIT is used.

The replacement of restorations continues to be a common clinical procedure, imposing a heavy burden on health care expenditure across the globe.[1,4,27] Fractures and secondary caries are the main reasons for the failure of direct composite restorations in posterior teeth.[2,3] Prevention and control of secondary caries are therefore of paramount importance: patient-related risk factors should be appropriately managed and materials selection and restorative procedures have to be carried out with due diligence and care.[1,12] Furthermore, current detection methods for secondary caries lesions are best used in combination, not on their own, at specific thresholds to avoid false-positive diagnoses.[28] In addition, contemporary caries excavation techniques such as selective caries removal, which frequently have distinctive radiographic features, should be taken into account to avert invasive and costly overtreatment and ensure the best patient-centered outcome.[29]

The advantages of restoration refurbishment and repair over replacement are legion: most importantly, the lifespan of a partially defective restoration can often be prolonged through refurbishment or repair.[8,13,30] When a restoration is replaced, some sound tooth structure is inevitably removed and the cavity is frequently enlarged.[11-13] Therefore, the possibilities of restoration refurbishment and repair need to be exhausted. Replacement restorations should be deemed indicated only when, based on a

Table 1: *Mean Results (SD, 95% CI) Regarding Composite Residues (Volume, Height, and Percentage of Removal), Defect Volume and Depth, and Duration (ext.)*

Operator and Technique	Volume Defect (mm^3)	Maximum Depth Defect (mm)	Time (s)
Operator 1: conventional technique	5.77 (1.89; 4.77-6.78) D	0.99 (0.18; 0.89-1.08) B	328.50 (168.78; 238.56-418.44) A,B
Operator 1: FIT	3.73 (1.83; 2.76-4.71) B,C	0.58 (0.18; 0.48-0.67) A	163.19 (69.79; 126.00-200.37) C
Operator 2: conventional technique	3.29 (1.56; 2.46-4.12) A,B,C	0.90 (0.32; 0.73-1.70) B	329.06 (174.75; 235.94-422.18) A,B
Operator 2: FIT	1.80 (1.24; 1.14-2.47) A	0.44 (0.16; 0.35-0.52) A	195.13 (88.99; 147.71-242.54) B,C
Overall: conventional technique	4.53 (2.12; 3.77-5.30) C,D	0.94 (0.26; 0.85-1.04) B	328.78 (169.00; 267.85-389.71) A
Overall: FIT	2.77 (1.83; 2.11-3.43) A,B	0.51 (0.18; 0.44-0.57) A	179.16 (80.32; 150.20-208.11) C

meticulous assessment, the possibility of refurbishment or repair has been ruled out.[30-32] The results of the present study suggest that FIT makes it easier to selectively remove composite and contributes to the preservation of sound dental hard tissue. Thus, FIT may be a useful tool for the repair and replacement of composite restorations.

The operators in the present study aimed at removing composite as completely as possible. In clinical settings, however, the replacement of defective composite restorations does not necessarily require the complete removal of old composite. For instance, it may frequently be advisable to leave composite near the pulp chamber to reduce pulp irritation.[33] Adequate bond strengths between the composite of the old restoration and new composite are obtainable when mechanical and adhesive surface pretreatments are performed.[9,34,35] The application of appropriate repair techniques obviates the need for complete composite removal, and FIT may facilitate to deliberately leave composite close to the pulp.

To determine the appropriate pretreatment and bonding protocols, one must know whether a cavity is bounded by composite or dental hard tissues or both.[6] Composite detection with FIT is straightforward and swift.[15] Therefore, FIT used for cavity inspection seems to be a useful aid to select the proper pretreatment method. The present study indicates that composite removal procedures hardly ever achieve the complete removal of restoration material. When replacing direct composite restorations, airborne particle abrasion with aluminum oxide may, consequently, be recommended as cavity pretreatment in most cases.

The present study has certain inherent limitations that demand careful consideration. First, the direct composite restorations had no visible defects or imperfections whatsoever, and spectrophotometric shade selection ensured an exact color match. In contrast, restorations that are replaced in clinical practice are usually (partially) defective.[30] Common features of intraoral aging such as marginal staining may facilitate the removal procedure. The present study simulated challenging conditions, and therefore its results may not directly translate to clinical settings where composite removal is straightforward owing to substantial defects and/or conspicuous color differences. However, in the setup of the present study, patient-related factors that can complicate restorative interventions (limited mouth opening and suchlike) were absent. Arguably, composite removal procedures in clinical settings frequently present an even bigger challenge than in the present study.

Second, in the present study a custom micromotor that emitted blue light at a wavelength of 405 nm through the illumination source of the handpiece was used in the FIT group. This allowed the fluorescence-aided detection of composite while the handpiece was in use. Such a setup offers a seamless workflow and hence time savings.[19] However, currently there are no micromotors or handpieces with integrated FIT commercially available. There are some devices on the market that emit light with the wavelengths required for FIT (eg, D-Light Pro, GC Corporation, Tokyo, Japan; SiroInspect, Dentsply Sirona). When one uses such devices for composite removal procedures, fluorescence-aided detection and composite removal usually occur in separate steps. As a consequence, removal procedures using FIT in this manner may be slightly less expeditious than in the present study. Further research is needed to assess what impact the use of currently available equipment has on the treatment duration of fluorescence aided composite removal.

Third, a couple of young dentists with unimpaired eyesight performed the composite removal procedures in the present study. The completeness and selectivity of composite removal are, to a degree, dependent on the operator.[19] In addition, near visual acuity under simulated clinical conditions varies between individuals and decreases with advancing

age.[36] In the present study, the small convenience sample of operators was biased toward young dental professionals. It would therefore be desirable that subsequent investigations are undertaken with a more representative sample of dentists to evaluate the replicability of the present study.

CONCLUSIONS

Within the limitations of this *in vitro* study, FIT improved the selectivity and completeness of composite removal, and composite removal procedures using FIT were more expeditious compared with the conventional technique. FIT may thus contribute to tooth substance preservation when the repair or replacement of defective direct composite restorations is indicated.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethikkommission Nordwest- und Zentralschweiz. The approval code for this study is EKNZ UBE-15/111.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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References

1. Lynch CD, Opdam NJ, Hickel R, Brunton PA, Gurgan S, Kakaboura A, Shearer AC, Vanherle G, & Wilson NHF (2014) Guidance on posterior resin composites *Journal of Dentistry* **42(4)** 377-383.
2. Demarco FF, Corrêa MB, Cenci MS, Moraes RR, & Opdam NJM (2012) Longevity of posterior composite restorations *Dental Materials* **28(1)** 87-101.
3. Ástvaldsdóttir Á, Dagerhamn J, van Dijken JWV, Naimi-Akbar A, Sandborgh-Englund G, Tranaeus S, & Nilsson M (2015) Longevity of posterior resin composite restorations in adults: a systematic review *Journal of Dentistry* **43(8)** 934-954.
4. Eltahlah D, Lynch CD, Chadwick BL, Blum IR, & Wilson NHF (2018) An update on the reasons for placement and replacement of direct restorations *Journal of Dentistry* **72** 1-7.
5. Estay J, Martin J, Viera V, Valdivieso J, Bersezio C, Vildosola P, Mjör IA, Andrade MF, Moraes RR, Moncada G, Gordan VV, & Fernandez E (2018) 12 years of repair of amalgam and composite resins *Operative Dentistry* **43(1)** 12-21.
6. Hickel R, Brushaver K, & Ilie N (2013) Repair of restorations: criteria for decision making and clinical recommendations *Dental Materials* **29(1)** 28-50.
7. Casagrande L, Laske M, Bronkhorst EM, Huysmans MCDNJM, & Opdam NJM (2017) Repair may increase survival of direct posterior restorations: a practice based study *Journal of Dentistry* **64** 30-36.
8. Martins BMC, Silva EJNLD, Ferreira DMTP, Reis KR, & Fidalgo TKDS (2018) Longevity of defective direct restorations treated by minimally invasive techniques or complete replacement in permanent teeth *Journal of Dentistry* **78** 22-30.
9. Kanzow P, Wiegand A, Gostemeyer G, & Schwendicke F (2018) Understanding the management and teaching of dental restoration repair *Journal of Dentistry* **69** 1-21.
10. Bush MA, Hermanson AS, Yetto RJ, & Wieczkowski G, JR (2010) The use of ultraviolet LED illumination for composite resin removal *General Dentistry* **58(5)** e214-e218.
11. Brantley CF, Bader JD, Shugars DA, & Nesbit SP (1995) Does the cycle of rerestoration lead to larger restorations *Journal of the American Dental Association* **126(10)** 1407-1413.
12. Nedeljkovic I, Teughels W, De Munck J, Van Meerbeek B, & Van Landuyt KL (2015) Is secondary caries with composites a material-based problem *Dental Materials* **31(11)** e247-e277.
13. Kanzow P, Wiegand A, & Schwendicke F (2016) Cost-effectiveness of repairing versus replacing composite or amalgam restorations *Journal of Dentistry* **54** 41-47.
14. Yi I, Chan KH, Tsuji GH, Staninec M, Darling CL, & Fried D (2016) Selective removal of esthetic composite restorations with spectral guided laser ablation *Proceedings of the Society of Photo-optical Instrumentation Engineers* **Feb 13** 9692.
15. Meller C, Connert T, Löst C, & ElAyouti A (2017) Reliability of a fluorescence-aided identification technique (FIT) for detecting tooth-colored restorations *Clinical Oral Investigations* **21(1)** 347-355.
16. Meller C & Klein C (2015) Fluorescence of composite resins *Dental Materials Journal* **34(6)** 754-765.
17. Schott TC & Meller C (2018) A new fluorescence-aided identification technique (FIT) for optimal removal of resin-based bracket bonding remnants after orthodontic debracketing *Quintessence International* **49(10)** 809-813.
18. Salomao FM, Rocha RS, Franco LM, Sundfeld RH, Bresciani E, & Fagundes TC (2019) Auxiliary UV light devices for removal of fluorescent resin residues after bracket debonding *Journal of Esthetic and Restorative Dentistry* **31(1)** 58-63.
19. Dettwiler C, Meller C, Eggmann F, Saccardin F, Kühl S, Filippi A, Krastl G, Weiger R, & Connert T (2018) Evaluation of a fluorescence-aided identification tech-

nique (FIT) for removal of composite bonded trauma splints *Dental Traumatology* **34(5)** 353-359.

20. Ammannato R, Ferraris F, & Marchesi G (2015) The "index technique" in worn dentition *International Journal of Esthetic Dentistry* **10(1)** 68-99.
21. Soliman S, Preidl R, Karl S, Hofmann N, Krastl G, & Klaiber B (2016) Influence of cavity margin design and restorative material on marginal quality and seal of extended Class II resin composite restorations in vitro *Journal of Adhesive Dentistry* **18(1)** 7-16.
22. Mehl A, Gloger W, Kunzelmann KH, & Hickel R (1997) A new optical 3-D device for the detection of wear *Journal of Dental Research* **76(11)** 1799-1807.
23. Dexter FC & Stearns EI (1948) Example of metamerism: extreme example of color change with varying illuminant *Journal of the Optical Society of America* **38(9)** 816.
24. Meller C & Klein C (2012) Fluorescence properties of commercial composite resin restorative materials in dentistry *Dental Materials Journal* **31(6)** 916-923.
25. Da Rosa Rodolpho PA, Donassollo TA, Cenci MS, Loguercio AD, Moraes RR, Bronkhorst EM, Opdam NJM, & Demarco FF (2011) 22-Year clinical evaluation of the performance of two posterior composites with different filler characteristics *Dental Materials* **27(10)** 955-963.
26. Opdam NJM, van de Sande FH, Bronkhorst E, Cenci MS, Bottenberg P, Pallesen U, Gaengler P, Lindberg A, Huysmans MCDNJM, & van Dijken JW (2014) Longevity of posterior composite restorations *Journal of Dental Research* **93(10)** 943-949.
27. Mjör IA & Toffenetti F (2000) Secondary caries *Quintessence International* **31(3)** 165-179.
28. Schwendicke F, Brouwer F, Paris S, & Stolpe M (2016) Detecting proximal secondary caries lesions *Journal of Dental Research* **95(2)** 152-159.
29. Signori C, Gimenez T, Mendes FM, Huysmans M-CDNJM, Opdam NJM, & Cenci MS (2018) Clinical relevance of studies on the visual and radiographic methods for detecting secondary caries lesions: a systematic review *Journal of Dentistry* **75** 22-33.
30. Wilson N, Lynch CD, Brunton PA, Hickel R, Meyer-Lueckel H, Gurgan S, Pallesen U, Shearer AC, Tarle Z, Cotti E, Vanherle G, & Opdam N (2016) Criteria for the replacement of restorations *Operative Dentistry* **41(Supplement 7)** S48-S57.
31. Brunton PA, Ghazali A, Tarif ZH, Loch C, Lynch C, Wilson N, & Blum IR (2017) Repair vs replacement of direct composite restorations *Journal of Dentistry* **59** 62-67.
32. Rauber GB, Taguchi C, Padilha A, Re Silveira RC de, Bernardon JK, & Baratieri LN (2018) Color repair of a composite resin restoration *Operative Dentistry* **44(1)** 1-7.
33. Mjör IA & Odont D (2001) Pulp-dentin biology in restorative dentistry. Part 2 *Quintessence International* **32(7)** 537-551.
34. Blunck U (2013) Pretreatment of composite resin surfaces for repair *Journal of Adhesive Dentistry* **15(6)** 592.
35. Loomans BAC, Cardoso MV, Roeters FJM, Opdam NJM, Munck J de, Huysmans MCDNJM, & van Meerbeek B (2011) Is there one optimal repair technique for all composites *Dental Materials* **27(7)** 701-709.
36. Eichenberger M, Perrin P, Neuhaus KW, Bringolf U, & Lussi A (2013) Visual acuity of dentists under simulated clinical conditions *Clinical Oral Investigations* **17(3)** 725-729.

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Characterization and Comparative Analysis of Voids in Class II Composite Resin Restorations by Optical Coherence Tomography

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Clinical Relevance

In addition to the layering technique, the use of flowable resin-based composites may also result in void formation in restorations.

SUMMARY

Purpose: This study aimed to characterize and analyze the number of voids and the percentage of void volume within and between the layers of class II composite restorations made using the bulk fill technique or the incremental technique by optical coherence tomography (OCT).

Methods and Materials: Class II cavities (4×4×2 mm) were prepared in 48 human third molars (n=24 restorations per group, two class II cavities per tooth). Teeth were divided into four groups and restored as follows: group 1 (FOB), bulk filled in a single increment using Filtek One Bulk Fill (3M Oral Care); group 2 (FXT), incrementally filled using four oblique layers of Filtek Z350 XT (3M Oral Care); group 3 (FBF+FXT), bulk filled in a single increment using Filtek Bulk Fill Flowable Restorative (3M Oral Care) covered with two oblique layers of Filtek Z350 XT (3M Oral Care), and group 4 (FF+FXT), incrementally filled using Filtek Z350 XT Flow (3M Oral Care) covered with two oblique layers of Filtek Z350 XT (3M Oral Care). After the restorative procedure, specimens were immersed into distilled water and stored in a hot-air oven at 37°C. Forty-eight hours later, thermal cycling was conducted (5000 cycles, 5°C to 55°C). Afterward, OCT was used to detect the existence of voids and to

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calculate the number of voids and percentage of voids volume within each restoration. Data were submitted to chi-square and Kruskal-Wallis tests (α=0.05). Comparisons were made using the Dunn method.

Results: Voids were detected in all groups, ranging from 0.000002 (FBF+FXT and FF+FXT) to 0.32 mm^3 (FBF+FXT). FF + FXT presented voids in all of the restorations and had a significantly higher number of voids per restoration when compared to the other groups ($p<0.05$), but restorations with the presence of voids were significantly higher only when compared to FXT ($p<0.05$). FBF + FXT presented a significantly higher percentage of voids volume than that of FXT ($p<0.05$). When comparing restorations made using high-viscosity resin-based composites (FOB and FXT), no significant differences regarding number of voids or percentage of voids volume were detected ($p\geq0.05$).

Conclusions: The use of flowable resin-based composites can result in an increased number of voids and percentage of voids volume in restorations, and this appears to be more related to voids present inside the syringe of the material than to the use of incremental or bulk fill restorative techniques.

INTRODUCTION

Resin-based composites (RBCs) are materials indicated for use in different types of dental treatments in all areas of the mouth, and due to their versatility, their use is growing continuously.[1] Since their first appearance, several investigations have been developed to achieve improvements concerning their clinical behavior,[1] but there are still controversies regarding which is the best restorative technique that would minimize the incorporation of voids during the restorative procedures in order to avoid reducing the mechanical properties of the final restoration.[2-4]

The incremental restorative technique, which consists of inserting RBC increments up to 2 mm thick,[5] has been commonly executed to ensure a restoration with an appropriate degree of polymerization with reduced cytotoxicity and improved physical properties.[6] However, it may also present some disadvantages related to the formation of voids and the absence of union between the layers as well as the difficulties associated with restoring large, deep, and difficult-to-access cavities, which may increase the time needed for the restorative procedure.[7] Therefore, a new generation of these materials has been developed, the so-called bulk fill RBCs, designed to restore medium to deep cavities in posterior teeth with large increments of up to 4- to 5-mm depth.[8] Manufacturers claim that bulk fill RBCs minimize the incorporation of voids,[9] and studies have reported the same pattern, declaring a lower presence of voids within the mass of the RBC restoration with the use of the bulk fill restoration technique due to decreased handling during the restorative procedure,[3,10,11] especially in deeper cavities.[12] In contrast, a higher presence of porosity in greater thicknesses of the material has also been reported,[13] as has a lower incidence of gaps in a larger amount of small increments,[2] supporting the use of the incremental technique.

Voids within a restoration, also referred to as porosities or bubbles, are caused by air entrapment and can be incorporated into RBCs during their manipulation when performing a restorative procedure or even while being manufactured. Such voids can be located as follows: on the restoration surface, leading to a greater retention of organic residue and causing an appearance similar to marginal staining; in the restoration's bulk, negatively affecting its aesthetic and mechanical properties;[14] or at the tooth-restoration interface, forming gaps at the restoration's margin.[15]

Diverse techniques have been performed for the evaluation of porosity, including conventional methods, such as microscopy on sectioned samples,[16] or radiographic analyses of specimens.[11] However, in an attempt to avoid destruction and improve the resolution of the internal structure images, new technologies have been introduced.[17] Optical coherence tomography (OCT) is a nondestructive[18] technique that allows noncontact cross-sectional and optical imaging.[19] Since its introduction in dentistry in 1998,[20] OCT has expanded its application in laboratory research as well as clinical practice, enabling the characterization of oral structures and restorative materials.[20-23] In this way, OCT continues to be used in research for the characterization of composites, aiming at the evaluation of internal and marginal adaptation[24,25] and, furthermore, the detection of defects in restorations.[21,25] In fact, OCT analysis has also been presented as a valid method for the characterization of voids in RBCs by previous work that compared OCT results with data obtained by microcomputed tomography,[25] another technique recognized for the evaluation of porosities in restorations.[6]

Table 1: *Restorative Materials Used in This Study and Technical Information Provided by Manufacturer (3M Oral Care)*

Material	Composition
Filtek Z350 XT	Bis-GMA, UDMA, TEGDMA, and bis-EMA Fillers: a combination of nonagglomerated/nonaggregated 20-nm silica filler, nonagglomerated/nonaggregated 4- to 11-nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20-nm silica and 4- to 11-nm zirconia particles). The inorganic filler loading is about 72.5% by weight (55.6% by volume).
Filtek One Bulk Fill Restorative	AFM (dynamic stress-relieving monomer), AUDMA, UDMA, and 1,12-dodecane-DMA Fillers: a combination of a nonagglomerated/nonaggregated 20-nm silica filler, a nonagglomerated/nonaggregated 4- to 11-nm zirconia filler, an aggregated zirconia/silica cluster filler (comprised of 20-nm silica and 4- to 11-nm zirconia particles), and a ytterbium trifluoride filler consisting of agglomerate 100-nm particles. The inorganic filler loading is about 76.5% by weight (58.5% by volume).
Filtek Z350 XT Flow	Bis-GMA, TEGDMA, and bis-EMA Fillers: a combination of 5-nm diameter of nonagglomerated/nonaggregated silica nanofiller, 5-10 nm diameter of nonagglomerated/nonaggregated zirconia nanofiller, loosely bound agglomerated zirconia/silica nanocluster, consisting of agglomerates of 5- to 20-nm primary zirconia/silica particles and a cluster particle size range of 0.6 to 1.4 microns. The inorganic filler loading is about 65% by weight (55% by volume).
Filtek Bulk Fill Flow	BisGMA, UDMA, bisEMA, and Procrylat Fillers: a combination of zirconia/silica with a particle size range of 0.01 to 3.5 microns and ytterbium trifluoride filler with a range of particle sizes from 0.1 to 5.0 microns. The inorganic filler loading is approximately 64.5% by weight (42.5% by volume).

Abbreviations: Bis-GMA, bisphenol A diglycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; bis-EMA, bisphenol A ethoxylated dimethacrylate; AFM, addition-fragmentation chain transfer monomer; AUDMA, aromatic urethane dimethacrylate; UDMA, urethane dimethacrylate; DMA, dimethylacetamide.

It is known that the presence of voids within restorations negatively affects the flexural strength of the material and determines a lower resistance to fatigue and wear[26,27] due to their contribution to the initiation and propagation of cracks, leading to failure of the restoration as it is subjected to mechanical and external loads when functioning.[28] However, the advantages of using a certain restorative technique, improving the quality of a restoration by reducing the presence of voids, are still unclear and must be verified.

Thus, the purpose of this *in vitro* study was to characterize and analyze the presence of voids inside class II restorations made using the bulk fill technique compared with restorations made using the incremental technique through OCT, a nondestructive technique. The null hypotheses of this study were 1) that there is no significant difference in the number of voids among restorations made using incremental technique compared with those made with bulk fill technique and 2) that there is no significant difference in the percentage of voids volume among restorations made using incremental technique compared with those made with bulk fill technique.

METHODS AND MATERIALS

Specimen Selection and Preparation

Forty-eight freshly extracted, sound, caries-free human third molars were selected, cleaned, and stored at 37°C. In order to obtain a regular enamel surface, proximal, mesial, and distal surfaces of the third molars were polished using a polishing machine (023-011263, EcoMET, Buehler, Lake Bluff, IL, USA). Teeth were divided into four groups (n=24 restorations per group, two class II cavities per tooth, where mesial and distal restorations were observed separately), according to the RBC and restorative technique used (Table 1). Prior to any preparation, high-density vinyl polysiloxane silicone (VPS) was manufactured to serve as a matrix for each tooth, which aided the material placement in the restorative procedure. A VPS matrix was used since by positioning the tooth inside a silicone mold, it was possible to standardize the restorative procedure, especially the composite's cervical adaptation. After that, teeth were removed from their matrices, and two slot class II preparations were made at each proximal surface with a 4-mm depth, 4-mm length, and 2-mm width (Fig. 1). Measurements of the preparations were checked with a digital caliper (500-196-30, Mitutoyo Corp, Kanagawa, Japan).

Restorative Procedures

Prepared teeth were repositioned into the VPS matrix for the restorative procedure, which was performed by a single operator in order to standardize this procedure. All restorative procedures were performed by the same operator as well. The self-etch adhesive system ClearFill SE Bond (Kuraray, Tokyo, Japan) was applied according to manufacturer's instructions. The restorative materials and

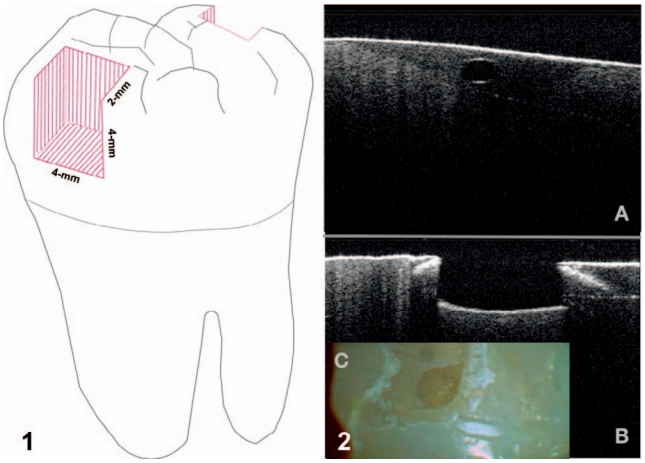

Figure 1. *Tooth cavities. Two slot class II preparations made at each proximal surface, with 4-mm depth, 4-mm length, and 2-mm width.*

Figure 2. *Void detection. (A): OCT image representing what was considered a void inside the restoration. (B): OCT image representing what was considered a void on the proximal surface of the restoration. (C): Image of a restoration with a superficial void on the proximal face. OCT, optical coherence tomography.*

techniques were as follows: group 1 (FOB), Filtek One Bulk Fill (3M Oral Care, A2 shade), bulk filled using a single 4-mm increment; group 2 (FXT), Filtek Z350 XT (3M Oral Care, A2 Body shade), incrementally filled using four oblique 2-mm layers; group 3 (FBF+FXT), Filtek Bulk Fill Flow (3M Oral Care, A2 shade), bulk filled using a 2-mm single layer covered with two 2-mm oblique layers of Filtek Z350 XT; and group 4 (FF+FXT), Filtek Z350 XT Flow (3M Oral Care, A2 shade), incrementally filled using a 2-mm layer, also covered with two 2-mm oblique layers of Filtek Z350 XT. Each RBC increment was photoactivated for 20 seconds with the light-curing unit Radii-cal (SDI, Victoria, Australia), which presents a radiant exitance of 1200 mW/cm^2. After restoration, in order to ensure an adequate polymerization of the RBCs, teeth were removed from the VPS matrix, and afterward a polyester matrix was placed on the occlusal and proximal surfaces of restorations; each occlusal, mesial, and distal surface was photopolymerized for 20 seconds. Once the restorative procedure was completed, specimens were immersed into distilled water and stored in a hot-air oven at 37°C (Fanem 502, Fanem, São Paulo, Brazil). Forty-eight hours later, thermal cycling was conducted using 5000 cycles (5°C to 55°C, 30-second dwell time), equivalent to approximately six months of thermal alterations in the oral cavity,[28] using a thermal cycling machine (Nova Etica 521/4D, Nova Etica, São Paulo, Brazil) to simulate intraoral thermal alterations.[29]

OCT Analysis and Definition of Voids

After thermal cycling, OCT was performed (OCT930 SR, Thorlabs Inc, Newton, NJ, USA). The equipment's light source emitted a central wavelength of 930 nm and 2 mW of optical power with a maximum image depth in resin composite of about 0.7 mm with axial resolution of 0.4 and lateral resolution 6.0 μm. In order to compensate for the limited penetration and to make the evaluation of all the RBC increments possible, scanning of a representative portion of the restoration was made with teeth positioned with the proximal surface facing up. The SR Scan program (Thorlabs Inc) was used to perform the tomographic scan, with parameters of 2000 A-scan (columns) for a width of 2.5 mm (range). Based on the low-coherence light scattered from the sample micron/level resolution subsurface, cross-sectional images of 2000 × 512 pixels, corresponding to 2.5 × 0.7 mm, were obtained by analyzing the interference pattern.[19] To standardize the evaluation, specimens were always scanned starting from the cervical to the coronal portion of the restoration and double-checked without considering either the restoration interface or the adhesive layer. Voids in this study were defined as spherical or ellipsoidal defects, and two types of voids were observed: 1) voids inside the restoration, as a defect with a surrounding light margin, which is produced when the light transverses the air trapped inside the void, reflecting a portion of light and producing a higher signal intensity,[25] or 2) voids on the proximal surfaces, as a semispherical or semispheroidal irregularity on the top of the image, which corresponded to air trapped between the silicone matrix and the composite during the restorative procedure (Fig. 2). When a void was visually detected, a scan was made around it to define the largest visible diameter. The volume of each one was also calculated by establishing a model in which it was assumed that two types of voids could be observed: those with a similar height and width, close to a spherical shape, and those with one of the two dimensions of greater size (height or width), corresponding to the ellipsoidal format. Therefore, the volume of the spherical voids was calculated using the formula to obtain the volume of the sphere ($4/3\pi r^3$) and the volume of the ellipsoidal voids with the formula to obtain the volume of the ellipsoid ($4/3\pi a^2 b$). Moreover, total volume of the voids was calculated by restoration, and with these values, the percentage of voids volume of the restoration was determined, considering 11.2 mm^3 as the total restoration volume. This volume corresponds to the preparation's dimensions (4-mm length

Table 2: *Percentage of Restorations With Voids per Group and Total Number of Voids*[a]

Group	Percentage of Restorations With Voids (Restorations With Voids/24)	Number of Voids	
		Total	Median (Interquartile Range, Q3-Q1)
Group 1 (FOB) Filtek One Bulk Fill	83.3%	44	2 (3-1) A
Group 2 (FXT) Filtek Z350XT	75.0%	26	1 (2-0.75) A
Group 3 (FBF+FXT) Filtek Bulk Fill Flowable + FXT	79.2%	53	2 (3-1) A
Group 4 (FF+FXT) Filtek Z350XT Flow + FXT	100.0%	176	7 (10-5) B

[a] *Different letters represent statistical significant differences among groups regarding number of voids.*

and 4-mm height) and the penetration depth of the OCT (0.7 mm). Images were formatted keeping the original pixel distance (512) using ImageJ software (1.8.0_172, National Institutes of Health, Bethesda, MD, USA). Then measurements used to obtain the dimension values for volume calculation of voids were taken using the same software. To calculate the measurements of the X-axis, the measurement was performed directly on the formatted image, and on the Y-axis, image distortion was considered by the material's refractive index. The value was corrected, dividing it by the refractive index of the composite resin: 1.6.[30]

Syringe Samples and Characterization of Restorative Materials

One sample of each resin material used in the different restorative techniques was extracted directly from their manufactured syringe, avoiding manipulation. High-viscosity materials were removed from the syringe with a clean cut made with a number 10 surgical blade (Solidor, Lamedid, Suzhou, China), and flowable materials were dispensed on a glass slab. Then syringe samples were light cured according to the manufacturer's instructions, followed by an individual scan by OCT. This step was performed to evaluate the presence of any flaws in the material when directly extracted from the syringes with no further manipulation.

Statistical Analysis

After a descriptive analysis of the number, percentage of voids volume, and shape and location of voids per restoration, data regarding the presence or absence of voids inside each restoration per group was analyzed by the chi-square test to verify if there were differences between restorative techniques (α=0.05). The number of voids and the percentage of voids volume per restoration and per group were organized into tables and analyzed with the Liliefors test for normality. Due to a nonnormal data distribution, the Kruskal-Wallis test and comparisons with the Dunn method were applied (α=0.05).

RESULTS

OCT analysis detected the presence of voids in all groups. The percentage of restorations with voids and the total number of voids per group are presented in Table 2. FF + FXT had the highest number of voids, registering a total of 176. The shape and location of the voids are described in Table 3. All groups presented more spherical voids than ellipsoidal voids, with the exception of FXT, which presented the same number of spherical and ellipsoidal voids. Moreover, the number of voids inside restorations was greater than that of voids located on the surface of proximal surfaces. Voids with diameters of 0.01 mm (FF+FX and FBF+FXT) to 1.07 mm (FBF+FXT) were observed. Individual volume observed among the voids remained within 0.000002 to 0.32 mm^3 (Table 4). The percentage of voids volume

Table 3: *Shape and Location of Voids (Total per Group) Detected by Optical Coherence Tomography*

Group	Shape of Voids		Location of Voids	
	Ellipsoidal	Spherical	Superficial	Internal
Group 1 (FOB) Filtek One Bulk Fill	7	37	7	37
Group 2 (FXT) Filtek Z350XT	13	13	10	16
Group 3 (FBF+FXT) Filtek Bulk Fill Flowable + FXT	16	37	20	33
Group 4 (FF+FXT) Filtek Z350XT Flow + FXT	81	95	13	163

Table 4: *Range of Void Size and Percentage of Void Volume per Group*[a]

Group	Range of Void Size (mm^3)	Percentage of Voids Volume	
		Range (%)	Median (Interquartile Range, Q3-Q1)
Group 1 (FOB) Filtek One Bulk Fill	0.00001-0.076	0.001-0.7	0.005 (0.019-0.0003) AB
Group 2 (FXT) Filtek Z350XT	0.00001-0.059	0.001-0.5	0.002 (0.008-0.0002) A
Group 3 (FBF+FXT) Filtek Bulk Fill Flowable + FXT	0.000002-0.32	0.003-2.9	0.032 (0.138-0.0039) B
Group 4 (FF+FXT) Filtek Z350XT Flow + FXT	0.000002-0.03	0.002-0.24	0.009 (0.031-0.0053) AB

[a] *Different letters represent statistical significant differences among groups regarding percentage of voids volume.*

determined ranged from 0.001% (FOB and FXT) to a maximum of 2.9% (FBF+FXT) of the total restoration.

FF + FXT presented voids within all restorations (100%), being significantly greater than FXT ($p<0.0149$) and presenting no significant differences when compared with the other groups ($p\geq0.05$). The percentage of restorations with porosities was at least 75% per group, and considering all 96 restorations evaluated, voids were detected in 84.4% of them.

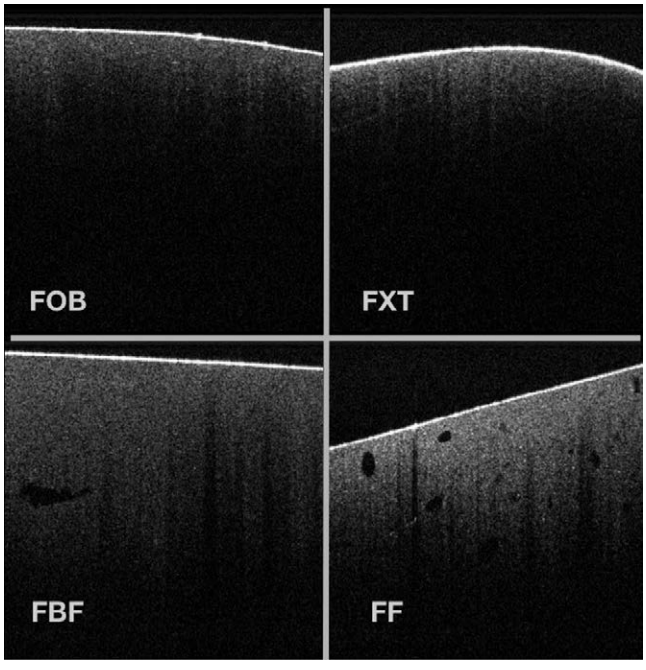

Figure 3. *OCT images of syringe samples representing each restorative material used in the study. FOB- and FXT-labeled figures show homogeneous and void-free characteristics. By comparing figures labeled FBF and FF, a higher number of small voids can be observed in FF, while a larger, single void can be seen in FBF, representing a higher void volume but a small number. OCT, optical coherence tomography; FOB, Filtek One Bulk Fill; FXT, Filtek Z350XT; FBF, Filtek Bulk Fill Flowable; FF, Filtek Z350XT Flow.*

The Kruskal-Wallis test detected significant differences among the groups regarding number of voids ($p<0.0001$) and percentage of voids volume ($p=0.0158$). The Dunn multiple comparison test determined that considering the number of voids, FF + FXT was significantly higher than the other three groups ($p<0.05$), while considering the percentage of voids volume, FBF + FXT was significantly higher only when compared to FXT ($p<0.05$). There were no significant differences when comparing with the other groups ($p\geq0.05$).

The images of syringe samples (Fig. 3) showed that high-viscosity RBCs FOB and FXT were homogeneous and void free. In contrast, nonmanipulated samples of flowable RBCs, FBF, and FF presented defects in their structure. In FBF, a large defect could be observed in the material's internal region. In addition, there were several smaller defects within the FF material, presenting characteristic void images inside.

DISCUSSION

The first null hypothesis was rejected since FF + FXT number of voids was significantly higher than the other three groups. The second null hypothesis was also rejected since the FBF + FXT percentage of voids volume was higher than FXT.

All groups presented voids within restorations in the OCT evaluation. The high presence of voids within restorations (84.4%) has already been reported, describing values of presence of voids ranging from 86.4% to 100% of examined samples scanned by an electron microscope.[2] Regarding the shape of the voids, although a different study measured only spherical voids,[25] two types of shapes were observed and defined in this study—ellipsoidal and spherical—in order to get a more accurate volume measure. Some studies associate an ellipsoidal shape with the

voids located within increments of composite and a spherical shape with those located inside an increment of the material.[2] This study could not verify this characterization since ellipsoidal voids were observed in restorations made with both incremental and bulk fill techniques for both high viscosity and flowable materials. In addition, voids inside the restoration were more frequent compared with voids located on the surface of the proximal surface.

The percentage of voids volume varied from 0.001% (FOB and FXT) to a maximum of 2.9% (FBF+FXT), in accordance with results obtained by a previous study in which values ranging from 0.1% to 2% were registered.[25]

The analysis of number of voids did not confirm that a greater number of voids is responsible for the greater percentage of voids volume in the restorations. However, those discrepancies can be explained by comparing the difference in the size of voids detected in each group since the FBF + FXT presented voids with an average size of 0.01 mm^3, much larger than those detected in FF + FXT, with an average size of 0.0005 mm^3. This discrepancy demonstrates the importance of determining the volume of the voids and not only their quantity when evaluating the porosity of the restorations since large discrepancies in size could lead to misinterpretation of the results. Moreover, clinically, a large void is mechanically worse for a restoration than a small void even if the actual number of voids is the same, differing only in volume.

The presence of voids in restorations may be related to the operative technique, the operator's ability, and the consistency of the material.[31,32] Since in the present study all the restorations were made by the same operator, the possibility of incorporating voids was equal for all groups, thus avoiding influencing, negatively or positively, the results of any group. However, regarding the operative technique factor, it was observed that the groups that presented the highest quantity of voids and percentage of voids volume, respectively, belonged to different types of composites (incremental technique with conventional flowable RBC [FF+FXT] and bulk technique fill with bulk fill flowable RBC [FBF+FXT]), although the type of manipulation was the same: 2 mm of a flowable RBC followed by incremental layering of a conventional high-viscosity RBC. Both techniques were performed with the same amount and size of increments since cavity preparation depth did not allow for greater thickness of bulk fill flowable composite, as it had to be covered with two oblique layers of high-viscosity RBC.[33]

The results of this study disagree with the premise that the bulk fill technique allows a decreased void incorporation within restorations, attributed to the possibility of inserting the material into cavities using a single portion. Nevertheless, they agree with the results of studies that showed that a similar percentage of voids volume was detected when incremental and bulk fill techniques were evaluated.[34] Although new insertion techniques for bulk fill RBCs, such as sonic insertion of the material, continue to be developed, the previously mentioned technique has not been confirmed to be advantageous, as a recent study performed with microcomputed tomography showed that restorations performed with the sonicated technique presented greater void formation when compared with the conventional technique.[4]

Considering the viscosity of the materials used in the present study, it can be observed that restorations made with lower-viscosity RBCs presented a higher number of voids and a higher percentage of voids volume (FF+FXT and FBF+FXT) than restorations performed with high-viscosity RBCs. Studies have also reported a higher incidence of voids and percentage of voids volume attributed to the intrinsic porosity of low-viscosity RBCs that cannot be controlled or modified by the operator because it is limited by their stipulated restorative protocols.[35]

In order to help understand the porosity results obtained in this study, syringe samples of the resin materials used in the different restorative techniques were extracted directly from their syringes, avoiding manipulation and photoactivation. After being individually analyzed using OCT, it was possible to observe that the high-viscosity RBCs FOB and FXT were homogeneous and void free. In contrast, the flowable RBCs presented defects in their structure. In the FBF, a defect can be observed in the internal region of the material; although it does not have the defined characteristics of the definition of a void, it has the potential to cause a defect of rounded shape and of great size once manipulated for its use as a restorative material. In addition, there are several smaller defects in the structure of the FF material, presenting characteristic void images inside. This fact might be related to how the flowable materials are disposed inside their syringes or to how they leave the syringe in the needles, which is directly related to the manufacturer and not to the operator and can jeopardize the resin composite restoration mass by incorporating voids inside the material.

Although conclusions cannot be made from the analysis of only one sample of material, the images suggest a correlation with the results obtained in this study. Since the FF + FXT group presented the highest number of voids and the FBF + FXT group presented the worst behavior when analyzing the percentage of voids volume, it can be assumed from the voids detected by the OCT that for low-viscosity materials, characteristics of the restorative material are more determinant for porosity than the restorative technique.[36] The explanation for such an assumption can be attributed to the manufacturing process of these resins, which may generate porosities intrinsic to the material. As the presence of voids in a restoration can result in less fatigue resistance, reduction of flexural strength, and surface irregularity,[26,37] it becomes even more critical in the case of flowable bulk fill RBCs since the reduced time required to fill a large cavity using the bulk fill restorative technique demands larger thickness of the material. In contrast, flowable conventional RBCs are indicated for preventive restorations and liners or to restore small lesions due to their reduced mechanical properties.[38]

High-viscosity materials were homogeneous in the syringe samples, suggesting that the technique, whether incremental or bulk fill, was responsible for the incorporation of voids within restorations. Further studies need to be developed to determine the intrinsic porosity of composite resin materials inside their syringes. Likewise, RBCs must be used following the technique recommended by their manufacturers to avoid failures in the restorative technique, with special caution regarding the use of flowable resins given their potential to present greater initial porosities.

CONCLUSIONS

Within the limitations of this laboratory study, it is possible to conclude that the use of flowable RBCs can lead to an increased number and percentage of voids volume in restorations. This appears to be more related to voids present inside the syringe of the material than to the incremental or bulk fill restorative technique performed.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Comitê de Ética em Pesquisa FOUSP. The approval code for this study is 81332517.9.0000.0075.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Ferracane JL (2011) Resin composite—State of the art *Dental Materials* **27(1)** 29-38.
2. Samet N, Kwon KR, Good P, & Weber HP (2006) Voids and interlayer gaps in class 1 posterior composite restorations: A comparison between a microlayer and a 2-layer technique *Quintessence International* **37(10)** 803-809.
3. Jarisch J, Lien W, Guevara PH, Greenwood WJ, & Dunn WJ (2016) Microcomputed tomographic comparison of posterior composite resin restorative techniques: Sonicated bulk fill versus incremental fill *General Dentistry* **64(5)** 20-23.
4. Hirata R, Pacheco RR, Caceres E, Janal MN, Romero MF, Giannini M, Coelho PG, & Rueggeberg FA (2018) Effect of sonic resin composite delivery on void formation assessed by micro-computed tomography *Operative Dentistry* **43(2)** 144-150.
5. Sabbagh J, McConnell RJ, & McConnell MC (2017) Posterior composites: Update on cavities and filling techniques *Journal of Dentistry* **57** 86-90.
6. Ferracane JL, Hilton TJ, Stansbury JW, Watts DC, Silikas N, Ilie N, Heintze S, Cadenaro M, & Hickel R (2017) Academy of Dental Materials guidance—Resin composites: Part II—Technique sensitivity (handling, polymerization, dimensional changes) *Dental Materials* **33(11)** 1171-1191.
7. Abbas G, Fleming GJ, Harrington E, Shortall AC, & Burke FJ (2003) Cuspal movement and microleakage in premolar teeth restored with a packable composite cured in bulk or in increments *Journal of Dentistry* **31(6)** 437-444.
8. Roggendorf MJ, Kramer N, Appelt A, Naumann M, & Frankenberger R (2011) Marginal quality of flowable 4-mm base vs. conventionally layered resin composite *Journal of Dentistry* **39(10)** 643-647.
9. Opdam NJ, Roeters JJ, Joosten M, & Veeke O (2002) Porosities and voids in class I restorations placed by six operators using a packable or syringable composite *Dental Materials* **18(1)** 58-63.
10. Bicalho AA, Pereira RD, Zanatta RF, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2014) Incremental filling technique and composite material—Part I: Cuspal deformation, bond strength, and physical properties *Operative Dentistry* **39(2)** E71-E82.

11. Soares CJ, Rosatto C, Carvalho VF, Bicalho AA, Henriques J, & Faria ESAL (2017) Radiopacity and porosity of bulk-fill and conventional composite posterior restorations—Digital X-ray analysis *Operative Dentistry* **42(6)** 616-625.
12. Chadwick RG, McCabe JF, Walls AW, & Storer R (1989) The effect of placement technique upon the compressive strength and porosity of a composite resin *Journal of Dentistry* **17(5)** 230-233.
13. Lagouvardos P, Nikolinakos N, & Oulis C (2015) Volume fraction and location of voids and gaps in ultraconservative restorations by X-ray computed micro-tomography *Dental Research Journal* **12(6)** 520-527.
14. Sarrett DC (2005) Clinical challenges and the relevance of materials testing for posterior composite restorations *Dental Materials* **21(1)** 9-20.
15. Peutzfeldt A & Asmussen E (2004) Determinants of in vitro gap formation of resin composites *Journal of Dentistry* **32(2)** 109-115.
16. Ferracane JL, Hilton TJ, Stansbury JW, Watts DC, Silikas N, Ilie N, Heintze S, Cadenaro M, & Hickel R (2017) Academy of Dental Materials guidance—Resin composites: Part II—Technique sensitivity (handling, polymerization, dimensional changes) *Dental Materials* **33(11)** 1171-1191.
17. Bakhsh TA, Sadr A, Shimada Y, Tagami J, & Sumi Y (2011) Non-invasive quantification of resin-dentin interfacial gaps using optical coherence tomography: Validation against confocal microscopy *Dental Materials* **27(9)** 915-925.
18. Huang D, Swanson EA, Lin CP, Schuman JS, Stinson WG, Chang W, Hee MR, Flotte T, Gregory K, Puliafito CA, & Fujimoto JG (1991) Optical coherence tomography *Science* **254(5035)** 1178-1181.
19. Fercher AF (2010) Optical coherence tomography—Development, principles, applications *Zeitschrift für Medizinische Physik* **20(4)** 251-276.
20. Machoy M, Seeliger J, Szyszka-Sommerfeld L, Koprowski R, Gedrange T, & Wozniak K (2017) The use of optical coherence tomography in dental diagnostics: A state-of-the-art review *Journal of Healthcare Engineering* **2017** 7560645.
21. Shimada Y, Sadr A, Sumi Y, & Tagami J (2015) Application of optical coherence tomography (OCT) for diagnosis of caries, cracks, and defects of restorations *Current Oral Health Reports* **2(2)** 73-80.
22. Ishibashi K, Ozawa N, Tagami J, & Sumi Y (2011) Swept-source optical coherence tomography as a new tool to evaluate defects of resin-based composite restorations *Journal of Dentistry* **39(8)** 543-548.
23. Sampaio CS, Rodrigues RV, Souza-Junior EJ, Freitas AZ, Ambrosano GM, Pascon FM, & Puppin-Rontani RM (2016) Effect of restorative system and thermal cycling on the tooth-restoration interface—OCT evaluation *Operative Dentistry* **41(2)** 162-170.
24. Borges BC, de Assuncao IV, de Aquino CA, de Melo Monteiro GQ, & Gomes AS (2016) Marginal and internal analysis of preheated dental fissure-sealing materials using optical coherence tomography *International Dental Journal* **66(1)** 23-28.
25. Nazari A, Sadr A, Saghiri MA, Campillo-Funollet M, Hamba H, Shimada Y, Tagami J, & Sumi Y (2013) Non-destructive characterization of voids in six flowable composites using swept-source optical coherence tomography *Dental Materials* **29(3)** 278-286.
26. Kwon SR, Oyoyo U, & Li Y (2014) Influence of application techniques on contact formation and voids in anterior resin composite restorations *Operative Dentistry* **39(2)** 213-220.
27. Kim EH, Jung KH, Son SA, Hur B, Kwon YH, & Park JK (2015) Effect of resin thickness on the microhardness and optical properties of bulk-fill resin composites *Restorative Dentistry and Endodontics* **40(2)** 128-135.
28. Gale MS & Darvell BW (1999) Thermal cycling procedures for laboratory testing of dental restorations *Journal of Dentistry* **27(2)** 89-99.
29. Ernst CP, Cortain G, Spohn M, Rippin G, & Willershausen B (2002) Marginal integrity of different resin-based composites for posterior teeth: An in vitro dye-penetration study on eight resin-composite and compomer-/adhesive combinations with a particular look at the additional use of flow-composites *Dental Materials* **18(4)** 351-358.
30. Meng Z, Yao XS, Yao H, Liang Y, Liu T, Li Y, Wang G, & Lan S (2009) Measurement of the refractive index of human teeth by optical coherence tomography *Journal of Biomedical Optics* **14(3)** 034010.
31. Feilzer AJ, De Gee AJ, & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.
32. Opdam NJ, Roeters JJ, Peters TC, Burgersdijk RC, & Teunis M (1996) Cavity wall adaptation and voids in adhesive class I resin composite restorations *Dental Materials* **12(4)** 230-235.
33. Chesterman J, Jowett A, Gallacher A, & Nixon P (2017) Bulk-fill resin-based composite restorative materials: a review *British Dental Journal* **222(5)** 337-344.
34. Almeida LJ, Penha KJS, Souza AF, Lula ECO, Magalhaes FC, Lima DM, & Firoozmand LM (2017) Is there correlation between polymerization shrinkage, gap formation, and void in bulk fill composites? A muCT study *Brazilian Oral Research* **31** e100.
35. Balthazard R, Jager S, Dahoun A, Gerdolle D, Engels-Deutsch M, & Mortier E (2014) High-resolution tomography study of the porosity of three restorative resin composites *Clinical Oral Investigations* **18(6)** 1613-1618.
36. Drummond JL (2008) Degradation, fatigue, and failure of resin dental composite materials *Journal of Dental Research* **87(8)** 710-719.
37. Lynch CD, Opdam NJ, Hickel R, Brunton PA, Gurgan S, Kakaboura A, Shearer AC, Vanherle G, & Wilson NH (2014) Guidance on posterior resin composites: Academy of Operative Dentistry—European Section *Journal of Dentistry* **42(4)** 377-383.
38. Baroudi K & Rodrigues JC (2015) Flowable resin composites: A systematic review and clinical considerations *Journal of Clinical Diagnostic Research* **9(6)** Ze18-Ze24.

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Enamel Etching for Universal Adhesives: Examination of Enamel Etching Protocols for Optimization of Bonding Effectiveness

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Clinical Relevance

Conventional phosphoric acid etching with reduced etching times and polyalkenoic acid etching for 15 seconds are potential optimal etching protocols to improve enamel bonding effectiveness with universal adhesives, unlike phosphoric acid ester monomer etching.

SUMMARY

Objective: The purpose of this study was to evaluate whether different enamel etching methods with reduced etching times would improve the bonding effectiveness of universal adhesives.

Methods and Materials: Three enamel etching methods, phosphoric acid ester monomer (PPM) etching, phosphoric acid (PPA) etching, and polyalkenoic acid (PLA) etching, and three universal adhesives, G-Premio Bond (GP), Prime&Bond elect (PE), and Scotchbond Universal Adhesive (SU), were evaluated. Initial bond strengths and fatigue strengths of universal adhesives to ground enamel and ground enamel etched for less than one, five, 10, and 15 seconds using different etching methods were determined. The bonded fatigue specimens were loaded using a sine wave at a frequency

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of 20 Hz for 50,000 cycles or until failure occurred with a staircase method. Atomic force micrograph (AFM) observations along with measurements of surface Ra roughness and modified surface area of enamel with different etching protocols were also conducted.

Results: The bond fatigue durability of universal adhesives to enamel with PPA etching from less than one to 15 seconds and PLA etching for 15 seconds was significantly higher than that to ground enamel. The bond fatigue durability to enamel with PPM etching was not increased compared with ground enamel. The surface Ra roughness and surface area obtained with AFM of enamel increased after PPA and PLA etching, and those values were significantly higher than those of ground enamel. Furthermore, surface Ra roughness and surface area with PPA etching were significantly higher than those with PLA etching. However, surface Ra roughness and surface area of enamel with PPM etching were similar to those of ground enamel regardless of etching time.

Conclusion: PPA etching for less than one to 15 seconds and PLA etching for 15 seconds improve universal adhesive bonding, surface Ra roughness, and surface area of enamel. However, PPM etching is not effective, regardless of etching time, in improving bonds strengths, increasing surface roughness, and increasing surface area.

INTRODUCTION

A recent trend in simplifying and streamlining adhesive systems has been the use of universal adhesives applied in either etch-and-rinse or self-etch modes.[1] Scotchbond Universal Adhesive (3M Oral Care, St Paul, MN, USA) was introduced in 2012 as the first commercial universal adhesive. Other manufacturers later released similar universal adhesives with various distinctive characteristics such as the ability to be used with various substrates,[2] shortened application times,[3] or various levels of tooth substrate wetness.[4] It has been reported that the bond durability of most universal adhesives to enamel in etch-and-rinse and self-etch modes,[5] and dentin in self-etch mode,[6] is lower than that of two-step self-etch adhesives. Nevertheless, the clinical use of universal adhesives is rapidly increasing due to their versatility. Therefore, the optimal conditions for universal adhesive application, including the use of enamel etchants, deserve continued investigation.

A systematic review and meta-analysis of the bond strength (micro-shear and micro-tensile bond strengths) of universal adhesives by Rosa and others[7] reported that, although the dentin bond strength of universal adhesives is not influenced by the bonding strategy used, the enamel bond strength is higher in etch-and-rinse mode than in self-etch mode. These results may indicate that universal adhesives are best used in etch-and-rinse mode. On the other hand, it has been reported in clinical studies of universal adhesives (Scotchbond Universal Adhesive [3M Oral Care] and Tetric N-Bond Universal [Ivoclar Vivadent, Schaan, Liechtenstein]) over three years by Loguercio and others[8,9] that clinical results are not dependent on the bonding strategies used for resin composite restorations in noncarious cervical lesions and that the use of etch-and-rinse mode had only a minor effect for universal adhesives. These clinical studies[8,9] may suggest that the higher laboratory enamel bond strength of universal adhesive in etch-and-rinse mode does not have a strong impact on the clinical results of adhesively bonded resin composite restorations. Based on clinical results, the use of universal adhesives in self-etch mode may be preferable for clinicians because of the simplified process and cost effectiveness, as phosphoric acid etching is not used. The incongruity between the laboratory and clinical studies means that it is still unclear whether best practice with universal adhesives should be etch-and-rinse or self-etch mode. This conflict cannot be easily resolved as both laboratory studies and relatively short-term clinical studies have important limitations.

Many clinicians still prefer to use etch-and-rinse mode for adhesive systems with the standard etching protocol of 30%-40% phosphoric acid (PPA) for 15 seconds.[10] The advantages of the use of PPA etching for enamel include increases in the surface wettability,[11] surface roughness,[12] and surface free-energy,[13] leading to improved bonding, even though the surface hardness of enamel decreases.[14] On the other hand, PPA etching of dentin leads to decreased wettability and increased hydrophobicity of the surface compared with ground dentin due to the aggressive demineralization of the smear layer and the superficial layer of dentin.[15] In addition to these unfavorable characteristics of the adherent for bonding, which are well known, Tay and others[16] reported that the hydrophobicity of demineralized dentin leads to osmosis of water content from deeper

dentin, causing weaker bonding due to osmotic blisters and the hydrolysis of the adhesive itself. Furthermore, PPA etching of dentin activates endogenous collagenolytic proteases associated with the degradation of the interface between the adhesive and dentin.[17] In this way, clinicians who prefer to use universal adhesives in etch-and-rinse mode may believe that doing so has disadvantages due to the etchant's influence on enamel and dentin, even if it has many advantages for enamel bonding.

Recent clinical studies[8,9] suggest that the use of universal adhesives in etch-and-rinse mode does not have a strong and immediate effect on clinical results of resin composite restorations. However, a recent review of laboratory results[7] does suggest a strong effect on the enamel bond strength and no effect on dentin bonding of universal adhesives in etch-and-rinse mode. Taken together, these studies suggest that it may be possible to develop optimal protocols of universal adhesives in etch-and-rinse mode that preserve benefits for enamel bonding while minimizing negative influences on other tooth substrates. That is, a lower level of demineralization of enamel and dentin may be sufficient to achieve the clinical benefits while minimizing damage to both types of tooth substrate. A previous study[17] suggested that aggressive demineralization may create a potential vulnerability for acidic attacks and secondary caries formation, although the need for some degree of etching is undeniable. One possibility is to use a weaker acid, and another is to reduce the etching time below 15 seconds. Thus, evaluations of optimal protocols, such as the use of alternative etching methods and reduced etching times with universal adhesives, may be desirable.

Several alternative etching methods are currently available. Polyalkenoic acid (PLA), which is a family of complex acids including polyacrylic, polyitaconic, and polymaleic acids, is used in etching for cavity cleansing and conditioning in glass ionomer cement restorations.[18] In these restorative procedures, PLA etching promotes the formation of irregularities on the surface of the substrate, forming an intermediate layer that facilitates ion exchange between the glass ionomer matrix and the calcium in the partially demineralized smear layer.[19] In addition, PLA etching forms insoluble salts with calcium due to its high molecular weight that serve as receptors for primary chemical bonds between glass ionomer cement and the carboxyl groups of polyalkenoic acid.[20] Alternatively, another method for etching has been a newly developed phosphoric acid ester monomer (PPM) etching. Whereas PPA has three hydroxyl groups, a PPM has at least one of these groups replaced with an ester; thus, the monomer can simultaneously demineralize and also bond to substrates. PLA and PPM etching can remove the smear layer and modify enamel and dentin surfaces with less demineralization than PPA etching, minimizing the unfavorable effects for bonding to tooth substrates. Because of these characteristics, PLA and PPM are potentially attractive for application with universal adhesives in etch-and-rinse mode. Accordingly, a comparison between different etching methods and conventional PPA etching for universal adhesives in terms of enamel bond durability may be valuable.

The purpose of this laboratory study was to investigate the central hypothesis of whether different etching methods with reduced etching times would improve the enamel bonding effectiveness of universal adhesives. The two null hypotheses tested were as follows: 1) there would be no differences in bond durability of universal adhesives to enamel among different etching protocols; and 2) different etching protocols would not influence enamel surface morphology.

METHODS AND MATERIALS

Study Materials

Three universal adhesives, 1) G-Premio Bond (GP, GC, Tokyo, Japan); 2) Prime&Bond elect (PE, Dentsply Sirona, Milford, DE, USA); and 3) Scotchbond Universal Adhesive (SU, 3M Oral Care), and three etchants, 1) a PLA etchant (Enamel Conditioner, Shofu, Kyoto, Japan); 2) a PPM etchant (Multi Etchant, Yamakin, Tokyo, Japan); and 3) a PPA etchant (Ultra-Etch, Ultradent Product, South Jordan, UT, USA), were evaluated. Z100 Restorative (3M Oral Care) was used as the resin composite for the bonding procedures. The study materials are listed in Table 1 with associated components.

Specimen Preparation

Sectioned buccal and lingual halves of de-identified extracted human molar teeth with the apical portions removed were mounted in 25-mm brass rings using an acrylic resin (Bosworth Fastray, Keystone Industries, Myerstown, PA, USA). Flat enamel surfaces were prepared on the mounted buccal or lingual surfaces by wet grinding using a gradually increasing grit sequence (180, 320, 600, 1200, 2000 and 4000 grit) of silicon carbide papers (Struers, Cleveland, OH, USA) in a grinder-polisher (Ecomet 4, Buehler, Lake Bluff, IL, USA). These

Table 1: *Materials Used in This study*

Materials	Type of Material (Code)	Components	Manufacturer
G-Premio Bond	Universal adhesive (GP)	MDP, 4-MET, MEPS, methacrylate monomer, acetone, water, silica, initiator	GC
Prime&Bond elect	Universal adhesive (PE)	Dipentaerythritol pentaacrylate monophosphate, polymerizeable dimethacrylate resin, polymerizeable trimethacrylate resin, diketon, organic phosphine oxide, stabilizers, cetylamine hydrofluoride, acetone, water	DENTSPLY Caulk
Scotchbond Universal Adhesive	Universal adhesive (SU)	Bis-GMA, HEMA, decamethylene dimethacrylate, ethyl methacrylate, propenoic acid, methyl-reaction products with decanediol and phosphorous oxide, copolymer of acrylic and itaconic acid, dimethylaminobenzoate, methyl ethyl ketone, ethanol, water, silane treated silica, initiator	3M Oral Care
Enamel Conditioner	Etchant (PLA)	Polyalkenoic acids, thickener, pigment	Shofu
Multi Etchant	Etchant (PPM)	M-TEG-P, thickener, pigment	Yamakin
Ultra-Etch	Etchant (PPA)	35% Phosphoric acid, glycol, cobalt aluminate blue spinel	Ultradent Products
Z100 Restorative	Resin composite	Bis-GMA, TEGDMA, silane treated ceramic, benzotriazolyl methylphenol	3M Oral Care

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate, MDP, methacryloyloxydecyl dihydrogen phosphate; MEPS, methacryloyloxyalkyl thiophosphate methylmethacrylate; M-TEG-P, 11-methacryloyloxy-4-ethyleneglycol dihydrogen phosphate; TEGDMA: triethylene glycol dimethacrylate; 4-MET, 4-methacryloyloxyethyl trimellitate.

surfaces were then washed with an air-water spray and air-dried using a dental three-way syringe at a distance of 5 cm above the surface and an air pressure of 2.5 kgf/cm^2.

One control group per adhesive was prepared by rinsing ground enamel with an air-water spray for 10 seconds and air drying without the application of etching agents. Four etching time groups were prepared for each etching method and adhesive: 1) ground enamel with etching agent applied and then immediately rinsed with an air-water spray for 10 seconds and air dried (less than one-second group); 2) ground enamel with etching agent for five seconds and then rinsed with an air-water spray for 10 seconds and air dried (five-second group); 3) ground enamel with etching for 10 seconds and then rinsed with an air-water spray for 10 seconds and air dried (10-second group); and 4) ground enamel with etching agent for 15 seconds and then rinsed with an air-water spray for 10 seconds and air dried (15-second group). The specimens were prepared under ambient laboratory conditions of 23 ± 2°C and 50 ± 10% relative humidity.

Initial Bond Strength Tests

Stainless steel molds with an inner diameter of 2.38 mm, an outer diameter of 4.8 mm, and a height of 2.6 mm were used to bond a resin composite to enamel surfaces in all groups. The mold-enclosed method was used to minimize the impact of repeated application of force to the resin in the sample and to make the applied force as close to a pure shear force as possible. The bonding side surfaces of the metal molds were prepared with a releasing agent (3% solution of paraffin in hexane) to chemically isolate the bonded enamel/resin composite interface. The enamel surfaces of the different etching protocols were then treated with the universal adhesives according to the manufacturers' instructions (Table 2). A fixture was used to position and hold the molds over the bonding surfaces as the resin composite was placed into the mold using a condenser to an approximate height of 2.5 mm. The resin composite was photo-cured for 40 seconds at a standardized distance of 1 mm using a quartz-tungsten halogen (QTH) curing unit (Spectrum 800 Curing Unit, Dentsply Sirona) set at 800 mW/cm^2. The bonded

Table 2: *Application Protocol for Tested Adhesives*

Adhesive	Application Protocol
GP	Adhesive applied to air-dried enamel/dentin surface for 10 seconds. Strong stream of air applied over the liquid adhesive for five seconds or until adhesive no longer moved and the solvent had completely evaporated. Adhesive light cured for 10 seconds.
PE	Adhesive applied to air-dried enamel/dentin surface with rubbing for 20 seconds. Gentle stream of air applied over the liquid for at least five seconds. Adhesive light cured for 10 seconds.
SU	Adhesive applied to air-dried enamel/dentin surface with rubbing action for 20 seconds, and then medium air pressure applied to surface for five seconds. Adhesive light cured for 10 seconds.

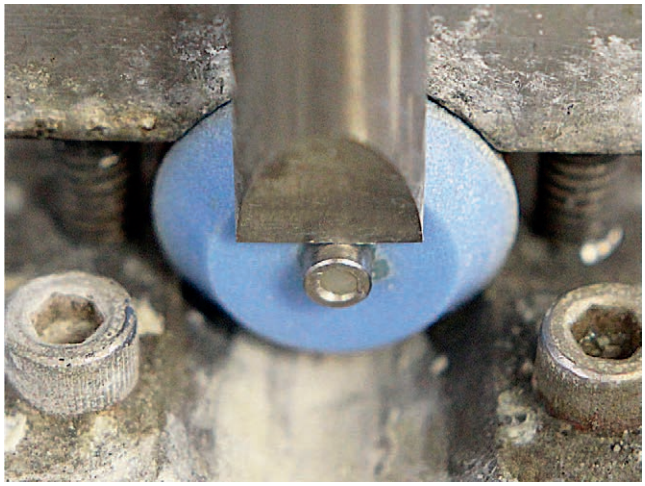

Figure 1. *Figure illustrating the "Mold-Enclosed" bonding jigs.*

specimens were stored in 37°C distilled water for 24 hours before initial bond strength testing.

Shear bond strength tests were carried out on all groups with the stainless steel mold described above (mold-enclosed method). A chisel-shaped metal rod was used to apply the load on the stainless steel molds immediately adjacent to the flat ground enamel surfaces (Figure 1). The specimens (n=15) were loaded to failure using an all-electric dynamic test instrument (ElectroPuls E1000, Instron, Norwood, MA, USA) with a crosshead speed of 1 mm/min. Initial shear bond strengths (MPa) were calculated from the peak load at failure divided by the bonded surface area.

Bond Fatigue Strength Test

A staircase method was used to perform the bond fatigue strength tests using the all-electric dynamic test instrument. Twenty specimens (n=20) were prepared for all groups for each of the adhesives being tested. Subsequently, the specimens were stored in 37°C distilled water for 24 hours prior to testing. Tsujimoto and others[21,22] reported that the bond fatigue strength, using the mold enclosed system used in this study, was not influenced by the frequency rate (2 or 20 Hz) or the numbers of cycles (50,000, 100,000, or 1,000,000 cycles); thus, the fatigue load was applied using a sine wave at a frequency of 20 Hz for 50,000 cycles or until failure occurred. The initial peak load for bond fatigue strength testing for each of the adhesives was set at a level approximately half of the initial shear bond strength determined for each group and the lower load limit was set at 0.4 N. Subsequent specimen loading was adjusted upward or downward approximately 10% from the previous load depending on specimen survival or failure. This procedure was repeated for the 20 specimens in each test group. The test specimens were immersed in room temperature water (23±2°C) during bond fatigue strength testing to minimize the influence of any temperature changes on the bonded assemblies during testing. The bond fatigue strength and standard deviation were calculated using the formula described by Draughn.[23]

Failure Mode Analysis

Bond failure sites were assessed after the initial bond strength and fatigue strength tests by a single experienced individual using an optical microscope (MZ16, Leica Microsystems, Heerbrugg, Switzerland) at 20× magnification. The failure modes were assessed on the percentage of substrate area (adhesive, resin composite, or enamel) observed on both the debonded resin composite cylinders and the enamel bonding sites. The failure modes were classified as follows: 1) adhesive failure at the interface; 2) cohesive failure in resin composite; 3) cohesive failure in enamel; or 4) mixed failure.

Atomic Force Microscopy Evaluation

Six enamel specimens were prepared for each of the previously described groups for atomic force microscopy (AFM) evaluation. Each specimen was imaged in three different locations near the center of the specimen. Prior to measurement, the specimens were blown with dried air in a sweeping motion for approximately five seconds at 0.55 MPa to remove any dust particles and surface debris. AFM evaluations were performed using a scanning probe microscope/AFM (5420 SPM/AFM Microscope, Agilent Technologies, Santa Clara, CA, USA) in an acoustical and mechanical isolation chamber under ambient laboratory conditions (23±2°C and 50±10% relative humidity). Micrographs were obtained in constant force contact mode with a silicon nitride (Si_3N_4) cantilever (tip radius of ≤10 nm and spring constant of 0.2 N/m; BudgetSensors, Sofia, Bulgaria) at 512 lines per image at a rate of three to four lines per second. In this mode, the AFM is in constant feedback with the cantilever to maintain a constant deflection by modulating the AFM/specimen separation with piezoelectric motors. A schematic diagram of the AFM is shown in Figure 2.

Micrographs (30×30 μm) were analyzed, quantitatively and qualitatively, with image analysis software (Gwyddion, Central European Institute of Technology, Brno, Czech Republic). Enamel surface

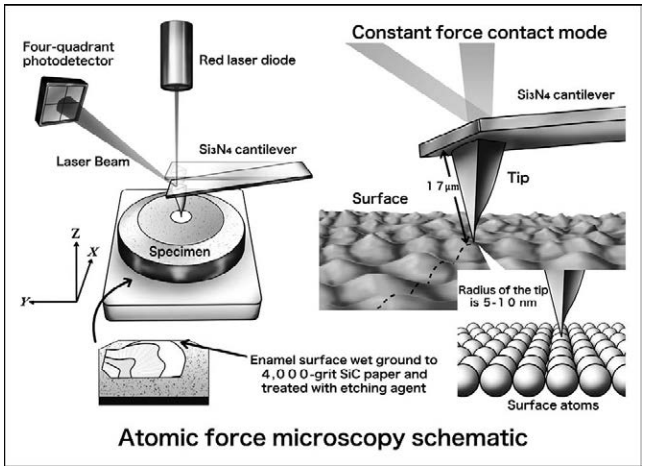

Figure 2. *Schematic illustration of AFM, where a flexible cantilever with an atomically sharp tip is systematically swept across the surface of a sample. A laser beam positioned at the back of the aluminum coated, reflective cantilever captures three-dimensional topography changes, which are reflected in changes to the laser beam position on a four-quadrant photodetector monitored by a specialized computer system.*

roughness was quantified in terms of Ra (nm), the arithmetic average of the absolute values of the profile height deviations from the mean line, recorded within an equivalent imaging area. Among several parameters for measurement of surface roughness, the average Ra is most commonly reported.[24-26] Similarly, the geometric surface area (μm^2) was obtained, which measures the modified surface area (above the anticipated 900 μm^2 for a flat surface) due to height variations across the surface. Experienced investigators assessed qualitative intra- and intergroup differences.

Statistical Analysis

The initial bond strength, surface Ra roughness, and surface area data obtained with AFM were analyzed using a three-way analysis of variance (ANOVA; factors: 1) etching method, 2) etching time and 3) adhesive) followed by Tukey's *post hoc* honest significant differences test with a significance level of $\alpha = 0.05$. Fisher's exact test was used to statistically analyze the failure mode after initial bond strength and fatigue strength testing with a significance level of $\alpha = 0.05$. These statistical analyses were conducted using a commercial statistical software package (SPSS Statistics, International Business Machines, Armonk, NY, USA). The bond fatigue strength data were analyzed using a modified *t*-test with Bonferroni correction and significance level of $\alpha = 0.05$ (custom program).

RESULTS

Initial Bond Strength

The results for the effects of etching protocols on the initial bond strength of the universal adhesives to enamel are shown in Table 3. The three-way ANOVA revealed that the main factors 1) etching method (F=23.231, p<0.001), 2) etching time (F=18.112, p<0.001), and 3) adhesive (F=8.112, p=0.012) significantly affected these values, and etching method and etching time were the most influential. In addition, an interaction between etching method and etching time (F=3.112, p=0.032) was observed, but there was no significant interaction between the other factors.

The initial bond strengths of the universal adhesives increased immediately after PPA etching and were significantly higher (p<0.05) than those of the control, but the values were not influenced by etching times (p>0.05). The initial bond strength of the universal adhesives with PLA etching gradually increased with the increase of etching times, and the values with PLA etching for 15 seconds were similar to those with PPA etching at all etching times.

The initial bond strengths of universal adhesives with PPM etching did not increase and were similar (p>0.05) to those of the control regardless of pre-etching time. Furthermore, the initial bond

Table 3: *Effects of Etching Protocols on the Initial Shear Bond Strength (MPa) of the Universal Adhesives to Enamel*[a]

Etching Time (s)	G-Premio Bond			Prime&Bond elect			Scotchbond Universal		
	PPA	PLA	PPM	PPA	PLA	PPM	PPA	PLA	PPM
Control	25.4 (3.3)^{a}			26.9 (4.2)^{a}			27.1 (3.8)^{a}		
<1	32.5 (4.3)^{b,A}	27.5 (3.4)^{a,B}	19.9 (3.8)^{b,C}	37.1 (4.3)^{b,A}	29.3 (4.7)^{a,B}	24.2 (4.5)^{a,C}	42.2 (5.3)^{b,A}	35.4 (4.2)^{b,B}	24.9 (3.8)^{a,C}
5	33.1 (4.1)^{b,A}	27.7 (4.6)^{a,B}	20.5 (3.7)^{b,C}	38.2 (3.9)^{b,A}	28.3 (5.7)^{a,B}	24.0 (5.5)^{a,B}	42.1 (4.3)^{b,A}	36.5 (4.9)^{b,B}	24.2 (5.1)^{a,C}
10	32.9 (3.8)^{b,A}	28.0 (3.4)^{a,B}	20.7 (3.5)^{b,C}	39.2 (3.2)^{b,A}	31.2 (5.0)^{a,b,B}	22.6 (4.5)^{a,C}	43.2 (4.4)^{b,A}	38.0 (5.4)^{b,B}	25.1 (4.4)^{a,C}
15	33.7 (4.1)^{b,A}	32.3 (4.3)^{b,A}	21.3 (4.0)^{b,B}	39.0 (4.3)^{b,A}	37.1 (5.3)^{b,A}	22.3 (3.5)^{a,B}	43.7 (4.3)^{b,A}	41.3 (4.7)^{c,A}	24.1 (4.7)^{a,B}

[a] *Values in parentheses are standard deviations. Same superscript lowercase letter in same column indicates no significant difference (p>0.05). Same superscript capital letter within individual rows indicates no significant difference (p>0.05).*

Table 4: *Effects of Etching Protocols on the Bond Fatigue Strength (MPa) of the Universal Adhesives to Enamel*[a]

Etching Time (s)	G-Premio Bond			Prime&Bond elect			Scotchbond Universal		
	PPA	PLA	PPM	PPA	PLA	PPM	PPA	PLA	PPM
Control	12.1 (1.3)[a]			13.2 (1.2)[a]			13.7 (1.4)[a]		
<1	15.5 (1.3)[b,A]	13.0 (1.2)[a,B]	9.7 (1.0)[b,C]	18.1 (1.3)[b,A]	13.3 (1.5)[a,B]	11.5 (1.2)[a,B]	20.1 (1.3)[b,A]	16.7 (1.2)[b,B]	12.1 (1.2)[a,C]
5	16.2 (1.1)[b,A]	13.1 (1.2)[a,B]	9.9 (1.4)[b,C]	18.2 (1.4)[b,A]	13.5 (1.1)[a,B]	11.6 (1.5)[a,B]	20.4 (1.3)[b,A]	17.7 (1.2)[b,B]	12.3 (1.1)[a,C]
10	16.1 (1.2)[b,A]	13.3 (1.3)[a,B]	10.1 (1.5)[b,C]	19.2 (1.2)[b,A]	14.9 (1.0)[a,b,B]	11.3 (1.3)[a,C]	21.0 (1.4)[b,A]	18.3 (1.4)[b,B]	12.4 (1.4)[a,C]
15	16.5 (1.1)[b,A]	16.4 (1.2)[b,A]	10.8 (1.0)[b,B]	19.0 (1.0)[b,A]	18.1 (1.3)[b,A]	11.3 (1.5)[a,B]	21.7 (1.3)[b,A]	20.2 (1.7)[c,A]	12.3 (1.3)[a,B]

[a] *Values in parentheses are standard deviations. Same superscript lowercase letter in same column indicates no significant difference (p>0.05). Same superscript capital letter within individual rows indicates no significant difference (p>0.05).*

strengths of universal adhesives in the control group were not influenced ($p>0.05$) by the type of adhesive.

Bond Fatigue Strength

The results for the effects of etching protocols on the bond fatigue strength of the universal adhesives to enamel are shown in Table 4. The bond fatigue strengths of the universal adhesives increased significantly, immediately after PPA etching, and were significantly higher ($p<0.05$) than those of the control group, regardless of etching time.

The bond fatigue strengths of GP and PB with PLA pre-etching for 15 seconds were significantly higher ($p<0.05$) than those of the control and other etching time groups. On the other hand, the bond fatigue strengths of SU with PLA etching increased significantly with increased etching times and were significantly higher ($p<0.05$) than those of the control group. There were no statistically significant differences in bond fatigue strengths of universal adhesives between those with PLA etching for 15 seconds and those with PPA etching, regardless of adhesive.

However, the bond fatigue strengths of the universal adhesives with PPM etching did not increase and were similar ($p>0.05$) to those of the control group, regardless of etching time. The bond fatigue strength of universal adhesives in the control was not influenced by the type of adhesive.

Failure Mode Analysis

The failure mode analyses for initial bond strength and fatigue strength testing are shown in Tables 5 and 6, respectively. Failure mode was overwhelmingly adhesive failure. Fisher's exact tests did not reveal statistically significant differences ($p>0.05$) in failure mode depending on the type of adhesive, the etchant, or the etching time. All adhesives with PPM etching showed exclusively adhesive failure at all etching times. Similarly, only a single case of nonadhesive failure was observed for etching times below 10 seconds, which was also the only case of nonadhesive failure for GP. Other cases showed a small number of cohesive failure in enamel, and two cases of mixed failure were observed overall, but these other failure modes were not significant; in no case were fewer than 85% of failures classified as adhesive failure. This is normal for studies of enamel bonding using the mold-enclosed method.

AFM Observations

Representative three-dimensional topographic images of enamel surfaces obtained by AFM are shown in Figures 3A through 3M. In the control (Figure 3a), periodic grooves made by polishing were observed, and a smear layer was not clearly observed on the ground surfaces. There were clear morphologic differences between enamel with PPA and PLA etching and the control. Topographic

Table 5: *Effect of Etching Protocols on Surface Roughness (nm) and Surface Area (μm^2) of Enamel Surfaces*[a]

Etching Time (s)	Surface Roughness			Surface Area		
	PPA	PLA	PPM	PPA	PLA	PPM
Control	10.9 (2.4)[a]			901.5 (1.2)[a]		
<1	139.5 (28.3)[b,A]	23.0 (6.2)[b,B]	8.4 (1.9)[a,C]	1150.8 (65.4)[b,A]	912.4 (4.7)[b,B]	902.1 (1.6)[a,C]
5	154.3 (19.8)[b,A]	23.9 (6.9)[b,B]	9.0 (2.7)[a,C]	1178.5 (74.6)[b,A]	911.5 (4.2)[b,B]	902.2 (2.8)[a,C]
10	173.9 (25.5)[b,c,A]	23.4 (6.3)[b,B]	9.2 (1.5)[a,C]	1202.2 (38.5)[b,c,A]	910.9 (2.5)[b,B]	900.9 (0.4)[a,C]
15	194.3 (28.2)[c,A]	24.8 (8.0)[b,B]	9.0 (3.8)[a,C]	1223.4 (42.1)[c,A]	912.2 (3.3)[b,B]	903.0 (0.1)[a,C]

[a] *Values in parentheses are standard deviations. Same superscript lowercase letter in same column indicates no significant difference (p>0.05). Same superscript capital letter within individual rows indicates no significant difference (p>0.05).*

Table 6: *Failure Mode Analysis of Debonded Specimens After Shear Fatigue Strength Tests*[a]

Etching Time (s)	G-Premio Bond			Prime&Bond Elect			Scotchbond Universal		
	PPA	PLA	PPM	PPA	PLA	PPM	PPA	PLA	PPM
Control	[100/0/0/0][a]			[100/0/0/0][a]			[100/0/0/0][a]		
<1	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]
5	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]
10	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[86/0/14/0][a,A]	[93/0/7/0][a,A]	[100/0/0/0][a,A]	[86/7/7/0][a,A]	[100/0/00][a,A]	[100/0/0/0][a,A]
15	[93/0/7/0][a,A]	[100/0/0/0][a,A]	[100/0/0/0][a,A]	[86/0/14/0][a,A]	[93/0/7/0][a,A]	[100/0/0/0][a,A]	[86/7/7/0][a,A]	[93/0/7/0][a,A]	[100/0/0/0][a,A]

[a] *Percentage of failure mode [adhesive failure/cohesive failure in resin/cohesive failure in enamel/mixed failure]. Same small letter in same column indicates no significant difference (*$p > 0.05$*). Same capital letter within individual rows indicates no significant difference (*$p > 0.05$*).*

images of enamel surfaces with PPA (Figures 3b through 3e) and PLA (Figures 3f through 3i) etching showed micro-irregularities that were different between PPA and PLA etching, and the degree of demineralization for enamel with PLA etching was weaker than those with PPA etching, regardless of etching time. The degree of demineralization appeared to increase with an increase of etching time for both PPA and PLA etching. On the other hand, topographic images of enamel surfaces with PPM etching (Figures 3j through 3m) did not show any morphologic differences compared with those of the control, independent of etching time.

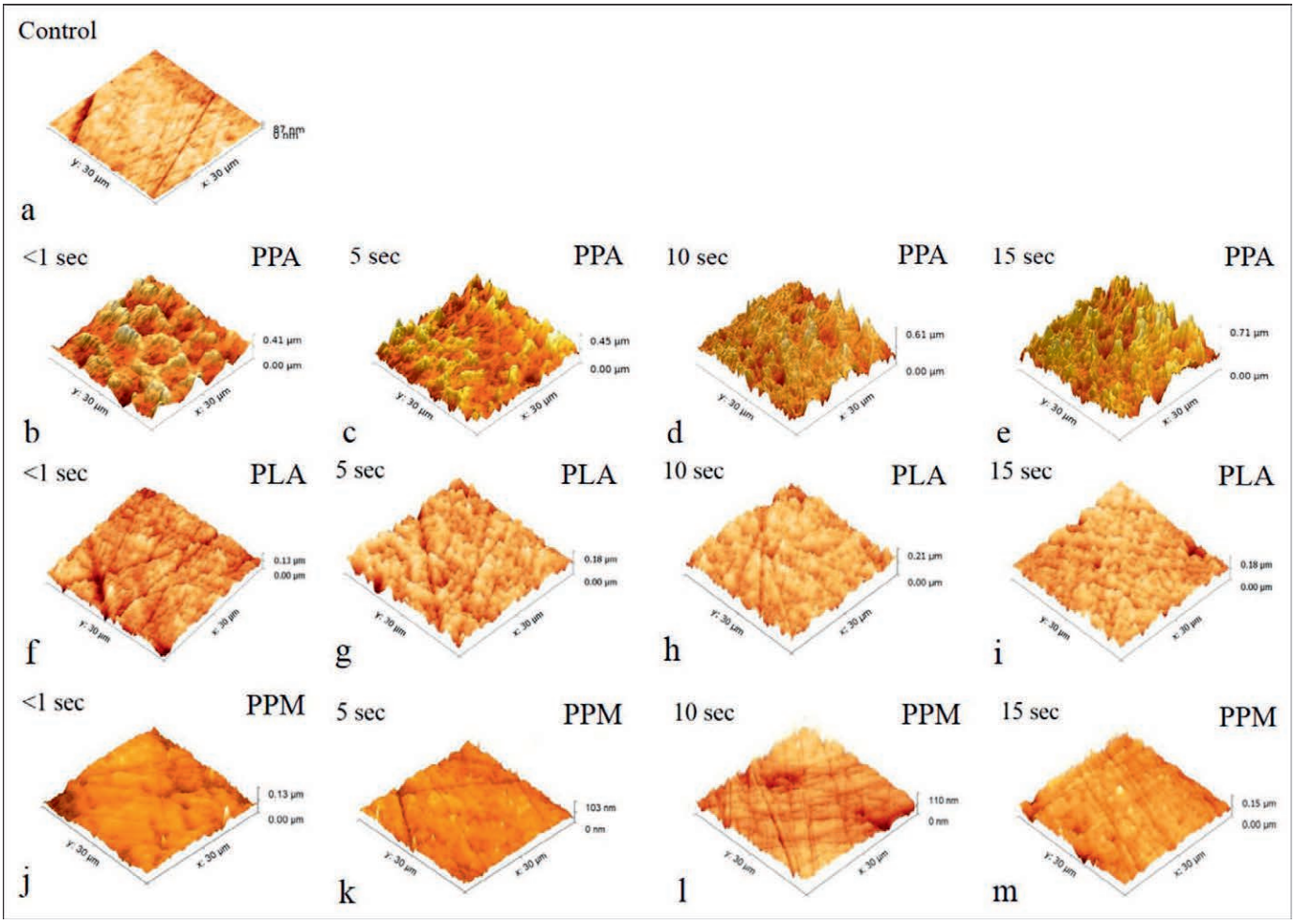

Figure 3. *False color three-dimensional topographic images (30 × 30 μm) of enamel surfaces obtained by constant force atomic force microscopy. Micrograph heights were normalized for accurate visual inspection, where the vertical scale bar shows the maximum feature height in the given micrograph. (a): Control; (b-e): PAA etched <1 to 15 sec; (f-i): PLA etched <1 to 15 sec; and (j-m): PPM etched <1 to 15 sec.*

Table 7: *Effect of Etching Protocols on Surface Roughness and Surface of Enamel Surfaces*[a]

Etching Time (s)	Surface Roughness (nm)			Surface Area (μm^2)		
	PPA	PLA	PPM	PPA	PLA	PPM
Control	10.9 (2.4)[a]			901.5 (1.2)[a]		
<1	139.5 (28.3)[b,A]	23.0 (6.2)[b,B]	8.4 (1.9)[a,C]	1150.8 (65.4)[b,A]	912.4 (4.7)[b,B]	902.1 (1.6)[a,C]
5	154.3 (19.8)[b,A]	23.9 (6.9)[b,B]	9.0 (2.7)[a,C]	1178.5 (74.6)[b,A]	911.5 (4.2)[b,B]	902.2 (2.8)[a,C]
10	173.9 (25.5)[b,c,A]	23.4 (6.3)[b,B]	9.2 (1.5)[a,C]	1202.2 (38.5)[b,c,A]	910.9 (2.5)[b,B]	900.9 (0.4)[a,C]
15	194.3 (28.2)[c,A]	24.8 (8.0)[b,B]	9.0 (3.8)[a,C]	1223.4 (42.1)[c,A]	912.2 (3.3)[b,B]	903.0 (0.1)[a,C]

[a] *Values in parentheses are standard deviations. Same superscript lowercase letter in same column indicates no significant difference (p>0.05). Same superscript capital letter within individual rows indicates no significant difference (p>0.05).*

Surface Roughness and Geometric Surface Area Measurements

The surface roughness (Ra, nm) and surface area (μm^2) obtained with AFM are shown in Table 7. Significantly higher ($p<0.05$) surface roughness and surface area of enamel with the PPA and PLA etching were observed compared with those in the control, but the values of surface roughness and surface area of enamel with PPA etching were significantly higher ($p<0.05$) than those with PLA etching. The surface area of the enamel with PPA etching increased significantly ($p<0.05$) with increased etching time. Surface roughness of the enamel with PPA etching appeared to increase with increased etching time, but this was not significant ($p>0.05$). Although the surface roughness and surface area of enamel with PLA etching were also increased immediately after etching, those values were not influenced by the etching time. On the other hand, the surface roughness and surface area of enamel with PPM etching were not increased compared with the control, and there was no statistically significant difference ($p>0.05$) in the values of surface roughness and surface area between PPM etching and the control.

DISCUSSION

One aspect of this study confirms the effect of reduced PPA etching times on enamel bonding with universal adhesives. PPA etching for 15 seconds is generally recommended by manufacturers when universal adhesives are used in etch-and-rinse mode, based on earlier research results by Barkmeier and others[12,27,28] and Uno and Finger.[29] Those studies reported that PPA etching times in excess of 15 seconds did not increase bond strength to enamel even though the surface roughness of enamel continued to increase. However, the universal adhesives investigated herein are different from the adhesive systems used in those studies. In addition, a recently published article by Tsujimoto and others[30] on enamel adhesion with single-step self-etch adhesives and Stape and others[31] on dentin with universal adhesives revealed that PPA etching with a reduced etching time of three seconds is a potential protocol to improve the bonding of adhesives. Thus, the question of optimal PPA etching time for universal adhesives was revisited.

In the present study, the bond fatigue durability of universal adhesives to enamel with PPA etching was significantly higher than that to ground enamel, but the value was not statistically increased depending on the etching time. In the results for surface characteristics obtained from AFM, surface roughness (67.8-85.3 nm) and surface area (921.9-943.9 μm^2) were increased immediately after PPA etching compared with those of ground enamel (surface Ra roughness: 10.1 nm; surface area: 901.5 μm^2) and micro-irregularities of the surfaces were seen regardless of etching times. Previous studies[24-26] reported that the surface roughness of enamel with PPM etching obtained by AFM showed 160-321 nm; thus, the surface Ra roughness observed in the present study was relatively low compared with earlier studies. However, the surface Ra roughness of the baseline of the previous studies[24-26] was 30-50 nm, whereas that of the baseline of the present study was 10.1 nm. Moreover, slight differences in methodologies used to measure Ra roughness, including image size and resolution, strongly impact subsequent values. In the present study, an enamel surface ground with 4000-grit silicon carbide papers was used as baseline to minimize the influence of any directionality or inhomogeneities of the surface grooving or scratches created by the abrasives on bond fatigue strength testing. Therefore, a lower baseline surface Ra roughness of enamel was found in the present study, but the resultant increase in Ra was similar in magnitude to the earlier studies. In addition, Tsujimoto and others[30] reported that PPA etching with reduced etching times of under 15 seconds can improve the bond durability of simpli-

fied adhesives and the interfacial characteristics of enamel to an adequate level. These findings are consistent with the results of the present study. Thus, the results of this study clearly indicate that PPA etching is still the gold standard for improving enamel bonding, even with universal adhesives, and that PPA etching with reduced etching time for universal adhesives, used in etch-and-rinse mode, may be a potential protocol to improve bonding effectiveness of universal adhesives for use in the clinic. Although, both the study by Stape and others[31] and the present study revealed the effectiveness of PPA etching with reduced etching time for universal adhesives, it is necessary to garner additional data on bonding to unground enamel before making clinical recommendations.

The fact that PPA etching with reduced etching times may be effective for enamel bonding with universal adhesives suggests that different, weaker types of acids or acidic agents may also be effective, particularly if the etching times are extended. This is worth consideration because strong acids may inflict considerable damage on tooth substrates. Therefore, the effect of different etching methods (both PLA and PPM etching) on enamel bonding with universal adhesives, including the effect of reduced etching times, was evaluated.

PLA etching has been widely used over a long period for restoration with glass ionomer cement, and this combination has been extensively investigated.[18,19,32] However, there appear to have been no investigations of whether PLA etching can be used with resin composites, much less any investigation of whether they can be used with universal adhesives. The PLA etchant used in this study is mainly composed of polyacrylic acid and has a pH of approximately 1.5. The pH of PLA is higher than that of PPA etching agent (pH$<$1.0), which may have accounted for the less aggressive demineralization of enamel. The present study showed that the bond fatigue durability of all universal adhesives to enamel with PLA etching for 15 seconds was significantly higher than that to ground enamel and was similar to enamel with PPA etching. In addition, although PLA etching did change the Ra values and surface area of enamel immediately after etching, as with PPA etching, the values measured were significantly lower than those for enamel with PPA etching. Therefore, even with less demineralization of enamel, PLA etching was able to effectively improve the enamel bond fatigue durability of all universal adhesives if applied for 15 seconds. Consequently, PLA etching for 15 seconds may be a potential protocol to improve the enamel bonding of universal adhesives with less damage to enamel than with PPA etching.

On the other hand, the PPM etchant used in this study is mainly composed of 11-methacryloyloxy-4-ethyleneglycol dihydrogen phosphate (11-M-TEG-P). These 11-M-TEG-P monomers can remove the smear layer, demineralize tooth substrates, and chemically bond to hydroxyapatite. For universal adhesives, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) is a key technological factor for chemical bonding with tooth substrates regardless of the bonding strategies used.[34] The idea behind using PPM etching is to enhance the chemical bonding with universal adhesives by supplementing the chemical bonding capacity of MDP to tooth substrates by applying a PPM in two separate steps. In the present study, the bond fatigue durability of PE and SU was not improved by PPM etching, and the bond fatigue durability of GP to enamel with PPM etching was even decreased, regardless of etching time. One plausible rationale for this observation may be that enamel with PPM etching had already reacted with the 11-M-TEG-P in the PPM etchant, and thus the 10-MDP in the universal adhesive could not react with the hydroxyl groups of enamel, leading to a lack of improvement, or even a deterioration, in the bond fatigue durability of the adhesives. Future work is warranted to further elucidate this issue. In addition, measurements of surface Ra roughness and surface area obtained with AFM did not increase with PPM etching, and the morphology of enamel with PPM etching was similar to that of ground enamel. This apparent lack of demineralization may also be related to the enamel bond fatigue durability not being increased by PPM etching. Therefore, the use of PPM etching with universal adhesives for any etching times was not an effective protocol for improving enamel bond fatigue durability in this study.

From the overall results of this study, the null hypotheses that 1) there would be no differences in bond durability of universal adhesives to enamel among different etching protocols, and 2) different etching protocols would not influence enamel surface morphology were both rejected. This study suggests that it may be possible to use universal adhesives with etching protocols for PPA etching with reduced etching times or PLA etching for 15 seconds, but it is imperative that additional data be generated for unground enamel before making any clinical recommendations. However, under the experimental conditions of this study, PPM etching was not effective

in improving enamel bonding with universal adhesives.

CONCLUSION

The results of this study suggest that 1) enamel bond durability with universal adhesives is different depending on the etching protocol and 2) different etching protocols influence enamel surface morphology. Overall, the results show that the enamel bonding of universal adhesives was improved with etching protocols of phosphoric acid etching for reduced etching times from less than one to 15 seconds or with polyacrylic acid etching for 15 seconds. However, phosphoric acid ester monomer etching was not effective in improving bonding with universal adhesives regardless of etching time.

Acknowledgement

The authors thank Jason M. Moody for technical contributions.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Creighton University School of Dentistry. The approval code for this study is 760765-1.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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References

1. Vermelho PM, Reis AF, Ambrosano GMB, & Giannini M (2017) Adhesion of multimode adhesives to enamel and dentin after one year of water storage *Clinical Oral Investigations* **21(5)** 1707-1715.
2. Siqueira F, Cardenas AM, Gutierrez MF, Malaquias P, Hass V, Reis A, Loguercio AD, & Perdigão J (2016) Laboratory performance of universal adhesive systems for luting CAD/CAM restorative materials *Journal of Adhesive Dentistry* **18(4)** 331-340.
3. Saikaew P, Matsumoto M, Chowdhury A, Carvalho RM, & Sano H (2018) Does shortened application time affect long-term bond strength of universal adhesives to dentin? *Operative Dentistry* **43(5)** 549-558.
4. Amsler F, Peutzfeldt A, Lussi A, & Flury S (2015) Bond strength of resin composite to dentin with different adhesive systems: Influence of relative humidity and application time *Journal of Adhesive Dentistry* **17(3)** 249-256.
5. Suda S, Tsujimoto A, Barkmeier WW, Nojiri K, Nagura Y, Takamizawa T, Latta MA, & Miyazaki M (2018) Comparison of enamel bond fatigue durability between universal adhesives and two-step self-etch adhesives: effect of phosphoric acid pre-etching *Dental Materials Journal* **23(2)** 244-255.
6. Tsujimoto A, Barkmeier WW, Takamizawa T, Watanabe H, Johnson WW, Latta MA, & Miyazaki M (2017) Comparison between universal adhesives and two-step self-etch adhesives in terms of dentin bond fatigue durability in self-etch mode *European Journal of Oral Sciences* **125(3)** 215-222.
7. Rosa WL, Piva E, & Silva AF (2015) Bond strength of universal adhesives: a systematic review and meta-analysis *Journal of Dentistry* **43(7)** 765-776.
8. Loguercio AD, de Paula EA, Hass V, Luque-Martinez I, Reis A, & Perdigão J (2015) A new universal simplified adhesive: 36-Month randomized double-blind clinical trial *Journal of Dentistry* **43(9)** 1083-1092.
9. Loguercio AD, Luque-Martinez IV, Fuentes S, Reis A, & Muñoz MA (2018) Effect of dentin roughness on the adhesive performance in non-carious cervical lesions: a double-blind randomized clinical trial *Journal of Dentistry* **69** 60-69.
10. Frankenberger R, Lohbauer U, Roggendorf MJ, Naumann M, & Taschner M (2008) Selective enamel etching reconsidered: better than etch-and-rinse and self-etch? *Journal of Adhesive Dentistry* **10(5)** 339-344.
11. Retief DH, Middleton JC, & Jamison HC (1985) Optimal concentration of phosphoric acid as an etching agent. Part III: enamel wettability studies *Journal of Prosthetic Dentistry* **53(1)** 42-46.
12. Barkmeier WW, Erickson RL, Kimmes NS, Latta MA, & Wilwerding TM (2009) Effect of enamel etching time on roughness and bond strength *Operative Dentistry* **34(2)** 217-222.
13. Tsujimoto A, Iwasa M, Shimamura Y, Murayama R, Takamizawa T, & Miyazaki M (2010) Enamel bonding of single-step self-etch adhesives: influence of surface energy characteristics *Journal of Dentistry* **38(2)** 123-130.
14. Zafar MS, & Ahmed N (2015) The effects of acid etching time on surface mechanical properties of dental hard tissues *Dental Materials Journal* **3(3)** 315-320.
15. Yamauchi K, Tsujimoto A, Jurado CA, Shimatani Y, Nagura Y, Takamizawa T, Barkmeier WW, Latta MA, & Miayzaki M (2019) Etch & rinse vs. self-etch mode for dentin bonding effectiveness of universal adhesives *Journal of Oral Sciences* in press.
16. Tay FR, & Pashley DH (2003) Have dentin adhesives become too hydrophilic? *Journal of Canadian Dental Association* **69(11)** 726-731.
17. Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, & De Stefano Dorigo E (2008) Dental adhesion review: aging and stability of the bonded interface *Dental Materials* **24(1)** 90-101.
18. Inoue S, Abe Y, Yoshida Y, De Munck J, Sano H, Suzuki K, Lambrechts P, & Van Meerbeek B (2004) Effect of conditioner on bond strength of glass-ionomer adhesive to dentin/enamel with and without smear layer interposition *Operative Dentistry* **29(6)** 685-692.

19. Hoshika S, De Munck J, Sano H, Sidhu SK, & Van Meerbeek B (2015) Effect of conditioning and aging on the bond strength and interfacial morphology of glass-ionomer cement bonded to dentin *Journal of Adhesive Dentistry* **17(2)** 141-147.
20. Fukuda R, Yoshida Y, Nakayama Y, Okazaki M, Inoue S, Sano H, Suzuki K, Shintani H, & Van Meerbeek B. (2003) Bonding efficacy of polyalkenoic acids to hydroxyapatite, enamel and dentin *Biomaterials* **24(11)** 1861-1867.
21. Tsujimoto A, Barkmeier WW, Erickson RL, Takamizawa T, Latta MA, & Miyazaki M (2018) Influence of number of cycles on shear fatigue strength of resin composite bonded to enamel and dentin using adhesives in self-etch mode *Dental Materials Journal* **37(1)** 113-121.
22. Tsujimoto A, Barkmeier WW, Erickson RL, Fischer NG, Markham MD, Takamizawa T, Latta MA, & Miyazaki M (2018) Shear fatigue strength of resin bonded to dentin under cyclic fatigue at physiological frequency *European Journal of Oral Sciences* **126(4)** 316-325.
23. Draughn RA (1979) Compressive fatigue limits of composite restorative materials *Journal of Dental Research* **58(3)** 1093-1096.
24. Loyola-Rodriguez JP, Zavala-Alonso V, Reyes-Vela E, Patiño-Marin N, Ruiz F, & Anusavice KJ (2010) Atomic force microscopy observation of the enamel roughness and depth profile after phosphoric acid etching *Journal of Electron Microscopy* **59(2)** 119-125.
25. Cerci BB, Roman LS, Guariza-Filho O, Camargo ES, & Tanaka OM. Dental enamel roughness with different acid etching times: atomic force microscopy study *European Journal of General Dentistry* **1(3)** 187-191.
26. Hashimoto Y, Hashimoto Y, Nishiura A, & Matsumoto N (2012) Atomic force microscopy observation of enamel surfaces treated with self-etching primer *Dental Materials Journal* **32(1)** 181-188.
27. Barkmeier WW, Gwinnett AJ, & Shaffer SE (1985) Effects of enamel etching time on bond strength and morphology *Journal of Clinical Orthodontics* **19(1)** 36-38
28. Barkmeier WW, Shaffer SE, & Gwinnett, AJ (1986) Effects of 15 vs 60 second enamel acid conditioning on adhesion and morphology *Operative Dentistry* **11(3)** 111-116.
29. Uno S & Finger WJ (1995) Effect of acid etchant composition and etch duration on enamel loss and resin composite bonding *American Journal of Dentistry* **8(4)** 165-169.
30. Tsujimoto A, Barkmeier WW, Takamizawa T, Watanabe H, Johnson WW, Latta MA, & Miyazaki M (2016) Influence of duration of phosphoric acid pre-etching on bond durability of universal adhesives and surface free-energy characteristics of enamel *European Journal of Oral Sciences* **124(4)** 377-386.
31. Stape THS, Wik P, Mutluay MM, Al-Ani AAS, & Tezvergil-Mutluay A (2018) Selective dentin etching: A potential method to improve bonding effectiveness of universal adhesives *Journal of the Mechanical Behavior of Biomedical Materials* **86** 14-22.
32. Cardoso MV, Delmé KI, Mine A, Neves Ade A, Coutinho E, De Moor RJ, & Van Meerbeek B (2010) Towards a better understanding of the adhesion mechanism of resin-modified glass-ionomers by bonding to differently prepared dentin *Journal of Dentistry* **38(11)** 921-929.
33. Cardoso MV, Delmé KI, Mine A, Neves Ade A, Coutinho E, De Moor RJ, & Van Meerbeek B (2010) Towards a better understanding of the adhesion mechanism of resin-modified glass-ionomers by bonding to differently prepared dentin *Journal of Dentistry* **38(11)** 921-929.
34. Makishi P, André CB, Ayres A, Martins AL, & Giannini M (2016) Effect of storage time on bond strength and nanoleakage expression of universal adhesives bonded to dentin and etched enamel *Operative Dentistry* **41(3)** 305-317.

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Influence of Spectroscopic Techniques on the Estimation of the Degree of Conversion of Bulk-fill Composites

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Clinical Relevance

Clinicians should understand that degree of conversion (DC) of bulk-fill composites varies between flowable and sculptable materials. The technique of measurement significantly influences the reported values of DC.

SUMMARY

Objectives: To compare the degree of conversion (DC) of different flowable and sculptable bulk-fill composites (BFC), at 0- and 4-mm depths from the surface, by Fourier transform infrared (FTIR), attenuated total reflection FTIR (ATR-FTIR), and FT-Raman spectroscopic techniques.

Methods and Materials: Six BFC were investigated, including three sculptable composites (Admira Fusion [Voco], Aura Bulk Fill [SDI], and X-tra Fill [Voco]) and three flowable composites (Venus Bulk Fill [Heraeus], Filtek [3M], and X-tra Base [Voco]). Three molds of each composite were light cured as specified by the manufacturer. For each mold, slices corresponding to 0-mm (surface) and 4-mm depth were analyzed by spectroscopic techniques: ATR-FTIR, FTIR, and FT-Raman. The spectra of uncured composite material were used as an analytical control for background subtraction of the treated composite. The area and amplitude of the reference peaks (1607 and 1637 cm^{-1}) were obtained to calculate the DC percentage at 0- and 4-mm depth. A Kruskal-Wallis nonparametric test was used for materials, and paired comparisons were made using Mann-Whitney nonparametric test. Wilcoxon's rank test was used for comparison between spectro-

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scopic methods and between 0- and 4-mm depth in each composite. Significance was accepted at $p<0.05$.

Results: FTIR showed significantly lower DC values, both in areas and amplitudes of the peaks, when compared with the results reported by different BFC. Differences between the surface and 4-mm depth were detected more precisely by FT-Raman. ATR-FTIR obtained DC values significantly higher than those obtained by FTIR.

Conclusions: The vibrational spectroscopy method significantly influenced DC measurements of the flowable and sculptable BFC explored.

INTRODUCTION

Composites have become the most commonly used restorative materials in clinical dentistry.[1] A major drawback of resin composites is polymerization shrinkage, which reportedly occurs in the range of 2-5 vol%.[2] Polymerization shrinkage causes stress at the tooth-restoration interface as the elastic modulus of the composite increases during curing. This shrinkage stress is thought to be related to most clinical problems that cause failure of the restorations.[1,3-5]

Bulk-fill composite (BFC) has been introduced to overcome the drawbacks of the rather time-consuming incremental technique. Manufacturers claim that BFCs generate lower polymerization shrinkage stress and have better light transmission properties than conventional composites, which allows them to be used for increments of up to 4- to 5-mm thickness.[5] The capacity for polymerization in depth of BFC has been studied using the scraping test according ISO 4049 specifications,[6,7] microhardness,[6-12] or the degree of conversion (DC).[8,11,13-16] DC is a key material feature of dental resin composites, because it affects both physical and mechanical polymer properties as well as biocompatibility.[17,18] This DC parameter represents the percentage of unreacted carbon double bonds (C=C) in the cured material in relation to the uncured material.[19]

Vibrational spectroscopic techniques, such as Fourier transform infrared (FTIR), attenuated total reflection FTIR (ATR-FTIR), and FT-Raman spectroscopies, have been used to detect and analyze the structural and chemical composition of various materials. Although FT-Raman, FTIR, and ATR-FTIR are complementary spectroscopic techniques, they are dependent on different selection rules and polarization properties. While FTIR spectroscopy is based on the absorption of light, Raman is based on the scattering of light by vibrating molecules (ie, inelastic scattering of vibrating molecules), and ATR-FTIR uses a property of total internal reflection resulting in an evanescent wave. All of these spectroscopic methods are suitable for the analysis of the capacity or polymerization efficiency of dental resins, usually expressed as DC.[16,20,21] Nevertheless, results of DC measurements of dental composites seem to vary for similar materials and technical procedures depending on the sample preparation and the method of spectra analysis: considering overlapped or hidden peaks or the procedure for calculating the amplitude or the areas of the DC reference peaks.[20,22-26] For instance, Pianelli and others[27] found that the DC calculated using the area ratio after deconvolution appeared weaker than that obtained using the height ratio with or without deconvolution. On the other hand, the opposite was found by Khalil and others[26] using FT-Raman spectroscopy.

Many brands are introducing novel BFC in clinical dentistry. Owing to differences in rheological properties and application techniques, BFCs are further classified as either low viscosity (flowable), allowing better adaptation to cavity walls, or high viscosity (sculptable) material types.[17] Although much effort has been made to quantify the DC of bulk composites, the influence of the spectroscopic method used has been scarcely studied. In our study, we compared the polymerization characteristics of the main currently available flowable and sculptable BFC at 0-mm and 4-mm depth curing conditions.

Therefore, the main objectives were to evaluate (1) the influence of the spectroscopy technique (FTIR, ATR-FTIR, and FT-Raman) and the spectrometric analysis method in the calculation of the DC values in BFC and (2) the influence of different types of flowable or sculptable BFC and the curing-depth conditions.

METHODS AND MATERIALS

Sample Preparation

Six BFC were investigated (Table 1), including three flowable (VBF, FBF, and XbBF) and three sculptable (AFBF, ABF, XfBF) composites. Uncured material was inserted into cylindrical stainless-steel molds with an internal hole of 4 mm in diameter and 5 mm height (divided into three parts: 1 mm, 3 mm, and 1 mm; Figure 1).

Table 1: *Bulk-fill Dental Composites Used in the Study*

Material	Code	Shade	Manufacturer	Type	Resin Matrix
Venus Bulk Fill	VBF	Universal	Heraeus Kulzer GmbH, Hanau, Germany	Flowable bulk-fill	UDMA, EBPDMA
Filtek Bulk Fill	FBF	Universal	3 M ESPE, ESPE, St. Paul, MN, USA	Flowable bulk-fill	Bis-GMA, Bis-EMA, UDMA
X-tra Base	XbBF	Universal	Voco GmbH, Cuxhaven, Germany	Flowable bulk-fill	UDMA, Bis-EMA
Admira Fusion	AFBF	A1	Voco GmbH, Cuxhaven, Germany	Sculptable bulk-fill ORMOCER	Aromatic and aliphatic dimethacrylates, methacrylate-functionalized polysiloxane
Aura Bulk Fill	ABF	Universal	SDI Limited Bayswater, Bayswater, Victoria, Australia	Sculptable bulk-fill	UDMA, Bis-EMA, Bis-GMA, TEGDMA
X-tra Fill	XfBF	Universal	Voco GmbH, Cuxhaven, Germany	Sculptable bulk-fill	Bis-GMA, UDMA, TEGDMA

Abbreviations: Bis-EMA, ethoxylated bisphenol-A-dimethacrylate; Bis-GMA, bisphenol-A-glycidyldimethacrylate; EBPDMA, ethoxylated bisphenol A dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

The molds were slightly overfilled with the material, and the excess was then extruded, oxygen inhibition prevented and a smooth surface obtained by applying a transparent polyester Mylar strip on the top and bottom of the mold and pressing firmly with a glass slab. Care was taken to minimize entrapped air while uncured materials were placed in the mold. The mold sample was then light cured from the top (ie, in contact with the mold surface, 0-mm depth) as specified by the manufacturer (Table 1), using a light-emitted diode curing unit (1200 mW/cm^2; Bluephase 20i, IvoclarVivadent, Schaan, Liechtenstein). All samples were stored dry at 37°C in

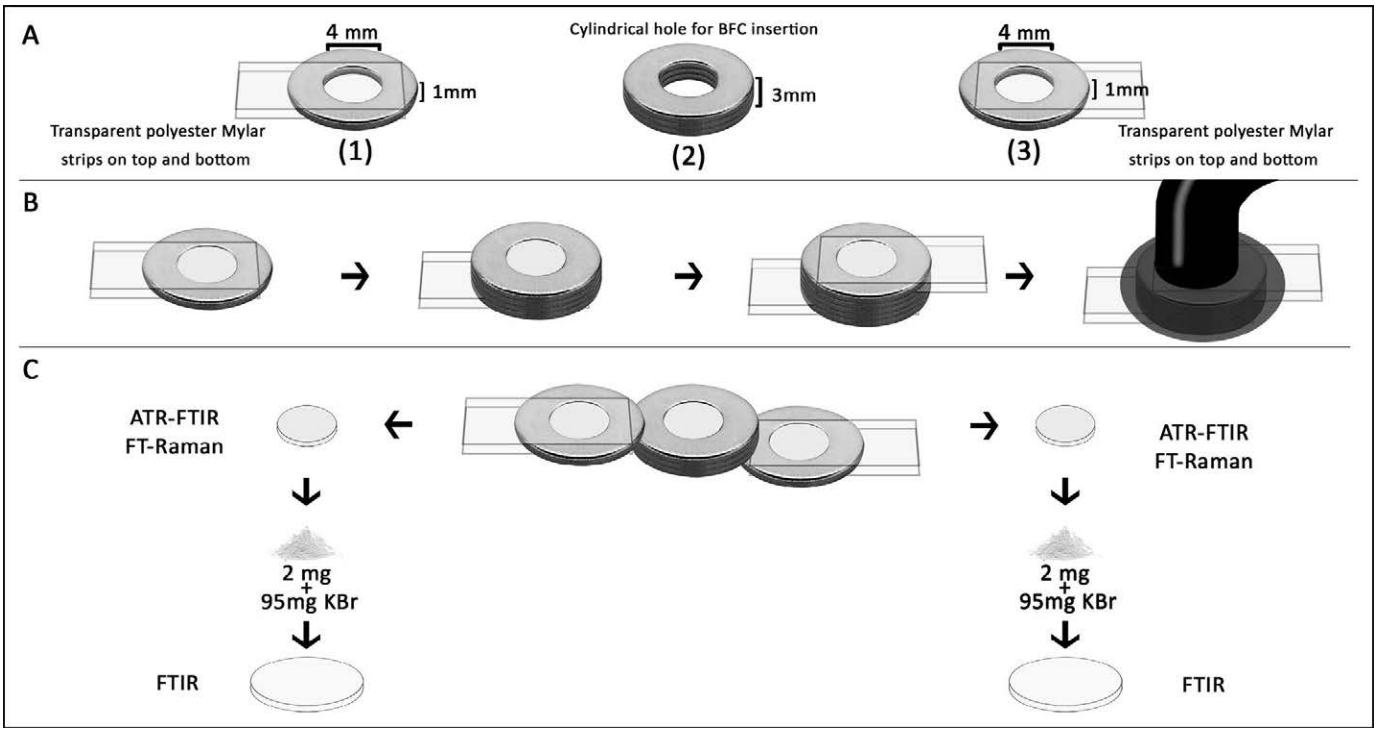

Figure 1. *Sample preparation for Fourier transform infrared (FTIR), attenuated total reflection–Fourier transform infrared (ATR-FTIR), and FT-Raman spectroscopic analyses. (A): Cylindrical stainless-steel mold with an internal hole of 4 mm in diameter and 5 mm in height divided into three parts: (1) and (3) 1-mm thick with transparent polyester Mylar strips at the top and bottom and (2) 3-mm thick. (B): Sequence of placement of the bulk-fill composites and polymerization. (C): After being stored dry at 37°C in the dark for 24 hours, they were unmolded, and two slices (0- and 4-mm depth, left and right, respectively) were obtained and analyzed using ATR-FTIR and FT-Raman. Then, these slices were pulverized, and 2 mg was mixed with 95 mg of potassium bromide to obtain disks under pressure that were analyzed using FTIR. For further details, see the "Methods and Materials" section.*

Table 1: *Bulk-Fill Dental Composites Used in the Study (ext.)*

Material	Filler		Curing Time
	Composition	Load (wt%/vol %)	
Venus Bulk Fill	Ba-Al-F-Silicate glass, YbF_3, SiO_2	65/38	20 s
Filtek Bulk Fill	Ytterbium trifluoride filler (sizes range from 0.1 to 5.0 μm) and zirconia/silica (size range of 0.01 to 3.5 μm)	64.5/42.5	20 s
X-tra base	Barium glass ceramic, fumed silica (size 3.5 μm)	75/60 (58)	10 s
Admira Fusion	Ba-Al-glass, pyrogenic SiO_2	84/-	20 s
Aura Bulk Fill	Silica, silinated barium glass particles, Ultra High Density pre-polymerized filler barium glass particles.	82/65	20 s
X-tra fill	Barium glass ceramic, fumed silica (size 3.5 μm)	86/70.1	10 s

darkness for 24 hours after irradiation prior to further analyses.

Three molds of each BFC were processed, and two slices from each one of these molds, at 0- and 4-mm depth from the surface, were obtained. Accordingly, three samples of each uncured composite material were also analyzed. Three spectra from each sample were recorded using different spectroscopic techniques.

In Vitro Analysis

Fourier transform–Raman spectroscopy (FT-Raman)—Spectra were obtained using a JASCO NRS-5100 spectrometer (Jasco Inc, Easton, MD, USA) coupled to an optical Olympus microscope and equipped with a charge-coupled device detector (1024×256 pixels) cooled by a Peltier-effect module. FT-Raman spectra were excited using a 785-nm red diode laser kept at 500 mW. Spectra were acquired between 1500 and 1750 cm^{-1} with a resolution of 1 cm^{-1}, an exposure time of 10 seconds, and 10 accumulations.

ATR-FTIR—Samples were analyzed using an FTIR JASCO 6200 spectrometer equipped with a diamond-tipped ATR accessory (ATR Pro ONE, Jasco). Samples were placed on the ATR crystal holder covering the entire crystal surface. All spectra were acquired between 600 and 4000 cm^{-1}, with a spectral resolution of 2 cm^{-1}, acquisition time of 10 seconds, and 10 accumulations.

FTIR—Each sample was powdered, and 2 mg was mixed with 95 mg of FTIR-grade potassium bromide (KBr) and pressed under a vacuum at 9 metric tons for 10 minutes.

Infrared spectral data were collected on an FTIR spectrometer (JASCO 6200) coupled with a transmission sample holder. The spectra were acquired in the absorption mode between 600 and 4000 cm^{-1}, at 2 cm^{-1} resolution over 1024 scans. A reference disk (97 mg of KBr) was used for background correction every 10 spectra acquisitions.

Spectral Analysis

Spectral analyses were carried out for the calculation of the DC for all the composite materials at 0 and 4 mm of depth. The DC of each composite was calculated by comparing the area and amplitude of particular peaks in the spectra derived from the uncured and cured resin.

After using a standard baseline technique,[24] a region of the spectra between 1575 and 1660 cm^{-1} was selected, and two peaks were considered for DC calculation (Figure 2): 1607 cm^{-1} (internal standard aromatic carbon double bond, C=C) and 1637 cm^{-1} (methacrylate C=C). For AFBF, based on Ormocer technology, reference peaks were considered at 1584 cm^{-1} (C=C) and at 1638 cm^{-1} (methacrylate C=C). The DC was calculated as follows:

$$\%\mathrm{DC}_{area/amplitude} = \left[1 - \frac{(1637\ \mathrm{cm}^{-1}/1607\ \mathrm{cm}^{-1})_{after\ curing}}{(1637\ \mathrm{cm}^{-1}/1607\ \mathrm{cm}^{-1})_{before\ curing}}\right] \times 100$$

Overlapping peaks were resolved, and their amplitudes and integrated areas were measured using curve-fitting software (Peakfit v4.12, Systat Software, Chicago, IL, USA). The second derivative method was used to resolve the peak calculations within the spectral region. Peak amplitude and position were allowed to vary within 5% and ±2 cm^{-1}, respectively. The degree of smoothing was set at 10% (Savitzky-Golay algorithm), and a mixed Gaussian-Lorentzian function was used to fit the contours (ie, curve shape and width) of the bands, allowing for a detailed and quantitative analysis of

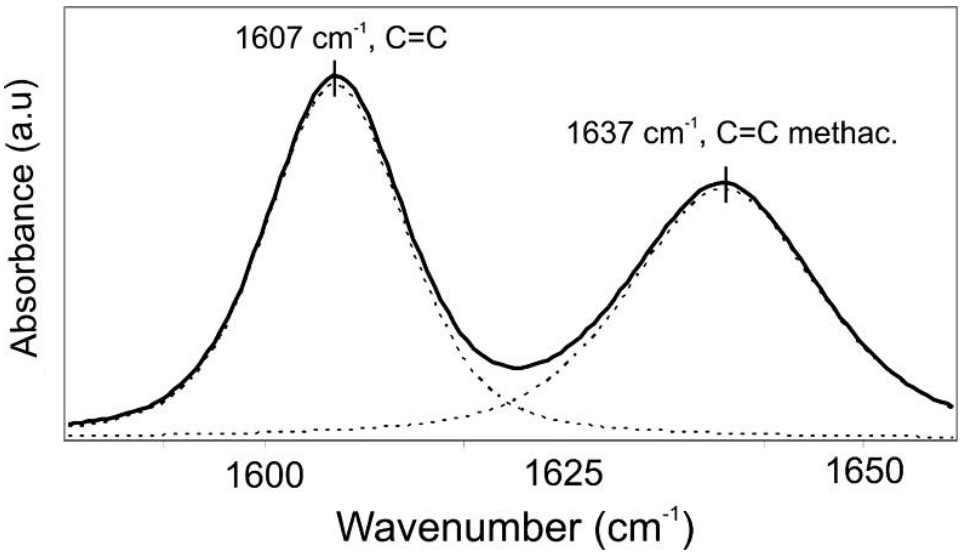

Figure 2. *Curve-fitting analysis for an average FT-Raman spectrum from bulk-fill composites (except for AFBF). Area and amplitude for the two reference peaks were calculated: 1607 cm^{-1} (internal standard aromatic carbon double bond, C=C) and 1637 cm^{-1} (methacrylate C=C).*

DC values. Curve fitting was accepted when r^2 reached values up to 0.995.

Statistical Analysis

After exploring the data distribution (Shapiro-Wilk test), nonparametric statistics were applied. A Kruskal-Wallis nonparametric test was used for these materials, and subsequent paired comparisons were made using a Mann-Whitney nonparametric test. Wilcoxon's rank test was used for comparisons between different spectroscopic methods and between 0- and 4-mm depths in each composite. Significance was accepted at $p<0.05$.

RESULTS

Comparisons Between Vibrational Spectroscopy Methods

Table 2 summarizes the DC measurements by FT-Raman, ATR-FTIR, and FTIR on the surface (0 mm) and at 4-mm depth for the six bulk-fill dental composites analyzed.

When the ratio of the integrated areas was used to determine the DC at 24 hours postcure, ATR-FTIR obtained higher percentages of conversion than the other vibrational spectroscopy methods, although the results for each composite were highly variable.

In the following section, "=" means "not significant" and "<" and ">" mean "significantly lower" and "significantly higher," respectively. At 0-mm depth for flowable BFC, the DC values obtained for VBF by integrated areas were ATR-FTIR=FT-Raman>FTIR, the DC values of FBF were ATR-FTIR>FT-Raman>FTIR ($p<0.012$ for all comparisons), and the DC values of XbBF were ATR-FTIR=FTIR>FT-Raman. ATR-FTIR also gave the highest DC values for the three sculptable BFC, although the DC for AFBF was ranked ATR-FTIR>FT-Raman>FTIR ($p<0.030$); for ABF, the DC was ATR-FTIR>FT-Raman=FTIR; and for XfBF, the DC ranged from 84% to 47%, with the ranking ATR-FTIR>FT-Raman>FTIR ($p<0.020$).

At 4-mm depth, among flowable BFC, VBF and FBF showed similar behavior to that obtained at 0-mm depth. The exception was XbBF, for which ATR-FTIR=FT-Raman>FTIR ($p<0.015$). The highest DC was also obtained with ATR-FTIR for the three sculptable BFC, with similar relations between methods as those described at 0-mm depth, except for AFBF, which showed a DC with ATR-FTIR>FTIR>FT-Raman ($p<0.030$).

When the relation between the amplitude of the reference peaks was considered (Table 2), the DC values for the flowable BFC were highly material dependent at 0-mm depth, VBF obtained a higher DC with FT-Raman compared with ATR-FTIR=FTIR, FBF showed significantly ($p=0.008$) higher DC values for FT-Raman=ATR-FTIR compared with FTIR, and XbBF showed a higher DC by ATR-FTIR>FT-Raman=FTIR. Among the sculptable BFC, ABF and XfBF showed similar DC using ATR-FTIR and FT-Raman, and these values were higher than FTIR values, although the differences were significant only for ABF ($p=0.008$). AFBF achieved similar results with the three spectroscopic methods, at between 55.10% and 56.56%, with no significant differences ($p>0.600$).

Table 2: *Degree of Conversion Values (Mean [SD]) Obtained for Relative Area or Amplitude by FT-Raman, ATR-FTIR, and FTIR at Surface (0 mm) and 4-mm Depth for Six Bulk-fill Dental Composites (BFC)*[a]

	Flowable BFC			Sculptable BFC		
	VBF	**FBF**	**XbBF**	**AFBF**	**ABF**	**XfBF**
0 mm						
Area						
FT-Raman	65.21 (2.82)^{2,a}	60.36 (1.67)^{3,a}	50.27 (8.25)^{4,a}	68.80 (8.20)^{1,a}	54.04 (7.31)^{3,4,a}	65.57 (5.71)^{1,2,a}
ATR-FTIR	67.37 (7.78)^{3,a}	71.54 (6.56)^{2,3,b}	68.19 (6.29)^{2,3,b}	76.43 (7.46)^{1,2,b}	81.79 (5.27)^{1,b}	84.07 (3.22)^{1,b}
FTIR	59.46 (5.07)^{1,b}	46.63 (7.96)^{3,c}	59.55 (7.63)^{2,b}	56.61 (6.18)^{1,c}	54.33 (7.02)^{1,2,a}	47.32 (9.16)^{2,3,c}
Amplitude						
FT-Raman	73.01 (2,01)^{1,A}	63.13 (1.59)^{2,A}	54.34 (3.08)^{3,A}	56.56 (10.38)^{2,3,A}	57.06 (6.83)^{2,3,A}	58.12 (3.87)^{3,A}
ATR-FTIR	65.16 (7.26)^{1,B}	58.76 (6.35)^{1,2,A}	59.51 (4.20)^{2,3,B}	56.15 (9.43)^{3,A}	65.69 (10.80)^{1,2,3,A}	63.75 (4.23)^{1,2,A}
FTIR	60.41 (3.87)^{1,B}	47.79 (3.76)^{2,B}	49.83 (6.27)^{2,A}	55.10 (7.40)^{1,2,A}	43.95 (3.17)^{3,B}	46.05 (6.96)^{2,3,B}
4 mm						
Area						
FT-Raman	73.95 (7.21)^{1,a}	62.65 (2.86)^{2,a}	58.72 (2.47)^{3,a}	50.79 (7.33)^{2,3,a}	48.83 (4.77)^{2,3,a}	57.20 (8.32)^{2,3,4,a}
ATR-FTIR	78.73 (9.50)^{1,a}	71.97 (5.47)^{3,b}	56.09 (8.46)^{3,a}	74.44 (8.59)^{1,2,b}	80.62 (5.97)^{1,b}	79.70 (5.37)^{1,b}
FTIR	55.87 (4.63)^{1,b}	41.64 (5.97)^{3,c}	48.91 (3.88)^{2,b}	61.37 (7.91)^{1,c}	56.51 (6.87)^{1,c}	48.77 (5.84)^{2,c}
Amplitude						
FT-Raman	75.53 (1.38)^{1,A}	65.51 (1.77)^{2,A}	60.01 (1.56)^{3,A}	44.01 (8.01)^{5,A}	52.20 (4.46)^{4,A}	52.21 (4.26)^{4,A}
ATR-FTIR	76.79 (7.27)^{1,A}	66.09 (8.65)^{2,A}	52.94 (4.25)^{4,B}	58.95 (6.51)^{3,B}	67.57 (8.86)^{1,2,B}	62.04 (2.83)^{2,3,B}
FTIR	56.21 (3.40)^{2,B}	43.44 (6.23)^{3,B}	46.73 (4.98)^{3,C}	60.78 (4.47)^{1,B}	44.20 (5.40)^{3,C}	45.50 (5.35)^{3,C}

[a] *Different numbers in rows represent significant differences between materials. They are ordered from greater to lower values. Different lowercase letters in columns represent significant differences in the comparison between techniques, in the area relation, for each BFC. Different uppercase letters in columns represent significant differences in the comparison between techniques, in the amplitude relation, for each BFC.*

At 4-mm depth, VBF and FBF showed similar DC using FT-Raman and ATR-FTIR, while FTIR obtained significantly lower DC values ($p<0.008$ in all comparisons). DC values for XbBF were ranked FT-Raman>ATR-FTIR>FTIR ($p<0.030$). Among the sculptable materials, ABF and XfBF had the highest DC values using ATR-FTIR>FT-Raman>FTIR ($p<0.040$), while for AFBF, the methods were ranked FTIR=ATR-FTIR>FT-Raman.

Comparisons Between Methods of Analysis: Relation of Integrated Areas vs Amplitude of Reference Peaks

Figure 3 summarizes the differences between the mean DC calculated for each BFC with relation to areas or amplitudes of the reference peaks, at 0- and 4-mm depth. Using ATR-FTIR, at 0-mm depth, all the BFC behaved better when the relation of areas was applied, except VBF, which had a similar DC using both methods of analysis. At 4-mm depth, only sculptable BFC had a significantly higher DC by areas ratio, with differences in DC with respect to amplitudes ratio next to 20% for XfBF. With FT-Raman, we obtained higher DC values using the relation of amplitudes for flowable BFC. Differences were significant for VBF and FBF at 0-mm depth ($p=0.008$ in both materials) and for FBF only at 4-mm depth ($p=0.008$). Sculptable composites AFBF and XfBF had higher DC values (about 12% and 7%, respectively) using the relation of areas rather than amplitudes at 0-mm depth ($p=0.008$) and at 4-mm depth differences were about 14% for AFBF ($p=0.008$) and 5% for XfBF ($p=0.139$); ABF demonstrated the opposite results, with significantly higher DC values (about 3%-4%, $p=0.008$) using the amplitudes ratio. Finally, by FTIR, no significant differences were found between the DC using areas or amplitudes ratios, except for XbBF at 0-mm depth ($p=0.021$) and ABF at 0- and 4-mm depth ($p=0.008$ both).

Comparisons Between BFC Materials

When we compared the relative efficiency of cure, the impact of the vibrational spectroscopy technique and the methodology for spectral analysis were evident, along with that of material. Significant differences are represented by different numbers within rows in Table 2 and are ordered from higher to lower values. At 4-mm depth, flowable BFC had a better DC than sculptable BFC using FT-Raman spectroscopy, but using ATR-FTIR, ABF, and XfBF (two sculptable BFC) achieved the best results. At 4-

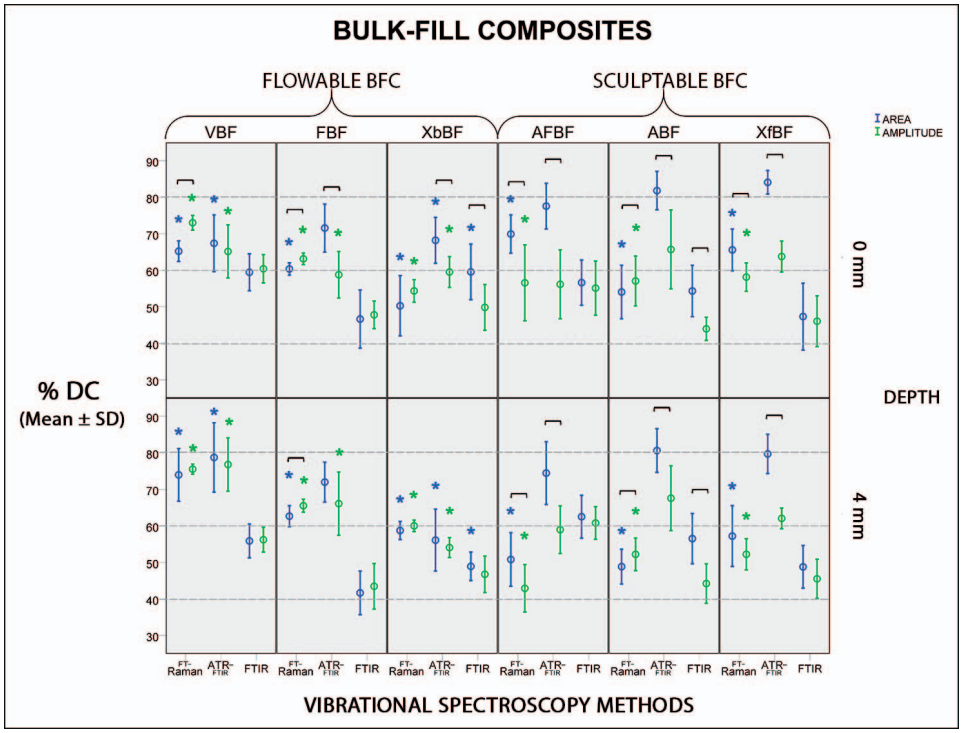

Figure 3. *Degree of conversion values (mean ± SD) obtained for relative area (blue lines and boxes) or amplitude (green lines and boxes) by FT-Raman, attenuated total reflection–Fourier transform infrared, and Fourier transform infrared at the surface (0 mm) and 4-mm depth for six bulk-fill dental composites. Square bracket indicates statistically significant differences between area and amplitude. Asterisk (*) indicates significant differences between 0 and 4 mm for area and amplitude separately, in each technique.*

mm depth, XbBF showed a drop in DC with respect to that obtained at 0-mm depth, in FTIR using areas and amplitudes relations. At 4-mm depth, VBF gave the most consistent results, with DC ranging from 73% to 78% using FT-Raman and ATR-FTIR and more than 55% by FTIR.

Comparison as a Function of Depth

It was not possible to identify a homogenous trend in the function of depth for all BFC, nor for flowable or sculptable composites as a group, for all vibrational spectroscopic methods. When considering both methods of analysis (ratio of areas and ratio of amplitudes; Figure 3), sculptable BFC did not show any differences in DC values between 0- and 4-mm depth by FTIR or ATR-FTIR. However, FT-Raman spectroscopy showed a significant reduction in the DC at 4 mm with respect to the surface. Flowable BFC showed the opposite behavior, with an increase in the DC at 4-mm depth with respect to the surface by FT-Raman spectroscopy, and for VBF, this increase was also detected by ATR-FTIR. XbBF was an exception, showing a drop in DC at 4 mm with respect to 0 mm in ATR-FTIR and FTIR.

DISCUSSION

This study analyzed the influence of spectroscopic technique (FTIR, ATR-FTIR, and FT-Raman) and the method of spectral analysis on the estimation of the DC for six bulk-fill (flowable and sculptable) composites. The results revealed that the BFC had significant differences in their DC values depending on the vibrational spectroscopy technique used and as a function of the method of spectral analysis (ie, the calculation of integrated areas or the amplitude of the reference peaks). Differences in nominal mean DC values were detected for selected materials, calculated at 0- and 4-mm depth with respect to the surface, and in comparisons between materials, depending on the spectroscopic technique and the method of spectral analysis.

Other factors influencing the DC measurements and related to the experimental conditions were controlled in our study, such as the distance of the light guide to the specimen surface,[28,29] which was set at 0 mm, and light curing from the top in contact with a transparent polyester Mylar strip. A plastic film on the top and bottom of the mold of the specimens was used because it minimizes light attenuation[28] and provides a smooth surface for FT-Raman analysis. Although the impact of such attenuation on the DC was not considered in the present study, the real DC values of the BFC would not be lower than those estimated. Temperature during manipulation has been shown to increase the DC of some BFC.[17] In the present study, the manipulation was performed at laboratory temperature (22°C) and specimens were stored at 37°C in

darkness for 24 hours after irradiation.[14,16,30] Although each compound has its own polymerization kinetics, after 24 hours, polymerization should be considered sufficient when using the curing time recommended by the manufacturers.[8,14,16] The thickness of the specimens is also known to impact the DC features of composites.[31] We used 4- to 5-mm-thick specimens because BFC are designed to be suitable for placement in such increments, and we also considered this thickness to be clinically relevant.[14,18,32]

Methodological standardization for spectral analyses is crucial for comparison between quantitative measurements in material characterization. In the present study, we performed detailed spectroscopic analyses of different molecular constituents in several composite resins. We employed a specific procedure during data acquisition and spectral analyses by FTIR, ATR-FTIR, and FT-Raman spectroscopies, using identical spectrometric methodology to avoid possible analytical data deviation due to the analytical procedure. The area and amplitude ratios between the two main reference peaks (C=C absorption bands at 1607 cm^{-1} and 1637 cm^{-1}) were used to determine the DC of dental resins.[24] The only exception was applied to the AFBF composite, an Ormocer-based sculptable composite, which consists of large and precondensed molecules of an inorganic matrix with a high degree of cross-linking.[33] In this case, the peak position at 1588 $\pm$ 4 cm^{-1} was considered as the reference peak, as proposed by several authors using different vibrational spectroscopic analyses.[34] These reference peaks were resolved by a second derivative methodology and fitted to a mixed Gaussian-Lorentzian function for detailed quantitative measurements. This spectrometric method was used by Gauthiers and others[25] on a mix of polymers and demonstrated the best correlation with the molar ratio of monomers in several calibration mixes using FTIR techniques.

In the present study, the use of area or amplitude to calculate DC had a different impact depending on whether FT-Raman or infrared methods (FTIR and ATR-FTIR) were applied. FT-Raman rendered higher mean DC values using the relative amplitude of the peaks rather than the area for flowable BFC; the opposite was found for sculptable BFC. In general, infrared methods produced higher DC values using the area rather than the amplitude relations. Rueggeberg and others[24] concluded that when using an appropriate method of subtraction of the baseline, no significant differences in DC were obtained between the ratio of amplitudes or areas of the peaks. This standard baseline method is well suited for FT-Raman spectroscopy.[25,27,35] Nevertheless, Khalil and others,[26] who also used FT-Raman spectroscopy, obtained higher DC values using the ratio of the areas. The most obvious impact of the spectroscopy technique applied was the finding of different behaviors of the DC at 4 mm with respect to the surface (ie, 0-mm depth) for flowable and sculptable BFC depending on whether the FT-Raman or IR techniques (FTIR and ATR-FTIR) were used. Flowable BFC had significantly higher DC values at 4-mm depth when determined by FT-Raman, and the opposite was found for high-density (sculptable) BFC, that is, a significant drop in their DC at 4 mm with respect to that obtained at 0 mm. The different DC values observed in some other studies based on sample specimen preparation,[32] storage,[18] or spectroscopy method[16] make it difficult to compare their results with the present study.

ATR-FTIR is the method most often used to study the DC of BFC. We observed a significant increase in the DC for VBF and a nonsignificant increase for FBF at depth (4 mm) with respect to the surface (0 mm) when using FT-Raman and ATR-FTIR measurements, while FTIR analyses underestimated DC values at depth. XbBF showed the opposite results with a fall in the DC at 4-mm depth with respect to the surface using ATR-FTIR. Similar results were reported by Zorzin and others,[11] with DC values of 65.24% at the surface and 62.53% at 4 mm. Czasch and Ilie[8] reported a DC of 64.9% at 0.1 mm and a slightly higher DC at 4-mm depth (66.1%) for VBF 5 minutes after curing. Marovic and others[15] attributed the increase in DC at depth with respect to the surface to heat formation due to the exothermic nature of free radical bulk polymerization, which gave rise to an increase in the DC in deeper parts of a bulky specimen, mainly in composites with a lower filler content. This could explain the differences in behavior between VBF with 65 wt% filler (38 vol%) and XbBF with 75 wt% (58 vol%). An increase in filler content reduces the DC.[34] Interestingly, regular composites that presented high light transmission showed no correlation between thickness and DC.[36,37] Among the BFC included in the present study, XfBF is the most studied sculptable BFC. The DC of XfBF varies from 70%[17] to 47.25%[38] at the surface. Using FTIR, Tarle and others[12] observed an increase from 72% at 1-mm depth to 73.9% at 4-mm depth, and these values were higher than the DC values found in the present study.

From a clinical point of view, DC is an important factor to consider when choosing a restorative

material. According to other authors, the DC of the BFC used in the current study is strongly material dependent.*

Focusing on individual materials, it is difficult to determine the possible interactions between composition and method of spectroscopy and analysis in the DC of BFC. There is no consensus on the minimal %DC requirements for the main restorative materials currently in use. However, it has been postulated that a conversion of at least 55% is desirable for occlusal layers.[38,40] In our study, if FTIR was applied as the spectroscopic technique to calculate DC, only three BFC would be clinically acceptable. In this case, it should also be taken into account that the state of aggregation of the BFC (powdered samples) as well as the absorption mode analyses for the FTIR technique may also influence the spectrometric measurements of DC values. Using FT-Raman, the DC of sculptable BFC at 4-mm depth ranged from 48% to 57%, making them unsuitable for clinical application, while the ATR-FTIR peak area ratio results would make them highly recommendable, with DC values ranging from 74% to 80%. It is interesting to note that the differing chemistry of the monomeric resin formulations and filler characteristics (type of volume fraction, density, and particle size and distribution) contribute to significant differences in the DC between restorative composites.[37] The DC of different monomer systems decreases in the following order: triethylene glycol dimethacrylate>urethane dimethacrylate (UDMA)>ethoxylated bisphenol-A-dimethacrylate (Bis-EMA)>bisphenol-A-glycidyldimethacrylate (Bis-GMA).[41] The DC is mainly related to the glass transition temperature (T_g) of the unreacted monomer.[41,42] Nevertheless, differences in T_g between conventional composites and BFC were only small in previous studies.[43,44]

In current BFC technology, two approaches have been adopted to manage high in-depth conversion: the first is by increasing translucency[11] by increasing filler size and matching the refractive index of the filler and resin matrix.[45] The second way is by enhancing curing by adding or combining new photoinitiators, for example, alpha diketone initiators such as camphorquinone, phenylpropanedione, and acylphosphine oxide and germanium-based compounds such as bis-(4-methoxybenzoyl)diethylgermane, which can be irradiated using visible light.[38,46] The light absorption characteristics of the photoinitiators were associated with polymerization and thus could influence the DC and be used as parameters for determining the polymerization efficiency of BFC.[47] The manufacturers of the BFC included in this study did not report the inclusion of such photoinitiators in their brochures or safety data sheets. In the current study, VBF obtained the most consistent results, with DC at 4-mm depth ranging from 73% to 78% using FT-Raman and ATR-FTIR analyses and from 55.87% to 56.21% by FTIR for the area and amplitude ratios, respectively. This high DC is consistent with previous reports[13,15,28] and could be explained by the relatively low filler load (38 vol%), very high translucency,[16] and the absence of Bis-GMA in VBF. Experimental composites containing UDMA had, on average, 1.2 and 1.3 times higher conversion at 1- and 4-mm depth, respectively, than those containing Bis-GMA, as well as improved handling properties.[48] FBF has a more complex monomer composition (see Table 1 for resin matrix composition). The DC of FBF was found to significantly decrease at 4-mm depth, which was attributed to the Zr present in the filler, as this has been shown to decrease translucency due to resin/filler refractive index mismatch.[16,28] The content of 50-60 wt% of silane-treated ceramic filler could also account for the lower DC.[34] In the opinion of Alshali and others,[13] the low DC found 24 hours after curing for XbBF (62%) and FBF (50%) was related to the presence of Bis-EMA in their monomer composition. The stiff central phenyl ring core of this monomer can significantly restrict the mobility of UDMA monomers and decrease their reactivity and ultimate conversion value. In our opinion, the lower irradiance of the specimens (600 mW/cm^2) in the study of Alshali and others[13] could also partially explain the lower values found in their study with respect to the present study. The sculptable BFC included in our study are composed of 84 wt% (AFBF) and 85 wt% (XfBF) filler. XfBF demonstrated excellent light transmission through 6-mm specimens, which can be attributed to the increased filler size and potentially improved matching between the refractive indices of filler particles and the resin matrix.[12] A DC of 73.9% was reported by Tauböck and others[17] for XfBF at 4-mm depth using slight experimental modifications and FTIR analyses. In the present study, the DC values of XfBF at 4-mm depth ranged from 79.70% using ATR-FTIR and the area ratios to 45.50% using the FTIR intensities ratio.

In summary, the results of this study reveal the impact of the spectroscopic technique and spectral methodology for calculation of the DC values of BFC materials. Based on our findings, the hypothesis of

* *References 8, 12, 14, 15, 17, 28, 29, 36, 39.*

equality between FT-Raman and IR spectroscopic techniques and spectrometric analysis to calculate the DC must be rejected. The use of a standard methodology of analysis revealed relevant differences between the DC values obtained for the current BFC. Overall, this study provides a complete analytical data set based on different spectroscopic methods to evaluate the features of DC materials at depth for flowable and sculptable composites. The differences obtained indicate that the information on DC should be considered with caution when selecting BFC restorative materials in the clinic.

CONCLUSIONS

The spectroscopic technique (FT-Raman, ATR-FTIR, and FTIR) influences DC measurements of BFC materials, with higher DC values in most cases for ATR-FTIR calculations. The spectrometric methodology (ie, using peak area or amplitude estimation) causes significant variations in DC values depending on the vibrational technique used. The DC also differed significantly among flowable and sculptable composites. Furthermore, the DC showed different behaviors according to depth, with higher values for flowable composites at 4 mm and for sculptable composites at the surface.

The observed differences mean that information on DC should be considered with caution when selecting BFC restorative materials. This information should also be considered when evaluating the DC features associated with different dental resin composites in the clinic (ie, composition, mechanical, biocompatibility properties).

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Kwon Y, Ferracane J, & Lee I-B (2012) Effect of layering methods, composite type, and flowable liner on the polymerization shrinkage stress of light cured composites *Dental Materials* **28(7)** 801-809, https://doi.org/10.1016/j.dental.2012.04.028.
2. Kleverlaan CJ & Feilzer AJ (2005) Polymerization shrinkage and contraction stress of dental resin composites *Dental Materials* **21(12)** 1150-1157, https://doi.org/10.1016/j.dental.2005.02.004.
3. Ferracane JL (2005) Developing a more complete understanding of stresses produced in dental composites during polymerization *Dental Materials* **21(1)** 36-42, https://doi.org/10.1016/j.dental.2004.10.004.
4. Ferracane JL & Hilton TJ (2016) Polymerization stress—Is it clinically meaningful? *Dental Materials* **32(1)** 1-10, https://doi.org/10.1016/j.dental.2015.06.020.
5. Kim RJ-Y, Kim Y-J, Choi N-S, & Lee I-B (2015) Polymerization shrinkage, modulus, and shrinkage stress related to tooth-restoration interfacial debonding in bulk-fill composites *Journal of Dentistry* **43(4)** 430-439, https://doi.org/10.1016/j.jdent.2015.02.002.
6. Garcia D, Yaman P, Dennison J, & Neiva GF (2015) Polymerization shrinkage and depth of cure of bulk fill flowable composite resins: Commentary *Journal of Esthetic and Restorative Dentistry* **27(4)** 232-233, https://doi.org/10.1111/jerd.12178.
7. Benetti A, Havndrup-Pedersen C, Honoré D, Pedersen M, & Pallesen U (2015) Bulk-fill resin composites: polymerization contraction, depth of cure, and gap formation *Operative Dentistry* **40(2)** 190-200, https://doi.org/10.2341/13-324-L.
8. Czasch P & Ilie N (2013) *In vitro* comparison of mechanical properties and degree of cure of bulk fill composites *Clinical Oral Investigations* **17(1)** 227-235, https://doi.org/10.1007/s00784-012-0702-8.
9. Alrahlah A, Silikas N, & Watts DC (2014) Post-cure depth of cure of bulk fill dental resin-composites *Dental Materials* **30(2)** 149-154, https://doi.org/10.1016/j.dental.2013.10.011.
10. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, & Leloup G (2014) Physico-mechanical characteristics of commercially available bulk-fill composites *Journal of Dentistry* **42(8)** 993-1000, https://doi.org/10.1016/j.jdent.2014.05.009.
11. Zorzin J, Maier E, Harre S, Fey T, Belli R, Lohbauer U, Petschelt A, & Taschner M (2015) Bulk-fill resin composites: Polymerization properties and extended light curing *Dental Materials* **31(3)** 293-301, https://doi.org/10.1016/j.dental.2014.12.010.
12. Tarle Z, Attin T, Marovic D, Andermatt L, Ristic M, & Tauböck TT (2015) Influence of irradiation time on subsurface degree of conversion and microhardness of high-viscosity bulk-fill resin composites *Clinical Oral Investigations* **19(4)** 831-840, https://doi.org/10.1007/s00784-014-1302-6.
13. Alshali RZ, Silikas N, & Satterthwaite JD (2013) Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals *Dental Materials* **29(9)** e213-e217, https://doi.org/10.1016/j.dental.2013.05.011.
14. Al-Ahdal K, Ilie N, Silikas N, & Watts DC (2015) Polymerization kinetics and impact of post polymerization on the degree of conversion of bulk-fill resin-composite at clinically relevant depth *Dental Materials* **31(10)** 1207-1213, https://doi.org/10.1016/j.dental.2015.07.004.
15. Marovic D, Tauböck TT, Attin T, Panduric V, & Tarle Z (2015) Monomer conversion and shrinkage force kinetics of low-viscosity bulk-fill resin composites *Acta Odontolgica Scandinavica* **73(6)** 474-480, https://doi.org/10.3109/00016357.2014.992810.
16. Par M, Gamulin O, Marovic D, Klaric E, & Tarle Z (2015) Raman spectroscopic assessment of degree of conversion

of bulk-fill resin composites—Changes at 24 hours post cure *Operative Dentistry* **40(3)** E92-E101, https://doi.org/10.2341/14-091-L.

17. Tauböck TT, Tarle Z, Marovic D, & Attin T (2015) Preheating of high-viscosity bulk-fill resin composites: Effects on shrinkage force and monomer conversion *Journal of Dentistry* **43(11)** 1358-1364, https://doi.org/10.1016/j.jdent.2015.07.014.
18. Fronza BM, Rueggeberg FA, Braga RR, Mogilevych B, Soares LES, Martin AA, Ambrosano G, & Giannini M (2015) Monomer conversion, microhardness, internal marginal adaptation, and shrinkage stress of bulk-fill resin composites *Dental Materials* **31(12)** 1542-1551, https://doi.org/10.1016/j.dental.2015.10.001.
19. Shin WS, Li XF, Schwartz B, Wunder SL, & Baran GR (1993) Determination of the degree of cure of dental resins using Raman and FT-Raman spectroscopy *Dental Materials* **9(5)** 317-324, https://doi.org/10.1016/0109-5641(93)90050-Z.
20. Ferracane JL & Greener EH (1984) Fourier transform infrared analysis of degree of polymerization in unfilled resins—Methods comparison *Journal of Dental Research* **63(8)** 1093-1095, https://doi.org/10.1177/00220345840630081901.
21. Tarumi H, Imazato S, Ehara A, Kato S, Ebi N, & Ebisu S (1999) Post-irradiation polymerization of composites containing bis-GMA and TEGDMA *Dental Materials* **15(4)** 238-242, https://doi.org/10.1016/S0109-5641(99)00040-8.
22. Park S-H (1996) Comparison of degree of conversion for light-cured and additionally heat-cured composites *Journal of Prosthetic Dentistry* **76(6)** 613-618, https://doi.org/10.1016/S0022-3913(96)90439-4.
23. Silikas N, Eliades G, & Watts D. (2000) Light intensity effects on resin-composite degree of conversion and shrinkage strain *Dental Materials* **16(4)** 292-296, https://doi.org/10.1016/S0109-5641(00)00020-8.
24. Rueggeberg FA, Hashinger DT, & Fairhurst CW (1990) Calibration of FTIR conversion analysis of contemporary dental resin composites *Dental Materials* **6(4)** 241-249, https://doi.org/10.1016/S0109-5641(05)80005-3.
25. Gauthier MA, Stangel I, Ellis TH, & Zhu XX (2005) A new method for quantifying the intensity of the C=C band of dimethacrylate dental monomers in their FTIR and Raman spectra *Biomaterials* **26(33)** 6440-6448, https://doi.org/10.1016/j.biomaterials.2005.04.039.
26. Khalil SKH, Allam MA, & Tawfik WA (2007) Use of FT-Raman spectroscopy to determine the degree of polymerization of dental composite resin cured with a new light source *European Journal of Dentistry* **1(2)** 72-79.
27. Pianelli C, Devaux J, Bebelman S, & Leloup G (1999) The micro-Raman spectroscopy, a useful tool to determine the degree of conversion of light-activated composite resins *Journal of Biomedical Materials Research* **48(5)** 675-681, https://doi.org/10.1002/(SICI)1097-4636(1999)48:5<675::AID-JBM11>3.0.CO;2-P.
28. Bucuta S & Ilie N (2014) Light transmittance and micromechanical properties of bulk fill vs. conventional resin based composites *Clinical Oral Investigations* **18(8)** 1991-2000, https://doi.org/10.1007/s00784-013-1177-y.
29. Li X, Pongprueksa P, Van Meerbeek B, & De Munck J (2015) Curing profile of bulk-fill resin-based composites *Journal of Dentistry* **43(6)** 664-672, https://doi.org/10.1016/j.jdent.2015.01.002.
30. Truffier-Boutry D, Demoustier-Champagne S, Devaux J, Biebuyck J-J, Mestdagh M, Larbanois P, & Leloup G (2006) A physico-chemical explanation of the post-polymerization shrinkage in dental resins *Dental Materials* **22(5)** 405-412, https://doi.org/10.1016/j.dental.2005.04.030.
31. Musanje L & Darvell BW (2006) Curing-light attenuation in filled-resin restorative materials *Dental Materials* **22(9)** 804-817, https://doi.org/10.1016/j.dental.2005.11.009.
32. Pongprueksa P, De Munck J, Duca RC, Poels K, Covaci A, Hoet P, Godderis L, Van Meerbeek B, & Van Landuyt KL (2015) Monomer elution in relation to degree of conversion for different types of composite *Journal of Dentistry* **43(12)** 1448-1455, https://doi.org/10.1016/j.jdent.2015.10.013.
33. VOCO GmbH Admira Fusion. Scientific compendium; Retrieved online August 4, 2017 from: http://www.voco.com/en/product/Admira-Fusion/SC_EN_Admira-Fusion.pdf.
34. Halvorson RH, Erickson RL, & Davidson CL (2003) The effect of filler and silane content on conversion of resin-based composite *Dental Materials* **19(4)** 327-333, https://doi.org/10.1016/S0109-5641(02)00062-3.
35. Mezzomo Collares F, Freitas Portella F, Castelo Branco Leitune V, & Werner Samuel SM (2013) Discrepancies in degree of conversion measurements by FTIR *Brazilian Oral Research* **27(6)** 453-454, https://doi.org/10.1590/S1806-83242013000600002.
36. Garoushi S, Vallittu P, Shinya A, & Lassila L (2016) Influence of increment thickness on light transmission, degree of conversion and micro hardness of bulk fill composites *Odontology* **104(3)** 291-297, https://doi.org/10.1007/s10266-015-0227-0.
37. Finan L, Palin WM, Moskwa N, McGinley EL, & Fleming GJP (2013) The influence of irradiation potential on the degree of conversion and mechanical properties of two bulk-fill flowable RBC base materials *Dent Mater* **29(8)** 906-912, https://doi.org/10.1016/j.dental.2013.05.008.
38. Papadogiannis D, Tolidis K, Gerasimou P, Lakes R, & Papadogiannis Y (2015) Viscoelastic properties, creep behavior and degree of conversion of bulk fill composite resins *Dental Materials* **31(12)** 1533-1541, https://doi.org/10.1016/j.dental.2015.09.022.
39. Ilie N, Keßler A, & Durner J (2013) Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites *Journal of Dentistry* **41(8)** 695-702, https://doi.org/10.1016/j.jdent.2013.05.008.
40. Ferracane JL, Mitchem JC, Condon JR, & Todd R (1997) Wear and marginal breakdown of composites with various degrees of cure *Journal of Dental Research*

76(8) 1508-1516, https://doi.org/10.1177/00220345970760081401.

41. Sideridou I, Tserki V, & Papanastasiou G (2002) Effect of chemical structure on degree of conversion in light-cured dimethacrylate-based dental resins *Biomaterials* **23(8)** 1819-1829, https://doi.org/10.1016/S0142-9612(01)00308-8.
42. Morgan DR, Kalachandra S, Shobha HK, Gunduz N, & Stejskal EO (2000) Analysis of a dimethacrylate copolymer (Bis-GMA and TEGDMA) network by DSC and 13C solution and solid-state NMR spectroscopy *Biomaterials* **21(18)** 1897-1903, https://doi.org/10.1016/S0142-9612(00)00067-3.
43. Engelhardt F, Hahnel S, Preis V, & Rosentritt M (2016) Comparison of flowable bulk-fill and flowable resin-based composites: An *in vitro* analysis *Clinical Oral Investigations* **20(8)** 2123-2130, https://doi.org/10.1007/s00784-015-1700-4.
44. Ilie N, Bucuta S, & Draenert M (2013) Bulk-fill resin-based composites: An *in vitro* assessment of their mechanical performance *Operative Dentistry* **38(6)** 618-625, https://doi.org/10.2341/12-395-L.
45. Ilie N & Stark K (2014) Curing behaviour of high-viscosity bulk-fill composites *Journal of Dentistry* **42(8)** 977-985, https://doi.org/10.1016/j.jdent.2014.05.012.
46. Arikawa H, Takahashi H, Kanie T, & Ban S (2009) Effect of various visible light photoinitiators on the polymerization and color of light-activated resins. *Dental Materials Journal* **28(4)** 454-460, https://doi.org/10.4012/dmj.28.454.
47. Ogunyinka A, Palin WM, Shortall AC, & Marquis PM (2007) Photoinitiation chemistry affects light transmission and degree of conversion of curing experimental dental resin composites *Dental Materials* **23(7)** 807-813, https://doi.org/10.1016/j.dental.2006.06.016.
48. Walters NJ, Xia W, Salih V, Ashley PF, & Young AM (2016) Poly(propylene glycol) and urethane dimethacrylates improve conversion of dental composites and reveal complexity of cytocompatibility testing *Dental Materials* **32(2)** 264-277, https://doi.org/10.1016/j.dental.2015.11.017.

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Properties of New Glass-Ionomer Restorative Systems Marketed for Stress-Bearing Areas

D Fuhrmann • D Murchison • S Whipple • K Vandewalle

Clinical Relevance

The newer glass-ionomer restorative materials marketed for posterior stress-bearing areas may not provide any significant advantage in mechanical properties over other conventional glass-ionomer materials.

SUMMARY

Objectives: The purpose of this study was to evaluate the properties (fracture toughness, surface hardness) of newer conventional glass-ionomer restorative materials that are marketed for posterior stress-bearing areas compared with more traditional glass-ionomer restorative materials marketed for non–load-bearing areas and composite-resin restorative materials.

Methods and Materials: Notched-beam fracture toughness specimens were created in a mold with each tested material (Equia Forte, GC America, with and without a surface coating of Equia Forte Coat; Ketac Universal, 3M/ESPE; ChemFil Rock, Dentsply; Fuji IX GP Extra, GC; Ionostar Molar, VOCO; Filtek Z250, 3M/ESPE; Filtek Supreme Ultra, 3M/ESPE) and fractured using a universal testing machine after 24 hours of storage. Hardness values were determined on the surface of the fracture toughness specimens using a hardness tester. Data were analyzed with a one-way ANOVA and Tukey's post hoc test per property (alpha=0.05).

Results: The composite-resin restorative materials had significantly greater fracture toughness than the glass-ionomer materials. There was no significant difference in fracture toughness between the glass-ionomer materials. The use of a resin coating significantly increased the surface hardness of the newer glass ionomer marketed for stress-bearing areas.

Conclusions: Fracture toughness was not improved with the newer glass-ionomer restorative materials marketed for stress-bearing areas compared to the conventional glass-ionomer materials, however a resin coating provided greater surface hardness.

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INTRODUCTION

For years, clinicians and researchers have sought to find a material to replace missing or weakened tooth structure that best mimics the characteristics of enamel, the hardest and most mineralized substance in the human body.[1] Teeth are subjected to many dynamic conditions ranging from different working movements, thermal insults and challenges, oscillations of pH levels, and parafunctional behaviors. Restorative materials differ in mechanical properties that allow them to withstand forces placed on them during function. Technique sensitivity and operator skill in placement affect long-term clinical success for materials. With these factors in mind, there is not currently one ideal restorative material.[2]

Amalgam is still considered an excellent choice for posterior restorations. Its use has declined for several reasons to include perceived adverse effects of mercury and esthetic factors. In 2013, a global agreement was signed at the Minamata Convention that dictated the phase down in the use of amalgam.[3,4] Composites are clearly more esthetic in appearance than amalgam. However, they wear faster than tooth structure and are subject to possible postoperative sensitivity and microleakage.[2] Compared with use of amalgam, placing composites using a 2-mm incremental technique is more time consuming.[5] Newer low-viscosity flowable bulk-fill composites and higher viscosity conventional bulk-fill composites have been marketed for 4 mm or greater depth of cure. These bulk-fill composite resins tend to be more translucent than traditional composites to allow for greater depth of cure. Bulk placement is more time efficient; however, further clinical research is still necessary to compare the advantages and disadvantages with the gold standard approach of incremental layering.[6]

The use of conventional glass-ionomer cements (GICs) has long been advocated for the primary dentition, for noncarious cervical lesions, and for use in atraumatic restorative treatment approaches. The use of GICs was popularized in the 1970s; however, their use was limited due to poor abrasion resistance, low tensile strength, poor esthetics, and low final hardness.[7,8] GICs exhibited a wear rate five times higher than amalgam and three times higher than composite-resin materials.[9] GIC has many highly advantageous properties such as a similar coefficient of thermal expansion to enamel and dentin, formation of direct chemical adhesion to tooth structure, biocompatibility, bulk placement, and fluoride uptake and release.[7,10] Even with these attributes, the properties of GIC may not be sufficient to overcome the limitations in areas of heavier occlusion.[11] Manhart and others reported that fractures in conventional GIC restorations caused an annual failure rate of 7.2%, which was higher that amalgam (3.0%) or composite restorations (2.2%) in posterior areas.[12]

Newer formulations of glass ionomers have been developed over the last decade with the purpose of mimicking the wear, strength, polishability, and esthetics of composite resins. Equia Fil (GC America, Alsip, IL, USA) is a high-viscosity conventional GIC. The company optimized the polyacid and particle size distribution, which reportedly created a higher cross-linkage of the GIC matrix. Combined with a nanofilled coating (Equia Coat, GC America), it was marketed to yield a restorative material indicated for posterior stress-bearing restorations.[13] Equia Fil was formerly known as Fuji IX GP Extra and Equia Coat was formerly known as G-Coat Plus. The Equia Fil formulation showed significant improvements in fracture strength once coated with Equia Coat.[14] Resin coatings have been implemented to seal surface defects and potentially limit abrasive wear and early material fracture while the GIC matures and reaches peak strength.[10] The fluoride uptake/release potential may be substantially reduced with the application of a surface coating.[15] However, it has been suggested that the fluoride release may be effective in reducing secondary caries in the marginal gap of the tooth–restoration interface and not in the exposed surface of the GIC intended to be protected by the resin coating.[14,16]

The latest generation of conventional GICs marketed for load-bearing restorations consists of products like Equia Forte (GC America) and Ketac Universal Aplicap (3M/ESPE, St Paul, MN, USA). Equia Forte contains a new higher-molecular-weight polyacrylic acid to reportedly create an even higher strength restorative material compared with Equia Fil. The Equia Forte Coat application incorporates a newer multifunctional monomer, which reportedly produces a tougher resin matrix to extend the indications to include stress-bearing class 2 restorations.[17] 3M ESPE recently introduced Ketac Universal Aplicap, which they claim may be used in class 1 and class 2 stress-bearing restorations as long as there is at least one additional support outside the restoration area. For class 2 restorations, they also recommend that the isthmus must be less than half the intercuspal distance. Ketac Universal contains a copolymer of acrylic and maleic acids.[18]

In the present study, these new GIC materials marketed for load-bearing restorations were compared with other GICs marketed for non–load-bearing class 1 and class 2 restorations (Fuji IX GP Extra), non–occlusion-bearing class 1 restorations (IonoStar Molar, VOCO America, Indian Land, SC, USA), semipermanent restoration of class 1 and 2 preparations in posterior teeth (ChemFil Rock, Dentsply, York, PA, USA), and two composite-resin restorative materials, Filtek Z250 and Filtek Supreme Ultra (3M/ESPE). Fuji IX GP Extra is a high-viscosity conventional glass ionomer that was later remarketed as Equia Fil.[19] Ionostar Molar is a new glass-ionomer restorative with improved characteristics that reportedly provides immediate packability.[20] ChemFil Rock is a zinc-reinforced glass-ionomer restorative material. The manufacturer claims that the inclusion of zinc-oxide enhances the setting reaction and increases strength and toughness.[21] The hybrid composite, Filtek Z250 (3M ESPE), and nanocomposite, Filtek Supreme (3M ESPE), were selected based on their different size and distribution of their filler particles.[22]

There is a lack of research to substantiate the marketing claims for the newest generation GICs in load-bearing areas. The purpose of this study was to test various mechanical properties (fracture toughness and surface hardness) of newer GICs marketed for stress-bearing areas compared with more conventional GICs marketed for non–load-bearing areas and composite-resin restorative materials. The null hypotheses tested was that there would be no differences in (1) fracture toughness or (2) surface hardness based on type of restorative material.

METHODS AND MATERIALS

Fracture toughness was determined using a single-edge notched-beam method. To prepare each specimen, a knife-edged split (2.5 × 5.0 × 25.0 mm) stainless-steel mold (Sabri, Downers Grove, IL, USA) was placed on a plastic strip–covered glass slide. Ten specimens of each restorative material were made by inserting the restorative material into the mold until completely filled. Then, the top surface of the mold was covered with a second plastic strip and glass slide to ensure that the end of the specimen was flat and parallel to the opposite surface of the mold. For the composite materials, one side of the specimen was exposed to a light polymerization unit (Bluephase 20i, Ivoclar Vivadent, Amherst, NY, USA) in three separate overlapping increments for 20 seconds each. The adequacy of the light unit's intensity was assessed prior to specimen preparation using a radiometer (Bluephase Meter, Ivoclar Vivadent). Next, the mold was turned over, and the opposite side of the specimen was exposed to the light in a similar manner as described before. The glass-ionomer specimens were allowed to chemically cure for 10 minutes before removal from the mold. One group of Equia Forte specimens were covered with Equia Forte Coat and light cured per the manufacturer's instructions. A resin coat was only applied to the Equia Forte specimens because no other manufacturer of the GIC materials tested in this study recommended the use of a surface coat.

Then, the specimens were stored for 24 hours in humidified air at 37°C in a laboratory oven prior to testing. The height, h, width, w, and the notch depth, a, of the specimens were measured with an electronic digital caliper. The specimens were then fractured using a universal testing machine (Model 5543, Instron, Canton, MA, USA) at a crosshead speed of 1.0 mm/min, with the notch on the tensile side and the loading pin aligned with the notch. Fracture toughness (K_{IC}) was calculated from measurements using the equation:

$$K_{IC} = \frac{3(a/w)^{1/2}[1.99 - a/w(1 - a/w)(2.15 - 3.93a/w + 2.7(a/w)^2]FS}{2(1 + 2a/w)(1 - a/w)^{3/2}hw^{3/2}}$$

where S is the span distance (20 mm) between supports and F is the maximum force at fracture.

The fractured specimens from the fracture toughness test were used as the specimens for the surface hardness test. Surface hardness was determined using a Knoop Hardness tester (Leco, LM300AT, St Joseph, MI, USA) under a load of 200 g for 10 seconds. A mean of three measurements was taken from each of the specimens per group.

Statistical Analysis

The mean and standard deviation was calculated for each of the restorative materials for each property. Data were analyzed with a one-way ANOVA and Tukey's post hoc test per property (α=0.05) using statistical software (IBM SPSS, version 24, Chicago, IL, USA).

RESULTS

A significant difference was found between the materials based on fracture toughness ($p<0.001$) or surface hardness ($p<0.001$; Table 1). Filtek Z250 had significantly greater fracture toughness

Table 1: *Fracture Toughness and Surface Hardness of the Tested Materials*[a]

Material	Property [Mean, SD)]	
	Fracture Toughness, MPa·m$^{1/2}$	Knoop Hardness, kg/mm^2
Filtek Z250 (3M/ESPE)	1.21 (0.12) A	81.3 (1.9) A
Filtek Supreme Ultra (3M/ESPE)	0.82 (0.20) B	72.8 (1.9) B
Equia Forte and Coat (GC)	0.40 (0.04) C	83.5 (4.5) A
ChemFil Rock (Dentsply)	0.39 (0.03) C	59.4 (0.8) E
Fuji IX GP Extra (GC)	0.38 (0.03) C	70.2 (1.0) BC
Ketac Universal (3M/ESPE)	0.36 (0.04) C	69.9 (2.6) BC
IonoStar Molar (VOCO)	0.35 (0.04) C	62.7 (1.7) D
Equia Forte (GC)	0.33 (0.04) C	69.1 (1.2) BC

[a] *Groups with the same letter per column are not significantly different (p>0.05).*

(1.21±0.12 MPa$*$m$^{1/2}$) than Filtek Supreme Ultra (0.82±0.20 MPa*m$^{1/2}$), which was significantly greater than glass-ionomer materials. There was no significant difference in fracture toughness between the glass-ionomer materials (range, 0.40-0.33 MPa$*$m$^{1/2}$). Equia Forte with Equia Forte Coat (83.5±4.5 kg/mm^2) and Filtek Z250 (81.3±1.9 kg/mm^2) had the greatest surface hardness and were not significantly different from each other. Values for Filtek Supreme Ultra (72.8±1.9 kg/mm^2), Fuji IX GP Extra (70.2±1.0 kg/mm^2), Ketac Universal (69.9±2.6 kg/mm^2), and Equia Forte (uncoated) (69.1±1.2 kg/mm^2) were not significantly different from each other. ChemFil Rock (59.4±0.8 kg/mm^2) had the lowest surface hardness, which was significantly lower than IonoStar Molar (62.7±1.7 kg/mm^2).

DISCUSSION

With the public desire for esthetic dentistry, it is clear the future will continue to evolve with novel materials being marketed, including newer glass ionomers, all of which would be more esthetic than amalgam. The current study compared fracture toughness and surface hardness of various glass ionomers with two widely used composites.

Mechanical properties, like fracture toughness, contribute to the performance of restorative materials. In the current study, significant differences were found between the glass ionomers and the composites. According to Ferracane, there may be some threshold for mechanical properties below which failure would be more likely, limiting the use of certain materials, such as glass ionomers, in stress-bearing areas. Fracture of the restorative material can be a primary reason for failure. The fracture toughness test relates to chipping and bulk fracture and may be the most critical laboratory factor in the estimation of resistance to intraoral fracture.[23] This current study found that all the tested glass ionomers had relatively low fracture toughness, including the new GIC materials marketed for stress-bearing areas. Hardness has a limited correlation to wear.[23] In a study by Faria and others, an inverse relationship between surface hardness and wear resistance was observed. In other words, a higher surface hardness may correlate with less wear.[24]

The first null hypothesis was rejected. This current study found a significant difference in fracture toughness between the two different composite resins and the five different GICs tested, with Filtek Z250 significantly more resistant to fracture than Filtek Supreme Ultra, both of which were significantly stronger than the GICs. The new glass-ionomer restorative materials marketed for stress-bearing areas, Equia Forte, with or without a resin coating, and Ketac Universal, displayed no significant difference in fracture toughness compared with the other GICs. The incorporation of the new multifunctional monomer in Equia Forte did not produce a significantly tougher matrix, at least based on the results of this study. This study found no statistically significant increase in fracture toughness with or without a resin coating of the Equia Forte. In the literature, however, the effect of resin coating on fracture toughness is somewhat equivocal, with one laboratory study showing no benefit[25] and others showing a significant increase.[14,26] In this current study, the application of the resin coating slightly increased the surface dimensions (width, height and notch length) compared with the other noncoated specimens. However, the increase was less than 100 μm in any dimension and the effect on fracture toughness values would be offset in the formula calculation. The results of this current study compare favorably with results of a laboratory study by Ilie and others and highlight the

difference between highly cross-linked restorative resin composites and cement matrices.[27] The fracture toughness of over 69 restorative materials in ten material categories were evaluated. The lowest fracture toughness was found with the GIC materials, followed by microfilled composite resins, resin-modified GICs and flowable compomers, which were not significantly different from each other. The ormocers, packable, and microhybrid composite resins performed statistically similar, reaching the highest fracture toughness.[27]

Differences were found in surface hardness based on restorative material. Therefore, the second null hypothesis was also rejected. More variability was observed between the GICs when tested for surface hardness. Application of the nanofilled resin coat (Equia Forte Coat) to the Equia Forte specimen resulted in a surface hardness comparable to that of the microhybrid composite resin Filtek Z250 and harder than Filtek Supreme Ultra and all of the other GICs. No difference in hardness was observed between Fuji IX GP Extra, Ketac Universal, and Equia Forte (without the resin coat). The incorporation of the new multifunctional monomer in Equia Forte had a relatively minimal effect on surface hardness, at least based on the results of this current study. The results of this study differ somewhat from a previous laboratory study by Al-Angari and others, comparing various GICs.[28] In that study, Fuji IX GP Extra had significantly greater surface microhardness than Equia Fil with a resin surface coating. In this current study, however, the surface hardness of Equia Forte with a resin coat was significantly greater than Fuji IX GP Extra, as expected. Previous laboratory studies have shown an increase of surface hardness after 6 months with resin coated GICs compared to GICs without application of a resin coating.[29] In a clinical study by Turkun and Kanik, the resin coating protected the margins of Equia Fil and created a regular and glossy surface; however, the coatings were worn away in nearly all of the restorations after six months.[9] Similar to this current study, the study by Al-Angari and others found that ChemFil Rock had the lowest surface hardness value.[28] The zinc reinforcement of ChemFil Rock did not provide any apparent increase in fracture toughness or surface hardness in this current study. ChemFil Rock and IonoStar Molar exhibited relatively low surface hardness and they are only marketed for non–load-bearing occlusal restorations in posterior teeth.

Although no clinical studies are available evaluating Equia Forte, a recent six-year clinical study by Turkun and Kanik found greater color match, marginal adaptation, anatomic form, and retention rates with Equia Fil, marketed for stress-bearing areas, compared with another conventional glass-ionomer restorative material, marketed for non–stress-bearing areas (Riva SC, SDI, Baywater, Australia).[9] However, in a recent study by Klinke and others, the number of unsatisfactory Equia Fil restorations according to the FDI World Dental Federation criteria was determined to be relatively high in two-surface class 2 restorations and even higher in three-surface class 2 restorations, with observable chipping and fractures likely related to lower strength properties.[30] In a four-year study by Gurgan and others, failure rates for a composite resin (Gradia Direct, GC America) in class 1 and 2 restorations were 0% compared with 7.7% for Equia Fil.[13] Basso and others found greater chipping and failures in the marginal proximal crest of wider restorations in their four-year clinical study of Equia Fil.[7] Conclusions from a retrospective clinical study suggested that Equia Fil should be limited to class I and smaller class II restorations if used in load-bearing restorations.[8] Radiographically, progressive loss of GIC material (Fuji IX GP) in proximal areas just below the contacts in class 2 restorations was commonly observed in a six-year retrospective study.[31]

GICs have been shown to be effective for the treatment of class V lesions, for use in primary teeth, and for use in atraumatic restorative techniques. They have been considered for use in non–stress-bearing locations and for a nonpermanent restoration in stress-bearing areas. Recently, GICs with newer formulations, such as Equia Forte and Ketac Universal, have been marketed for stress-bearing areas. This study observed lower fracture toughness and generally less hardness of the GICs compared with the composites tested. Application of an unfilled resin coating may improve the surface hardness to allow for the GIC to mature. Further developments are needed in the formulation of GIC restorative materials to predictably extend their indications in larger stress-bearing areas in posterior teeth. Clinical studies need to be conducted to determine the effectiveness of both formulation changes, as well as resin surface coating for newer GICs as definitive restorative materials.

CONCLUSIONS

The newer GIC restorative materials marketed for posterior stress-bearing areas did not demonstrate any advantage in fracture toughness over other

conventional GIC materials. However, the use of a resin coating significantly increased the surface hardness of the newer GIC, Equia Forte, to be similar to microhybrid composite-resin materials tested.

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The views expressed in this article are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, Uniformed Services, University of the Health Sciences, or the United States government.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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References

1. Galo R, Contente M, Galafassi D, & Borsatto M (2015) Hardness and modulus of elasticity of primary and permanent teeth after wear against different dental materials *European Journal of Dentistry* **9(4)** 587-593.
2. Abesi F, Safarcherati H, Sadati J, & Kheirollahi H (2011) In vitro wear of Ionofil Molar AC quick glass-ionomer cement *Indian Journal of Dental Research* **22(5)** 731-737.
3. Kielbassa A, Glockner G, Wolgin M, & Glockner K (2016) Systematic review on highly viscous glass-ionomer cement/resin coating restorations (Part I): do they merge Minamata Convention and minimum intervention dentistry? *Quintessence International* **47(10)** 813-823.
4. Kielbassa A, Glockner G, Wolgin M, & Glockner K (2017) Systematic review on highly viscous glass-ionomer cement/resin coating restorations (part II): do they merge Minamata Convention and minimum intervention dentistry? *Quintessence International* **48(1)** 9-18.
5. Olegario I, Hesse D, Bönecker M, Imparato J, Braga M, Mendes F, & Raggio D (2016) Effectiveness of conventional treatment using bulk-fill composite resin versus atraumatic restorative treatments in primary and permanent dentition: a pragmatic randomized clinical trial *BMC Oral Health* **17(1)** 34-41.
6. Edrees N, Amer H, Abdelaziz K, & Alajam W (2017) Benefits and drawbacks of bulk-fill dental composites: a systematic review *European Journal of Pharmaceutical and Medical Research* **4(10)** 124-137.
7. Basso M, Brambilla E, Goñe Benites M, Giovannardi M, & Ionescu A (2015) Glass ionomer cement for permanent dental restorations: a 48-months, multi-centre, prospective clinical trial *Stomatology Education Journal* **2(1)** 25-35.
8. Friedl K, Hiller KA, & Friedl K (2011) Clinical performance of a new glass ionomer-based restoration system: a retrospective cohort study *Dental Materials* **27(10)** 1031-1037.
9. Turkun L & Kanik O (2016) A prospective six-year clinical study evaluating reinforced glass ionomer cements with resin coating on posterior teeth: quo vadis? *Operative Dentistry* **41(6)** 587-598.
10. Klinke T, Daboul A, Turek A, Frankenberger R, Hickel R, & Biffar R (2016) Clinical performance during 48 months of two current glass ionomer restorative systems with coatings: a randomized clinical trial in the field *Trials* **17(1)** 239-252.
11. Zoergiebel J & Ilie N (2013) Evaluation of a conventional glass ionomer cement with new zinc formulation: effect of coating, aging and storage agents *Clinical Oral Investigations* **17(2)** 619-626.
12. Manhart J, Chen H, Hamm G, & Hickel R (2004) Buonocore Memorial Lecture. Review of the clinical survival of direct and indirect restorations in posterior teeth of the permanent dentition *Operative Dentistry* **29(5)** 481-508.
13. Gurgan S, Kutuk Z, Ergin E, Oztas S, & Cakir F (2015) Four-year randomized clinical trial to evaluate the clinical performance of a glass ionomer restorative system *Operative Dentistry* **40(2)** 134-143.
14. Lohbauer U, Krämer N, Siedschlag G, Schubert EW, Lauerer B, Müller FA, Petschelt A, & Ebert J (2011) Strength and wear resistance of a dental glass-ionomer cement with a novel nanofilled resin coating *American Journal Dentistry* **24(2)** 124-128.
15. Hattab FN & Amin WM (2001) Fluoride release from glass ionomer restorative materials and the effects of surface coating *Biomaterials* **22(12)** 1449-1458.
16. Eichmiller FC & Marjenhoff WA (1998) Fluoride-releasing dental restorative materials *Operative Dentistry* **23(5)** 218-228.
17. GC America. EQUIA® Forte Bulk Fill, Fluoride Releasing, Glass Hybrid Restorative System. Retrieve online August 2, 2016 from: http://www.gcamerica.com/products/operatory/EQUIA_Forte/.
18. 3M ESPE Ketac Universal Aplicap, Technical Product Profile. Retrieve online September 13, 2016 from: http://solutions.3mae.ae/wps/portal/3M/en_AE/3M_ESPE/Dental-Manufacturers/Products/Dental-Restorative-Materials/Glass-Ionomer/Glass-Ionomer-Restorative-Aplicap/.
19. GC Fuji IX GP Extra. Retrieved online December 5, 2016 from: http://www.gcamerica.com/products/operatory/GC_Fuji_IX_GP_extra/index.php.
20. Ionostar Molar. Retrieved online December 5, 2016 from: http://www.voco.com/au/product/ionostar-molar/index.html.
21. ChemFil Rock. Retrieved online December 5, 2016 from: https://www.dentsplysirona.com/en-gb/products/restorative/direct-restoration/glass-ionomers.html/Restorative/Direct-Restoration/Glass-Ionomers/Classic-Glass-Ionomers/ChemFil-Rock/p/DET-60606596/c/1000418.html.
22. Lien W & Vandewalle KS (2009) Physical properties of a new silorane-based restorative system *Dental Materials* **26(4)** 337-344.
23. Ferracane JL (2013) Resin-based composite performance: are there some things we can't predict? *Dental Materials* **29(1)** 51-58.

24. Faria A, Benassi U, Rodrigues R, Ribeiro R, & Mattos M (2007) Analysis of the relationship between the surface hardness and wear resistance of indirect composites used as veneer materials *Brazilian Dental Journal* **18(1)** 60-64.

25. Bagheri R, Taha NA, Azar MR, & Burrow MF (2013) Effect of G-Coat Plus on the mechanical properties of glass-ionomer cements *Australian Dental Journal* **58(4)** 448-453.

26. Bonifácio CC, Werner A, & Kleverlaan CJ (2012) Coating glass-ionomer cements with a nanofilled resin *Acta Odontologica Scandinavica* **70(6)** 471-477.

27. Ilie N, Hickel R, Valceanu A, & Huth K (2012) Fracture toughness of dental restorative materials. *Clinical Oral Investigations* **16(2)** 489-498.

28. Al-Angari S, Hara A, Chu T, Platt J, Eckert G, & Cook B (2014) Physicomechanical properties of a zinc-reinforced glass ionomer restorative material *Journal of Oral Sciences* **56(1)** 11-16.

29. Faraji F, Heshmat H, & Banava S (2017) Effect of protective coating on microhardness of new glass ionomer cement: nanofilled coating versus unfilled resin *Journal of Conservative Dentistry* **20(4)** 260-263.

30. Klinke T, Daboul A, Schwahn C, Frankenberger R, Hickel R, & Biffar R (2016) Dental practitioner recruitment for a randomized clinical trial in the field to evaluate the performance of a new glass ionomer restoration material *Trials* **10(17)** 73-80.

31. Scholtanus J & Huysmans M (2007) Clinical failure of class-II restorations of a highly viscous glass-ionomer material over a 6-year period: a retrospective study *Journal of Dentistry* **35(2)** 156-162.

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Clinical Effects of Desensitizing Prefilled Disposable Trays in In-office Bleaching: A Randomized Single-blind Clinical Trial

LM Martins • LA Lima e Souza • E Sutil • LM da Silva • JOS Silva • A Reis • AD Loguercio

Clinical Relevance: The prefilled disposable tray can be used to decrease self-reported tooth sensitivity without influencing the bleaching efficacy.

https://doi.org/10.2341/18-149-C

Clinical Evaluation of Noncarious Cervical Lesions of Different Extensions Restored With Bulk-fill or Conventional Resin Composite: Preliminary Results of a Randomized Clinical Trial

AMO Correia • ALB Jurema • MR Andrade • ALS Borges • E Bresciani • TMF Caneppele

Clinical Relevance: Regular nanofilled and regular bulk-fill resin composites showed good clinical performances for restoring noncarious cervical lesions of different sizes after 1 year.

https://doi.org/10.2341/18-256-C

Fatigue Failure Load of a Bonded Simplified Monolithic Feldspathic Ceramic: Influence of Hydrofluoric Acid Etching and Thermocycling

LF Guilardi • GKR Pereira • AS Vallau • IA Silva • JC Giordani • LF Valandro • MP Rippe

Clinical Relevance: Defects introduced by hydrofluoric acid etching can propagate when the assembly is subjected to aging and fatigue stimuli, impairing its mechanical performance.

https://doi.org/10.2341/19-069-L

A Two-year Clinical Comparison of Three Different Restorative Materials in Class II Cavities

H Balkaya • S Arslan

Clinical Relevance: Both bulk-fill and conventional composite resins showed a clinically successful performance in Class II restorations over a two-year period, unlike the high-viscosity glass ionomer.

https://doi.org/10.2341/19-078-C

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The prefilled disposable tray can be used to decrease self-reported tooth sensitivity without influencing the bleaching efficacy.

SUMMARY

Objectives: This study aimed to evaluate the desensitizing effect of a prefilled disposable tray containing potassium nitrate and fluoride on the self-reported tooth sensitivity (TS) and the bleaching efficacy of 40% hydrogen peroxide bleaching agent used for in-office bleaching in comparison with potassium nitrate and fluoride gel applied in a conventional-delivered tray system in an equivalence clinical trial.

Methods and Materials: Seventy-eight patients, with a right maxillary canine darker than A3, were selected for this single-blind (evaluators), randomized clinical trial. Teeth were bleached in two sessions with a one-week interval in between. Before in-office bleaching, the prefilled disposable tray or conventional tray containing potassium nitrate and fluoride was used for 15 minutes. Subsequently, the bleaching agent was applied in two 20-minute applications (per the manufacturer's directions) in each session. The color change was evaluated by subjective (Vita Classical and Vita Bleachedguide) and objective (Easyshade Advance Spectrophotometer) methods at baseline and 30 days after the first bleaching session. TS was recorded for up to 48 hours using a 0-10 visual analog scale. The absolute risk was evaluated by chi-square test, while

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the intensity of TS was evaluated by McNemar test (α=0.05). Color change in shade guide units and ΔE was analyzed by Student *t*-test for independent samples (α=0.05).

Results: Significant whitening was observed in both groups after 30 days of clinical evaluation. The use of different methods of desensitizer in a tray did not influence the absolute risk and intensity of TS ($p>0.05$), although a tendency of lower risk of TS with the prefilled disposable tray containing potassium nitrate and fluoride was observed.

Conclusion: The use of a prefilled disposable tray containing potassium nitrate and fluoride before the application of the in-office bleaching product did not affect the whitening degree and decreased self-reported TS when compared with a conventional-delivered tray system.

INTRODUCTION

Tooth bleaching represents the most common elective dental procedure for treatment of discolored teeth,[1] and according to Dutra and others,[2] an estimated more than 1 million Americans whiten their teeth annually, driving nearly $600 million in revenues for dental offices. A great part of the success of the bleaching therapy is that this procedure is a very conservative, simple, and low-cost procedure.[3]

Unfortunately, tooth sensitivity (TS) is the most frequently reported side effect associated with bleaching, particularly with in-office bleaching protocols that employ relatively high concentrations of hydrogen peroxide.[4-6] Even though the sensitivity will be only transient and will be resolved a few days after the end of the bleaching procedure, it is an unpleasant experience[7,8] and can be severe and irritating enough to lead patients to withdraw from treatment in some cases.[9]

This adverse effect has motivated clinicians and researchers to develop strategies for the prevention of bleaching-induced TS.[1] Several approaches, such as administration of analgesics, anti-inflammatories, antioxidants, and corticosteroids,[10-13] have failed to minimize this side effect caused by bleaching products. The most useful and effective agent for the management of bleaching-induced TS is potassium nitrate associated or not associated with sodium fluoride when compared with a placebo group.[14-17]

A recent systematic review[18] showed that usually a 3%-5% potassium nitrate gel was applied for 10-30 minutes prior to the in-office bleaching procedure in a tray or directly in the office.[14-17] Only recently, as a response to the demand for an alternative way of bleaching, a new product has become available on the market: a prefilled disposable tray containing potassium nitrate (UltraEZ, Ultradent Products Inc, South Jordan, UT, USA). Prefilled disposable tray systems are comfortable and have a low cost, as the professional does not need to fabricate a custom bleaching tray (impression, model buildup, tray fabrication, etc), and the procedure can be done at home[19] or in the office in the waiting room immediately before in-office bleaching. However, to the extent of our knowledge, no clinical studies have been performed comparing potassium nitrate–based products delivered via different methods, which was the main objective of the present study.

Therefore, the aim of this equivalent clinical trial was to compare bleaching-induced TS between the application of a prefilled disposable tray containing a desensitizer agent prior to in-office bleaching and a conventionally delivered tray system with a desensitizer agent. The following null hypotheses were tested: 1) the preventive use of desensitizer agents in different trays will not affect the absolute risk and intensity of bleaching-induced TS, and 2) the preventive use of desensitizer agents in different trays will not affect the color change after bleaching.

METHODS AND MATERIALS

Study Design

This was a randomized, single-blinded (evaluators) equivalence trial with an equal allocation rate between groups. This clinical trial was approved by the Local University Ethics Committee (59645816.3.0000.5020). The study was also registered on the Clinical Trials website and took place within the dental clinics of the two universities from September 2015 to February 2016.

Inclusion and Exclusion Criteria

Participants were examined in a dental chair after dental prophylaxis with pumice and water to check whether they met the study's eligibility criteria. To be included in this study, participants had to be aged 18 years or older and have good general and oral health. Participants had to have at least one shade A3 or darker canine as assessed by a value-oriented shade guide (VITA classical, Vita Lumin, Vita Zahnfabrik, Bad Säckingen, Germany) and had to have at least six anterior maxillary sound teeth. Participants with restorations on the labial surface

of their anterior teeth and noncarious cervical lesions, with full crowns or veneers, visible cracks, gingival recession, endodontically treated teeth, spontaneous tooth pain, or internal tooth discoloration were excluded from this study. Patients who had teeth that had fluorosis, patients who were pregnant or lactating, and patients who had bruxism habits were also excluded from this study.

Sample Size Calculation

The sample size calculation was based on the absolute risk of TS, the primary outcome of the study. If there was truly no difference between the standard and experimental treatment, then 78 patients would be required to be 90% sure that the limits of a two-sided, 90% confidence interval (CI) would exclude a difference between the standard and experimental group of more than 30%.

Random Sequence Generation and Allocation Concealment

Seventy-eight participants were selected according to the inclusion and exclusion criteria for bleaching with Opalescence Boost (Ultradent Products Inc). One group applied a desensitizer agent in a conventional-delivered tray system (Desensitizer KF2%, FGM Ind, Joinville, SC, Brazil), and the other applied the desensitizer in a prefilled disposable tray (UltraEZ, Ultradent Products Inc). A third operator, not involved in the research protocol, conducted the randomization procedure by using computer-generated tables. A blocked randomization (block sizes of two) was used with an equal allocation ratio (www.sealedenvelope.com). The same operator placed the identification groups in sequentially numbered, opaque, and sealed envelopes. Once the participant was eligible for the procedure and had completed all baseline assessments, the operator could open the envelope. Neither the participant nor the operator knew the group allocation before this stage.

Study Intervention

To maintain the allocation concealment before starting the bleaching procedure, a custom-fitted tray was made for all patients. For this purpose, an alginate impression of each subject's maxillary and mandibular arch was made and filled with dental stone. To produce study models, block-out material to the labial surfaces of teeth was not applied. A 1-mm soft vinyl material, provided by the manufacturer, was used to fabricate the custom-fitted tray for the desensitizer gel. The excess material on the labial and lingual surfaces was cut 1 mm from the gingival junction.

Before each bleaching session, subjects were instructed to wear the conventional-delivered tray system containing 5% potassium nitrate and 2% sodium fluoride desensitizing gel (Desensibilize KF 2%) or the prefilled disposable tray containing less than 5% potassium nitrate and less than 1% sodium fluoride desensitizing gel (UltraEZ, Ultradent Products Inc) for 15 minutes according to the randomization of patients.

Immediately after tray removal, subjects were instructed to wash the tray and brush their teeth as usual before the in-office bleaching was performed. For this purpose, the gingival tissue was isolated with a light-cured resin dam (Opal Dam, Ultradent Products Inc). The 40% hydrogen peroxide gel Opalescence Boost (Ultradent Products Inc) was applied in two 20-minute applications (manufacturer's directions) to all maxillary incisors, canines, and premolars of the same patient (Table 1). After seven days, this procedure was repeated using the same protocol. All participants were instructed to brush their teeth at least three times a day using fluoridated toothpaste (Colgate, Colgate-Palmolive, SP, Brazil).

TS Evaluation

Patients were asked to record their perception of TS during the first and second bleaching sessions using the five-point Numeric Rating Scale (NRS; 0 = *none*, 1 = *mild*, 2 = *moderate*, 3 = *considerable*, and 4 = *severe*). Subjects were asked to record their experience with TS during the treatment up to one hour after bleaching, from one hour to 24 hours after bleaching, and from 24 hours to 48 hours after bleaching. They were also asked to record whether or not they experienced TS during the 30-day period after bleaching. As two bleaching sessions were performed, the highest NRS score obtained in both bleaching sessions was considered for statistical purposes.

Color Evaluation

Color was recorded before the bleaching procedure and seven days and 30 days after the end of the bleaching treatment using an objective method (Easyshade Advance spectrophotometer, Vident, Brea, CA, USA) and a subjective method (value-oriented shade guide Vita Classic and Vita Bleachedguide). Color evaluation was done in a room under artificial lighting conditions without interference

Table 1: *Products, Composition, and Application Regimens*

Products	Composition[a]	Groups	Application Regimen
Opalescence Boost	Gel: 40% hydrogen peroxide, 20% water and desensitizing agents (3% potassium nitrate and 1.1% fluoride)	Conventional delivered-tray group: Desensitizer KF2 was applied for 15 minutes in a conventional-delivered tray system before the start of the in-office bleaching application (contains 2% sodium fluoride and 5% potassium nitrate)	1. Dry teeth and apply Opal Dam to dental arch slightly overlapping enamel (building the barrier 4- to 6-mm high and 1.5- to 2.0-mm thick) and interproximal spaces. 2. Light cure Opal Dam for 20 seconds per arch using a scanning motion. Carefully check the resin cure with an instrument. 3. Attach both syringes before mixing. Press the plunger of the red syringe in, pushing all the contents into the clear syringe. Forcefully press the small clear stem completely into the larger clear stem. Then press the clear plunger completely into the red syringe. To activate, press the chemical from the red syringe into the clear syringe with thumbs. Reverse action, and mix a minimum of 25 times on each side. 4. Press all mixed gel into the RED syringe. Separate the two syringes and attach the Micro 20ga FX tip onto the red syringe. 5. Apply a 0.5- to 1.0-mm-thick layer of Opalescence Boost to the labial surface of the tooth and slightly onto the incisal surfaces. 6. Leave gel on for 20 minutes. 7. Suction off using a surgical aspirator tip. Do not use water. 8. Repeat gel application for another 20 minutes (40 minutes total). 9. At the end, suction all the gel off, then wash and apply suction. 10. Remove gingival barrier by lifting it from one end.
		UltraEZ prefilled tray group: UltraEZ was applied for 15 minutes before the start of the in-office bleaching application (prefilled disposable tray contains <5% sodium hydrogen, <1% sodium fluoride, and <5% potassium nitrate)	

[a] *According to the manufacturer.*

from outside light. For both devices, color was checked at the middle third of the canine.

For the objective shade evaluation, an impression of the maxillary arch with high-putty silicon paste (Clonage, Nova DFL, Rio de Janeiro, RJ, Brazil) was taken, and a window on the labial surface of the silicon guide was created using a metal device with a 6-mm radius. The purpose of this was to standardize the area for color evaluation in all recall periods with the spectrophotometer. Color was determined using the parameters of the digital spectrophotometer on which the following values were indicated: L*, a*, and b*, where L* represented luminosity (the value from 0 [black] to 100 [white]), and a* and b* represented color along the red-green axis and the color along the yellow-blue axis, respectively. The difference between the baseline and each recall period (ΔE^*) was calculated using the following formula: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. For the subjective evaluation, the 16 tabs of the shade guide (VITA classical, Vita Lumin, Vita Zahnfabrik, Bad Säckingen, Germany) and the 29 tabs of the shade guide (Vita Bleachedguide, Vita Lumin, Vita Zahnfabrik, Bad Säckingen, Germany) were arranged from whitest to darkest. For calibration purposes, 10 participants whom we did not include in the study sample participated in the training phase. The two examiners scheduled these patients for bleaching and evaluated their teeth against the shade guide at baseline and once again two days after. The two evaluators presented superior color-matching competency according to the ISO/TR 28642.[20] This means that they had an agreement of at least 85% (Kappa statistic) before beginning the study evaluation (85% of correctly matched pairs of tabs in shade guides). If disagreements occurred during the evaluation, they needed to reach a consensus before the participant was dismissed.

Statistical Analysis

The analysis followed the intention-to-treat protocol and involved all the participants who were randomly assigned in the study. The statistician was blinded to study groups. The color change (primary outcome) was used to determine the efficacy of the bleaching treatment. The color change (ΔShade Guide Unit [SGU] and ΔE) between the baseline and 30 days was calculated for each group. The ΔE and ΔSGU data were subjected to paired Students' *t*-test. We compared the study group's absolute risk of TS using the chi-square test. The CI for the effect size was calculated. The comparison of the TS intensity

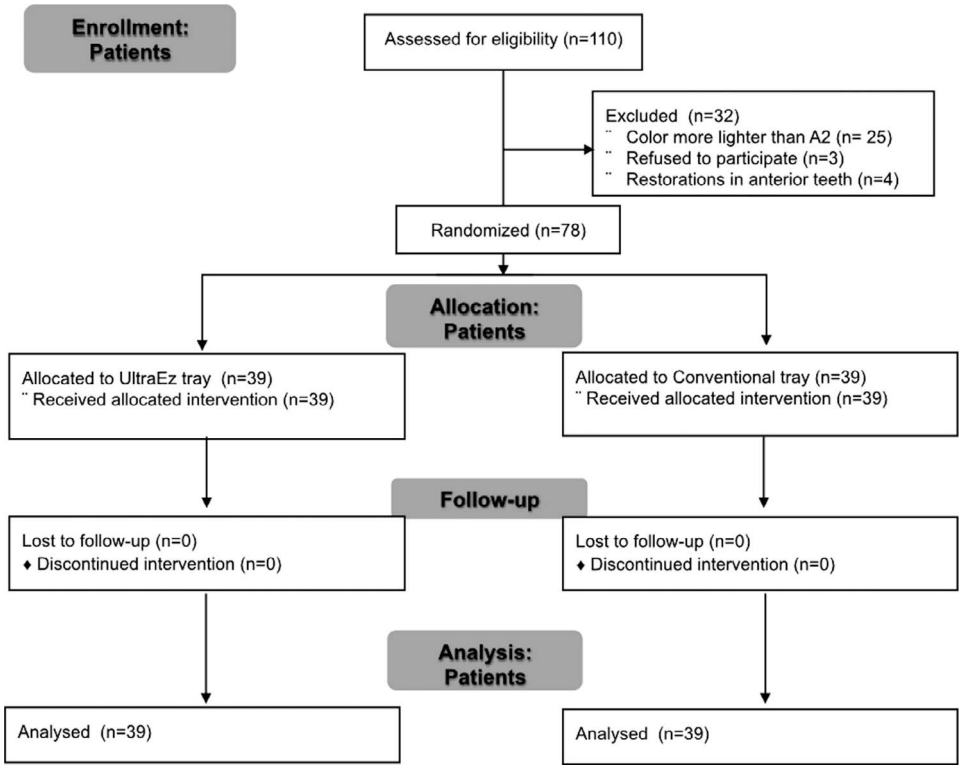

Figure 1. *CONSORT flow diagram detailing the recruitment and enrollment of the clinical trial.*

among time assessments for each group was performed using the McNemar test. The comparison of the intensity of TS among each group for different assessment points (during and following the bleaching process) was performed by applying the McNemar test. In all statistical tests, the alpha was preset at 0.05.

RESULTS

A total of 110 participants were examined in a dental chair to determine if they met the inclusion and exclusion criteria. A total of 79 patients were included in this clinical study (Figure 1). The mean age (years) of the participants and the baseline SGU are described in Table 2. One can observe comparable data among treatment groups by ensuring the comparability of the baseline features. None of the patients discontinued the intervention or presented adverse effects during the intervention. No medication and/or desensitizer were necessary to be prescribed or applied to the participants from this study for the relief of bleaching-induced TS.

Tooth Sensitivity

A total of 26 patients (absolute risk: 67%, 95% CI: 51% to 79%; Table 3) reported pain in the conventional-delivered tray group. Thirty-three patients (absolute risk: 84%, 95% CI: 70% to 93%; Table 3) reported pain in the prefilled disposable tray group. No significant difference was observed between the risks of TS of the two study groups (p=0.37; Table 3).

The TS intensity of both bleaching protocols was statistically similar (p=0.38), and the overall TS intensity at different assessment points is reported in Table 4. In all time assessments, the mean difference ranged from −0.01 to −0.03 and was not clinically important (Table 4). Most of the TS complaints occurred within the first 24 hours after bleaching and moved closer to zero after 24 hours.

Table 2: *Baseline Characteristics of the Participants Included in This Clinical Trial*

Characteristic	UltraEZ Prefilled Tray	Conventional-Delivered Tray
Age, y, mean ± SD	25.8 ± 6.5	24.5 ± 5.6
Baseline color, mean ± SD [median; interquartile range]		
SGU Vita Classical	10.1 ± 2.8 [9; 9-11]	10.0 ± 2.5 [9; 9-11]
SGU Vita Bleachedguide	11.3 ± 1.9 [10; 9-12]	11.0 ± 2.4 [10; 10-13]

Table 3: *Comparison of the Number of Patients Who Experienced Tooth Sensitivity (TS) at Least Once During the Bleaching Regimen in Both Groups Along With Absolute Risk and Risk Ratio*[a]

Treatment	Number of Participants With TS		Absolute Risk[a] (95% CI)	Risk Ratio (95% CI)
	Yes	No		
UltraEZ Prefilled Tray	26	13	67 (51-79) A	1.27 (0.97-1.64)
Conventional-delivered tray	33	06	84 (70-93) A	

[a] *McNemar test (p=0.27). Risks identified with different letters are statistically different.*

Color Change

The intra- and interexaminer kappa values were 0.95 to 0.88 for Vita Classical and 0.91 to 0.91 for Vita Bleachedguide, respectively. A whitening of approximately 7 to 9 SGUs and a ΔE of approximately 12 were detected for both groups 30 days after bleaching (Table 5). No statistically significant difference was observed between the study groups ($p>0.12$). For all color measurements, the mean difference ranged from −0.2 to 1.9 and was not clinically important (Table 5).

DISCUSSION

The results of the present study showed that no significant difference in terms of absolute risk and TS intensity was observed for a desensitizer gel containing nitrate potassium and fluoride applied in a conventional tray or in a prefilled disposable tray.

Regarding both substances used, the exact action mechanism of potassium nitrate and sodium fluoride for reducing bleaching-induced TS is not well understood. It is likely that fluoride prevents TS due to deposition of fluoride crystals in the exposed dentinal tubules.[21-23] However, in the present study, patients presenting teeth with visible cracks were excluded, mainly because enamel surface cracks or craze lines are potential factors in increasing bleaching-induced TS,[24] and this helps to explain the lack of preventive action of fluoride when applied alone before bleaching.[25]

On the other side, it is likely that potassium ions are the active component, and potassium nitrate works by reducing dentinal sensory nerve activity due to the depolarizing activity of the $K+$.[21,26-28] However, for this purpose, it was required that the potassium nitrate could be transported within the pulp chamber, preferably before hydrogen peroxide penetration.

Although the more useful protocol for preventing bleaching-induced TS is the topical application of substances containing potassium nitrate,[18,19,29] only recently have two papers shown that potassium nitrate penetrates the pulp chamber.[1,30] According to these papers, the transport within the tooth may be facilitated by the low molecular weight (101.10 g/mol) and water solubility of potassium nitrate gel.[1,30] Kwon and others[30] showed that this penetration occurs within the first five minutes after application, similar to hydrogen peroxide penetration,[31,32] and this is why nitrate potassium desensitizer gel needs to be applied before or together with the bleaching application.

Several factors may be involved in the potassium nitrate penetration, and one of them is material viscosity. The company that markets the prefilled disposable tray also produces the same product in syringes, and this is the most used product when applied in conventional-delivered tray systems.[19,27] However, according to Kwon and others,[1] UltraEZ (Ultradent Products Inc) in a syringe is the most viscous product when compared with other products in the market, and this characteristic negatively affects the penetration of the nitrate potassium. Unfortunately, no information is available regarding

Table 4: *Tooth Sensitivity Intensity (Means ± Standard Deviations) at the Different Assessment Points for Both Study Groups and the Statistical Comparison Along With the Effect Size (95% Confidence Interval) as Well as the* p*-Value of the Pairwise Comparison*[a]

Time Assessment	NRS Scale		Mean Difference (95% CI)
	UltraEZ Prefilled Tray	Conventional-Delivered Tray	
Up to 1 h	1.3 ± 1.7 A	1.4 ± 2.3 A	−0.1 (−1.01 to 0.81)
1 h to 24 h	1.8 ± 2.2 A	2.0 ± 1.3 A	−0.2 (−1.01 to 0.61)
24 h to 48 h	0.4 ± 0.9 B	0.7 ± 1.5 B	−0.3 (−0.86 to 0.26)

[a] *McNemar test (p=0.53) was applied for comparison of time assessments between groups, and McNemar test (p=0.38) was applied for comparison of time assessments within each group. Means identified with the same letters are statistically similar.*

Table 5: *Color Change in Shade Guide Units (SGU) and ΔE (Means ± Standard Deviations) Between Baseline and 30 Days After Bleaching for the Two Treatment Groups Along With the Effect Size (95% Confidence Interval) as Well as the p-Value of the Pairwise Comparison*[a]

Color Evaluation Tools	UltraEZ Prefilled Tray	Conventional-Delivered Tray	Mean Difference (95% CI)	*p*-Value[a]
ΔSGU (Vita Classical)	7.4 ± 2.8 A	7.6 ± 2.5 A	−0.2 (−1.40 to 1.00)	0.72
ΔSGU (Vita Bleachedguide)[b]	8.4 ± 3.4 B	9.3 ± 3.5 B	−0.9 (−2.46 to 0.66)	0.35
ΔE	12.8 ± 4.5 C	10.9 ± 4.3 C	1.9 (−0.09 to 3.89)	0.13

[a] *Student t-test. Means identified with the same letters are statistically similar.*
[b] *A numerical system using numbers 1 through 15 (corresponding to the 15 tabs) was used.*

the viscosity of Desensitizer KF2, although the manufacturer indicated that this is a low-viscosity gel, probably similar to other gels available in the market.[1]

Another factor that must be taken into account is the amount of gel applied. The proper dosage of potassium nitrate for maximum efficacy is still unknown, but it is expected that within certain parameters, a higher dosage of gel means a higher amount of gel inside the pulp chamber.

The amount of bleaching gel present in a prefilled disposable tray is about 60 mg, as per manufacturer descriptions. To the extent of the authors' knowledge, no studies have measured the amount of desensitizer gels inside the trays; however, different studies have measured the amount of bleaching gels, and these quantities range between 500 and 900 mg per application, mainly because the application depends on the patients' subjective interpretation regarding the amount of gel to insert in the tray.[33,34] Once again, a lower effectiveness of prefilled disposable trays is expected.

However, according to Kwon and others,[1] the most important factor in terms of potassium nitrate penetration is the product concentration. Although the exact concentration of nitrate potassium is a manufacturer property, according to the MSDS of each manufacturer, both delivered methods (conventional-delivered tray system and prefilled disposable tray) containing similar concentrations of potassium nitrate and sodium fluoride (Table 1) help to explain the similar results between both products.

The cause of bleaching-induced TS is not completely understood. According to the "hydrodynamic hypothesis,"[35] thermal and tactile stimuli are effective for evaluating dental sensitivity when dentin is exposed.[36] Since the hydrodynamic theory of dentin sensitivity enjoys wide acceptance as the explanation of dentinal sensation, many authors view bleaching-related pain as a form of dentin sensitivity.[37] Major differences distinguish bleaching-related pain from dentin hypersensitivity.

Although pain in bleached teeth can be evoked by thermal or other stimuli, most patients complain of tingling or shooting pain (zingers)[4] without provoking stimuli. Pain during and following bleaching treatments can affect intact teeth lacking dentin exposure, which is in sharp contrast to dentin sensitivity, in which pain occurs in teeth with exposed dentin. Recently, Markowitz[38] showed an alternative hypothesis related to the bleaching-induced TS. According to this author,[39] bleaching-induced TS arises as a consequence of peroxide penetrating the tooth structure, causing direct activation of a neuronal receptor and not through the hydrodynamic mechanism.

This fact, along with the current need of reporting patient-centered outcomes, led the authors of the present study to use self-reported pain, as done in clinical trials of bleaching that evaluate TS as the primary outcome.[5,10,12,15,19,24,29,39] However, we cannot rule out the fact that there are other methods described in the literature to evaluate TS that are not employed in the present study, and this can be considered one of the study limitations. Among them, we can cite the Schiff Cold Air and Yeaple Probe Tactile methods, commonly used in studies that evaluate dentin hypersensitivity.[40,41] To the extent of the authors' knowledge, they have not been used in bleaching studies yet, but they need to be included in future bleaching studies to determine if they can add significant information.

Regarding the color evaluation, despite one recent clinical study suggesting that potassium nitrate pretreatment may negatively affect whitening efficacy,[15] the results of the present study showed significant whitening at the end of the bleaching protocol, with the use of three different instruments, which is in accordance with the findings of several other clinical trials that evaluated color change.[3,5,10-18] As both desensitizer gels used were colorless, no significant interaction with tooth color was expected.

In the present study, color measurement was performed only in canines because these teeth are darker than incisors,[42,43] and this allows for more sensitive color change evaluation. An earlier article showed that a significantly stronger overall increase in lightness was observed for canines after treatment when compared with incisors. At the end of the bleaching treatment, teeth become more homogeneous in terms of lightness values.[44] In agreement with these findings, Ontiveros and others[45] observed that whitening of canines was 1.4-1.6 times more pronounced that of incisors. A recent study of the literature demonstrated that a higher degree of whitening occurs in teeth with a darker baseline color.[3] By using canines as the reference for color evaluation, the recruitment of patients becomes easier. Patients with incisors of color A3 are rare, but patients with canines of color A3 are quite common.

Although canines did not have a flat labial face, the use of custom molds enabled standardization of the area for color evaluation, thereby securing a correct angulation for placement of the spectrophotometer tip in all recall periods of this study. However, it worth mentioning that at least two previously published clinical studies did not show any significant variations in the color measurement with the spectrophotometer when measured in incisors or canines.[44,45]

Finally, if the use of a prefilled disposable tray to apply the desensitizer agent is taken into account, this new system has an advantage in comparison with the use of conventional custom trays. The professional does not need to fabricate a personalized custom tray (impression, model buildup, tray fabrication, and so on), and the procedure can be done in the waiting room of the office before dental care or even at home, in the same way that the prefilled disposable trays containing hydrogen peroxide can be used.[39]

CONCLUSIONS

The use of a prefilled disposable tray containing potassium nitrate and fluoride before the application of the in-office bleaching product did not affect the whitening degree and did not decrease self-reported TS, which was similar to the use of a conventional-delivered tray system.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Amazonas. The approval code for this study is 59645816.3.0000.5020.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Kwon S, Dawson D, Schenck D, Fiegel J, & Wertz P (2015) Spectrophotometric evaluation of potassium nitrate penetration into the pulp cavity *Operative Dentistry* **40(6)** 614-621.
2. Dutra A, Frary J, & Wise R (2004) Higher-order needs drive new growth in mature consumer markets *Journal of Business Strategy* **25(5)** 26-34.
3. Rezende M, Loguercio AD, Kossatz S, & Reis A (2016) Predictive factors on the efficacy and risk/intensity of tooth sensitivity of dental bleaching: a multi regression and logistic analysis *Journal of Dentistry* **45** 1-6.
4. Haywood VB (2005) Treating sensitivity during tooth whitening *Compendium of Continuing Education in Dentistry* **26(9 Supplement 3)** 11-20.
5. Matis B, Cochran M, & Eckert G (2009) Review of the effectiveness of various tooth whitening systems *Operative Dentistry* **34(2)** 230-235.
6. Nathanson D (1997) Vital tooth bleaching: sensitivity and pulpal considerations *Journal of American Dental Associaction* **28(Supplement)** 41S-44S.
7. Hewlett ER (2007) Etiology and management of whitening-induced tooth hypersensitivity *Journal of California Dental Association* **35(7)** 499-506.
8. Markowitz K (2010) Pretty painful: why does tooth bleaching hurt? *Medical Hypotheses* **74(5)** 835-840.
9. do Amaral FL, Sasaki RT, da Silva TC, Franca FM, Florio FM, & Basting RT (2012) The effects of home-use and in-office bleaching treatments on calcium and phosphorus concentrations in tooth enamel: an *in vivo* study *Journal of American Dental Association* **143(6)** 580-586.
10. Charakorn P, Cabanilla LL, Wagner WC, Foong WC, Shaheen J, Pregitzer R, & Schneider D (2009) The effect of preoperative ibuprofen on tooth sensitivity caused by in-office bleaching *Operative Dentistry* **34(2)** 131-135.
11. de Paula EA, Kossatz S, Fernandes D, Loguercio AD, & Reis A (2014) Administration of ascorbic acid to prevent bleaching-induced tooth sensitivity: a randomized tri-

ple-blind clinical trial *Operative Dentistry* **39(2)** 128-135.

12. de Paula EA, Loguercio AD, Fernandes D, Kossatz S, & Reis A (2013) Perioperative use of an anti-inflammatory drug on tooth sensitivity caused by in-office bleaching: a randomized, triple-blind clinical trial *Clinical Oral Investigation* **17(9)** 2091-2097.
13. Rezende M, Bonafe E, Vochikovski L, Farago PV, Loguercio AD, Reis A, & Kossatz S (2016) Pre- and postoperative dexamethasone does not reduce bleaching-induced tooth sensitivity: a randomized, triple-masked clinical trial *Journal of American Dental Association* **147(1)** 41-49.
14. Bonafé E, Loguercio AD, Reis A, & Kossatz S (2014) Effectiveness of a desensitizing agent before in-office tooth bleaching in restored teeth *Clinical Oral Investigations* **18(3)** 839-845.
15. Palé M, Mayoral JR, Llopis J, Vallès M, Basilio J, & Roig M (2014) Evaluation of the effectiveness of an in-office bleaching system and the effect of potassium nitrate as a desensitizing agent *Odontology* **102(2)** 203-210.
16. Reis A, Dalanhol A, Cunha T, Kossatz S, & Loguercio A (2011) Assessment of tooth sensitivity using a desensitizer before light-activated bleaching *Operative Dentistry* **36(1)** 12-17.
17. Tay LY, Kose C, Loguercio AD, & Reis A (2009) Assessing the effect of a desensitizing agent used before in-office tooth bleaching *Journal of the American Dental Association* **140(10)** 1245-1251.
18. Wang Y, Gao J, Jiang T, Liang S, Zhou Y, & Matis BA (2015) Evaluation of the efficacy of potassium nitrate and sodium fluoride as desensitizing agents during tooth bleaching treatment—a systematic review and meta-analysis *Journal of Dentistry* **43(8)** 913-923.
19. Leonard RH, Smith LR, & Caplan DJ (2004) Desensitizing agent efficacy during whitening in an at-risk population *Journal of Esthetic and Restorative Dentistry* **16(1)** 49-55.
20. International Organization for Standardization (2011) ISO/TR 28642. Dentistry-guidance on color measurement. Geneva, Switzerland: International Organization for Standardization.
21. Bartold P (2006) Dentinal hypersensitivity: a review *Australian Dental Journal* **51(3)** 212-218.
22. Fugaro JO, Nordahl I, Fugaro OJ, Matis BA, & Mjor I (2004) Pulp reaction to vital bleaching *Operative Dentistry* **29(4)** 363-368.
23. Tal M, Oron M, Gedalia I, & Ehrlich J (1976) X-ray diffraction and scanning electron microscope investigations of fluoride-treated dentine in man *Archives of Oral Biology* **21(5)** 285-290.
24. Özcan M, Abdin S, & Sipahi C (2014) Bleaching induced tooth sensitivity: do the existing enamel craze lines increase sensitivity? A clinical study *Odontology* **102(2)** 197-202.
25. Armênio RV, Fitarelli F, Armênio MF, Demarco FF, Reis A, & Loguercio AD (2008) The effect of fluoride gel use on bleaching sensitivity: a double-blind randomized controlled clinical trial *Journal of the American Dental Association* **139(5)** 592-597.
26. Haywood VB (2005) Treating sensitivity during tooth whitening *Compendium of Continuing Education in Dentistry* **26(9 Supplement 3)** 11-20.
27. Haywood VB, Caughman WF, Frazier KB, & Myers ML (2001) Tray delivery of potassium nitrate—fluoride to reduce bleaching sensitivity *Quintessence International* **32(2)** 105-109.
28. Hodosh M (1974) A superior desensitizer—potassium nitrate *Journal of the American Dental Association* **88(4)** 831-832.
29. Haywood VB, Cordero R, Wright K, Gendreau L, Rupp R, Kotler M, Littlejohn S, Fabyanski J, & Smith S (2005) Brushing with a potassium nitrate dentifrice to reduce bleaching sensitivity *Journal of Clinical Dentistry* **16(1)** 17-22.
30. Kwon SR, Dawson DV, & Wertz PW (2016) Time course of potassium nitrate penetration into the pulp cavity and the effect of penetration levels on tooth whitening efficacy *Journal of Esthetic and Restorative Dentistry* **28(S1)** S14-S22.
31. Kwon SR, Li Y, Oyoyo U, & Aprecio RM (2012) Dynamic model of hydrogen peroxide diffusion kinetics into the pulp cavity *Journal of Contemporary Dental Practice* **13(4)** 440-445.
32. Ilan R, Yarom T, & Israel L (1991) Effect of bleaching time and temperature on the radicular penetration of hydrogen peroxide *Dental Traumatology* **7(5)** 196-198.
33. Dahl JE & Becher R (1995) Acute toxicity of carbamide peroxide and a commercially available tooth-bleaching agent in rats *Journal of Dental Research* **74(2)** 710-714, 10.
34. Li Y (1996) Biological properties of peroxide-containing tooth whiteners *Food and Chemical Toxicology* **34(9)** 887-904.
35. Brännström M (1986) The hydrodynamic theory of dentinal pain: sensation in preparations, caries, and the dentinal crack syndrome *Journal of Endodontics* **12(10)** 453-457.
36. Pashley DH (1986) Dentin permeability, dentin sensitivity, and treatment through tubule occlusion. *Journal of Endodontics* **12(10)** 465-474.
37. Swift EJ Jr (2005) Tooth sensitivity and whitening. *Compendium Continuing Education in Dentistry* **26(9 Supplement 3)** 4-10.
38. Markowitz K (2010) Pretty painful: why does tooth bleaching hurt? *Medical Hypotheses* **74(5)** 835-840.
39. Carlos N, Bridi E, Amaral F, França F, Turssi C, & Basting R (2017) Efficacy of home-use bleaching agents delivered in customized or prefilled disposable trays: a randomized clinical trial *Operative Dentistry* **42(1)** 30-40.
40. Trushkowsky RD & Garcia-Godoy F (2014) Dentin hypersensitivity: differential diagnosis, tests, and etiology *Compendium of Continuing Education in Dentistry* **35(2)** 99-104.

41. Gillam DG (2013) Current diagnosis of dentin hypersensitivity in the dental office: an overview *Clinical Oral Investigation* **17(Supplement 1)** 21-29.

42. Hasegawa A, Motonomi A, Ikeda I, & Kawaguchi S (2000) Color of natural tooth crown in Japanese people *Color Research in Applications* **25(1)** 43-48.

43. Hasegawa A, Ikeda I, & Kawaguchi S (2000) Color and translucency of *in vivo* natural central incisors *Journal of Prosthetic Dentistry* **83(4)** 418-423.

44. Wetter NU, Branco EP, Deana AM, & Pelino JE (2009) Color differences of canines and incisors in a comparative long-term clinical trial of three bleaching systems. *Lasers in Medicine Science* **24(6)** 941-947.

45. Ontiveros JC, Eldiwany MS, & Paravina R (2012) Clinical effectiveness and sensitivity with overnight use of 22% carbamide peroxide gel. *Journal of Dentistry* **40(Supplement 2)** e17-e24.

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Clinical Evaluation of Noncarious Cervical Lesions of Different Extensions Restored With Bulk-fill or Conventional Resin Composite: Preliminary Results of a Randomized Clinical Trial

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Clinical Relevance

Regular nanofilled and regular bulk-fill resin composites showed good clinical performances for restoring noncarious cervical lesions of different sizes after 1 year.

SUMMARY

Purpose: This randomized clinical trial evaluated the influence of the occlusogingival distance (OGD) of noncarious cervical lesions (NCCLs) on the clinical performance of a regular bulk-fill resin composite and a regular nanofilled resin composite.

Methods and Materials: A total of 140 restorations were randomly placed in 77 participants by one operator. NCCLs were divided into four groups (n=35) according to OGD (1.5 mm±10% or 3 mm±10%) and resin composites (Filtek

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Bulk Fill Posterior [B] or Filtek Z350 XT [C]) used: 1.5 mm-B, 1.5 mm-C, 3 mm-B, and 3 mm-C. A two-step self-etch adhesive (Clearfil SE Bond) was applied following manufacturer instructions in all restorative procedures. Restorations were polished 1 week after placement. Clinical evaluation was performed at baseline (7 days), 6 months, and 1 year by two calibrated examiners, according to the modified US Public Health Service criteria evaluating fractures/retention, marginal staining, marginal adaptation, recurrence of caries, anatomic form, postoperative sensitivity, and surface texture. The Kruskal-Wallis test was used for intergroup comparison in each follow-up; the Friedman analysis of variance, followed by the least significant difference test (multiple comparisons) was used for intragroup comparison between baseline and follow-up times (α=0.05).

Results: Two restorations were lost at 12 months (1 for 1.5 mm-B and 1 for 3 mm-B). The retention rates at 12 months were 100% for 1.5 mm-C, 97% for 1.5 mm-B, 100% for 3 mm-C; and 97% for 3 mm-B, with no statistical difference among the groups (p=0.570). At 12 months, a statistically significant difference was found among the follow-up times for the same group (1.5 mm-B, 1.5 mm-C, and 3 mm-B) regarding the marginal staining criterion; moreover, the 3 mm-C group showed a significant difference from 6 months. No significant difference was found for the other parameters.

Conclusion: Both resin composites showed acceptable clinical performance, and the OGD of NCCLs did not influence the clinical performance of resin composite restorations after 12 months.

INTRODUCTION

Noncarious cervical lesions (NCCLs) are the result of the loss of cervical hard dental tissue at the level of the cementoenamel junction.[1] The NCCLs result from the accumulation of tension, attrition, and biocorrosion of tooth structure.[2,3] Therefore, they may represent superficial or more profound defects, and they may present different forms (wedge-shaped, flat, concave, or acute angle) and dimensions (vertical and horizontal width).[1,4] Clinically, these morphologic features may influence the choice of treatment of NCCLs and the longevity of restoration.[4–6]

NCCLs should be restored to treat dental hypersensitivity, prevent against further loss of tooth tissues, and improve esthetics.[7,8] However, restorative procedures are challenging because of the nonretentive shape of the cavity, the presence of sclerotic dentin, and the location of dentin and cement margins that are unfavorable for adhesion.[7,9–11] Therefore, micromechanical or chemical retention preserving dental structure, good esthetics, and functional characteristics are essential aspects in the choice of restorative material.[1]

Adhesive materials, such as glass ionomer cements[12–14] and resin composites,[1,2,15] are indicated to replace the lost tissue in the cervical region. Despite the high retention rate, glass ionomers usually have poorer esthetics (higher surface roughness, lower color stability, and lower wear resistance), and inferior mechanical properties (poor strength and hardness) compared with those of resin composites.[13,14]

Resin composites have been widely used in the treatment of NCCLs. The development and improvement of these materials, especially with the addition of nanoparticles, has led to excellent physicomechanical properties, such as surface smoothness and reduction of polymerization shrinkage.[16,17] Retention, marginal staining, and marginal adaptation are considered important parameters for evaluating resin restorations in NCCLs[2,18,19] and are directly related to the stress produced in the tooth/restoration interface,[20,21] which is influenced by the characteristics of the composite and the cavity to be restored.[22] The results of some experimental studies showed a direct relationship between the marginal quality and cavity dimensions.[22,23] These findings suggest that more severe microleakages are expected in restorations with larger dimensions, consequently, with a larger volume.

The bulk-fill resins were developed to overcome issues such as volumetric shrinkage and polymerization shrinkage stress. Thus, a bulk-fill resin may be appropriate for restorative treatment of NCCLs to minimize polymerization shrinkage.[24–26] Other potential advantages are related to the simplification of the clinical technique, more compact fillings, and time savings.[26,27] Favorable results have been reported from *in vitro* studies evaluating the physicomechanical properties of these materials.[17,24,27–29] Recent studies with bulk-fill resins evaluated their clinical performance as the base or lining of Class I and II restorations.[30–35] In NCCLs, a flowable bulk-fill resin was reported to have a similar clinical

performance to conventional composite resins after 1 year of follow-up.[15]

The influence of various clinical characteristics and factors associated with NCCLs and the influence of technique and restorative material on the longevity of the treatment have been reported.[2,9,13,15,18,36–39] This research is of relevance because the treatment protocols for NCCLs, as for those analyzed in this study (the influence of the size of NCCLs), had not been tested previously, and other factors influencing the longevity of restorations have not yet been investigated. Thus, this randomized controlled clinical trial aimed to evaluate the influence of the occlusogingival distance (OGD) of NCCLs and the type of resin composite (regular bulk-fill or regular nanofilled) on the 1-year performance of resin composite restorations.

METHODS AND MATERIALS

Ethics Approval and Protocol Registration

The Institutional Review Board approved this study. The protocol was registered in the Brazilian Clinical Trials Registry (ReBEC-www.ensaiosclinicos.gov.br). This clinical investigation followed the Consolidated Standards of Reporting Trials statement (CONSORT).[40]

Study Design and Locations of Data Collection

This was a randomized, controlled, parallel, blind (participants and examiners) clinical trial and was conducted between September 2016 and July 2018 at the clinic of the School of Dentistry.

Recruitment and Eligibility Criteria

Individuals seeking treatment in a screening clinic in the Restorative Dentistry Department of the local university were recruited for the study. A total of 128 participants were examined by two calibrated dental students to determine whether they met the eligibility criteria. Participants had to be at least 18 years old and be in good general and oral health, with an acceptable oral hygiene level and with at least 20 teeth in occlusion. In addition, they had to have at least one NCCL to be restored that was deeper than 1 mm in vital canines or premolars without mobility, with an opposing and adjacent tooth, and have an OGD of 1.5 mm or 3 mm (±10%). Patients with poor oral hygiene, severe or chronic periodontal disease, orthodontic appliances, severe bruxism; smokers; or participants undergoing tooth whitening procedures were excluded from the study.

Before measurement of the OGD, a dental screening and dental prophylaxis (rubber cup + pumice + water) was done in all participants. Then, two-step silicone impressions (Express XT Putty Soft + Express XT Light Body Quick; 3M/ESPE, St Paul, MN, USA) were made of each tooth with NCCLs that met the inclusion criteria. Gingival retraction cords (Ultrapak #000 and Ultrapak #00; Ultradent Products, Inc, South Jordan, UT, USA) were used in the gingival sulcus to better visualize the margin of the lesion. The impressions were disinfected and poured with gypsum (Durone IV; Dentsply Sirona, York, PA, USA) 3 hours after removal. The excess material was removed from all surfaces, and the cast was scanned with an extraoral scanner (inEos Blue; Dentsply Sirona, Vienna, Austria). The digitalized data were transmitted to a computer-aided design software program (Rhinoceros 4.0; McNeel North America, Seattle, WA. USA) in which the OGD (distance between the most apical point of the gingival margin to the occlusal margin, tracing a line parallel to the long axis of the tooth) of the lesions, was analyzed. After determining the OGD, the restorative treatment was randomly defined according to the sequence generated.

Sample-Size Calculation

The sample size was estimated by Sealed Envelope online software (Sealed Envelope Ltd. 2012. Power calculator for binary outcome equivalence trial. [Online] Available from: https://sealedenvelope.com/power/binary-equivalence/ [Accessed May 02 2016]) An annual failure rate of 2.2% to two-step self-etch adhesives in NCCLs was considered.[10] Thus, after 2 years, the retention rate of this material will be approximately 95.6%. The minimal sample size was 33 restorations using an alpha of 0.05, a power of 80%, and a two-sided test to detect a difference among the groups of 15%. Considering the estimated dropout rate throughout the experimental period, 35 teeth per group was determined.

Randomization and Allocation Concealment

The randomization lists were prepared using a website (www.random.org) with an equal allocation ratio for all comparison groups: NCCLs with OGD 1.5 mm (±10%) restored with either Filtek Z350 XT [C] or Filtek Bulk Fill Posterior [B] (3M ESPE, St Paul, MN, USA); and NCCLs with OGD 3 mm (±10%) restored with either Filtek Z350 XT [C] or Filtek Bulk Fill Posterior [B], for a total of four groups (n=35): 1.5 mm-B, 1.5 mm-C, 3 mm-B, and 3 mm-C.

Table 1: *Information About Restorative Materials Used*

Material	Manufacturer	Composition
Clearfil SE Bond	Kuraray America, Inc, New York, NY, USA	Primer: 10-MDP, HEMA, DMA, catalyst, water. Bond: 10-MDP, HEMA, DMA, Bis-GMA, filler, catalyst.
Filtek Z350 XT	3M ESPE, St Paul, MN, USA	Filler: 78.5 wt% (59.5 vol%) silica, zirconia, aggregated zirconia/silica. Matrix: Bis-GMA, UDMA, TEGDMA, dimethacrylate.
Filtek Bulk Fill Posterior	3M ESPE	Filler: 76.5 wt% (58.4 vol%) Silica, zirconia, ytterbium trifluoride, aggregated zirconia/silica. Matrix: AUDMA, AFM, UDMA, DDDMA, EDMAB.

Abbreviations: AFM, addition-fragmentation monomer; AUDMA, aromatic urethane dimethacrylate; Bis-GMA, bisphenol-glycidyl methacrylate; DDDMA, 1, 12-dodecanediol dimethacrylate; DMA, dimethacrylate; EDMAB, ethyl 4-dimethyl aminobenzoate. HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate;

For the allocation, opaque and sealed envelopes were used and numbered by a person not involved in the study. Just before the restorative procedure began, the envelope was opened, which ensured allocation concealment. In cases where the participant presented lesions with the same OGD, the tooth located in the quadrant of the smaller number and more mesial in this quadrant was the first to be assigned, continuing similarly until all teeth meeting the inclusion criteria were restored.

Restorative Procedures

Before starting the restorative procedure, participants signed the informed consent form. The dental prophylaxis (rubber cup + pumice + water) was performed and verbal oral hygiene instructions provided.

The shape of the lesions (labeled as saucer-shaped, wedge-shaped, or mixed-shape) were analyzed by a computer-aided design software program (Rhinoceros 4.0). The presence of wear facets was recorded. Preoperative sensitivity was also evaluated by applying air for 10 seconds from a dental syringe placed 2 cm from the tooth surface.

An experienced dentist with more than 5 years of dental practice experience restored all teeth. Participants received restorations in previously selected lesions that met the inclusion criteria.

A predetermined procedure was performed, which included cavity cleaning with rubber cup, pumice and water, rinsing, and drying. A shade guide was used for shade selection. No additional retentive features or bevels were placed in the cavities as per the guidelines recommended by the American Dental Association.[41] The relative isolation method was performed in all procedures. For this, gingival retraction cord (Ultrapak #000; Ultradent Products, Inc), cotton rolls, and a saliva aspirator were used.

The cavities received the two-step self-etch adhesive Clearfil SE Bond (Kuraray America, Inc, New York, NY, USA), according to the manufacturer's instructions (Table 1). One coat of primer was applied on the entire lesion surface for 20 seconds. A gentle air stream was applied for approximately 5 seconds to evaporate the solvent. Then, the adhesive was applied and light cured for 10 s at 800 mW/cm^2 (Radii cal, SDI, Victoria, Australia).

The cavities were restored with two different resin composites (Table 1). Filtek Bulk Fill Posterior was placed in a single increment and light cured for 40 seconds using a calibrated light-curing unit (Radii cal, SDI) at 800 mW/cm^2 with a radiometer (Demetron; Kerr, Orange, CA, USA) for half the lesions according to the predetermined OGD (1.5 mm±10% and 3 mm±10%). Filtek Z350 XT was used in up to three increments for cavities with an OGD of 3 mm (±10%) and in a single increment for cavities with an OGD of 1.5 mm (±10%). Light-curing of each increment was performed for 20 seconds at 800 mW/cm^2.

Final contouring was carried out with 12-fluted tungsten carbide burs (FG Bur; KG Sorensen, Barueri, SP, Brazil) immediately after the restorative procedure. Polishing was performed with disks (Sof-Lex, 3M ESPE) 7 days after placement.

Calibration Procedures

For calibration, 10 representative photographs of each score for each criterion were examined by two independent experienced dentists. Also, 10 patients, not involved in this study, were evaluated on two consecutive days. Before the evaluations, an intra-examiner and interexaminer agreement of at least 85% was obtained.[42]

Blinding

The two examiners and all participants were blinded to the group allocation during all recalls, resulting in a double-blind trial design.

Table 2: *Category and Grade Descriptions for the Clinical Evaluation According to the Modified US Public Health Service Criteria*

Category	Grade	Criterion
Retention	Alfa	No loss of restorative material
	Charlie	Partial or complete loss of restorative material
Marginal staining	Alfa	No discoloration along the margin
	Bravo	Slight and superficial staining (removable, usually localized)
	Charlie	Deep staining cannot be polished away
Marginal adaptation	Alfa	Restoration is continuous with existing anatomic form, explorer does not catch
	Bravo	Detectable V-shaped defect in enamel only. Catches explorer going both ways
	Charlie	Detectable V-shaped defect to dentin-enamel junction
Recurrence of caries	Alfa	No evidence of caries contiguous with the margin
	Charlie	Evidence of presence of caries
Anatomic form	Alfa	Continuous with adjacent anatomy
	Bravo	Missing of restorative material without exposing the dentin or base
	Charlie	Missing restorative material sufficient to expose the dentin or base
Postoperative sensitivity	Alfa	No postoperative sensitivity directly after the restorative process and during the study period
	Charlie	Sensitivity present at any time during the study period
Surface Texture	Alfa	Surface texture similar to that of enamel
	Bravo	Surface texture similar to composite resin surface
	Charlie	Surface texture with porosities, catches explorer

Clinical Evaluation

Two examiners evaluated all the restorations independently. When disagreements occurred, a discussion led to a consensus. The parameters evaluated by each examiner were individually recorded in a standardized form during all recall times.

The restorations were evaluated at baseline (7 days) and at 6 and 12 months after restoration placement using the modified US Public Health Service (USPHS) criteria (Table 2). The primary measurable variable was restoration retention or presence of fractures, followed by the secondary measurable variables: marginal staining, marginal adaptation, recurrence of caries, anatomic form, postoperative sensitivity, and surface texture. Seven days after the restoration placement, postoperative sensitivity was evaluated. Air from a dental syringe positioned 2 mm from the tooth surface was applied for 10 seconds. These variables were ranked with the following scores: Alfa (acceptable restoration), Bravo (minor change of the restoration), and Charlie (unacceptable restoration).

Statistical Analysis

R statistical language R Studio (version 3.4.4, R Studio Team, Boston, MA, USA) was used for statistical analysis of each criterion. The descriptive statistical analyses included the evaluated restorations, not considering the dropouts. The inferential statistical analyses followed the intention-to-treat protocol suggested by CONSORT,[40] which involved all teeth initially randomized, including those that were not evaluated at the specified time. In this case, the missing data were included with the score of the last evaluation.

The Kruskal-Wallis test was performed to analyze all evaluated criteria among the groups at each evaluation time. The Friedman repeated analysis of variance assessed the difference in the performance of each group among three recall times (baseline, 6 months, and 12 months), followed by the least significant difference test for multiple comparisons, when applicable. The Cohen kappa statistics were used to test interexaminer agreement. A significance level of 5% was adopted in all tests.

RESULTS

Characteristics of Included Participants

In this study, 128 individuals were examined. Fifty-one did not fulfill the inclusion criteria, so they were not included. Thus, 77 participants were enrolled in the clinical trial. A total of 140 restorations were placed, 35 in each of the four groups (Figure 1). Details about the participants and characteristics of the NCCLs are presented in Table 3.

At baseline (7 days) and after 6 months, all participants were evaluated (Figure 1). After 12 months, the recall rate was 97.1% (136 restorations of 140). Four restorations were not evaluated

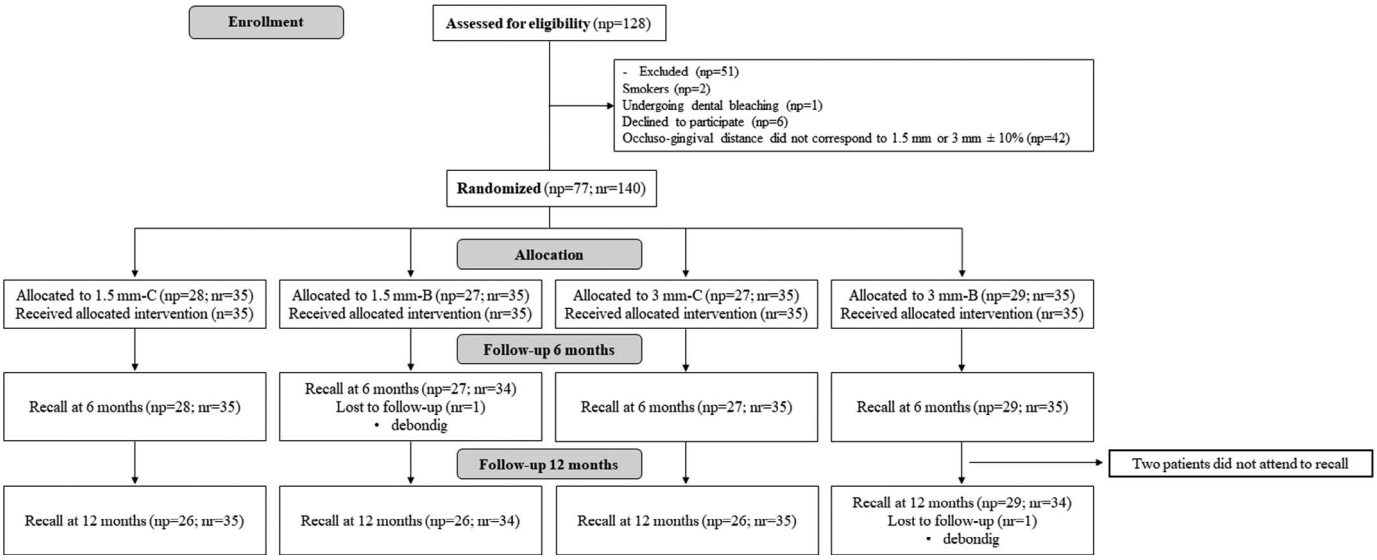

Figure 1. *Flow diagram with details about recruitment and allocation; 1.5 mm-C, NCCLs with OGD 1.5 mm restored with Filtek Z350 XT; 1.5 mm-B, NCCLs with OGD 1.5 mm restored with Filtek Bulk Fill Posterior; 3 mm-C, NCCLs with OGD 3 mm restored with Filtek Z350 XT; .3 mm-B, NCCLs with OGD 3 mm restored with Filtek Bulk Fill Posterior. NCCL, noncarious lesions; np, number of participants; nr, number of restorations; OGD, occlusogingival distance.*

because the participants did not attend during the follow-up period; of these, 2 restorations were in the 1.5 mm-C group, 1 restoration in the 1.5 mm-B group, and 1 restoration in the 3 mm-C group.

Overall Analysis

Two restorations were lost, one after 6 months and another after 12 months. According to the USPHS modified criteria, the 12-month retention rates (95% confidence interval) were 100% (90% to 100%) for 1.5 mm-C; 97% (85% to 99%) for 1.5 mm-B; 100% (90% to 100%) for 3 mm-C; and 97% (85% to 99%) for 3 mm-B, with no statistical difference among the groups (p=0.570) (Table 4). Also, no significant differences were found in marginal staining, marginal adaptation, recurrence of caries, anatomic form, postoperative sensitivity and surface texture among experimental groups.

The only significant difference among the follow-ups for the same group was found in marginal staining. In this parameter, the score Bravo increased significantly with time for 1.5 mm-C (p=0.049) and 1.5 mm-B groups (p=0.049). However, the only significant difference was found between the baseline and the 12-month follow-up. For the 3 mm-B group, three restorations had Bravo scores after 12 months of follow-up, showing a significant increase compared with the other recall times (p=0.049). In the 3 mm-C group, although more restorations rated as Bravo at the 12-month follow-up compared with those at the 6-month follow-up, there was no significant difference between them, only for baseline ($p<0.01$).

DISCUSSION

In a clinical trial, different parameters are used to determine the clinical performance of a restorative material or technique. In NCCLs, retention, marginal staining and marginal adaptation are the criteria that determine the longevity of the restorations.[2,18,19] These parameters are directly related to the stress produced at the tooth/restoration interface,[20,21] which is influenced by the nonretentive shape and geometry of the cavity. Also, the enamel bevel,[3,5] adhesion strategies, methods of photoactivation, viscosity of adhesive materials, application of low-viscosity resins and placement technique[3,43] contribute to the development of stress at the interface. The authors are unaware of a previous study that assessed the clinical performance of a regular bulk-fill resin composite in NCCLs besides the influence of the OGD of NCCLs. According to Aw et al.,[44] in a study of the prevalence of NCCLs, the OGD of evaluated NCCLs ranged from 1 to 4 mm in most of the lesions (91%). Then, two different sizes (small [1.5 mm±10%] or big [3 mm±10%] lesions) within that previously reported range were determined. The results of the present study showed that after 12 months, both resin composites exhibited acceptable clinical performances, despite marginal staining and regardless of the OGD.

Table 3: *Details Regarding Research Participants (Sex and Age), Characteristics, and Distribution of NCCLs*

Characteristics	Number of Participants, %			
Sex distribution				
Male	34 (44.1)			
Female	43 (55.8)			
Age distribution (years)				
21-40	12 (15.6)			
41-60	52 (67.5)			
61-80	13 (16.9)			
Characteristics of NCCLs	**Number of Lesions, %**			
	1.5 mm-C	**1.5 mm-B**	**3 mm-C**	**3 mm-B**
Tooth distribution				
Canines	7	7	9	7
Premolars	28	28	26	28
Arch distribution				
Maxillary	15	25	16	14
Mandibular	20	10	19	21
Shape				
Saucer-shaped	15	15	23	24
Wedge-shaped	16	17	7	3
Mixed-shape	4	3	5	8
Wear facets				
Yes	18	21	21	20
No	17	14	14	15
Preoperative sensitivity (air dry)				
Yes	18	22	20	15
No	17	13	15	20

Abbreviations: 1.5 mm-C, NCCLs with OGD 1.5 mm restored with Filtek Z350 XT; 1.5 mm-B, NCCLs with OGD 1.5 mm restored with Filtek Bulk Fill; 3 mm-C, NCCLs with OGD 3 mm restored with Filtek Z350 XT; 3 mm-B, NCCLs with OGD 3 mm restored with Filtek Bulk Fill; NCCL, noncarious cervical lesion.

A two-step self-etch adhesive was evaluated in this clinical trial because of the lower sensitivity of the technique and better clinical performance, with a failure rate of 2.2% after 24 months.[10] Clearfil SE Bond contains functional monomer 10-methacryloxydecyl phosphate (MDP), which bonds chemically with the hydroxyapatite of the tooth through its phosphate groups,[45] providing a more effective bond and more stability in water than other monomers, even without additional mechanical retention.[46] The good clinical performance of the restorative materials at 12 months may be attributed to these characteristics.

In this study, the retention rates were 100%, 97%, 100%, and 97% at 12 months for the 1.5 mm-C, 1.5 mm-B, 3 mm-C, and 3 mm-B groups, respectively, without statistical difference among the groups during this period (p=0.57). Also, no significant difference was detected for the other parameters among any groups (Table 4).

These results demonstrated that regular bulk-fill resin composite is a viable option for the restoration of NCCLs, with excellent performance after 12 months of clinical service. Similar findings have also been reported in previous clinical trials.[15] Recently, Canali et al.[15] assessed the 1-year clinical performance of a bulk-fill flowable and a regular nanofilled resin composite in NCCLs. The authors concluded that both resin composites showed acceptable clinical performances for the restoration of NCCLs. In a study with mathematical models, NCCLs restored with regular bulk-fill resin composite presented more favorable biomechanical behavior compared with another material and filling technique.[43] According to the manufacturer, Filtek Bulk Fill Posterior contains AUDMA (aromatic urethane dimethacrylate) and AFM (addition-fragmentation monomer), which are responsible for decreasing the number of reactive groups in the resin and for reducing the polymerization stress, respectively. Perhaps, this restorative protocol may decrease the restoration failure rates in NCCLs, improving clinical longevity. However, longer follow-ups are needed to understand the clinical performance of this material in NCCLs.

Restorations placed in lesions with 1.5 mm OGD showed no significant differences in retention rates compared with restorations placed in lesions with 3 mm OGD. A similar finding was reported in another clinical trial.[6] These authors suggested that only some characteristics predispose restorations to retentive failure, such as obtusely angled lesions with group function in posterior immobile teeth (especially molars) in male patients.

Irrespective of the resin composite and OGD, the only signs of degradation in this study presented as marginal staining after 12 months of clinical evaluation. In clinical trials, marginal staining has been associated with the presence of a marginal defect.[2,47] This defect may be the result of the marginal deterioration at the enamel side, since demineralization, in depth and extent, is restricted for mild self-etch adhesives; a chemical interaction is found between monomers and residual hydroxyapatite.[48] Indeed, some authors have reported that superficial discoloration is more frequent in the self-etch approach.[36,37] Furthermore, oral microflora and dietary habits of patients can be associated with marginal staining.[49] In the present study, despite a rather rapid development, the staining was considered clinically acceptable (Bravo), ac-

Table 4: *Number (Percentage) of Evaluated Restorations for Each Recall Time According to Experimental Group, Classified According to Modified US Public Health Service Criteria Compared by Kruskal-Wallis Test at* $p<0.05$

Evaluated Criteria	Score[a]	Baseline					6 months					12 months				
		1.5 mm-C	1.5 mm-B	3 mm-C	3 mm-B	*p*	1.5 mm-C	1.5 mm-B	3 mm-C	3 mm-B	*p*	1.5 mm-C	1.5 mm-B	3 mm-C	3 mm-B	*p*
Retention	Alfa	35 (100)	35 (100)	35 (100)	35 (100)	>0.99	35 (100)	34 (97.1)	35 (100)	35 (100)	0.39	33 (100)	33 (97)	34 (100)	34 (97.1)	0.57
	Charlie	–	–	–	–		–	1 (2.9)	–	–		–	1 (3)	–	1 (2.9)	
Marginal staining	Alfa	35 (100)	35 (100)	35 (100)	35 (100)	>0.99	33 (94.3)	32 (94.1)	33 (94.3)	35 (100)	0.55	29 (87.9)	29 (87.9)	25 (73.5)	31 (91.2)	0.08
	Bravo	–	–	–	–		2 (5.7)	2 (5.9)	2 (5.7)	–		4 (12.1)	4 (12.1)	9 (26.5)	3 (8.8)	
Marginal adaptation	Alfa	35 (100)	35 (100)	35 (100)	35 (100)	>0.99	34 (97.1)	34 (100)	35 (100)	35 (100)	0.39	32 (97)	33 (100)	34 (100)	33 (97)	0.57
	Bravo	–	–	–	–		1 (2.9)	–	–	–		1 (3)	–	–	1 (3)	
Recurrence of caries	Alfa	35 (100)	35 (100)	35 (100)	35 (100)	>0.99	35 (100)	34 (100)	35 (100)	35 (100)	>0.99	33 (100)	33 (100)	34 (100)	34 (100)	>0.99
Anatomic form	Alfa	35 (100)	35 (100)	35 (100)	35 (100)	>0.99	35 (100)	34 (100)	35 (100)	35 (100)	>0.99	33 (100)	33 (100)	34 (100)	34 (100)	>0.99
Postoperative sensitivity	Alfa	32 (91.4)	32 (91.4)	32 (91.4)	35 (100)	0.36	32 (91.4)	33 (97)	32 (91.4)	35 (100)	0.26	33 (100)	31 (93.9)	32 (94.1)	32 (94.1)	0.41
	Bravo	3 (8.6)	3 (8.6)	3 (8.6)	–		3 (8.6)	1 (3)	3 (8.6)	–		–	2 (6.1)	2 (5.9)	2 (5.9)	
Surface texture	Alfa	35 (100)	35 (100)	35 (100)	35 (100)	>0.99	34 (97.1)	32 (94.1)	35 (100)	34 (97.1)	0.55	32 (97)	32 (97)	32 (94.1)	33 (97)	0.87
	Bravo	–	–	–	–		1 (2.9)	2 (5.9)	–	1 (2.9)		1 (3)	1 (3)	2 (5.9)	1 (3)	

Abbreviations: 1.5 mm-C, NCCLs with OGD 1.5 mm restored with Filtek Z350 XT; 1.5 mm-B, NCCLs with OGD 1.5 mm restored with Filtek Bulk Fill; 3 mm-C, NCCLs with OGD 3 mm restored with Filtek Z350 XT; 3 mm-B, NCCLs with OGD 3 mm restored with Filtek Bulk Fill; NCCL, noncarious cervical lesion; OGD, occlusogingival distance.

[a] *Scores Alfa and Bravo show clinically acceptable restoration, while Charlie score indicates failure.*

cording to the modified USPHS criteria. In these situations, the restoration margins can be refinished and repolished without damage to improve esthetics. It should be emphasized that the discolorations were slight and superficial and required no intervention.

The stability and longevity of resin restorations in NCCLs are associated with etiologic factors and risk factors.[7,39,50] Also, technique and adhesive material are important to the clinical performance.[14,15,36–38] In the present study, OGD was not associated with restoration failure. The regular bulk-fill resin composite performed similarly and successfully compared with the nanofilled resin composite after a 1-year evaluation period. However, more extensive evaluations are necessary. These have been implemented to improve the understanding of the long-term performance of this material, mainly evaluating whether the OGD is important to the outcome.

CONCLUSION

This preliminary report on a 12-month evaluation had the following conclusions:

1. Both resin composites tested presented acceptable clinical results at the 12-month evaluation;
2. Filtek Bulk Fill Posterior showed similar retention rates to Filtek Z350 XT regardless of OGD;
3. The OGD did not affect the clinical performance of resin restorations.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Institute of Science and Technology of São José dos Campos UNESP Review Board. The approval code for this study is 1.734.858.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Stojanac IL, Premovic MT, Ramic BD, Drobac MR, Stojsin IM, & Petrovic LM (2013) Noncarious cervical lesions restored with three different tooth-colored materials: Two-year results *Operative Dentistry* **38(1)** 12-20.
2. Kubo S, Yokota H, Yokota H, & Hayashi Y (2010) Three-year clinical evaluation of a flowable and a hybrid resin

composite in non-carious cervical lesions *Journal of Dentistry* **38(3)** 191-200.

3. Kubo S, Yokota H, Yokota H, & Hayashi Y (2013) Challenges to the clinical placement and evaluation of adhesively-bonded, cervical composite restorations *Dental Materials* **29(1)** 10-27.
4. Sugita I, Nakashima S, Ikeda A, Burrow MF, Nikaido T, Kubo S, Tagami J, & Sumi Y (2017) A pilot study to assess the morphology and progression of non-carious cervical lesions *Journal of Dentistry* **57** 51-56.
5. Borges AL, Borges AB, Xavier TA, Bottino MC, & Platt JA (2014) Impact of quantity of resin, C-factor, and geometry on resin composite polymerization shrinkage stress in class V restorations *Operative Dentistry* **39(2)** 144-151.
6. Aw TC, Lepe X, Johnson GH, & Mancl LA (2005) A three-year clinical evaluation of two-bottle versus one-bottle dentin adhesives *Journal of the American Dental Association* **136(3)** 311-322.
7. Tay FR & Pashley DH (2004) Resin bonding to cervical sclerotic dentin: A review *Journal of Dentistry* **32(3)** 173-196.
8. Perez CR, Gonzalez MR, Prado NAS, de Miranda MSF, Macêdo MA, & Fernandes BMP (2012) Restoration of noncarious cervical lesions: when, why, and how *International Journal of Dentistry* **2012**(687058) 1-8.
9. Luque-Martinez IV, Mena-Serrano A, Muñoz MA, Hass V, Reis A, & Loguercio AD (2013) Effect of bur roughness on bond to sclerotic dentin with self-etch adhesive systems *Operative Dentistry* **38(1)** 39-47.
10. Peumans M, De Munck J, Mine A, & Van Meerbeek B (2014) Clinical effectiveness of contemporary adhesives for the restoration of non-carious cervical lesions. A systematic review *Dental Materials* **30(10)** 1089-1103.
11. Santos MJ, Ari N, Steele S, Costella J, & Banting D (2014) Retention of tooth-colored restorations in non-carious cervical lesions—A systematic review *Clinical Oral Investigations* **18(5)** 1369-1381.
12. van Dijken JW & Pallesen U (2008) Long-term dentin retention of etch-and-rinse and self-etch adhesives and a resin-modified glass ionomer cement in non-carious cervical lesions *Dental Materials* **24(7)** 915-922.
13. Fagundes TC, Barata TJ, Bresciani E, Santiago S, Franco EB, Lauris JR, & Navarro MF (2014) Seven-year clinical performance of resin composite versus resin-modified glass ionomer restorations in noncarious cervical lesions *Operative Dentistry* **39(6)** 578-587.
14. Celik EU, Tunac AT, & Yilmaz F (2019) Three-year clinical evaluation of high-viscosity glass ionomer restorations in non-carious cervical lesions: A randomised controlled split-mouth clinical trial *Clinical Oral Investigations* **23(3)** 1473-1480, http://dx.doi.org/10.1007/s00784-018-2575-y
15. Canali GD, Ignácio SA, Rached RN, & Souza EM (2019) One-year clinical evaluation of bulk-fill flowable vs. regular nanofilled composite in non-carious cervical lesions *Clinical Oral Investigations* **23(2)** 889-897.
16. Ferracane JL (2011) Resin composite—State of the art *Dental Materials* **27(1)** 29-38.
17. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, & Leloup G (2014) Physico-mechanical characteristics of commercially available bulk-fill composites *Journal of Dentistry* **42(8)** 993-1000.
18. Moretto SG, Russo EM, Carvalho RC, De Munck J, Van Landuyt K, Peumans M, Van Meerbeek B, & Cardoso MV (2013) 3-year clinical effectiveness of one-step adhesives in non-carious cervical lesions *Journal of Dentistry* **41(8)** 675-682.
19. Lawson NC, Robles A, Fu CC, Lin CP, Sawlani K, & Burgess JO (2015) Two-year clinical trial of a universal adhesive in total-etch and self-etch mode in non-carious cervical lesions *Journal of Dentistry* **43(10)** 1229-1234.
20. Ichim I, Li Q, Loughran J, Swain MV, & Kieser J (2007) Restoration of non-carious cervical lesions Part I. Modelling of restorative fracture *Dental Materials* **23(12)** 1553-1561.
21. Ichim I, Schmidlin PR, Kieser JA, & Swain MV (2007) Mechanical evaluation of cervical glass-ionomer restorations: 3D finite element study *Journal of Dentistry* **35(1)** 28-35.
22. Braga RR, Boaro LC, Kuroe T, Azevedo CL, & Singer JM (2006) Influence of cavity dimensions and their derivatives (volume and "C" factor) on shrinkage stress development and microleakage of composite restorations *Dental Materials* **22(9)** 818-823.
23. Pfeifer CSC, Braga RR, & Cardoso PEC (2006) Influence of cavity dimensions, insertion technique and adhesive system on microleakage of class V restorations *Journal of the American Dental Association* **137(2)** 197-202.
24. Bucuta S & Ilie N (2014) Light transmittance and micromechanical properties of bulk fill vs. conventional resin based composites *Clinical Oral Investigations* **18(8)** 1991-2000.
25. Kim RJ, Kim YJ, Choi NS, & Lee IB (2015) Polymerization shrinkage, modulus, and shrinkage stress related to tooth-restoration interfacial debonding in bulk-fill composites *Journal of Dentistry* **43(4)** 430-439.
26. Rosatto CM, Bicalho AA, Veríssimo C, Bragança GF, Rodrigues MP, Tantbirojn D, Versluis A, & Soares CJ (2015) Mechanical properties, shrinkage stress, cuspal strain and fracture resistance of molars restored with bulk-fill composites and incremental filling technique *Journal of Dentistry* **43(12)** 1519-1528.
27. Par M, Gamulin O, Marovic D, Klaric E, & Tarle Z (2015) Raman spectroscopic assessment of degree of conversion of bulk-fill resin composites—Changes at 24 hours post cure *Operative Dentistry* **40(3)** e92-e101.
28. Papadogiannis D, Tolidis K, Gerasimou P, Lakes R, & Papadogiannis Y (2015) Viscoelastic properties, creep behavior and degree of conversion of bulk fill composite resins *Dental Materials* **31(12)** 1533-1541.
29. Gamarra VSS, Borges GA, Júnior LHB, & Spohr AM (2018) Marginal adaptation and microleakage of a bulk-fill composite resin photopolymerized with different techniques *Odontology* **106(1)** 56-63.
30. van Dijken JW & Pallesen U (2016) Posterior bulk-filled resin composite restorations: A 5-year randomized controlled clinical study *Journal of Dentistry* **51** 29-35.

31. Bayraktar Y, Ercan E, Hamidi MM, & Çolak H (2017) One-year clinical evaluation of different types of bulk-fill composites *Journal of Investigative and Clinical Dentistry* **8(2)** 1-9.
32. Karaman E, Keskin B, & Inan U (2017) Three-year clinical evaluation of class II posterior composite restorations placed with different techniques and flowable composite linings in endodontically treated teeth *Clinical Oral Investigations* **21(2)** 709-716.
33. van Dijken JWV & Pallesen U (2017) Bulk-filled posterior resin restorations based on stress-decreasing resin technology: A randomized, controlled 6-year evaluation *European Journal of Oral Sciences* **125(4)** 303-309.
34. Yazici AR, Antonson SA, Kutuk ZB, & Ergin E (2017) Thirty-six-month clinical comparison of bulk fill and nanofill composite restorations *Operative Dentistry* **42(5)** 478-485.
35. Heck K, Manhart J, Hickel R, & Diegritz C (2018) Clinical evaluation of the bulk fill composite QuiXfil in molar class I and II cavities: 10-year results of a RCT *Dental Materials* **34(6)** e138-e147.
36. Perdigão J, Kose C, Mena-Serrano AP, De Paula EA, Tay LY, Reis A, & Loguercio AD (2014) A new universal simplified adhesive: 18-month clinical evaluation *Operative Dentistry* **39(2)** 113-127.
37. Peumans M, De Munck J, Van Landuyt K, & Van Meerbeek B (2015) Thirteen-year randomized controlled clinical trial of a two-step self-etch adhesive in non-carious cervical lesions *Dental Materials* **31(3)** 308-314.
38. Häfer M, Jentsch H, Haak R, & Schneider H (2015) A three-year clinical evaluation of a one-step self-etch and a two-step etch-and-rinse adhesive in non-carious cervical lesions *Journal of Dentistry* **43(3)** 350-361.
39. Oginni AO & Adeleke AA (2014) Comparison of pattern of failure of resin composite restorations in non-carious cervical lesions with and without occlusal wear facets *Journal of Dentistry* **42(7)** 824-830.
40. Schulz KF, Altman DG, Moher D, & CONSORT Group (2011) CONSORT 2010 statement: Updated guidelines for reporting parallel group randomised trials *International Journal of Surgery* **9(8)** 672-677.
41. American Dental Association Council on Scientific Affairs (2011) Acceptance program guidelines: dentin & enamel adhesive materials *American Dental Association* 1-12. Available from: www.ada.org
42. Cvar JF & Ryge G (2005) Reprint of criteria for the clinical evaluation of dental restorative materials *Clinical Oral Investigations* **9(4)** 215-232.
43. Correia AMO, Tribst JPM, Matos FS, Platt JA, Caneppele TMF, & Borges ALS (2018) Polymerization shrinkage stresses in different restorative techniques for non-carious cervical lesions *Journal of Dentistry* **76** 68-74.
44. Aw TC, Lepe X, Johnson GH, & Mancl LA (2002) Characteristics of noncarious cervical lesions: A clinical investigation. *Journal of the American Dental Association* **133(6)** 725-733.
45. Yoshihara K, Yoshida Y, Nagaoka N, Fukegawa D, Hayakawa S, Mine A, Nakamura M, Minagi S, Osaka A, Suzuki K, & Van Meerbeek B (2010) Nano-controlled molecular interaction at adhesive interfaces for hard tissue reconstruction *Acta Biomaterialia* **6(9)** 3573-3582.
46. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, Inoue S, Tagawa Y, Suzuki K, De Munck J, & Van Meerbeek B (2004) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **83(6)** 454-458.
47. Van Landuyt KL, Peumans M, Fieuws S, De Munck J, Cardoso MV, Ermis RB, Lambrechts P, & Van Meerbeek B (2008) A randomized controlled clinical trial of a HEMA-free all-in-one adhesive in non-carious cervical lesions at 1 year *Journal of Dentistry* **36(10)** 847-855.
48. Van Landuyt KL, Kanumilli P, De Munck J, Peumans M, Lambrechts P, & Van Meerbeek B (2006) Bond strength of a mild self-etch adhesive with and without prior acid-etching *Journal of Dentistry* **34(1)** 77-85.
49. Burrow MF & Tyas MJ (2012) Clinical investigation of G-Bond resin-based adhesive to non-carious cervical lesions over five years *Australian Dental Journal* **57(4)** 458-463.
50. Rocha AC, Da Rosa W, Cocco AR, Da Silva A, Piva E, & Lund RG (2018) Influence of surface treatment on composite adhesion in noncarious cervical lesions: Systematic review and meta-analysis *Operative Dentistry* **43(5)** 508-519.

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Fatigue Failure Load of a Bonded Simplified Monolithic Feldspathic Ceramic: Influence of Hydrofluoric Acid Etching and Thermocycling

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Clinical Relevance

Defects introduced by hydrofluoric acid etching can propagate when the assembly is subjected to aging and fatigue stimuli, impairing its mechanical performance.

SUMMARY

Objective: To evaluate the effect of hydrofluoric acid (HF) etching and thermocycling (Tc) on fatigue failure load of feldspathic ceramic restorations cemented with two resin cements.

Methods: Disc-shaped feldspathic ceramic (Vitablocs Mark II; Ø=10 mm, 1.0-mm thick) and G10 epoxy resin (Ø=10 mm, 2.5-mm thick) specimens were made and randomly allocated considering three factors: ceramic etching (ie, with vs without 10% HF plus silane application), resin cement (ie, self-adhesive [RelyX U200; U200] or conventional [Multilink Automix; MA]), and Tc (ie, with vs without 5-55°C/12,000 cycles). Adhesive cementation followed each manufacturer's instructions. The fatigue test (n=20) was based on the staircase approach (250,000 cycles; 20 Hz). Contact angle, surface topography, and fractography analysis were also executed. Specific statistical tests were employed for each outcome (α=0.05).

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Results: The interaction of HF and Tc factors decreased the fatigue resistance for both cements (U200 542.63>U200/HF-Tc 495.00; MA 544.47>MA/HF-Tc 506.84). Comparing the cements associated with HF or Tc, there was statistical superiority for MA (U200-Tc 537.37<MA-Tc 561.32; U200/HF 535.79<MA/HF 557.11), and no statistical difference was detected when only cement type or its association with HF-Tc was compared (U200 542.63=MA 544.47; U200/HF-Tc 495.00=MA/HF-Tc 506.84). The fracture always originated from defects at the ceramic-intaglio surface as radial cracks.

Conclusion: HF etching plus silane agent increased the ceramic surface free energy and its wettability, but it did not provide better results in terms of fatigue resistance compared with silane agent application only. The association of HF etching and aging significantly reduced the fatigue resistance of the material, regardless of the resin cement used.

INTRODUCTION

Hydrofluoric acid (HF) etching followed by silanization is the most widely accepted surface treatment for glass ceramics.[1] HF etching selectively dissolves the glassy matrix of the glass-ceramic surface, creating retentive micropores, pits, cracks, and grooves on the conditioned surface, favoring micromechanical retention.[2,3] A silane coupling agent is applied on the etched surface to promote a chemical bond between the inorganic phase of the ceramic (ie, silica) with the organic phase of the resin cement (ie, polymer).[4–6]

However, this has become increasingly debatable, as some studies have also shown no need for HF etching for enhancing adhesion between resin cements and glass ceramics,[7–9] and it has also been demonstrated that HF etching may even lead to a ceramic weakening effect.[10,11] This deleterious impact is explained by the fact that HF etching also creates and increases the flaw population at the ceramic bonding interface. By that, it might increase the ceramic susceptibillity to slow crack growth of critical defects under the constant masticatory stresses, since the ceramic fracture strength is inversely proportional to the largest or critical flaw present in the loaded restoration, as described by Griffith's law.[12] From the clinical standpoint, the above-mentioned weakening effect is particularly relevant when considering the evidence showing that the glass-ceramic fails from flaws on the ceramic intaglio surface.[13,14]

Thus, a relevant factor to be considered as a predictor of the restoration performance seems to be the capacity of the cement to completely fill in the introduced defects,[15] which has been scarcely discussed in the literature. Resin cements are indicated for adhesive luting of feldspathic ceramic restorations. They provide better adhesion (higher bond strength) to both ceramic and restorative foundations/substrates as well as superior mechanical and optical properties (greater color selection possibilities and higher color stability), high resistance to hydrolysis, and great inherent tensile strength.[16] The advent of self-adhesive resin cements in the attempt to provide simplification and easy handling techniques appeared as a promising alternative approach, and these cements have shown acceptable esthetic properties and bond strength results that are at least comparable with the conventional resin cements.[1]

In addition to understanding the population of defects and the fill-up potential of different cements on feldspathic ceramic restorations, another important condition involved in such a scenario is the subsequent degradation of the bonding interface. According to Lu and others,[17] aging the bonding interface in water promotes a reduced elastic modulus of the cements as well as an apparent reduction in the bond strength. This degradation of cement properties may be sufficient to redistribute stresses in the restorative set, thus reducing the ability of restorations to tolerate masticatory loads over time.

Few studies have evaluated the mechanical performance of feldspathic ceramics cemented to tooth substrates or analog materials under fatigue stimuli.[18,19] According to Strasser and others,[20] ceramic pretreatments (eg, HF etching and air abrasion) are fundamental to provide a better and more stable adhesion (ie, reduced susceptibility to interface degradation) and consequently better performance and long-term predictability for such restorations, being that its absence may increase the risk of premature failure.

Thus, considering the above assumptions, the lack of consensus on this issue, and the clinical appeal, this laboratory study investigated the following: does HF etching change the flaw population on the glass-ceramic intaglio surface and consequently affect the fatigue behavior of adhesively bonded simplified monolithic glass-ceramic restorations when subjected to aging?

The study aimed to evaluate the influence of HF etching and aging on the fatigue failure load of a computer-aided design/computer-aided manufacturing (CAD/CAM) monolithic feldspathic ceramic adhesively bonded to a dentin analog material using two resin cements (a self-adhesive and a conventional). The assumed hypotheses were the following: 1) HF etching will not promote different fatigue behavior compared with nonetching, 2) thermocycling (Tc) will reduce the fatigue failure load results, and 3) no significant difference will be found between the cements.

METHODS AND MATERIALS

Study Design

Restorative sets were fabricated through a simplified approach[3] using feldspathic ceramic discs (Vita Mark II) cemented to glass fiber reinforced epoxy resin discs (G10 substrate, dentin analog) with conventional (MA; Multilink Automix, Ivoclar Vivadent, Schaan, Liechtenstein) and self-adhesive (U200; RelyX U200, 3M ESPE, Seefeld, Germany) resin cements. The diameter of the specimens was determined as 10.0 mm because it resembles the average diameter of the first molar occlusal surface,[21] and the final thickness of the cemented set was 3.5 mm because it is equivalent to the average thickness between the occlusal surface and the pulp chamber roof of molar teeth.[22,23] The specimens were prepared and randomly (www.random.org) distributed considering the three factors under study: ceramic etching (ie, with vs without 10% HF etching plus silane application), resin cement (ie, self-adhesive [U200] or conventional [MA]), and Tc (ie, with vs without; 5-55°C/12,000 cycles; Table 1).

Production of Ceramic Discs

Prefabricated feldspathic ceramic blocks (Vitablocs Mark II for CEREC/inLab, Lot 45950, Vita Zahnfabrik, Bad Säckingen, Germany) were rounded into cylinders with a diamond drill (internal Ø=10 mm; Diamant Boart, Brussels, Belgium) coupled to an electric drill (SBE 1010 Plus, Metabo, Nürtingen, Germany) and then sectioned in a precision diamond saw (Isomet 1000, 15LC Diamond Disc, Buehler, Lake Bluff, IL, USA), resulting in disc-shaped specimens of 1.05 mm thickness. The procedures were executed with constant and abundant water cooling. The occlusal surfaces of the discs were sequentially polished (#400-, #600-, #800-, #1200-, and #2000-grit silicon carbide abrasives; Buehler) to obtain a smooth top surface, removing the defects introduced by cutting and obtaining a final thickness of 1.0 mm (±0.01 mm). The opposite surface (ie, cementation/intaglio surface) was kept as cut to resemble milled CAD/CAM surfaces. After, the discs were cleaned with isopropyl alcohol (78%; 10 minutes) in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras, Ribeirão Preto, Brazil) to remove any processing sediment (cutting and polishing procedures).

Table 1: *Study Experimental Design*

Resin Cement (Manufacturer)	Baseline		Aged[a]	
	No HF[c]	HF[b,c]	No HF[c]	HF[b,c]
RelyX U200 (3M ESPE)	U200	U200/HF	U200-Tc	U200/HF-Tc
Multilink Automix (Ivoclar Vivadent)	MA	MA/HF	MA-Tc	MA/HF-Tc

[a] *Thermocycling (Tc): 12,000 cycles between 5 and 55°C.*
[b] *Ten percent hydrofluoric acid etching for one minute.*
[c] *Silane coupling agent application: RelyX Ceramic Primer for the RelyX U200 cement and Monobond Plus for the Multilink Automix cement.*

Production of Dentin Analog Discs

Epoxy resin cylinders (±250-mm length×12.7-mm Ø; NEMA grade G10, Accurate Plastics Inc, New York, NY, USA) were rounded in a polishing machine (200- and 600-grit silicon carbide abrasives; EcoMet/AutoMet Polisher, Buehler) to obtain cylinders of 10 mm in diameter and then cut with a precision diamond saw (Isomet 1000, Buehler), as previously described in the section "Production of Ceramic Discs," with the exception that the thickness of this material was set to 2.7 mm. Both sides of the discs were sequentially polished (#400- and #600-grit SiC abrasives) to obtain smooth surfaces, removing the defects introduced by cutting until a final thickness of 2.5 mm was achieved.

Cementation Procedure

Prior to cementation procedures, the discs were randomly assigned into eight groups (n=25) according to the study factors (Table 1). Based on the two levels (with vs without) considered in the HF etching factor, half of the ceramic specimens were submitted only to an ultrasonic bath (distilled water, five minutes) and air dried for 30 seconds, remaining untouched until the cementation procedure, while the other half was etched with 10% HF (Condac 10 Porcelana, FGM, Joinville, Brazil) for one minute, rinsed (one minute), submitted to the ultrasonic bath protocol to remove any precipitates generated from acid etching, and then air dried for 30 seconds prior to the cementation procedure. The epoxy resin specimens were all etched with 10% HF (Condac 10

Porcelana, FGM) for one minute, rinsed (one minute), submitted to an ultrasonic bath (distilled water, five minutes) to remove the debris formed during acid etching, and air dried for 30 seconds prior to the cementation procedure.

The other cementation procedures were performed according to the manufacturer's instructions of the respective cement:

- Self-adhesive resin cement (U200): The silane-based primer (RelyX Ceramic Primer, 3M ESPE) was scrubbed in both substrates (ceramic and epoxy resin) for five seconds and air-dried until the solvent evaporation (10 seconds).
- Conventional resin cement (MA): Ceramic conditioning: The silane-based primer (Monobond Plus, Ivoclar Vivadent) was applied on the ceramic bonding surface, scrubbed for 15 seconds, then left to react for 45 seconds, and air dried. Epoxy resin conditioning: After the aforementioned HF etching, Multilink Primers A and B (Ivoclar Vivadent) were mixed in a 1:1 ratio, scrubbed on the epoxy resin surface for 30 seconds, and air dried for about five seconds to obtain a thin film.

After the primer applications, the respective cement was mixed (1:1 ratio) and applied on the epoxy resin, and the ceramic disc was seated under a load of 250 g. The cement excess was removed with a Microbrush, and light-curing was performed (1200 mW/cm^2; Radii-cal, SDI Limited, Bayswater, Australia) for 20 seconds at each region (occlusal and four axial surfaces: 0, 90, 180, and 270°). Prior to the fatigue test, the specimens were stored in distilled water (37°C) for four days. The specimens that were thermocycled were stored (distilled water, 37°C) for one day prior to the Tc and for an additional four days after Tc and prior to the fatigue test.

Thermocycling

The Tc subgroups (Table 1) were subjected to aging by Tc to stimulate the degradation of the bond interface. The specimens were subjected to 12,000 thermal cycles in water at temperatures of 5°C and 55°C, with a dwell time of 30 seconds and a transfer time of four seconds (Ethik Technology, Vargem Grande Paulista, São Paulo, Brazil).

Fatigue Failure Load Test: Staircase Method

First, to define the fatigue test parameters for each condition, a load-to-fracture test was executed in a universal testing machine (n=5; EMIC DL 2000, São José dos Pinhais, SP, Brazil) with a crosshead speed of 1 mm/min and incremental load until the auditory perception of cracking (ie, presence of radial cracks confirmed by light trans-illumination and visual inspection) by a single trained blinded operator (L.F.G.; ie, the researcher did not know the respective group at the moment of testing and inspection).

The fatigue test was run in an electric machine (Instron ElectroPuls E3000, Instron Corp, Norwood, MA, USA) following the staircase method described by Collins.[24] To better distribute the stress during testing and to avoid contact damage (Hertzian's cone cracks; fracture by surface contact damage), an adhesive tape (110 μm) was placed on the feldspathic top surface, and a nonrigid sheet (cellophane; 2.5 μm) was placed between the piston and the specimen.[19,25,26] The specimens were placed on a flat steel base and submerged in distilled water, and a stainless-steel hemisphere of 40 mm in diameter was used to apply the load on the center of the specimens' top surface.[14,27,28]

The fatigue test (n=20; staircase method) was run under a frequency of 20 Hz during 250,000 load pulses in each step, with a load amplitude ranging from a minimum of 10 N to the maximum load to failure for each specimen. The first specimen from each group was tested with an initial load close to the estimated fatigue failure load (~60% of the mean of load-to-fracture test) until the fracture or survival was observed at the number of predetermined cycles (250,000). Then, the next specimen was tested with a step size (~5% of initial load) higher (when the previous specimen survived) or lower (when the previous specimen failed) than the initial loading level. This procedure was repeated until at least 15 samples per group were tested after the start of the test, and according to Collins,[24] the test starts only after the first stair inversion (first different outcome obtained), with 15 specimens being required to get reliable results following this methodology.

Contact Angle Measurements

Additional ceramic samples (n=3) were obtained, treated for each evaluated condition (HF etching plus silane coupling agent application; HF application only; silane application only; and baseline, no HF and no silane application), and subjected to contact angle analysis through the sessile drop technique using a goniometer (Drop Shape Analysis, model DSA 30S, Krüss, Hamburg, Germany) connected to a software program (DSA3, V1.0.3-08, Krüss). A drop (11 μL) of deionized water was deposited on the ceramic-treated surface using a

Table 2: *Results From the Monotonic Load-to-Failure Test (n=5)*[a]

Group	Mean of Load-to-Failure Test (N)	Initial Load for Fatigue Test (N)	Step Size (N)	Mean Load for Fatigue Failure, L_f (SD) (N)[b]
U200	820.4	490	25	542.63 (21.88) Ab
U200-Tc	900.8	540		537.37 (21.88) Ab
U200/HF	863.9	520		535.79 (22.38) Ab
U200/HF-Tc	870.6	520		495.00 (22.05) Bb
MA	841.0	505	25	544.47 (28.03) Ab
MA-Tc	888.5	535		561.32 (41.23) Aa
MA/HF	897.8	540		557.11 (20.50) Aa
MA/HF-Tc	781.1	470		506.84 (42.79) Bb

[a] *The parameters defined to start the fatigue test (n=20; initial load for fatigue test=~60% from the mean of load to fracture; step size=~5% of initial load for fatigue test) based on the staircase method and mean load for fatigue failure (L_f and standard deviation [SD]) obtained through the fatigue test.*
[b] *Different uppercase letters indicate statistical differences depicted by one-way ANOVA and post hoc Bonferroni tests considering each cement individually (α=0.05). Different lowercase letters indicate statistical differences depicted by independent-samples* t*-tests for paired conditions between both cements (U200 vs MA,* p*=0.823; U200-Tc vs MA-Tc,* p*=0.034; U200/HF vs MA/HF,* p*=0.004; U200/HF-Tc vs MA/HF-Tc,* p*=0.293; α=0.05).*

syringe, and five seconds after dropping, the contact angle was measured for 10 seconds (series of 30 images per second).

Topographic Analysis

Additional ceramic samples (n=2) for each evaluated condition (with and without HF etching) were prepared as previously described, sputtered with a gold-palladium alloy under vacuum, and then examined under scanning electron microscopy (SEM; VEGA3 Tescan, Brno-Kohoutovice, Czech Republic) to evaluate their surface topography (500× and 2500× magnification) and defects created by HF etching in the cross-sectional view (500× and 3500× magnification). SEM images were obtained through the use of two detectors: secondary electron and back-scattering electron.

Fractographic Analysis

All failed specimens after the fatigue test were evaluated in a stereomicroscope (Stereo Discovery V20, Carl-Zeiss, Gottingen, Germany) to determine the presence and direction of radial cracks by light transillumination. Next, these specimens were sectioned in two halves, perpendicular to the direction of the cracks, in a high-precision diamond saw (Isomet 1000, Buehler). Then, the sectioned halves were reanalyzed in a stereomicroscope to determine the crack origin and its propagation direction, and the representative cracks were selected and sputtered with a gold-palladium alloy under vacuum for a descriptive analysis of higher resolution in SEM (as described for topographic analysis) with 1000× and 2500× magnification.

Data Analysis

All statistical analyses were performed using the IBM SPSS Statistics Program (v24 for Windows; IBM Corp; α=0.05).

First, a three-way analysis of variance (ANOVA) was used to determine the influence of each factor on the fatigue failure load of the restorative set and to elucidate any presence of interaction between the independent study variables (cement, HF etching, and Tc).

One-way ANOVA and post hoc Bonferroni tests were adopted to compare the effect of different conditions for each respective cement separately. *t*-Tests for independent samples were used between paired conditions (U200 vs MA; U200-Tc vs MA-Tc; U200/HF vs MA/HF; U200/HF-Tc vs MA/HF-Tc) to compare and depict the statistical differences for fatigue failure load between the cements exposed to the same study factor.

RESULTS

Fatigue Failure Load Test: Staircase Method

Based on the three-way ANOVA, there were statistically significant influences of the factors cement (p=0.006; MA>U200), Tc (p=0.000; without Tc>with Tc), and HF etching (p=0.000; without HF>with HF) and for the interaction HF × Tc (p=0.000) on the fatigue load results. No statistically significant influence was detected for other associations.

Analyzing each cement separately, the one-way ANOVA and post hoc Bonferroni analyses showed that both cements behaved similarly to the different

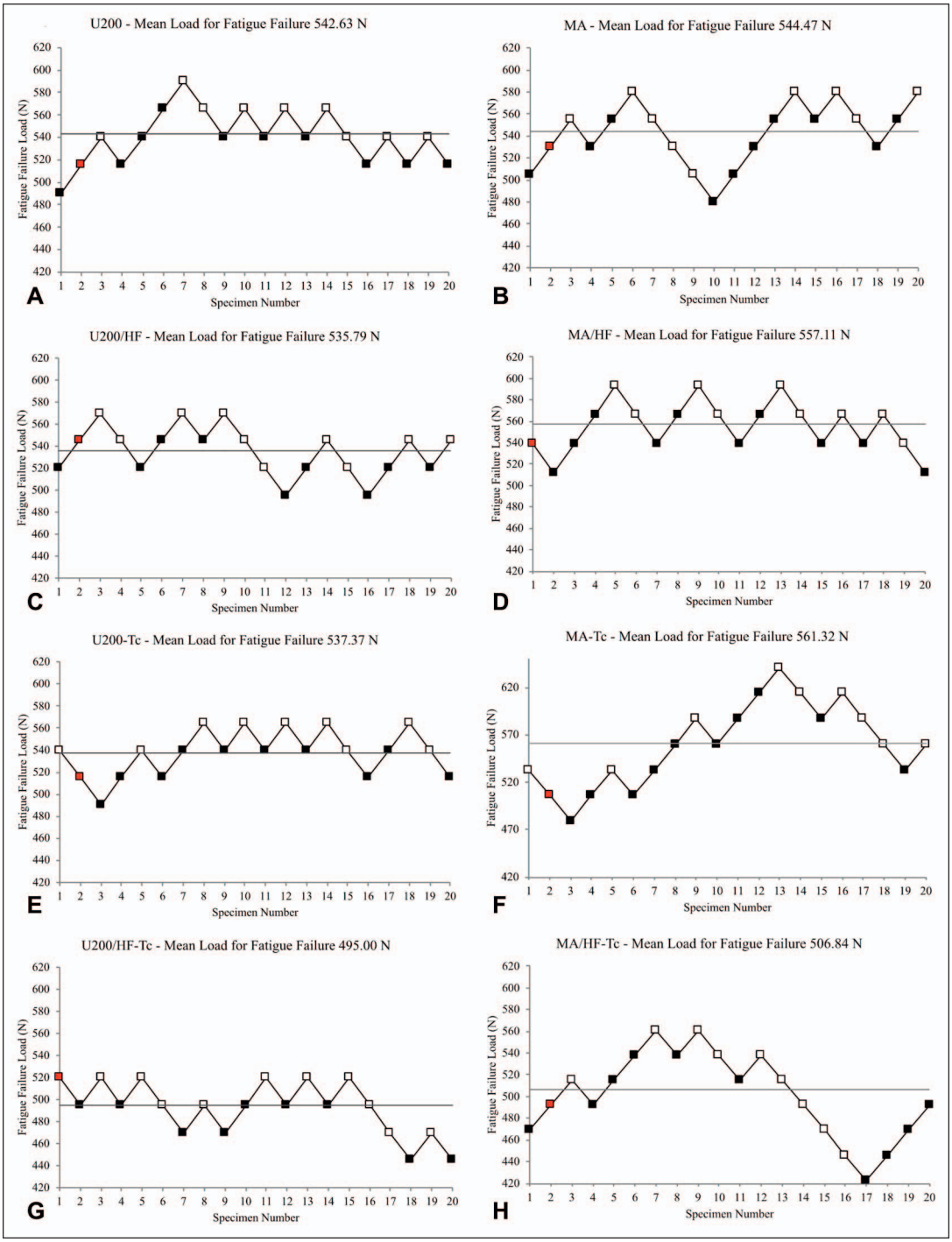

Figure 1. *Survival and failure patterns observed during staircase fatigue testing (250,000 cycles; 20 Hz). Horizontal lines indicate the mean load value, red marks the start of up-and-down characters, and solid marks represent survival and empty marks represent failure.*

conditions, being that the fatigue resistance was statistically reduced only when the HF and Tc factors were applied together (Table 2; Figure 1).

Comparing the same conditions between the cements through *t*-tests for independent samples, the MA cement yielded better results when the specimens were submitted to HF etching or Tc factors. There was no difference between the cements at baseline (without HF and without Tc) or for the association of factors (with HF and Tc; Table 2).

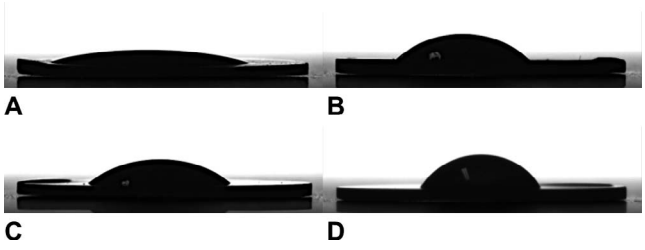

Figure 2. *Representative images and results of contact angle measurements of ceramic surfaces subjected to the following treatments: (A): Hydrofluoric acid etching (HF) plus silane coupling agent application (10.45±2.94). (B): Only HF application (44.15±13.70). (C): Only silane application (33.27±3.55). (D): Baseline (no treatment) (56.24±1.71).*

Contact Angle Measurements

The lowest contact angle was observed after HF etching plus silane application, indicating greater surface wettability. The highest contact angle (lowest wettability) was observed on the untreated surface (baseline group). Intermediate values were observed when HF etching only or silane only was used (Figure 2).

Topographic Analysis

Topographic and cross-sectioned SEM images of baseline (nonetched) and HF-etched (HF 10%; one minute) specimens are shown in Figure 3. Etched surfaces presented an irregular topography characterized by the presence of numerous micro irregularities, pits, grooves, and striations as a result of the glassy phase dissolution, known as a honeycomb-etched pattern (Figure 3A). The baseline images showed a smoother and more homogeneous surface without any relevant irregularity (Figure 3B).

Fractographic Analysis

Stereomicroscope and SEM analysis showed that all failures were radial cracks starting from the ceramic intaglio surface (Figure 4). Cracks due to contact damage between the piston and the ceramic surface were not found.

DISCUSSION

Our findings show that the HF etching condition associated with Tc had a significant deleterious effect on the load for fatigue failure regardless of the cement used, and HF etching prior to silanization did not enhance the fatigue resistance of the simplified ceramic restorations.

HF etching promotes a surface dissolution of the ceramic glass matrix, creating micro retentions that contribute to the mechanical bonding with dental substrate when adhesively bonded.[9,10,29] However, this has become debatable, since fatigue properties of all-ceramic systems can be related to the flaw population (size, number, and distribution) of the material,[12] and some studies have demonstrated that HF etching may lead to a decrease in the feldspathic ceramics strength by introducing defects on the surface that may not be completely filled by the resin cement.[10,30,31] In our study, the first hypothesis was accepted since HF etching did not promote different fatigue failure load results compared with baseline. Regarding the Tc factor, the second hypothesis was partially accepted since it reduced the fatigue failure load only for the HF-etched specimens.

Other authors have obtained similar results, corroborating that HF etching improves the bond strength by creating micromechanical interlocking but may lead to a weakening effect on the ceramic, thereby compromising the clinical performance of the restoration.[10,31] Moreover, recent studies corroborate these findings, showing worse or equal results for bond strength,[7] biaxial flexural strength,[11] and fatigue load[19] when conditioning glass-ceramic with HF prior to bonding, proving that this subject still requires future evaluations and considerations. In addition, HF has potentially hazardous toxicity known from other applications and these risks should also be considered when applied in dentistry.[32]

The topographic images in Figure 3C show the micro retentions, pits, and grooves created by the selective HF etching of the glass-ceramic vitreous matrix, which could lead to a decrease in the ceramic fatigue resistance, especially when the defects are not completely filled by the cement. Defects created by HF etching may also lead to stress concentration, which can result in ceramic premature fracture starting from the adhesive interface,[18,30] as observed in our study (Figure 4). This behavior is particularly important since sharp defects (as created by HF etching; Figure 3C) are more damaging than rounded defects. In this sense, it has to be emphasized that we used a high-concentration HF etchant (10% HF for one minute), which produces many more defects than in lower concentrations (for instance, 1% and 5%)[19] and makes the material more susceptible to the presence of unfilled defects after bonding and, consequently, to crack initiation and propagation under fatigue loads. Clinical[33,34] and laboratory studies[18] on failed glass-ceramic crowns have reported that the great majority of bulk fractures start from flaws on the ceramic-intaglio surface, where high tensile stress is concentrated.

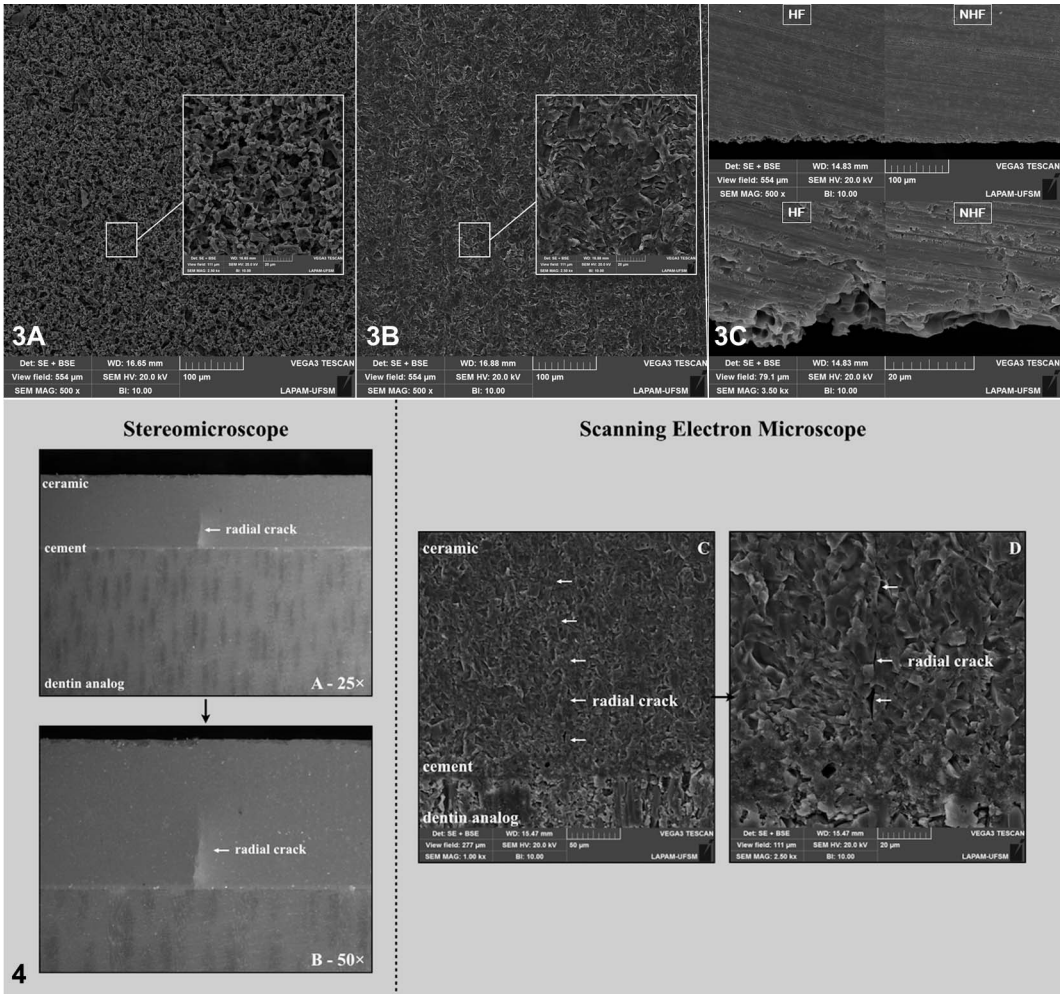

Figure 3. *SEM images of the ceramic surface with hydrofluoric acid (HF) etching (A: 500× and 2500×) and without hydrofluoric acid etching (B: 500× and 2500×). (C): Etching pattern in cross-sectioned specimens: on the left side is the HF-etched ceramic in 500× (top left) and 3500× (bottom left) magnification; on the right side is the nonetched (NHF) ceramic in 500× (top right) and 3500× (bottom right) magnification.*

Figure 4. *Radial crack was the failure pattern for the failed specimens in all groups: The images show the radial crack that indicates the origin and pattern of ceramic failure. (A, B): Stereomicroscope images of cut representative specimens where we can clearly see the radial crack (white arrows) starting at the intaglio-ceramic surface. (C): 1000× and (D): 2500× magnification: SEM images of a cut representative specimen with white arrows pointing to the radial crack.*

Adhesive cementation significantly increases the restorative material's fracture loads.[35] Using finite element analysis and fatigue testing for the lithium disilicate glass-ceramic, de Kok and others[36] proved that proper adhesion can better distribute stress during loading, increasing the material's resistance. In our study, the group treated with HF (which provides a surface with many more defects; Figure 3) had a statistically similar fatigue resistance to the nonetched surface (baseline; Table 2), corroborating the results found by de Kok and others[36] of the protective role of bonding in overcoming the ceramic's internal roughness effect.

Regarding the resin cement filling capacity, cements with low viscosity are more prone to penetrate the ceramic than cements with high viscosity.[37] According to Gamal and others,[38] self-adhesive resin cements have high viscosity and consequently are less capable of spreading onto the ceramic and substrate surfaces, which may compromise its wettability. The application of HF etching plus silane has been recommended to promote chemical bonding between inorganic molecules of the ceramic with organic molecules of these resin cements.[39,40] This sequence is already well established in the literature in terms of bond strength, as these procedures increase the surface energy of the

ceramic and the wettability of the resin cement, improving adhesion.[3,31] This greater wettability can be evidenced by the lower contact angle of the treated surfaces, as shown in Figure 2A. In our work, in terms of fatigue resistance, we can note that this procedure may not summarily be necessary, since the groups that were not submitted to HF etching had the same or even better results than the etched ones. However, it must be considered that this assertion is a finding only with respect to fatigue resistance. From this viewpoint, there is a need to develop novel surface-conditioning methods to address the problem related to the bond durability and defect fill-in potential[6] and to try to eliminate the use of HF to produce a less technique-sensitive and safer (ie, using a less hazardous material) bonding system.

As the conventional cement performed better than the self-adhesive when the HF etching and Tc factors were applied separately, the third hypothesis was partially accepted. According to Gamal and others,[38] that result could be explained by the high viscosity of the self-adhesive resin cement and less spreadability onto the substrate-ceramic surface, reducing its ability to infiltrate into surface irregularities.

When the materials are free to deform, they will expand or contract due to fluctuations in temperatures. By that, the temperature change during Tc is a deleterious factor for the adhesion between ceramic and dental substrate.[41] Because of the different coefficients of linear thermal expansion between the materials in the adhesive interface (ceramic/cement/substrate), they have different degrees of contraction and expansion during Tc, leading to micromechanical fatigue stresses in the adhesive interface, breaking adhesive bonds, and finally reducing the adhesion quality.[41] In the present study, aging (Tc 5-55°C/12,000 times) significantly affected the fatigue resistance for both cements only when the ceramic was previously etched with HF. This corroborates the findings of Venturini and others,[31] who hypothesized that when the micro retentions are not completely filled by the cement, this empty and unfilled space allows faster water absorption at the interface of the restoration with its consequent hydrolysis and degradation and finally a decrease in the material fatigue resistance.

The present study implemented a fatigue test under a wet environment, where constant loads with ranging intensity were applied until the failure of the specimens. This method mimics the oral environment and more closely simulates the masticatory stresses when compared with the static test.[35] However, the applied test setup (axial load) may not fully simulate all the forces to which the material is subjected in the oral environment, especially to lateral loads (sliding motion) that generate compressive, tensile, and shear stresses on the ceramic surface leading to the subsurface crack formation and propagation.[42] Another limitation may be the simplified restorative set (disc-shaped specimens), which does not completely simulate the anatomy of a molar crown.

In our study, the loads at initial radial cracks (overall mean equal to 534.83 N) exceeded the maximum bite forces during mastication (148.73 to 354.01 N)[43] but were far below the maximum bite forces reached in sleep associated bruxism (±800 N) and maximum voluntary bite forces during daytime (±1000 N).[44] Considering the subjects discussed above, we emphasize that our results should be carefully analyzed, and more *in vitro* and clinical findings can corroborate our results.

CONCLUSIONS

- The HF etching associated with a silane coupling agent increased the ceramic surface free energy (lower contact angle and consequently higher wettability), but it did not provide better results in terms of fatigue resistance compared with silane agent application only.
- When the feldspathic ceramics were HF etched, bonded (regardless of the cement used), and then subjected to aging, the fatigue failure load of the restorations was significantly reduced.
- HF etching and Tc factors applied alone did not lead to degradation of the ceramic fatigue resistance, regardless of the resin cement used (conventional [MA] or self-adhesive [U200]), being that the conventional cement performed better in both cases (only HF and only Tc).

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Conflict of Interest

The authors have no financial interest in any of the companies or products mentioned in this article.

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REFERENCES

1. Souza RO, Castilho AA, Fernandes VV, Bottino MA, & Valandro LF (2011) Durability of microtensile bond to nonetched and etched feldspar ceramic: self-adhesive resin cements vs conventional resin *Journal of Adhesive Dentistry* **13(2)** 155-162.
2. Aida M, Hayakawa T, & Mizukawa K (1995) Adhesion of composite to porcelain with various surface conditions *Journal of Prosthetic Dentistry* **73(5)** 464-469.
3. Chen C, Trindade FZ, de Jager N, Kleverlaan CJ, & Feilzer AJ (2014) The fracture resistance of a CAD/CAM resin nano ceramic (RNC) and a CAD ceramic at different thicknesses *Dental Materials* **30(9)** 954-962.
4. Söderholm KJ & Shang SW (1993) Molecular orientation of silane at the surface of colloidal silica *Journal of Dental Research* **72(6)** 1050-1054.
5. Özcan M & Volpato CA (2015) Surface conditioning protocol for the adhesion of resin-based materials to glassy matrix ceramics: how to condition and why? *Journal of Adhesive Dentistry* **17(3)** 292-293.
6. Matinlinna JP, Lung CYK, & Tsoi JKH (2018) Silane adhesion mechanism in dental applications and surface treatments: a review *Dental Materials* **34(1)** 13-28.
7. Peumans M, Valjakova EB, De Munck J, Mishevska CB, & Van Meerbeek B (2016) Bonding effectiveness of luting composites to different CAD/CAM materials *Journal of Adhesive Dentistry* **18(4)** 289-302.
8. Wille S, Lehmann F, & Kern M (2017) Durability of resin bonding to lithium disilicate and zirconia ceramic using a self-etching primer *Journal of Adhesive Dentistry* **19(6)** 491-496.
9. El-Damanhoury HM & Gaintantzopoulou MD (2018) Self-etching ceramic primer versus hydrofluoric acid etching: etching efficacy and bonding performance *Journal of Prosthodontic Research* **62(1)** 75-83.
10. Addison O & Fleming GJ (2004) The influence of cement lute, thermocycling, and surface preparation on the strength of a porcelain laminate veneering material *Dental Materials* **20(3)** 286-292.
11. Xiaoping L, Dongfeng R, & Silikas N (2014) Effect of etching time and resin bond on the flexural strength of IPS e.max Press glass ceramic *Dental Materials* **30(12)** e330-336.
12. Griffith AA (1921) The phenomena of rupture and flow in solids *Philosophical Transactions of the Royal Society of London* **221** 163-198.
13. Costa AK, Kelly RD, Fleming GJ, Borges AL, & Addison O (2015) Laminated ceramics with elastic interfaces: a mechanical advantage? *Journal of Dentistry* **43(3)** 335-341.
14. Monteiro JB, Oliani MG, Guilardi LF, Prochnow C, Rocha Pereira GK, Bottino MA, de Melo RM, & Valandro LF (2018) Fatigue failure load of zirconia-reinforced lithium silicate glass ceramic cemented to a dentin analog: effect of etching time and hydrofluoric acid concentration *Journal of the Mechanical Behavior of Biomedical Materials* **77** 375-382.
15. Thompson JY, Rapp MM, & Parker AJ (1998) Microscopic and energy dispersive x-ray analysis of surface adaptation of dental cements to dental ceramic surfaces *Journal of Prosthetic Dentistry* **79(4)** 378-383.
16. Peutzfeldt A, Sahafi A, & Flury S (2011) Bonding of restorative materials to dentin with various luting agents *Operative Dentistry* **36(3)** 266-273.
17. Lu C, Wang R, Mao S, Arola D, & Zhang D (2013) Reduction of load-bearing capacity of all-ceramic crowns due to cement aging *Journal of the Mechanical Behavior of Biomedical Materials* **17** 56-65.
18. Venturini AB, Prochnow C, May LG, Kleverlaan CJ, & Valandro LF (2018) Fatigue failure load of feldspathic ceramic crowns after hydrofluoric acid etching at different concentrations *Journal of Prosthetic Dentistry* **119(2)** 278-285.
19. Venturini AB, Prochnow C, Pereira GKR, Werner A, Kleverlaan CJ, & Valandro LF (2018b) The effect of hydrofluoric acid concentration on the fatigue failure load of adhesively cemented feldspathic ceramic discs. *Dental Materials* **34(4)** 667-675.
20. Strasser T, Preis V, Behr M, & Rosentritt M. Roughness, surface energy, and superficial damages of CAD/CAM materials after surface treatment *Clinical Oral Investigations* 2018;22(8):2787-2797.
21. Ferrario VF, Sforza C, Tartalia GM, Colombo A, & Serrao G (1999) Size and shape of the human first permanent molar: a Fourier analysis of the occlusal and equatorial outlines *American Journal of Physical Anthropology* **108(3)** 281-294.
22. Sulieman M, Addy M, & Rees JS (2005) Surface and intra-pulpal temperature rises during tooth bleaching: an in vitro study *Brazilian Dental Journal* **199(1)** 37-40.
23. Harris EF & Hicks JD (1998) A radiographic assessment of enamel thickness in human maxillary incisors *Archives of Oral Biology* **43(10)** 825-831.
24. Collins JA (1993) *Failure of Materials in Mechanical Design: Analysis, Prediction, Prevention* **2nd edition** Willey Interscience, John Wiley & Sons, New York NY.
25. Wachtman JB Jr, Capps W, & Mandel J (1972) Biaxial flexure tests of ceramic substrates *Journal of Materials Science* **7** 188-194.
26. Monteiro JB, Riquieri H, Prochnow C, Guilardi LF, Pereira GKR, Borges ALS, de Melo RM, & Valandro LF (2018) Fatigue failure load of two resin-bonded zirconia-reinforced lithium silicate glass-ceramics: effect of ceramic thickness *Dental Materials* **34(6)** 891-900.
27. Kelly JR (1999) Clinically relevant approach to failure testing of all-ceramic restorations. *Journal of Prosthetic Dentistry* **81(6)** 652-661.
28. Kelly JR, Rungruanganunt P, Junter B, & Vailati F (2010) Development of a clinically validated bulk failure test for ceramic crowns *Journal of Prosthetic Dentistry* **104(4)** 228-238.
29. Thordrup M, Isidor F, & Hörsted-Bindslev P (1999) A 3-year study of inlays milled from machinable ceramic blocks representing 2 different inlay systems *Quintessence International* **30(12)** 829-836.

30. Della Bona A, Anusavice KJ, & Hood JA (2002) Effect of ceramic surface treatment on tensile bond strength to resin cement *International Journal of Prosthodontics* **15(3)** 248-253.

31. Venturini AB, Prochnow C, May LG, Bottino MC, & Valandro LF (2015) Influence of hydrofluoric acid concentration on the flexural strength of a feldspathic ceramic *Journal of the Mechanical Behavior of Biomedical Materials* **48** 241-248.

32. Özcan M, Allahbeickaraghi A, & Dündar M (2012) Possible hazardous effects of hydrofluoric acid and recommendations for treatment approach: a review *Clinical Oral Investigations* **16(1)** 15-23.

33. Kelly JR, Giordano R, Pober R, & Cima MJ (1990) Fracture surface analysis of dental ceramics; clinically failed restorations *International Journal of Prosthodontics* **3(5)** 430-440.

34. Thompson JY, Anusavice KJ, Naman A, & Morris HF (1994) Fracture surface characterization of clinically failed all-ceramic crowns *Journal of Dental Research* **73(12)** 1824-1832.

35. Attia A, Abdelaziz KM, Freitag S, & Kern M (2006) Fracture load of composite resin and feldspathic all-ceramic CAD/CAM crowns *Journal of Prosthetic Dentistry* **95(2)** 117-123.

36. de Kok P, Pereira GKR, Fraga S, de Jager N, Venturini AB, & Kleverlaan CJ (2017) The effect of internal roughness and bonding on the fracture resistance and structural reliability of lithium disilicate ceramic *Dental Materials* **33(12)** 1416-1425.

37. Kelly JR, Nishimura I, & Campbell SD (1996) Ceramics in dentistry: historical roots and current perspectives *Journal of Prosthetic Dentistry* **75(1)** 18-32.

38. Gamal R, Gomaa YF, & Abdellatif MA (2017) Microtensile bond strength and scanning electron microscopic evaluation of zirconia bonded to dentin using two self-adhesive resin cements: effect of airborne abrasion and aging *Future Dental Journal* **3(2)** 55-60.

39. Fabianelli A, Pollington S, Papacchini F, Goracci C, Cantoro A, Ferrari M, & van Noort R (2010) The effect of different surface treatments on bond strength between leucite reinforced feldspathic ceramic and composite resin *Journal of Dentistry* **38(1)** 39-43.

40. Pollington S, Fabianelli A, & van Noort R (2010) Microtensile bond strength of a resin cement to a novel fluorcanasite glass-ceramic following different surface treatments *Dental Materials* **26(9)** 864-872.

41. Andreatta Filho OD, Araújo MA, Bottino MA, Nishioka RS, & Menezes MM (2005) Study of thermocycling effect on the bond strength between an aluminous ceramic and a resin cement *Journal of Applied Oral Science* **13(1)** 53-57.

42. Ren L & Zhang Y (2014) Sliding contact fracture of dental ceramics: principles and validation *Acta Biomaterialia* **10(7)** 3243-3253.

43. Takaki P, Vieira M, & Bommarito S (2014) Maximum bite force analysis in different age groups *International Archives of Otorhinolaryngology* **18(3)** 272-276.

44. Nishigawa K, Bando E, & Nakano M (2001) Quantitative study of bite force during sleep associated bruxism *Journal of Oral Rehabilitation* **28(5)** 485-491.

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A Two-year Clinical Comparison of Three Different Restorative Materials in Class II Cavities

H Balkaya • S Arslan

Clinical Relevance

Both bulk-fill and conventional composite resins showed a clinically successful performance in Class II restorations over a two-year period, unlike the high-viscosity glass ionomer.

SUMMARY

Objectives: The aim of this clinical study was to evaluate the clinical performance of Class II restorations of a high-viscosity glass ionomer material, of a bulk-fill composite resin, and of a microhybrid composite resin.

Methods and Materials: One hundred nine Class II restorations were performed in 54 patients using three different restorative materials: Charisma Smart Composite (CSC; a conventional composite resin), Filtek Bulk Fill Posterior Restorative (FBF; a high-viscosity bulk-fill composite), and Equia Forte Fil (EF; a high-viscosity glass ionomer). Single Bond Universal adhesive (3M ESPE, Neuss, Germany) was used for both conventional and bulk-fill composite resin restorations. The restorations were evaluated using modified US Public Health Service criteria in terms of retention, color match, marginal discoloration, anatomic form, contact point, marginal adaptation, secondary caries, postoperative sensitivity, and surface texture. The data were analyzed using the chi-square, Fisher, and McNemar tests.

Results: Eighty-four restorations were evaluated at two-year recalls. There were clinically acceptable changes in composite resin restorations (FBF and CSC). In addition, no statistically significant difference was observed between the clinical performances of these materials in terms of all criteria ($p>0.05$). However, there was a statistically significant difference between the EF group and the FBF and CSC groups in all parameters except for marginal discoloration, secondary caries, and postoperative sensitivity ($p<0.05$).

Conclusions: The tested bulk-fill and conventional composite resins showed acceptable clinical performance in Class II cavities. However, if EF is to be used for Class II restoration, its use should be carefully considered.

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INTRODUCTION

Direct restorations have been widely employed to restore posterior teeth because of their low cost, less

need for the removal of sound tooth substance when compared with indirect restorations, and acceptable clinical performance.[1–3] Recently, tooth-colored direct restorative materials have become very popular with advances in adhesive technology, the development of new dental materials, and increasing esthetic demands. However, there is still uncertainty about which is the ideal material for use in the restoration of posterior teeth in terms of long-term clinical success.

Composite resins have commonly been used for posterior restoration because of their acceptable esthetic qualities and their improved mechanical properties and because they allow more conservative cavity preparation design.[4,5] The main challenge with regard to the use of composite resins is polymerization shrinkage. This might cause negative results such as poor marginal adaptation, marginal discoloration, white line formation around the restoration, tubercule fractures, microleakage, secondary caries, and postoperative sensitivity.[6,7] Furthermore, conventional composite resins commonly need an incremental placement technique to avoid depth-of-cure limitations and to overcome polymerization shrinkage stress.[8,9] This placement technique has some drawbacks, such as voids remaining and contamination risk between layers, difficulty in the placement of layers in small cavities, and increased application time.[8,10] To overcome these problems, a new material class referred to as "bulk-fill composite resins" has been developed. It is claimed that bulk-fill composite resins can be polymerized up to 4-mm thickness in a single step without adversely affecting polymerization shrinkage, the adaptation of the cavities, and the degree of conversion during application. They also exhibit less polymerization shrinkage than conventional composite resins.[11]

Glass ionomer cement (GIC) can also be used as an alternative to composite resins in the conservative restoration of posterior caries lesions. GIC has some advantages such as physicochemical adhesion to tooth tissues, fluoride release, biocompatibility, low shrinkage, low marginal leakage, anticaries properties on the restoration edges, and increased remineralization in adjacent proximal caries.[12,13] However, conventional GIC also has disadvantages such as low fracture and abrasion resistance, inadequate color stability, moisture sensitivity, and poor esthetic properties. Some of these disadvantages weaken the physical properties of the material and restrict its use in areas exposed to intensive chewing forces.[14] In recent years, to reduce the moisture sensitivity of GIC in the early stages of hardening, to increase their abrasion resistance, and to enable them to be used in areas exposed to chewing forces, the materials have been strengthened by changing the powder/liquid ratio, particle size, and distribution; as a result, highly viscous glass ionomer cement (HVGIC) has been presented to the market.[14,15] The manufacturer suggests that these materials should be applied with surface-coating resins.[16] The application of surface-coating resins to the GIC surface enhances the surface brightness of the material; prevents the reduction in translucency of the material over time; fills the gaps caused by the material, finishing processes, and surface irregularities in such a way as to provide a smooth surface; reduces moisture sensitivity in the early stages of hardening; increases the resistance to fracture and abrasion; and improves mechanical properties.[16]

In the literature, several studies have compared the clinical performances of conventional composite and high-viscosity glass ionomer or conventional composite and bulk-fill composite, but to the best of our knowledge, this is the first study to compare the clinical performances of a high-viscosity glass ionomer and a bulk-fill composite resin. For this reason, the aim of this clinical study was to compare the clinical performances of a bulk-fill composite resin, a microhybrid composite resin, and a high-viscosity glass ionomer in Class II cavities using modified US Public Health Service (USPHS) criteria. The null hypothesis of this study was that there would be no difference between the two-year clinical performances of composite resin materials (conventional and bulk-fill) and HVGIC in Class II cavities.

METHODS AND MATERIALS

Before conducting the study, the research protocol was approved by the Faculty of Medicine Ethics Committee, Erciyes University, Kayseri, Turkey (approval 2017/44). In this randomized controlled clinical study, an HVGIC (Equia Forte Fil, GC, Tokyo, Japan), a bulk-fill composite resin (Filtek Bulk Fill Posterior Restorative, 3M ESPE, St. Paul, MN, USA), and a microhybrid composite resin (Charisma Smart Composite, Heraeus Kulzer, Hanau, Germany) were compared. The materials, compositions, and batch numbers are given in Table 1.

Study Design and Patient Selection

Patients attending our clinic for routine dental care were examined clinically and radiographically using bite-wing radiography. In this study, 80 patients

Table 1: *Materials, Compositions, and Batch Numbers*

Material and Manufacturer	Batch	Composition
Charisma Smart Composite, Heraeus Kulzer GmbH, Hanau, Germany	010501A	Bis-GMA, barium aluminum fluoride glass, silicon dioxide
Filtek Bulkfill Posterior Restorative, 3M-ESPE, St. Paul, MN, USA	N651351	Aromatic dimethacrylate (AUDMA), urethane dimethacrylate (UDMA), and 1,12-dodecane dimethacrylate (DDMA) Zirconia/silica and ytterbium trifluoride filler
Equia Forte Fil, GC, Tokyo, Japan	150213B	Powder: 95% strontium fluoro alumino-silicate glass, 5% polyacrylic acid Liquid: 40% aqueous polyacrylic acid Equia Forte coat: 40%-50% methyl methacrylate, 10%-15% colloidal silica, 0.09% camphorquinone, 30%-40% urethane methacrylate, 1%-5% phosphoric ester monomer
Single Bond Universal, 3M ESPE, Neuss, Germany	620318	10-MDP phosphate monomer, Vitrebond, copolymer, HEMA, Bis-GMA, dimethacrylate resin, silane, ethanol, water

were assessed for eligibility for participation, and 26 patients were excluded, either because they did not meet all the inclusion criteria or because they did not agree to attend follow-up visits (Figure 1). A total of 54 patients satisfying the inclusion criteria was selected. The inclusion criteria for the selection of patients for the study were as follows: 1) the patient had no systemic disease, 2) the patient was older than 18 years, 3) the patient had good periodontal status, 4) the teeth to be restored were vital, 5) the teeth to be restored had proximal contacts on both mesial and distal surfaces and were in occlusion with the antagonist teeth, and 6) the teeth had Class II caries lesions in the external and middle third of dentin thickness as determined radiographically. The exclusion criteria were as follows: 1) the existence of xerostomia and bruxism; 2) absence of adjacent and antagonist teeth; 3) extremely poor oral hygiene, severe or chronic periodontitis; 4) pregnant or lactating women; 5) teeth with any restoration, endodontic treatment, or periodontal and periapical pathology; and 6) patients who were undergoing orthodontic treatment. The volunteers participating in the study were informed about the research protocol and possible complications. Finally, an informed consent form was read and signed by the patients.

Restorative Procedures

In this study, 109 teeth in 54 patients (31 female, 23 male) were randomly restored by an experienced operator using three different restorative materials. The randomization of the restorative materials was done using a table of random numbers. The average age of the patients was 22 years (range: 20-32 years).

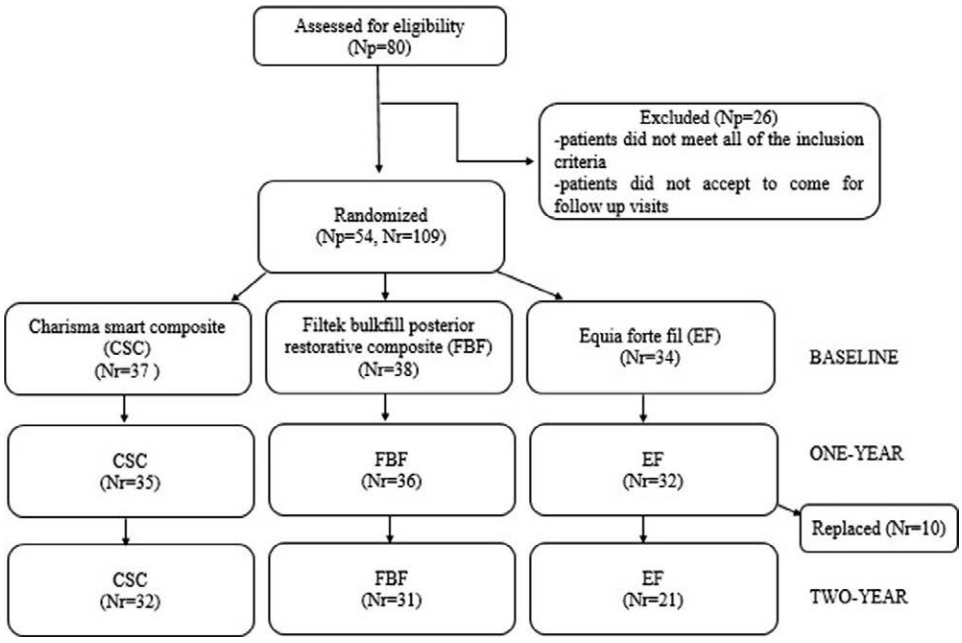

Figure 1. *Flow diagram. Np indicates number of patients; Nr, number of restorations.*

Initial bite-wing radiographs were taken before the treatment, and the most appropriate material color was selected before restorative procedures began. Local anesthesia was applied to patients who complained about pain or sensitivity to prevent discomfort during restorative procedures. Cavity preparations were performed using diamond fissure burs (Diamirsrl, Resia, Italy) at high speed with water cooling. Hand instruments and slow-speed tungsten carbide burs were used to remove caries. Conservative cavity design (Class II slot) was used, and beveling was not applied to the cavity walls to avoid the unnecessary loss of hard dental tissue. The outline shape of the cavity was limited to the removal of the caries lesion. Any additional retention was not prepared. The cavity preparations did not involve any cusps, all the gingival margins included sound enamel, and two surfaces cavities (MO or DO) were included in this study. $Ca(OH)_2$ cavity liner material (Dycal, Dentsply, Konstanz, Germany) was applied where needed as the base material (it was needed for only two restorations). Cotton pellets and suction were used to isolate the operative field. After an ivory-type matrix system (Ivory No. 1 matrix, Hahnenkratt, Königsbach-Stein, Germany) and wooden wedges were placed to the cavities, the cavities were disinfected with 0.2% chlorhexidine gluconate. All cavities were restored as follows.

Charisma Smart Composite Group—Single Bond Universal adhesive (3M ESPE, Neuss, Germany) was applied to the cavities according to the manufacturer's instructions and polymerized with a light-emitting diode light device (Valo, 1000 mW/cm^2, Ultradent, South Jordan, UT, USA) for 10 seconds. Charisma Smart Composite (CSC) was placed incrementally by using horizontal increments, not exceeding 2 mm in the cavity, and each layer was cured for 20 seconds. After removal of the matrix and wedges, the restorations were cured for an additional 10 seconds from the buccal and palatal/lingual sides.

Filtek Bulk Fill Posterior Restorative Group—Single Bond Universal adhesive was applied and polymerized as in group 1. Filtek Bulk Fill Posterior Restorative (FBF) was placed in bulk into the cavity, but at no more than 4 mm thick, and was cured for 20 seconds. After removal of the matrix and wedges, the restorations were cured for an additional 10 seconds from the buccal and palatal/lingual sides.

Equia Forte Fil Group—Cavity Conditioner (GC) was applied to the cavities for 10 seconds, washed, and gently dried. After isolation, an Equia Forte Fil (EF) capsule was placed in an automatic mixer and mixed for 10 seconds. The capsule was then placed in a special applicator and injected into the cavities. After the manufacturer's recommended setting time of 2.5 minutes, the restoration was finished, polished, and gently dried. Equia Forte Coat (GC) was applied to the restoration surfaces and cured for 20 seconds.

The finishing and polishing procedures were performed in the same appointment using high-speed fine diamonds (Meisinger Dental Burs, Hager & Meisinger GmbH, Neuss, Germany), Sof-Lex XT discs (3M ESPE), and yellow composite polishing rubbers (Nais, Sofia, Bulgaria).

Clinical Evaluation of the Restorations

In this study, all participating researchers were educated for calibration before the study was conducted. After the restoration placement, the patients were recalled after one week (baseline), six months, one year, and two years. The restorations were examined clinically using mirrors and probes, and bite-wing radiographs and intraoral photographs were taken from the patients. Dental floss was used to check the contact points. The restorations were evaluated by an experienced blinded investigator according to the modified USPHS criteria (Table 2). Intraobserver reliability was assessed using Cohen's Kappa, and it resulted in a Kappa value of 0.95.

Statistical Analysis

The data obtained were collected in a data pool, and statistical analyses were performed using the software program SPSS 22.0 (SPSS, Chicago, IL, USA). Frequency and rate values were used in the descriptive statistics of the data. For each parameter, chi-square and Fisher tests were used to compare the changes across different time points within each restorative material. In addition, the McNemar test was used to evaluate the difference between the materials. The level of significance was set at $\alpha=0.05$ for all tests.

RESULTS

A total of 109 restorations were placed in 54 patients. Fifty-one of these restorations (46.8%) were placed in premolars, and 58 (53.2%) were placed in molars. Eighty-four restorations were evaluated at the two-year recall with a 77% recall rate, while 103 restorations were evaluated at the one-year recall. None of the restorations showed any change up to six months. The number of evaluated restorations at baseline and at the one-year and two-year recall

Table 2: *Modified US Public Health Service Criteria Used in This Study*

Criteria	Alpha	Bravo	Charlie
Anatomic form	The restoration is continuous with the existing anatomic form	The continuity of restoration with teeth partially degraded but clinically acceptable	The continuity of restoration with teeth completely deteriorated, need to be replaced
Contact point	Normal contact point	No contact point but no periodontal irritation	No contact point, but there is a periodontal irritation finding/the patient wants to change the filling
Marginal adaptation	There is no visible evidence of a crevice along the margin into which the explorer will penetrate	There is visible evidence of a crevice along margin into which the explorer will penetrate or catch	The explorer penetrates the crevice, and dentin or base is exposed
Marginal discoloration	There is no discoloration anywhere on the margin between the restoration and the tooth structure	Discoloration is present but has not penetrated along the margin in a pulpal direction	Discoloration has penetrated along the margin in a pulpal direction
Secondary caries	No evidence of secondary caries	—	Evidence of secondary caries
Color match	The restoration matches the adjacent tooth structure in color and translucency	The mismatch in color and translucency is within the acceptable range	The mismatch in color and translucency is outside the acceptable range
Postoperative sensitivity	No postoperative sensitivity, after the restorative procedure and during the study	Slight sensitivity at any stage of the study	Severe sensitivity at any stage of the study
Retention	No loss of restorative material	—	Fracture and/or loss of restorative material
Surface texture	The surface of the restoration does not have any defects	The surface of the restoration has minimal defects	The surface of the restoration has severe defects

according to tooth type (premolar/molar) is given in Table 3. There was no significant difference between premolar and molar teeth for all parameters and all evaluation periods ($p>0.05$). The clinical evaluation scores of the restorations at baseline and at the one-year and two-year recalls are given in Table 4.

At the baseline evaluation, all CSC, FBF, and EF restorations were scored as "Alpha" for all criteria except color match. In the EF group, although the most appropriate material color was selected, eight restorations were scored as "Bravo" and 26 restorations scored as "Charlie" for the color match because of a lack of material translucency.

Color Match

At the one-year and two-year recalls, no significant color change occurred in the CSC and FBF groups ($p>0.05$). In the EF group, no color change was observed at the one-year evaluation when compared with baseline, while at the two-year recall, a slight improvement in color match of EF restorations was observed. Nevertheless, there was no statistically significant difference between the baseline, and the one-year and two-year color match of the EF group ($p>0.05$). There was a statistically significant difference between the EF group and the CSC and FBF groups for the color match ($p<0.05$), while there was no statistically significant difference between the CSC and FBF groups in any evaluation period ($p>0.05$).

Anatomic Form

Regarding the anatomic form criteria, there was a statistically significant difference between the EF

Table 3: *Distribution of the Materials According to Tooth Type at the Evaluation Periods*

	Baseline		One Year		Two Years	
	Premolar	Molar	Premolar	Molar	Premolar	Molar
Charisma Smart Composite	17	20	15	20	14	18
Filtek Bulkfill Posterior Restorative	18	20	16	20	14	17
Equia Forte Fil	16	18	15	17	12	9
Total	51	58	46	57	40	44

Table 4: *Baseline, One-Year, and Two-Year Clinical Evaluation of Restorations According to US Public Health Service Criteria*

Criterion	Baseline			One Year			Two Years		
	A	B	C	A	B	C	A	B	C
Anatomic form									
CSC	37	0	0	35	0	0	32	0	0
FBF	38	0	0	36	0	0	31	0	0
EF	34	0	0	26[a,b]	4	2	15[a,b]	5	1
Contact point									
CSC	37	0	0	35	0	0	32	0	0
FBF	38	0	0	36	0	0	31	0	0
EF	34	0	0	27[a]	0	5	14[a,b]	5	2
Marginal adaptation									
CSC	37	0	0	30	5	0	23[b]	9	0
FBF	38	0	0	34	2	0	27	4	0
EF	34	0	0	20[a,b]	10	2	10[a,b]	10	1
Marginal discoloration									
CSC	37	0	0	35	0	0	31	1	0
FBF	38	0	0	34	2	0	29	2	0
EF	34	0	0	31	1	0	20	0	1
Secondary caries									
CSC	37	—	0	35	—	0	32	—	0
FBF	38	—	0	36	—	0	31	—	0
EF	34	—	0	32	—	0	21	—	0
Color match									
CSC	37	0	0	35	0	0	32	0	0
FBF	38	0	0	35	1	0	31	0	0
EF	0[a]	8	26	0[a]	6	26	5[a]	10	6
Postoperative sensitivity									
CSC	37	0	0	35	0	0	32	0	0
FBF	38	0	0	36	0	0	31	0	0
EF	34	0	0	32	0	0	21	0	0
Retention									
CSC	37	—	0	35	—	0	32	—	0
FBF	38	—	0	36	—	0	31	—	0
EF	34	—	0	24[a,b]	—	8	15[a,b]	—	6
Surface texture									
CSC	37	0	0	35	0	0	30	2	0
FBF	38	0	0	36	0	0	31	0	0
EF	34	0	0	22[a,b]	9	1	11[a,b]	8	2

Abbreviations: A, Alpha; B, Bravo; C, Charlie; CSC, Charisma Smart Composite; EF, Equia Forte Fil; FBF, Filtek Bulkfill Posterior Restorative.
[a] *Significant difference between the restorative materials (*$p<0.05$*).*
[b] *Significant difference in comparison with baseline for each restorative material (*$p<0.05$*).*

group and the CSC and FBF groups ($p<0.05$), while there was no statistically significant difference between the CSC and FBF groups ($p>0.05$) at the one-year and two-year recall. However, no statistically significant difference was found between the one-year and the two-year clinical performances of the materials ($p>0.05$). For the EF group, the two-year results of the anatomic form showed a significant change compared with the baseline ($p<0.05$).

Contact Point

In terms of the contact point criteria, all of the restorations of the CSC and FBF groups scored as "Alpha," while 14 restorations were scored as "Alpha," five restorations were scored as "Bravo," and two restorations scored as "Charlie" because of marginal fracture or chipping in the EF group at the two-year evaluation. There was a statistically sig-

nificant difference between the EF group and the CSC and FBF groups ($p<0.05$), while there was no statistically significant difference between the CSC and FBF groups ($p>0.05$) at the one-year and two-year evaluation periods. Also, in the EF group, a statistically significant difference was found between the one-year and two-year evaluations ($p<0.05$). The two-year results regarding the contact point showed a significant change compared with the baseline only in the EF group ($p<0.05$).

Marginal Adaptation

In terms of the marginal adaptation criteria, there was a statistically significant difference between the EF group and the CSC and FBF groups ($p<0.05$), while there was no statistically significant difference between the CSC and FBF groups ($p>0.05$) at the one-year and two-year evaluations. However, at the two-year recall, a significant change was observed in the marginal adaptation of EF restorations when compared with the baseline and the one-year evaluation ($p<0.05$).

Marginal Discoloration

For marginal discoloration criteria, only two FBF restorations and one CSC restoration were scored as "Bravo," while one EF restoration was scored as "Charlie." In terms of the marginal discoloration criteria at the one-year and two-year evaluations, there was no statistically significant difference between the groups ($p>0.05$).

Secondary Caries and Postoperative Sensitivity

During the two-year evaluation period, no postoperative sensitivity or secondary caries were observed in any of the restored teeth, and all restorations were scored as "Alpha" for these criteria ($p>0.05$).

Retention and Survival Rate

In terms of the retention criteria, all evaluated CSC and FBF restorations were scored as "Alpha" in all the evaluation periods. In the EF group, eight restorations were scored as "Charlie" because of marginal fracture and material loss, which could be seen radiographically in the proximal area as a result of dissolution at the one-year recall. At the two-year recall, six additional EF restorations were scored as "Charlie" in terms of the retention criteria for the reasons mentioned above (Figures 2 and 3). However, a total loss of restorative material was not observed in any of the restorations during the two-year evaluation. In terms of the retention criteria, there was a statistically significant difference between the EF group and the CSC and FBF groups ($p<0.05$), while there was no statistically significant difference between the CSC and the FBF groups ($p>0.05$) at both the one-year and the two-year evaluation (Figures 4 and 5).

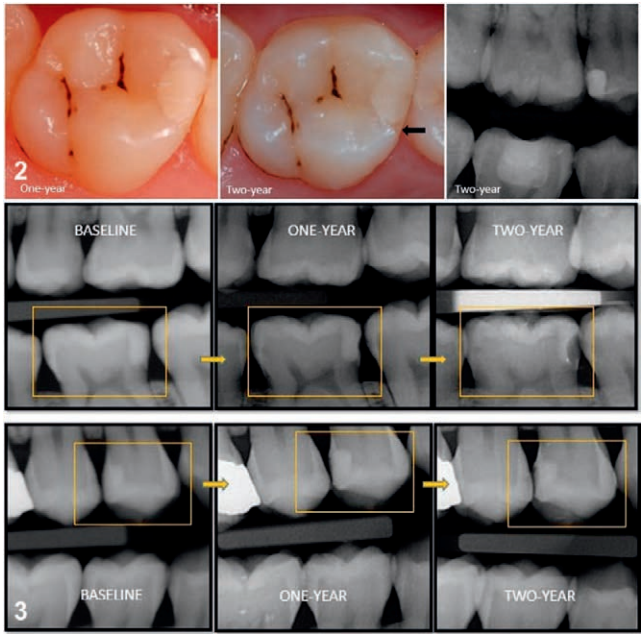

Figure 2. *One-year and two-year clinical appearance and two-year radiography of an Equia Forte Fil (EF) restoration. At the two-year evaluation, marginal fracture was observed.*

Figure 3. *One-year and two-year radiographies of Equia Forte Fil (EF) restorations. At the two-year evaluation, material loss in the proximal wall of the restorations was observed.*

After two years, the survival rates of the CSC and FBF groups were 100%, whereas the survival rate of the EF group was 54.3%. In total, 16 EF restorations (10 restorations at the one-year recall, six restorations at the two-year recall) had to be replaced or modified as a base due to marginal fracture and material loss in the proximal area during the two-year follow-up.

Surface Texture

During the two-year evaluation period, there was no significant change in the surface texture of the FBF and CSC restorations, unlike the EF restorations. In terms of the surface texture criteria, there was a statistically significant difference between the EF group and the CSC and FBF groups ($p<0.05$), while there was no statistically significant difference between the CSC and the FBF groups in the one-year and two-year evaluations ($p>0.05$).

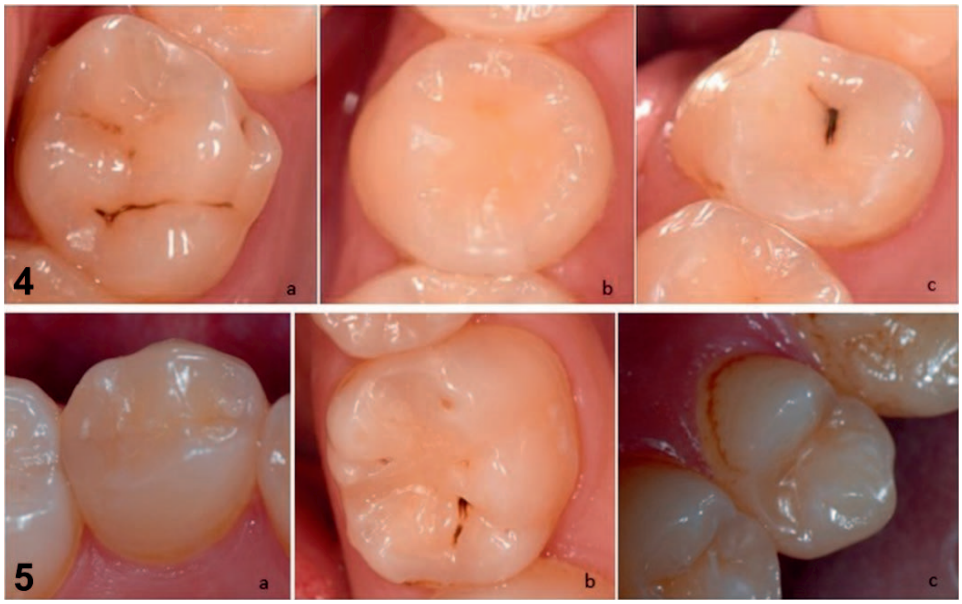

Figure 4. *Charisma Smart Composite (CSC) restorations scored as "Alpha" after the two-year recall.*

Figure 5. *Filtek Bulkfill Posterior Restorative (FBF) restorations scored as "Alpha" after the two-year recall.*

DISCUSSION

Bulk-fill restorative materials such as bulk-fill composite resins and HVGICs have recently become very popular materials in operative dentistry because they provide clinicians with an easy and quick application. The bulk application of restorative materials overcomes some challenges such as void formation and contamination risk between the layers and difficulty in the placement of layers in small cavities. In this clinical study, the clinical performances of three restorative systems were evaluated during a two-year period. The null hypothesis of this study was rejected because composite resin materials (conventional and bulk-fill) showed a significantly better clinical performance than HVGIC.

The retention rates represent the most important evaluation criteria to determine the clinical success of restorative materials. The American Dental Association requires a retention rate of at least 90% of the restorations after 18 months to obtain full acceptance.[17] In this study, the two-year survival rate of the CSC and the FBF restorations was 100%, while it was 54.3% for the EF restorations because 16 of 35 EF restorations required replacement at the end of two years. Based on this information, EF with low clinical success was considered unsuitable for the permanent restoration of Class II cavities, whereas bulk-fill and conventional composite resins were found to be clinically acceptable.

In the literature, no previous clinical study has attempted to compare the clinical performances of an HVGIC and a bulk-fill composite resin in Class II cavities. For this reason, it is not possible to make a direct comparison with previous studies. Çolak and others[18] and Bayraktar and others[19] evaluated the one-year clinical performances of Class II restorations made using either bulk-fill composite resin or conventional composite resin. The authors reported that the bulk-fill composite resins showed similar clinical performance when compared with conventional composite resin. The 12-month findings of the present study are consistent with their short-term data. Moreover, in another study comparing long-term clinical performances (10 years) of a hybrid composite resin and a bulk-fill composite resin in Class II cavities, the authors reported that both materials performed quite well clinically with no significant differences.[20] Although our evaluation period is two years, similarly, no difference was found between the clinical performances of bulk-fill composite and microhybrid composite. Yazıcı and others[21] evaluated the 36-month clinical performance of a nanofill composite resin and a bulk-fill composite resin in Class II restorations. According to their study results, at both 24 months and 36 months, the bulk-fill composite resin demonstrated better clinical performance in terms of marginal discoloration and marginal adaptation, while there was no difference between the materials in terms of other parameters. The data of the present study are not in agreement with the study of Yazıcı and others because no significant difference was found in terms of marginal discoloration or marginal adaptation of composite resin materials during the two-year period.

Gurgan and others[22] investigated the long-term clinical performance of the Equia restorative system on permanent posterior teeth in Class I and Class II caries lesions according to USPHS criteria and compared it with a microhybrid composite resin. The researchers reported that both restorative materials showed a clinically successful performance after 6 years. In another long-term clinical trial, the clinical performances of two different HVGICs (Equia Fil and Riva SC) were assessed using USPHS criteria.[23] The authors stated that Equia Fil showed acceptable clinical performance at the end of six years. However, Türkün and Kanik[23] did not assess the restoration as a fail since the restoration did not require a replacement, although it needed to be repaired. In our study, if the restoration needed to be repaired, it was evaluated as a fail. The restorations that had only a small degree of chipping that did not cause food impaction and did not require repair were maintained and monitored throughout the study.

In the literature, there are studies that evaluated the clinical and radiographic performances of Class II restorations involving HVGIC, and it was reported that concavity was seen radiographically on the proximal wall of the restorations at the 18-month recall.[24,25] In the present study, we also evaluated the clinical and radiographic performances of the restorations, and when any loss of restorative material (fracture and/or material loss in the proximal wall) was noted, these restorations were scored as "Charlie" in terms of the retention criteria. At the end of two years, the loss of material in the proximal wall that could be observed on radiographs was seen in a total of nine restorations (25.7%). We agree with previous studies on the cause of the occurrence of this material loss in the proximal wall of the restorations. It may be related to the inability of the protective resin to be applied effectively to the proximal wall of the glass ionomer restoration because it is not easy to access the proximal area. If the surface-coating agent cannot be applied effectively, the proximal area is unprotected from moisture contamination during the initial hardening phase, and the GIC may dissolve.[24,25] The use of metal matrices during the restorative procedure can also be another cause. GIC can adhere to metals chemically, and micro cracks may occur in GIC because of the force applied during the removal of the matrix. These micro cracks may make the material more susceptible to chemical attack.[25] Furthermore, it has also been reported that surface-coating agents wear over time.[24] In our study, deterioration in the surface texture was observed at the one-year and two-year evaluation of the EF restorations. We think that the wear of the surface-coating agent and the glass ionomer material may cause this deterioration.

In this study, the color match with the surrounding dental tissue of the restoration was a problem in the EF group. HVGICs have more translucency than conventional GICs. The HVGIC used in this study, also known as the glass hybrid system, has many color options. Even so, the color and translucency properties of HVGIC restorations were still not enough, and its color match was not as good as that of composite resin restorations up to one year. However, since the restorations were in the posterior region, and the patients were not disturbed by the appearance of the restorations, the replacing of the restoration was not considered in order to remedy the color mismatch. Also, it was known that the translucency of GICs would improve due to continuous maturation.[26] Our findings support this information. At the two-year evaluation, a slight improvement was observed in terms of the color match of the HVGIC restorations. We believe that this improvement in the color match is associated with increasing cement maturation. Consistent with our findings, Diem and others[27] reported that the color match of HVGIC restorations improved over the three years of the study (about 25% "good" at baseline, steadily increasing to about 80% "good" at three years) with improving translucency over time as the cement matures.

Secondary caries and postoperative sensitivity were not observed in any restoration during the two-year follow-up period. Chlorhexidine is commonly used as an antimicrobial agent for cavity disinfection, and it was used for cavity disinfection before the placing of the restorations in this study. Besides, EF is a fluoride-releasing restorative material, and it may have prevented the formation of caries under the restoration in this group. The absence of secondary caries could also be related to the good oral hygiene status of the patients. Cavity depth plays an important role in determining postoperative complications. The deeper the cavity, the greater the likelihood of postoperative sensitivity. In addition, operative trauma is another factor that may cause postoperative sensitivity.[28] In the present study, operative procedures (both cavity preparation and placement of the restoration) were done carefully by an experienced operator. The depth of cavities was mostly moderate in this study, and $Ca(OH)_2$ cavity liner material was applied in deeper cavities. In addition, a universal bond was used in the self-etch

mode in this study, and acid etching that removes the smear layer and requires technique sensitivity was not applied. All factors mentioned above might have had a favorable effect on postoperative sensitivity.

CONCLUSIONS

During a two-year follow-up period, a conventional microhybrid composite resin and a bulk-fill composite resin showed similar clinical performance, and these materials were found to be clinically more successful than a high-viscosity glass ionomer material. Moreover, within the limitations of this study, it can be concluded that the use of high-viscosity glass ionomer material as a permanent restorative material in Class II cavities is not appropriate.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Erciyes University, Faculty of Medicine Ethics Committee. The approval code issued for this study is 2017/44.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Demarco FF, Correa MB, Cenci MS, Moraes RR, & Opdam NJ (2012) Longevity of posterior composite restorations: not only a matter of materials *Dental Materials* **28(1)** 87-101.
2. Brunthaler A, Konig F, Lucas T, Sperr W, & Schedle A (2003) Longevity of direct resin composite restorations in posterior teeth *Clinical Oral Investigations* **7(2)** 63-70.
3. Manhart J, Chen H, Hamm G, & Hickel R (2004) Buonocore Memorial Lecture. Review of the clinical survival of direct and indirect restorations in posterior teeth of the permanent dentition *Operative Dentistry* **29(5)** 481-508.
4. Laegreid T, Gjerdet NR, Johansson A, & Johansson AK (2014) Clinical decision making on extensive molar restorations *Operative Dentistry* **39(6)** 231-240.
5. Lynch CD, Opdam NJ, Hickel R, Brunton PA, Gurgan S, Kakaboura A, Shearer AC, Vanherle G, & Wilson NH (2014) Guidance on posterior resin composites: Academy of Operative Dentistry–European Section *Journal of Dentistry* **42(4)** 377-383.
6. Ferracane JL & Hilton TJ (2016) Polymerization stress—is it clinically meaningful? *Dental Materials* **32(1)** 1-10.
7. Zorzin J, Maier E, Harre S, Fey T, Belli R, Lohbauer U, Petschelt A, & Taschner M (2015) Bulk-fill resin composites: polymerization properties and extended light curing *Dental Materials* **31(3)** 293-301.
8. Park J, Chang J, Ferracane J, & Lee IB (2008) How should composite be layered to reduce shrinkage stress: Incremental or bulk filling? *Dental Materials* **24(11)** 1501-1505.
9. Bicalho AA, Pereira RD, Zanatta RF, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2014) Incremental filling technique and composite material—part Ic Cuspal deformation, bond strength, and physical properties *Operative Dentistry* **39(2)** 71-82.
10. Abbas G, Fleming GJ, Harrington E, Shortall AC, & Burke FJ (2003) Cuspal movement and microleakage in premolar teeth restored with a packable composite cured in bulk or in increments *Journal of Dentistry* **31(6)** 436-444.
11. Garoushi S, Vallittu P, Shinya A, & Lassila L (2016) Influence of increment thickness on light transmission, degree of conversion and micro hardness of bulk fill composites *Odontology* **104(3)** 291-297.
12. Burke FJ (2013) Dental materials—what goes where? The current status of glass ionomer as a material for loadbearing restorations in posterior teeth. *Dental Update* **40(10)** 840-844.
13. Berg JH & Croll TP (2015) Glass ionomer restorative cement systems: an update *Pediatric Dentistry* **37(2)** 116-124.
14. Friedl K, Hiller KA, & Friedl KH (2011) Clinical performance of a new glass ionomer based restoration system: a retrospective cohort study *Dental Materials* **27(10)** 1031-1037.
15. Çelik EU, Tunac AT, & Yilmaz F (2018) A randomized, controlled, split-mouth trial evaluating the clinical performance of high-viscosity glass-ionomer restorations in noncarious cervical lesions: two-year results *Journal of Adhesive Dentistry* **20(4)** 299-305.
16. Bagheri R, Palamara J, Mese A, & Manton DJ (2017) Effect of a self-adhesive coating on the load-bearing capacity of tooth-colored restorative materials *Australian Dental Journal* **62(1)** 71-78.
17. Loguercio AD & Reis A (2008) Application of a dental adhesive using the self-etch and etch-and-rinse approaches: an 18-month clinical evaluation. *Journal of the American Dental Association* **139(1)** 53-61.
18. Colak H, Tokay U, Uzgur R, Hamidi MM, & Ercan E (2017) A prospective, randomized, double-blind clinical trial of one nano-hybrid and one high-viscosity bulk-fill composite restorative systems in class II cavities: 12 months results *Nigerian Journal of Clinical Practice* **20(7)** 822-831.
19. Bayraktar Y, Ercan E, Hamidi MM, & Çolak H (2017) One-year clinical evaluation of different types of bulk-fill composites *Journal of Investigative and Clinical Dentistry* **8(2)** 1-9.
20. Heck K, Manhart J, Hickel R, & Diegritz C (2018) Clinical evaluation of the bulk fill composite QuiXfil in molar class I and II cavities: 10-year results of a RCT *Dental Materials* **34(6)** e138-e147.
21. Yazici AR, Antonson SA, Kutuk ZB, & Ergin E (2017) Thirty-six-month clinical comparison of bulk fill and

nanofill composite restorations *Operative Dentistry* **42(5)** 478-485.

22. Gurgan S, Kutuk ZB, Ergin E, Oztas SS, & Cakir FY (2017) Clinical performance of a glass ionomer restorative system: a 6-year evaluation. *Clinical Oral Investigations* **21(7)** 2335-2343.
23. Türkün LS & Kanik Ö (2016) A prospective six-year clinical study evaluating reinforced glass ıonomer cements with resin coating on posterior teeth: quo vadis? *Operative Dentistry* **41(6)** 587-598.
24. Tal E, Kupietzky A, Fuks AB, Tickotsky N, & Moskovitz M (2017) Clinical performance of heat-cured high-viscosity glass ıonomer class ıı restorations in primary molars: a preliminary study *Journal of Clinical Pediatric Dentistry* **41(4)** 264-270.
25. Scholtanus JD & Huysmans MC (2007) Clinical failure of class-II restorations of a highly viscous glass-ionomer material over a 6-year period: a retrospective study *Journal of Dentistry* **35(2)** 156-162.
26. Mount GJ (2002) *An Atlas of Glass-İonomer Cements*. *A Clinician's Guide* **3rd edition** Martin Dunitz, London.
27. Diem VT, Tyas MJ, Ngo HC, Phuong LH, & Khanh ND (2014) The effect of a nano-filled resin coating on the 3-year clinical performance of a conventional high-viscosity glass-ionomer cement *Clinical Oral Investigations* **18(3)** 753-759.
28. Auschill TM, Koch CA, Wolkewitz M, Hellwig E, & Arweiler NB (2009) Occurrence and causing stimuli of postoperative sensitivity in composite restorations *Operative Dentistry* **34(1)** 3-10.

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