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**Clinical Research**

# *In Vitro* and *In Vivo* Evaluations of Three Computer-Aided Shade Matching Instruments

K Yuan • X Sun • F Wang
H Wang • J Chen

### Clinical Relevance

Shadepilot was the only instrument tested in the present study that showed high accuracy and reliability both *in vitro* and *in vivo*. As different L*a*b* values and shade matching results were reported using various instruments for the same tooth, a combination of the evaluated shade matching instruments and visual shade confirmation is recommended for clinical use.

### SUMMARY

**This study evaluated the accuracy and reliability of three computer-aided shade matching instruments (Shadepilot, VITA Easyshade, and ShadeEye NCC) using both *in vitro* and *in vivo* models. The *in vitro* model included the measurement of five VITA Classical shade guides. The *in vivo* model utilized three instruments to measure the central region of the labial surface of maxillary right central incisors of 85 people. The accuracy and reliability of the three instruments in these two evaluating models were calculated. Significant differences were observed in the accuracy of instruments both *in vitro* and *in vivo*. No significant differences were found in the reliability of instruments between and within the *in vitro* and the *in vivo* groups. VITA Easyshade was significantly different in accuracy between *in vitro* and *in vivo* models, while no significant difference was found for the other two instruments. Shadepilot was the only**

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**instrument tested in the present study that showed high accuracy and reliability both *in vitro* and *in vivo*. Significant differences were observed in the L*a*b* values of the 85 natural teeth measured using three instruments in the *in vivo* assessment. The pair-agreement rates of shade matching among the three instruments ranged from 37.7% to 48.2%, and the incidence of identical shade results shared by all three instruments was 25.9%. As different L*a*b* values and shade matching results were reported for the same tooth, a combination of the evaluated shade matching instruments and visual shade confirmation is recommended for clinical use.**

## INTRODUCTION

The accurate and reliable selection of tooth shade is critical for successful clinical restoration, particularly for esthetics. The traditional method used for visually selecting shades is comparing natural tooth color with commercial standard shade guides.[1,2] However, visual shade selection has been found to be unreliable and inconsistent.[2,3] This selection is a subjective process since visual color perception is the result of psychological and physiological responses that vary between individuals.[4,5] General variables include lighting conditions, the angle of perception, experience levels, age, and eye fatigue as well as physiological variables, such as color perception deficiencies, all of which may lead to discrepancies.[6-8] In addition, the double-layering effect of natural teeth created by enamel translucency in combination with dentin opacity make visual shade selection difficult.[9]

Limitations in visual shade selection have triggered the search for more accurate, consistent, and scientific shade matching methods, ultimately utilizing objective instrumentation. Instrumental color shade selection may have a number of potential advantages over visual shade selection because of its inherent objectivity and the potential for user-independent accuracy and reliability. At the present time, an increasing number of computer-aided shade matching instruments, which include colorimeters, spectrophotometers, digital color analyzers, or a combination of these, are commercially available for clinical use.[7,13] Measurements from these instruments can be rapidly exported to one or more of the dental shade-guide systems, and the results can also be reported as X, Y, Z tristimulus or as Commission International de l'Éclairage (CIE) L*a*b* values. This enables uniform and precise communication of color information between clinicians and technicians and promotes shade analyses, interpretation, and fabrication of restorations.[14] CIE L*a*b* is one of the standard color models used to describe colors in a three-coordinate system. The L* value defines the lightness of the color and can range between 0 (black) and 100 (white). The a* and b* values refer to the chromatic characteristics of the color. The a* value defines green (negative a*) to red (positive a*) colors, while the b* value defines blue (negative b*) to yellow (positive b*) colors.[2,14,15] Delta E ($\Delta E$) describes the color difference between two specimens and is calculated using the following formula[2]: $\Delta E = [(L^*_1 - L^*_2)^2 + (a^*_1 - a^*_2)^2 + (b^*_1 - b^*_2)^2]^{1/2}$.

The accuracy and reliability of color measuring and matching functions of shade matching instruments could be affected by the operating principles of the instrument that influence how the instrument handles light reflected from the tooth surface.[14] Spectrophotometers function by measuring spectral curve at the time that light is reflected or transmitted from a specimen.[8,15] In spectrophotometers, the intensity of light reflected from a specimen is measured for all visible spectrum wavelengths.[14,15] Ishikawa-Nagai and others[10-12] have reported computer-aided spectrophotometers as an excellent instrumental method for color matching in porcelain restorations.

Colorimeters have red, green, and blue filters that approximate the spectral function of a human eye. Using colorimeters, the X, Y, Z tristimulus or CIE L*a*b* values of a specimen can be measured after the reflected light has been processed through a series of filters.[8,14] Colorimeters are considered to be more reliable and accurate for color-difference measurement than spectrophotometers.[8] However, their reliability may be poor because of the aging of filters, and their accuracy can be affected by the object metamerism, which occurs when a pair of objects match under one light source but do not match under one or more other light sources.[16]

Although color measuring and matching performance of some types of computer-aided shade matching instruments have been reported,[7,14,15,17-19] these studies were based solely on either *in vitro* or *in vivo* models. Studies considering systematic evaluations using both *in vitro* and *in vivo* methods were not identified by the authors. Results from *in vivo* models may not reflect those obtained *in vitro* because of application-specific issues when the instruments are put into actual use, and thus issues with instrumental accuracy and reliability may remain. Therefore, the present study evaluated color

measuring and matching performance of three computer-aided shade matching instruments using both *in vitro* and *in vivo* models. The null hypothesis was that there is no difference in color measuring and matching performance of the tested instruments between and within these two models.

## MATERIALS AND METHODS

Three commercially available computer-aided shade matching instruments were evaluated (Table 1): Shadepilot (DeguDent GmbH, Hanau, Germany), VITA Easyshade (VITA Zahnfabrik, Bad Säckingen, Germany), and ShadeEye NCC (Shofu Inc, Kyoto, Japan). All the instruments were new and were operated by the same investigator following the manufacturers' instructions. The instruments were first allowed to warm up for 15 minutes. They were then calibrated following the manufacturers' specifications using the included standards before each measurement. Statistical analyses were completed using standard statistical software (SPSS Statistics version 17.0).

### In Vitro *Model*

For the *in vitro* model, color measurements were performed with the three shade matching instruments using a common clinically used shade guide (VITA Classical, VITA Zahnfabrik). Five new shade guides (a total of 80 shade tabs) were used. All shade tabs were cleaned with soap followed by pure ethanol (15 minutes) and finally with distilled water (15 minutes) using an ultrasonic cleaning device (VITA Sonic II, VITA Zahnfabrik). The shade tab to be measured was placed in the middle of a medium gingival colored matrix (Shofu Gumy, Shofu Inc), and an identically colored shade tab was placed on either side in an attempt to simulate an oral environment.[7,15] Shade measurements were performed in the central region of the shade tab inside a lightproof box.

To ensure study accuracy, each shade tab from five shade guides was measured once by each of the three instruments. Accurate measurements were defined as identical shade matches to the shade tab. The measuring accuracy of each instrument was calculated as a percentage of correct matches for a total of 80 measurements (corresponding to 80 shade tabs). For the reliability study, each shade tab was measured two nonconsecutive times within an interval of one hour. Reliability measurements were defined as identical repeated measurements, regardless of whether the measurements matched the actual shade tabs. Reliability was calculated as a percentage of the identical repeated measurements of the total shade tabs used.

Table 1: *Shade Matching Instruments Evaluated in the Present Study*

| Instrument | Manufacturer | Type |
|---|---|---|
| Shadepilot | DeguDent GmbH, Hanau, Germany | Spectrophotometer |
| VITA Easyshade | VITA Zahnfabrik, Bad Säckingen, Germany | Spectrophotometer |
| ShadeEye NCC | Shofu Inc, Kyoto, Japan | Colorimeter |

### In Vivo *Model*

For the *in vivo* model, the maxillary right central incisors of 85 people (45 men and 40 women, average age 33 ± 11 years) were measured. In general, the patients' teeth were normal and varied over a wide range of shades. The selected teeth had to have the least possible variation in surface characteristics and morphology. Thus, teeth with caries, tissue defects, heterogeneous staining, or extremely concave, convex, rough, or irregular surfaces were excluded.[14] All test subjects gave informed consent for the study, and the study protocol was approved by the local Ethics in Research Committee prior to the experiment.

Before measurements were performed, test subjects were requested to remove their makeup and to brush their maxillary anterior teeth for about one minute to remove soft deposits. Subjects were then instructed to position their heads against the headrest of the treatment chair and to open their mouth to a slight degree with their tongue remaining in a relaxed position. The tooth to be tested was dried using an air syringe, and tooth color was measured by each of the three instruments. The measurements were taken at the central region of the labial surface of each tooth. In the color matching procedure, the color data were exported into the VITA Classical shade guide system, while in the color measuring procedure, the data were transferred into the CIE L*a*b* value. For CIE L*a*b* value measurements, each tooth was measured three times, and average values were recorded.

The CIE L*a*b* values of shade tabs for the five VITA Classical shade guides used in the *in vitro* model were also measured by each instrument. For

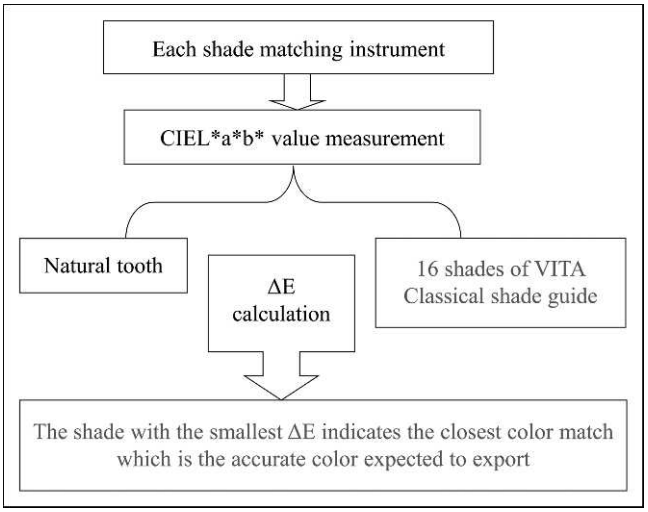


Figure 1. *Method for assessing accurate tooth color using the VITA Classical shade guide system*

each shade tab, three consecutive measurements were performed, and an average value was recorded. The final CIE L*a*b* values of the shade tabs with the same color mark, which represented the CIE L*a*b* values of the 16 different shades of VITA Classical shade guide, were represented as the mean value of these shade tabs.

Tooth color, in terms of the VITA Classical shade guide system, was calculated (Figure 1). The color differences (ΔE) between the tested tooth and the 16 different shades of the VITA Classical shade guide were calculated using the color difference formula from the data obtained.[2] The shade showing the smallest ΔE value indicated the closest color match to the tested tooth, and this shade should be considered the accurate color of the tested tooth, which the measuring instrument was expected to export. For each calculation, the CIE L*a*b* values of the tooth and the shade were provided by the same instrument. It is possible that different instruments would demonstrate different calculation results for the same measured tooth. Correct measurements were defined as identical shade matches to the accurate color. The accuracy of each instrument was calculated as a percentage of correct matches to the total number of measurements (n=85).

For the study of reliability, each tooth was measured twice within an interval of one hour. Reliability measurements were defined as identical repeated measurements, whether or not the measurements matched the accurate shades. The reliability of each instrument was calculated as a percentage of the identical repeated measurements of the total number of individual teeth measured.

## RESULTS

Accuracy data for each instrument in both the *in vitro* and the *in vivo* models are shown in Figure 2. The results of multiple comparisons using a chi-square test at the 0.05 level of significance with Bonferroni correction for differences in accuracy are shown in Table 2. For the *in vitro* model measuring shade tabs, the highest accuracy was found when

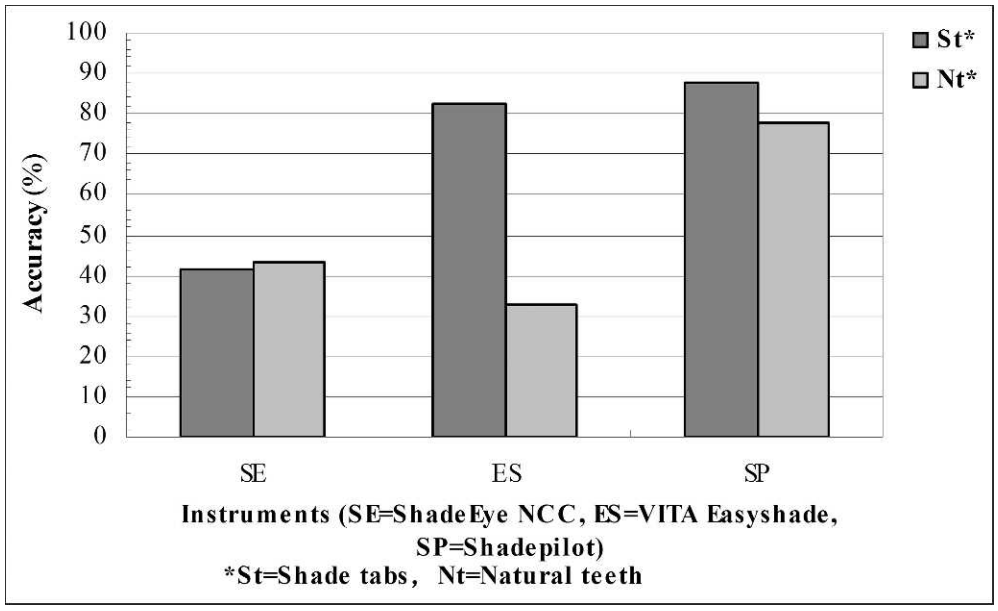


Figure 2. *Accuracy data for* in vitro *shade tab and* in vivo *natural tooth measurements using the three shade matching instruments*

Table 2: *Differences in Accuracy for Shade Tab and Natural Tooth Measurements*

| | Shade Tabs | | Natural Teeth | |
|---|---|---|---|---|
| | $\chi^2$ Value | *p* | $\chi^2$ Value | *p* |
| ShadeEye NCC vs VITA Easyshade | 28.85 | <0.001* | 2.02 | 0.115 |
| ShadeEye NCC vs Shadepilot | 37.31 | <0.001* | 20.72 | <0.001* |
| VITA Easyshade vs Shadepilot | 0.78 | 0.376 | 34.36 | <0.001* |

** Statistically significant difference set at p≤0.0167 following Bonferroni correction applied to the overall significance level of 0.05.*

using Shadepilot (87.5%), followed by VITA Easyshade (82.5%). The accuracy of ShadeEye NCC (41.3%) was significantly lower than the other two instruments ($p<0.001$). For the *in vivo* measurements using natural teeth, Shadepilot also demonstrated the highest accuracy (77.7%), while both ShadeEye NCC and VITA Easyshade showed significantly lower values (43.5% and 32.9%, respectively; $p<0.001$).

Differences in accuracy in comparing *in vitro* vs *in vivo* measurements of each instrument with a chi-square test are shown in Table 4. Only VITA Easyshade showed a significant difference in accuracy in comparing *in vitro* and *in vivo* measurements ($p<0.001$).

Reliability data for each instrument *in vitro* and *in vivo* are shown in Figure 3. The results of multiple comparisons assessed using the chi-square test at the significance level of 0.05 with Bonferroni correction for differences in reliability appear in Table 3. VITA Easyshade showed the highest reliability between the *in vitro* and *in vivo* methods, with 96.3% for shade tabs measurements and 92.9% for natural teeth measurements. Shadepilot demonstrated lower reliabilities (93.8% and 89.4%). The lowest reliability was found with ShadeEye NCC (87.5% and 88.2%). However, no statistically significant differences in reliability were found among all of the instruments between and within *in vitro* and *in vivo* models (Tables 3 and 4).

Table 3: *Differences in Reliability for Shade Tab and Natural Tooth Measurements*

| | Shade Tabs | | Natural Teeth | |
|---|---|---|---|---|
| | $\chi^2$ Value | *p* | $\chi^2$ Value | *p* |
| ShadeEye NCC vs VITA Easyshade | 3.01 | 0.083 | 1.10 | 0.293 |
| ShadeEye NCC vs Shadepilot | 1.84 | 0.175 | 0.06 | 0.808 |
| VITA Easyshade vs Shadepilot | 0.13 | 0.468 | 0.66 | 0.417 |

** Statistically significant difference set at p≤0.0167 following Bonferroni correction applied to the overall significance level of 0.05.*

Table 4: *Differences in Accuracy and Reliability for Shade Tabs vs Natural Teeth Measurements According to Each Instrument*

| Shade Tabs vs Natural Teeth | Accuracy | | Reliability | |
|---|---|---|---|---|
| | $\chi^2$ Value | *p* | $\chi^2$ Value | *p* |
| ShadeEye NCC | 0.09 | 0.767 | 0.02 | 0.885 |
| VITA Easyshade | 41.29 | <0.001* | 0.35 | 0.554 |
| Shadepilot | 2.76 | 0.097 | 1.00 | 0.318 |

** Statistically significant difference at the 0.05 level.*

The mean CIE L*a*b* values of the 85 natural teeth measured by the three instruments are shown in Figure 4. ShadeEye NCC showed the lowest values of tooth color parameters. Compared with Shadepilot, VITA Easyshade showed higher L* and b* values but lower a* values. Significant differences in the L*a*b* values among the three instruments were found (Table 5; Tukey HSD test, $p<0.001$).

The pair-agreement rates for VITA Classical shades exported by all instruments when color measurements were performed on natural teeth are shown in Figure 5. The pair-agreement rates of Shadepilot-ShadeEye NCC and Shadepilot-VITA Easyshade were 48.2% and 44.7% respectively, higher than ShadeEye NCC-VITA Easyshade (37.7%). No significant difference was found among the pair-agreement rates of the three instruments (chi-square test, $\chi^2=2.01$, $p=0.366$). The agreement rate for all three instruments was 25.9%.

## DISCUSSION

Accuracy and reliability are two important considerations when selecting shade matching instruments in both the laboratory and the clinic. The accuracy of

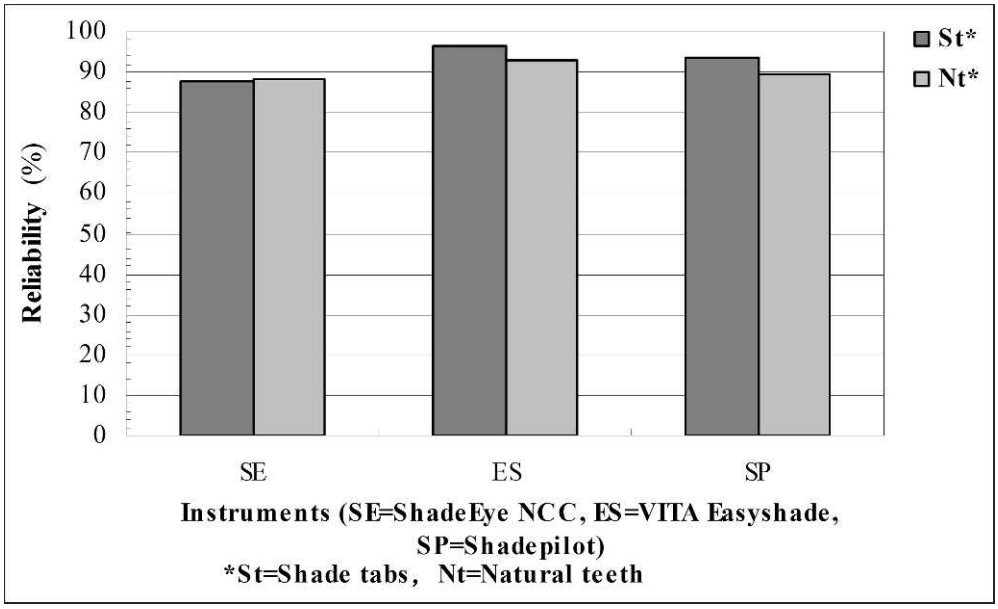


Figure 3. *Reliability data for* in vitro *shade tab and* in vivo *natural tooth measurements with the three shade matching instruments*

an instrument represents its ability to make a correct color match to a tested substrate, while reliability represents the consistency of the shade matching results when measuring the same substrate.[7] The results of the present study mandate rejection of the null hypothesis, which anticipated no differences in color measuring and matching performance of the three instruments. Shadepilot exhibited a significantly higher accuracy than ShadeEye NCC, both *in vitro* and *in vivo*, while VITA Easyshade demonstrated a significantly higher accuracy *in vitro* than *in vivo*. All instruments showed high reliability (over 87%). No differences in reliability were observed among the three tested instruments between and within the *in vitro* and *in vivo* models.

Kim-Pusateri and others[7,15] determined the accuracies and reliabilities of several instruments in an *in vitro* model using shade tabs as measuring standards. A similar *in vitro* model was used in the present study to evaluate color matching performance of the three instruments. In the present study, the accuracy and reliability of VITA Easyshade were 82.5% and 96.3%, respectively, while in the Kim-Pusateri study, comparatively higher values were reported (92.6% and 96.4%). A potential reason for the differences may result from the potential variability of the shade guides used in the two different experiments. Variation between shade guides has been reported, even in guides made by the same manufacturer.[15] The present study also verifies the fact that CIE L*a*b* values from the same kind of shade tab, including the five VITA classical shade guides utilized, were different. In addition, the irregular surface of shade tabs may affect the obtained results.

Significant differences in shade matching accuracy were determined among the three instruments studied. The reason for these differences may be due to a combination of factors, including the mechanisms by which the instruments use to illuminate the tooth, the instrument's ability to measure complex translucent objects, the internal design of the instrument, the software managing data collection and analysis, and also the inherent variability of shade guides.[2,14] Additionally, instrument type and the mechanism that each instrument uses to perform the measurements may be potential causes for discrepancies in the accuracy of color measurement. For example, Shadepilot and VITA Easyshade are spectrophotometers, which work with the full spectrum of light reflected from a tooth, while ShadeEye NCC is a colorimeter, which filters the incoming spectrum of reflected light. In addition, Shadepilot measures reflected light from a whole region of the tooth (complete-tooth measurement), while VITA Easyshade and ShadeEye NCC measure a specific spot on the tooth (spot measurement). Other differences in instrumental measurements involve the shape of the detecting head. Since shade tabs and natural teeth have surface anomalies and curve variations, their surfaces are typically not flat. Both VITA Easyshade and ShadeEye NCC have a flat terminal detecting head designed to measure flat surfaces. Therefore, they are prone to significant edge-loss effects, which

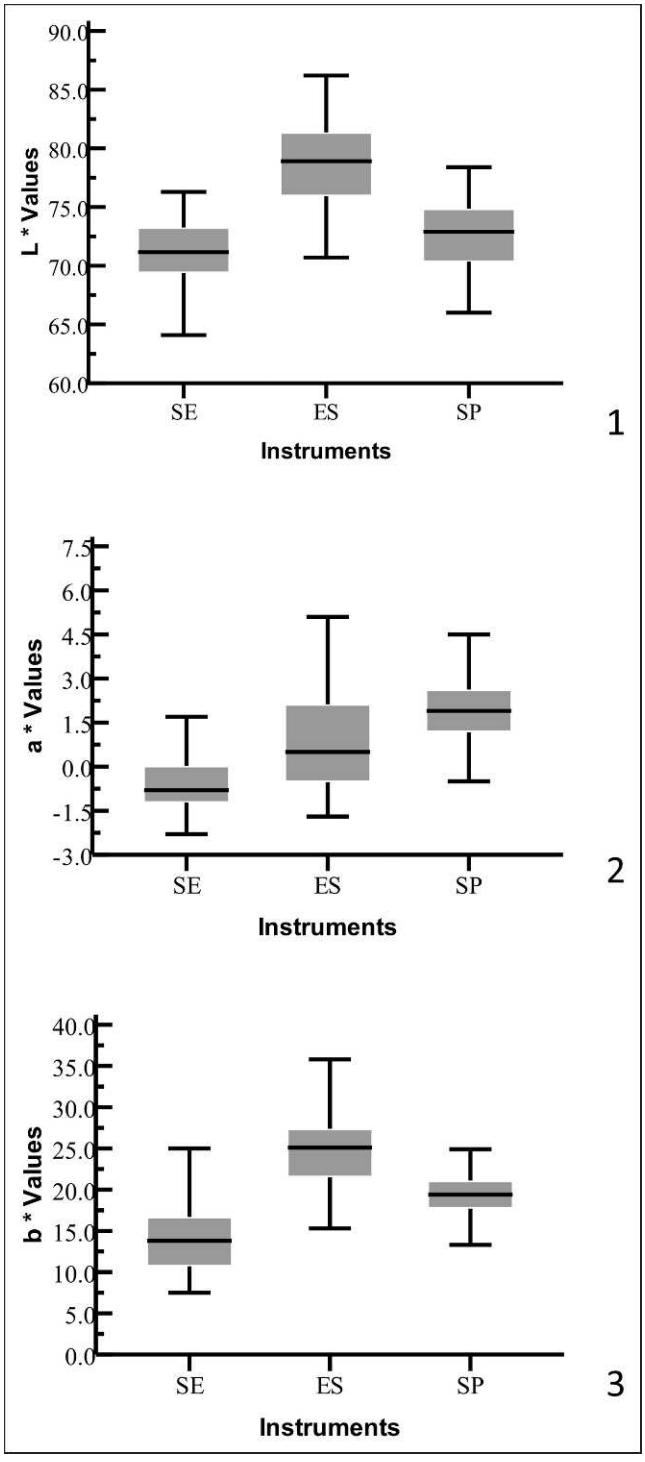


Figure 4. *Box plots of CIE L*a*b* values of 85 natural teeth measured using ShadeEye NCC (SE), VITA EasyShade (ES), and Shadepilot (SP). (1): L* values. (2): a* values. (3): b* values*

may lead to incorrect color measurements.[1,8,20] Alternatively, complete-tooth measurement does not cause edge loss; however, with this alternative method, it is difficult to control the measuring angle.

Of all three shade matching instruments included in the present investigation, VITA Easyshade showed significantly lower accuracy for *in vivo* natural tooth color measurements in comparison to VITA Easyshade's accuracy performance *in vitro*. This highlights the influence of the complex characteristics and variations of natural teeth in comparison to shade tabs and demonstrates that *in vitro* evaluations may be insufficient to evaluate the actual clinical performance of a computer-aided shade matching instrument. VITA Easyshade has a large terminal detecting head with a diameter of 6.0 mm, while ShadeEye NCC has a much smaller terminal detecting head with a diameter of only 2.5 mm. The larger the flat detecting head, the more difficult it becomes to contact the curved surface of a natural tooth and, consequently, the more space that remains between the edge of the detecting head and the tooth surface. This may result in loss of reflected light and incorrect color measurements. Furthermore, accurate *in vivo* color measurements are more difficult to achieve than *in vitro* color measurement.

For *in vivo* measurements, VITA Easyshade showed greater CIE L*a*b* values than the other two instruments, with the exception of the b* values obtained from Shadepilot. The cause of these differences may also be a combination of factors. The results of the present study reconfirm the results of a previous *in vitro* study,[14] in which 31 extracted anterior human teeth were measured using VITA Easyshade and ShadeEye NCC, and the results showed that VITA Easyshade provided greater CIE L*a*b* values than ShadeEye NCC. In clinical practice, different L*a*b* values would not affect the final color of the restoration if the extrapolation algorithm of the instrument were properly established to correctly transfer these color parameters to the standard color of a shade tab in a clinical guide.[14] As Shadepilot demonstrated high accuracies in both the *in vitro* and the *in vivo* models, it seems to have a more precise interpolation algorithm.

All the pair-agreement rates of VITA Classical Shades reported by the three instruments when color measurements were performed on natural teeth were lower than 50%. Low agreements for computer-aided shade matching instruments were also found by Hugo and others.[13] Although the measurements exported by each instrument were different, the unpaired results of the measurement of the same tooth were typically close to each another. For example, when measuring the same tooth, one instrument exported the result as A3,

Table 5: *Significant Differences Among CIE L*a*b* Values of Natural Teeth Measured Using Each Instrument Assessed with Tukey HSD Test*

| | L* | | a* | | b* | |
|---|---|---|---|---|---|---|
| | *p*** | 95% CI | *p*** | 95% CI | *p*** | 95% CI |
| ShadeEye NCC vs VITA Easyshade | <0.001 | (−8.20, −6.99) | <0.001 | (−1.72, −1.14) | <0.001 | (−12.24, −10.40) |
| ShadeEye NCC vs Shadepilot | <0.001 | (−2.16, −0.96) | <0.001 | (−2.79, −2.21) | <0.001 | (−6.17, −4.33) |
| VITA Easyshade vs Shadepilot | <0.001 | (5.44, 6.64) | <0.001 | (−1.36, −0.78) | <0.001 | (5.15, 6.70) |

** *The mean difference is statistically significant when* p< *0.05.*

while the other instrument may have exported the result as A3.5. These minor differences may due to the different L*a*b* values and interpolation algorithms of the instruments.

There are a few limitations with the present study. One limitation is that the measuring units of shade tabs and natural teeth were not repositioned by positioning guides; however, this may have only a slight influence on the results since all the color measuring reliabilities of the shade tabs and natural teeth were high and showed no significant differences. This study did not involve a comparison between instrumental and visual shade selection methodologies. Although instrumental color shade selection has a potential advantage over visual shade selection and is considered to be objective, as determined in the present study, shade measuring and matching results varied among the three instruments. Therefore, visual shade confirmation is still recommended when using the three tested shade matching instruments included in the present study.

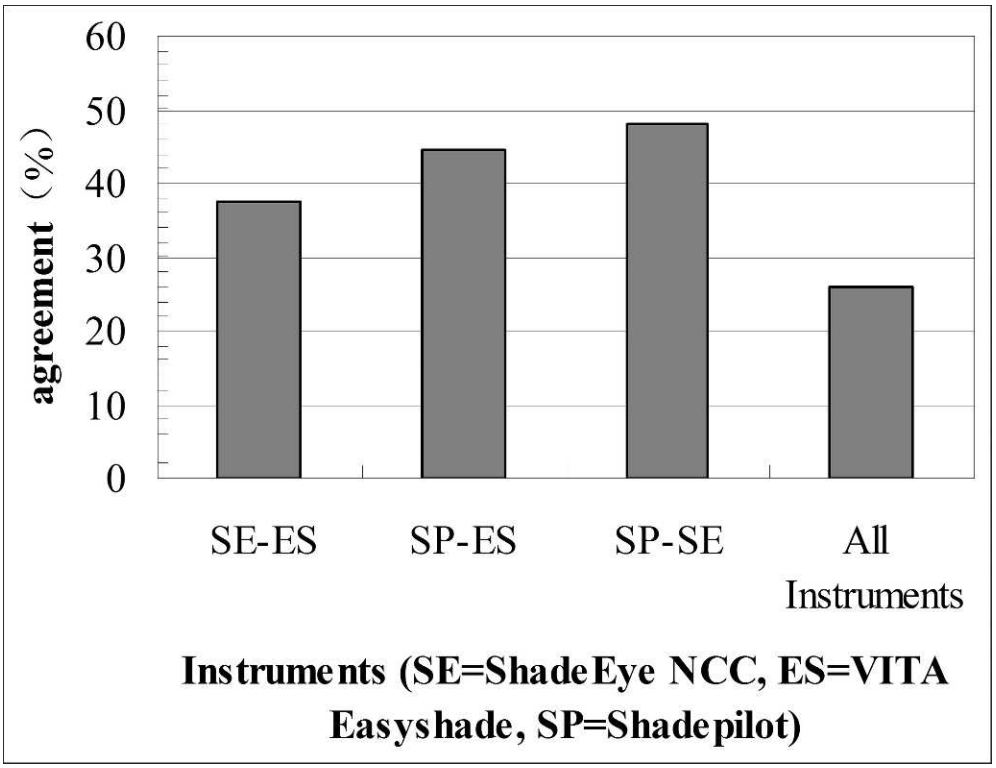


Figure 5. *Pair-agreement rates of* in vivo *natural tooth measurements*

## CONCLUSION

ShadeEye NCC, VITA Easyshade, and Shadepilot showed highly varied accuracy in a range from 32.9% to 87.5% for *in vitro* and *in vivo* models. All instruments had similarly high reliability (>87%). Shadepilot was the only instrument to show both high accuracy and reliability in the *in vitro* and *in vivo* models. VITA Easyshade showed a significantly lower accuracy *in vivo* than *in vitro*. In addition, the L*a*b* values of the same tooth measured by the three instruments were different, and the pair-agreement rates of shade matching results were less than 50%. Therefore, this study highlights the importance of *in vivo* assessment of shade matching instruments as well as the recommendation that color matching instrumentation measurements be combined with visual confirmation in the clinic.

### Conflict of Interest Declaration

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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# Effect of Gender, Experience, and Value on Color Perception

ME Miranda



**Clinical Relevance**

As a result of variations in accurate shade perception, considerations should be given to using multiple opinions to compensate for errors caused by gender or inexperience, especially when lighter shades are involved.


## SUMMARY

**Statement of the Problem: Precise shade matching can be one of the most difficult tasks for the dentist and some variables may influence the process of shade comparison.**

**Purpose of the Study: This study tested the differences in shade perception between genders, the influence of the observer's clinical experience, and the value of ceramics in correct shade selection.**

**Material and Methods: A total of 45 women and 54 men compared 16 pairs of ceramic disks according to shades. The $\chi^2$ and Fisher exact tests were used to analyze the results, adopting 5% as the level of statistical significance. An analysis of risk was also performed to evaluate the variables.**

**Results: The results indicated that there were statistical differences among gender, clinical experience, and shades in discriminating ceramics.**

**Conclusions: Men and observers with more clinical experience were more successful in discriminating shades, although darker shades were selected more correctly than the lighter ones.**


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## INTRODUCTION

Achieving a harmonious smile has been one of the great challenges in restorative treatments, and in the search for excellent esthetics, the target of study by many researchers has been to reproduce the shade of natural teeth. Studies continue to be conducted in the endeavor to simulate natural teeth with restorative materials, and over the years, new possibilities have arisen. Nevertheless, it is still a challenging task to select the ideal shade for an esthetic restoration.

In 1801, Thomas Young learned about the trichromatic nature of human vision, presupposing the hypothesis that this phenomenon would be the result of the eye containing three light-sensitive mechanisms. Today it is known that this mechanism is related to the presence of light-receptor cells in the human retina, the cones and rods, which absorb the light by means of photosensitive pigments and transform it into a stimulus so that the brain perceives the color.[1]

The evaluation of color occurs through the sequential processes of stimulation, sensation, and perception during color detection. These physical and psychological factors interact in order to enable color to be perceived.[2,3] The ability to select a shade varies from one individual to another and in the same individual at different times of visualization.[1,3,4] Due to variables in the environment and different personal evaluations and interpretations, dentists have found it challenging to select and communicate shades.

Shade selection depends on individual evaluation, which is subjective and consequently a clinically difficult process. Therefore, this is one of the reasons for developing standards for communicating shades and instruments that make it easier to measure them.[5]

At present, many methods are used to evaluate tooth shade. The comparison may be visual with the aid of scales, by means of instruments for measuring shade, or by digital image analysis.[6] For the majority of dental professionals, a standard visual scale with a series of values continues to be the most commonly used method of shade selection, rather than computerized methods.[5,7]

Chromatic anomalies are factors that influence correct shade selection, and these defects are more frequently found among men.[8,9] Approximately 8% of all men and 0.5% of all women have some degree of defective color vision.[9] This is one of the reasons women are considered more capable of matching shades than men are.[1,10,11]

It is known that men and women differ in their capacity to distinguish shades,[7,12,13] and traditionally women have been considered better at selecting shades than men.[11,14] The majority of studies have attached little or no importance to the observer's gender at the time of shade selection, even though this is a variable that can influence the end result of the selection process.[8,12,13]

Another variable to consider in shade matching is the observer's clinical experience, which can alter the capacity to reproduce the matching process.

The aim of this study was to investigate the importance of the observer's gender in the selection of shade. In addition, the study evaluated the extent to which the observer's clinical experience,[15-19] the ceramic shade,[20,21] and the value [5,21] had an influence on the choice of shade.

Therefore, the null hypotheses to be tested were as follows: 1) gender is not a factor that significantly influences shade selection in ceramic restorations; 2) the observer's clinical experience is not related to the correct choice of ceramic shade; and 3) the value of the tooth does not influence the dentist's ability to select the ceramic shade.

## METHODS AND MATERIALS

Informed patient consent and ethics commission approval (Application No. 05/058 at São Leopoldo Mandic–School of Dentistry) were obtained for any *in vivo* experiments involved in this research.

A pilot study was conducted with 10 individuals in order to determine the power analysis of the sample size. A total of 99 student volunteers from the dental school were asked to participate in the project: 45 women (11 undergraduate and 34 postgraduate students from the restorative dentistry department) and 56 men (13 undergraduate and 43 postgraduate students). To eliminate possible errors resulting from congenital defects of color visualization, (eg, hereditary disorder linked to X-chromosome), each participant was submitted to a color vision test, using the Ishihara Test for Color Blindness.[22] The exam was performed by a calibrated examiner from the restorative dentistry department before the experiment was conducted, which excluded two men from the sample group, so that 54 individuals remained in the men's group.

Thirty-two ceramic samples (NobelRondo Alumina, Nobel Biocare, Göteborg, Sweden) were manufactured by the same certified dental technician, comprising 16 different shades of body ceramics (A0, A1, A2, A3, A3.5, A4, B0, B1, B2, B3, B4, C1, C2, C3, D2, D3), which were equally duplicated for performing shade matching. The final dimensions of the disks after finishing were approximately 2.9 mm thick and 11.0 mm in diameter.

The participants were asked to match the shades of one set of ceramic disks with those of the other set. The sets were placed on a neutral gray background that had 18% reflectance. No time limit was imposed on the evaluation, but the volunteers were advised of the possibility of fatigue of the

Table 1: *Number of Cases Observed for the Study of the Association Between the Variables of Gender × Choice*

| Gender | Choice | | Total |
|---|---|---|---|
| | Correct | Wrong | |
| Female | 507 | 213 | 720 |
| Male | 654 | 210 | 864 |
| Total | 1161 | 423 | 1584 |

$\chi^2$_: $p$=0.0181
Fisher exact test: $p$=0.0028
Relative risk = 1.2171
Confidence interval = (1.034 to 1.433)

Table 2: *Number of Cases Observed for the Study of the Association Between the Variables of Experience × Choice*

| Experience | Choice | | Total |
|---|---|---|---|
| | Correct | Wrong | |
| Undergraduate: | 238 | 146 | 384 |
| Postgraduate | 923 | 277 | 1200 |
| Total | 1161 | 423 | 1584 |

$\chi^2$_: $p$<0.0001
Fisher exact test: $p$=6.847E-09
Relative risk = 1.6471
Confidence interval = (1.3976 to 1.9411)

retina cells if they stayed fixed on the samples for a long time. All the tests were performed on the premises of the São Leopoldo Mandic Postgraduation Center (Campinas, SP, Brazil) under the clinical lighting conditions that exist at the patient chair (fluorescent and natural light that comes through the window of the room) because it was the type of lighting most commonly found in dental offices and prosthesis laboratories.[23-26]

To analyze the association between the variables, the $\chi^2$ and Fisher exact tests were applied, adopting 5% as the level of statistical significance. Furthermore, an analysis of risk was performed, comparing the risk of wrong choices between the genders, among students with different levels of clinical experience, and between shades by the construction of intervals of confidence at a 95% probability.

## RESULTS

The results are presented in Tables 1, 2, and 3. The results of the statistical analysis revealed that, with regard to gender, there was an association between the variables ($\chi^2$_= 5.5890 with 1 *df*, $p$=1.81%), rejecting the null hypothesis that they are independent. In the analysis of risk, it was verified that, among women, the risk of wrong choice was 29.58% and among men, 24.31% (Table 1). Thus, gender was a factor with a significant influence on choosing the ceramic shade.

A significant difference was found between the experienced observer and the beginners (Table 2). From the $\chi^2$ test results ($\chi^2$= 33.1628, $p$<0.01%) and the Fisher exact test ($p$=6.847$^{-9}$), α dependence among the variables was shown. Thus, it was concluded that there was a significant association and that among undergraduate students, the risk of wrong choice was 38.02% and among the postgraduates, 23.08%.

Evaluating the influence of the shade of ceramic on the frequency of errors in the choice of shades (Table 3), the results of the $\chi^2$ test ($\chi^2$= 51.2835 , $p$<0.01%) showed statistical difference among the values. The Fisher exact test (p<0.01) showed that the frequency of errors rejected the null hypothesis that the variables are independent. In the risk analysis, it was verified that the risk of error in choice for the samples with light shades (A0, A1, A2, B0, B1, B2, C1, C2, D2) was 34.6%, and for the dark shades (A3, A3.5, A4, B3, B4, C3, D3) it was 18.69%,

Table 3: *Number of Cases Observed for the Study of the Association Between the Variables of Light Shade × Dark Shade × Number of Errors*

| Shade | Choice | | Total |
|---|---|---|---|
| | Correct | Wrong | |
| Light | 518 | 274 | 792 |
| Dark | 644 | 148 | 792 |
| Total | 1162 | 422 | 1584 |

$\chi^2$_: $p$<0.01%
Fisher exact test: $p$=2.578E-13
Relative risk = 1.8514
Confidence interval = (1.5557 to 2.2032)

indicating a greater probability of error in selecting the light ones.

## DISCUSSION

Selecting the shade of esthetic restorations is frequently based on visual perception by comparison with a shade guide in relation to the natural tooth, but there is great difficulty in transmitting the information obtained to the laboratory technician. In shade matching routinely done in dental offices, many uncontrolled variables are introduced into the evaluations made by human observers, such as the conditions of the environment, observer, or material evaluated.

In the current experiment, shade matching was performed using a fluorescent light source that, although not ideal, provided a standard necessary for the measurements, something not always possible with daylight. The results of some studies have shown a notable difference in correct shade selection under ideal lighting conditions.[9,23,24] Nevertheless, even the use of the ideal type of lighting does not guarantee precise shade matching, because the environment is not free of the natural light coming through the workroom window and mixing with the artificial light.[3]

The problems of shade perception are more common than one imagines and have different causes and variations. Because color blindness is a hereditary disorder linked to the X chromosome, men are 10 to 20 times more prone to having the deficiency than women,[1] which was also found in the current research, given that two men were excluded from the sample for reasons of daltonism (color blindness) as detected by the Ishihara test. The present data are in agreement with the data described by Barna and others[25] who, when examining several dentists with regard to color visualization defects, found that seven of the 50 participants had a chromatic anomaly. Moser and others[8] in evaluating a group of 670 dentists, verified that 10% had some type of deficiency related to shade perception. This condition affects the ability to discriminate shades, which is in agreement with other studies.[7,8]

Although women are considered more capable than men with regard to shade selection,[10,11,13,26] some studies have demonstrated that there were no statistical differences with regard to the observer's gender in the ability to select color.[20,27,28] The results found in the present research, however, are in conflict with the previously described reports, because male observers obtained a higher frequency of correct results (75.69%) in matching the ceramic shades when compared with the female observers (70.42%). Donahue and others,[12] when working with a similar methodology, found similar results, in which men showed slightly higher (63%) differences in comparison with women (58%) but without any statistical significance. Analyzing the space axes of shade and evaluating the dissimilarities perceived by each gender, Bimler and others[13] concluded that men are less sensitive to stimuli on the green-red axis, but in compensation, are more sensitive along the axis of brightness. Because the value of shade is considered the most critical component of color when matching shades,[26,29,30] this is perhaps one of the reasons for the current results.

Dentists and dental students are required to select the shades of esthetic restorations in their work environment. The question to be discussed is whether these professionals, irrespective of their years of clinical experience, are equally capable of performing this task. According to some studies, the evaluators' years of clinical experience did not positively affect the capacity of shade selection.[7,15,21,25,28] Nevertheless, other studies found that this factor was relevant in the frequency of right choices of ceramic shade, which is in agreement with the findings of this current study.[16-19,31] Experienced clinicians were statistically significantly better at recognizing pairs of equal shades correctly (76.92%) than were the novices (61.98%). Therefore, one can speculate that training is a preponderant factor in correct shade selection.

Analysis of the risk of wrong choice when selecting a light or dark shade was also performed, with verification that the probability of wrongly choosing a light shade was greater. These results are in partial agreement with the data described by Lagouvardos and others,[21] who verified that the shades at the beginning or end of each group evaluated (ceramic scale–Vita Classical, resin composite shade scale–Heraeus Kulzer, and extracted teeth) had more reliable results. The intermediate shades in the scales have little difference between them according to the color values ($\Delta E < 3.3$).[28] Small differences in shade may not be perceived when there are differences below 3.7 units of $\Delta E$, because these were evaluated as being equal in the oral cavity.[32]

A factor that could have affected the results obtained in this research was related to the use of fluorescent light. This could have been replaced by standardized corrected light to verify whether this

parameter is essential in pairing the shades, because in order to discriminate colors, this is known to be essential.[9,19,23-25]

From analysis of the data in the literature and the results of the current study, any type of formula for transmitting data to the laboratory is with error, because there are a series of factors, both biological and technical, that must be considered when shade perception is involved. It is proposed that, irrespective of gender, professionals should consider getting a second opinion in shade selection in order to obtain more reliable results. Given the small number of studies in the literature similar to the present study, particularly with regard to the observer's gender, further investigations on the same subject should be conducted to validate and confirm the conclusions of this analysis.

## CONCLUSIONS

1. Selection of the ceramic shade was influenced by the observer's gender, with the male gender tending to be more successful in discriminating the shades.
2. The participants with more clinical experience had a significantly higher number of correct choices than the novices.
3. For the shades evaluated, it was observed that darker shades were easier to distinguish than the lighter ones.

## CLINICAL IMPLICATIONS

As a result of variations in accurate shade perception, considerations should be given to using multiple opinions to compensate for errors caused by gender or inexperience, especially when lighter shades are involved.

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## Laboratory Research

# Comparative *In Vitro* Validation of VistaProof and DIAGNOdent Pen for Occlusal Caries Detection in Permanent Teeth

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### Clinical Relevance

Laser/light fluorescence devices were highly reliable for occlusal caries diagnosis in permanent teeth but not superior in accuracy to visual methods.


### SUMMARY

**Purpose: Current caries diagnostic tools are neither very accurate nor very reliable for the detection of carious lesions of different depths. Thus, the development of new devices and techniques is needed. The aim of this *in vitro* study was to validate a newer fluorescence device, VistaProof (VP), and compare it with DIAGNOdent Pen (DP), direct visual (DV) and indirect visual methods (IDV), with respect to accuracy and reliability for the detection of occlusal caries in permanent teeth.**

**Methods and Materials: One hundred seven sites on 41 occlusal surfaces of recently extracted premolars were selected and classified into lesion categories according to Ekstrand's clinical criteria, by direct and indirect visual examination. The fluorescence of the sites was also measured by the two devices, and the teeth were ground through the sites for histological evaluation of their lesion depth. One calibrated examiner of high reliability (intraclass correlation coefficient [ICC]>0.85) made all of the evaluations. Sensitivity, specificity, and accuracy of each detection method were estimated based on histological examination as the reference method, estimated using cut-off limits calculated on the basis of best agreement between the devices' values and histological examination. McNemar tests and receiver operating characteristic (ROC) curve**


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**analyses were used to compare the validity measures of all detection methods at $\alpha$=0.05, while the ICC was used to test the reproducibility of the methods based on a second measurement one week after the first.**

**Results: There was no statistically significant difference ($p>0.05$) between the accuracy of DP and VPs for both enamel and dentin lesions. The areas under the ROC curves (AUC) for the two devices were also found not to be different ($p>0.05$). The reliability of DP was statistically significantly better than VP ($p<0.05$).**

**Conclusion: The validity of both fluorescence devices were not found to be significantly different and not better than visual methods for the detection of noncavitated carious lesions.**

## INTRODUCTION

Despite the reduction in the prevalence of tooth decay,[1] the pattern of the reduction is not uniform for all dental surfaces, and the occlusal surface still remains the most susceptible site for caries development.[2,3] The diagnosis of occlusal caries, especially the noncavitated type, is a difficult and problematic task because of the superficial remineralization potential that can delay cavitation and because of the extensive use of fluorides that can slow the progress of the lesion, strengthen occlusal enamel, and mask dentinal caries.[4,5]

In the past decade, numerous studies have been published to improve existing dental caries detection techniques and to seek new noninvasive ones that could quantify the depth of carious lesions.[6-9] New nonharmful devices that measure the fluorescence of the hard dental tissues illuminated by light of specific wavelengths have been developed and recommended as caries detection aids.[10,11]

In 1998, a chairside portable device that uses a diode laser to measure differences in fluorescence intensity between normal and demineralized enamel tissue in smooth and fissured surfaces, DIAGNOdent (DIAGNOdent 2095, KaVo, Bibberach, Germany), was introduced for caries diagnosis. A new model, DIAGNOdent Pen (DIAGNOdent 2190, KaVo, Bibberach, Germany), with smaller-diameter tips has been developed for fissured, smooth, and approximal surface caries detection.[11-13] The device emits a red laser beam at a wavelength of 655 nm (1 mW maximum power) and measures the lesion's fluorescence (at 720-750 nm), produced by microbial metabolic products, the porphyrins.[14] Although the device functions with the same principle as the old one, its main difference is at the tip, which is rotatable around its long axis to facilitate use in approximal areas. A laser light is sent to the tooth through a single sapphire fiber tip, which at the same time collects the reflected light and filters the ambient and the fluorescent light entering the tip. In DIAGNOdent 2095, the light is transported to the angulated tip through a central fiber while fluorescence light is collected through additional fibers that are concentrically arranged around this central fiber. Results of *in vivo*[13,15] and *in vitro*[11,12,16] studies in permanent teeth have shown that this new model has better sensitivity but worse specificity than the previous one on occlusal surfaces and that the validity of the device to detect caries in relation to their lesion depth was very good.

An intraoral fluorescence camera (VistaProof, Dürr Dental, Bietigheim-Bissingen, Germany) that illuminates teeth with a violet light (405 nm) and captures the reflected light as a digital image was recently developed.[17] The reflected light is filtered for light below 495 nm and contains the green-yellow fluorescence of normal teeth with a peak at 510 nm as well as the red fluorescence of bacterial metabolites with a peak at 680 nm. Special software quantifies the green and red components of the reflected light on a scale from 0 to 3 as a ratio of red to green, showing the areas with a higher than healthy tooth ratio. A detailed description of this camera is given by Rodrigues and others.[11]

This device uses the same principle as the QLF device (Inspektor Research Systems BV, EG Amsterdam, The Netherlands) but presents differences from it. The illumination light of the QLF has an average wavelength of 380 nm (290-450 nm), and the reflected light is filtered at 520 nm, allowing only the fluorescence above this wavelength to be recorded. QLF has more sophisticated software that allows the user to select and analyze areas of interest, even those of low autofluorescence (highly light-scattering areas).

These four fluorescence devices (DIAGNOdent 2095, DIAGNOdent 2190, QLF, and VistaProof) have a similar function as they can analyze the fluorescence of porphyrins in bacterial waste. However, the first two collect data for the fluorescence of the lesion directly and in contact with the lesion, while the other two, being cameras, collect data indirectly from the fluorescence image map of the lesion. Several studies have reported on VistaProof,[11,17-23] and three of them compared it with

Table 1: *Criteria Used for Direct (DV) and Indirect (IDV) Visual Examination, Fluorescence Devices, and Histological Examinations*

| Score | DV[a] | IV[a] | DP+ | VPs+ | VPm− | Histology |
|---|---|---|---|---|---|---|
| D0 *Sound* | No caries | No indication of enamel lesion | <9 | <1.3 | <1 | No enamel demineralization |
| D1 *Early enamel caries* | Opacity or discoloration visible after air drying | Opacity or discoloration visible | 9-24 | 1.30 | 1.0-1.49 | Demineralization limited to the outer half of the enamel |
| D2 *Deep enamel caries* | Opacity or discoloration visible without air drying | Opacity or discoloration larger than the fissure width | 25–44 | 1.41 | 1.5-1.99 | Demineralization extending to the inner half of the enamel |
| D3 *Early dentin caries* | Grayish discoloration from the underlying dentin | Grayish discoloration from the underlying dentin | ≥44 | ≥1.59 | 2.0-2.49 | Demineralization involving the outer half of dentin |
| D4 *Deep dentin caries* | Cavitation exposing the dentin beneath | Cavitation exposing the dentin beneath | | | ≥2.5 | Demineralization involving the inner half of dentin |

*+, Cutoffs estimated from the data; −, cutoffs suggested by the manufacturer.*
*[a] Criteria based on the classifications from Ekstrand and others.[6]*

DIAGNOdent.[11,22,23] Studies comparing QLF and DIAGNOdent devices are limited,[24-26] and no study has yet published results on a comparison of VistaProof and QLF.

Only two *in vitro* studies comparing VistaProof to other diagnostic methods have been published.[11,17] For permanent teeth, results showed VistaProof to have similar sensitivity (0.86) to DIAGNOdent Pen (0.78) and visual examination (0.73) but lower specificity (0.63) than all other methods.[11] On primary teeth, the study of De Benedetto and others[23] showed no differences between VistaProof and DIAGNOdent Pen (intraclass correlation coefficient [ICC]vp=0.85, ICCdp=0.85) in intraexaminer reliability. No validity estimations were made in this study. Therefore, more research is needed to clarify its validity in detecting enamel and dentin lesions.

Visual examination is usually used as a control to compare different examination methods.[27,28] Visual inspection aided by magnification has been shown to increase the *in vitro* sensitivity for caries,[29] while indirect visual examination through photographs[30] or digital images[28,31] seemed to have greater potential for the detection of caries.

The aim of this *in vitro* study in permanent teeth was to investigate the reliability and accuracy of VistaProof and DIAGNOdent Pen in enamel and dentin occlusal caries detection and to compare them with that of classical visual methods. The null hypotheses were therefore that there are no differences in the reliability and accuracy of the two devices or between the devices and the control methods for occlusal caries detection in permanent teeth.

## METHODS AND MATERIALS

### Sample Selection and Preparation

Forty-one premolars macroscopically sound or with initial (noncavitated) occlusal caries lesions[1] were selected upon visual inspection with a magnifying loupe 2.5× from a pool of recently extracted human teeth that were stored in tap water from the day of their extraction so that each caries category could be equally represented. Teeth were cleaned with a rubber cup and an air-water syringe and dried for 5 seconds using compressed air, and then the sites were selected carefully to represent lesions according to the criteria of Ekstrand and others[6] (see Table 1). Teeth with open occlusal cavities (D4), hypoplastic fissures, occlusal restorations, occlusal fissure sealants, extensive occlusal staining, and approximal caries close to the marginal ridge were excluded from the sample. Two sites on the same surface but distinctly separate were selected on each upper premolar (pits in proximal grooves) and three on each lower (pits in proximal grooves and the central pit) with the separation being at least 2 mm apart. The teeth yielded a total of 107 examination sites on their occlusal surfaces. Figure 1 shows a lower

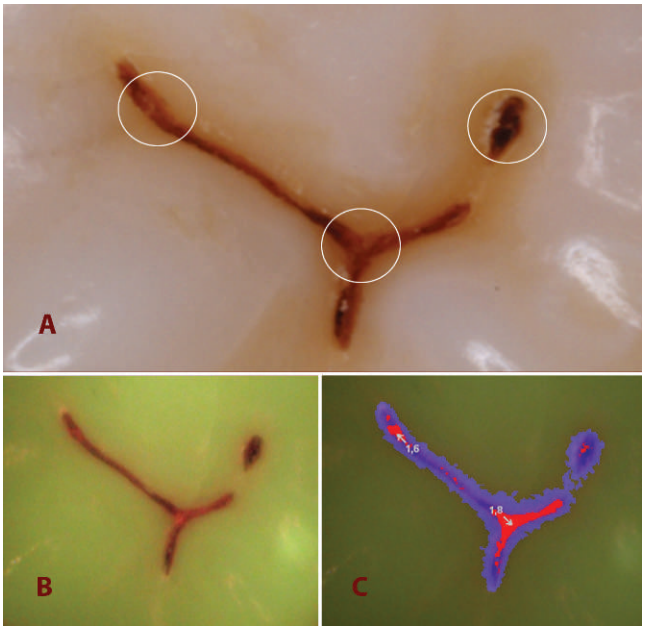


Figure 1. *(A): Original photo image of a second lower premolar with three selected occlusal sites. (B): Fluorescence image of the same occlusal surface. (C): Processed image for fluorescence analysis.*

premolar with three distinctly separate sites on its occlusal surface.

Examination sites on each tooth were recorded in digital photographs (3264 × 2448 pixels) using an Olympus digital camera at a magnification of 2×. A sketch of the surface with the selected areas was drawn on tracing paper for each tooth to assist the accurate histological preparation of the sites.

## Examination Methods

The teeth were continuously stored in tap water and were removed and air dried for two to three seconds for examination. Four different examination methods were employed; two fluorescent and two visual. To reduce bias, calibration of the examiner was performed prior to the examination, each tooth was selected randomly and blindly for each method, and no standard sequence in examination method was followed. The examiner was assisted by a second trained person, who provided the samples.

*Direct Visual Examination, DV (Control 1)*—The selected sites were evaluated visually according to the caries scoring system shown in Table 1, by one calibrated examiner, under standard lighting conditions from a dental unit light and an observation distance of 30 cm, without the use of any visual aids.

*Indirect Visual Examination, IDV (Control 2)*—For the indirect visual examination, digital photographs of all occlusal surfaces were evaluated randomly on a computer screen (1280 × 1024) and scored using the same criteria as for the DV examination (Table 1). The initial photos were taken with an Olympus digital camera (E-500, Olympus Corp., Tokyo, Japan) and an Olympus digital 50-mm macro lens (plus 2× teleconverter) at a magnification of 2× (jpeg, 24-bit color, 3264 × 2448 pixels size) and viewed on a monitor screen (HPL1950) at a magnification of 8×, for a final magnification of 16×.

*DIAGNOdent Pen Device, DP*—The device was used according to the manufacturer's instructions, using the cylindrical tip suitable for occlusal surfaces. Calibration of the device was performed separately for every tooth to obtain precise measurements, Calibration against the reference occurred after every 10 teeth to minimize calibration shift. The tip was placed perpendicularly to the occlusal site and was rotated around its long axis to record the highest value. Three consecutive recordings were taken for each examination site, and their mean value was recorded as the final value for that site.

*VistaProof device, VP*—The device was employed using the long-distance spacer for optimum image quality of the occlusal surface according to the manufacturer's instructions. The optic sensor was facing downward, and the spacer was vertical to the occlusal surface. The video signal was digitized by the software, and a picture of 720 × 576 pixels with a resolution of 72 pixels/inch was created. The software (DBSWIN, Dürr Dental, Bietigheim-Bissingen, Germany) shows the pit and fissure areas that emit fluorescence and quantifies the red and green components of fluorescence (Figure 2). The analyzed pictures were saved to the connected computer, and the corresponding fluorescent values for each site were recorded.

*Histological Examination, HIS (Gold Standard)*—Following laser/light fluorescence measurements, teeth were mounted in transparent acrylic resin blocks covering the whole tooth surface. Each block was ground longitudinally in a buccolingual direction on a polishing machine (ECOMET III grinder, Buehler Ltd, AG, Uzwil, Switzerland) using silicon carbide paper of increasing grit number (wet-or-dry Tri-M-ite: 180- to 1200-grit in sequence) until the first examination site was reached. At the examination site, the specimen was polished using polishing cloths (DP/OP polishing cloths, Struers A/S, Ballerup, Denmark) with α-alumina polishing suspension (5 μm, Struers A/S, Ballerup, Denmark) to achieve a very smooth surface for evaluation. Each polished cut was examined under a stereomicroscope (Leitz Elvar, Esselte Leitz GmbH & Co KG, Stuttgard,

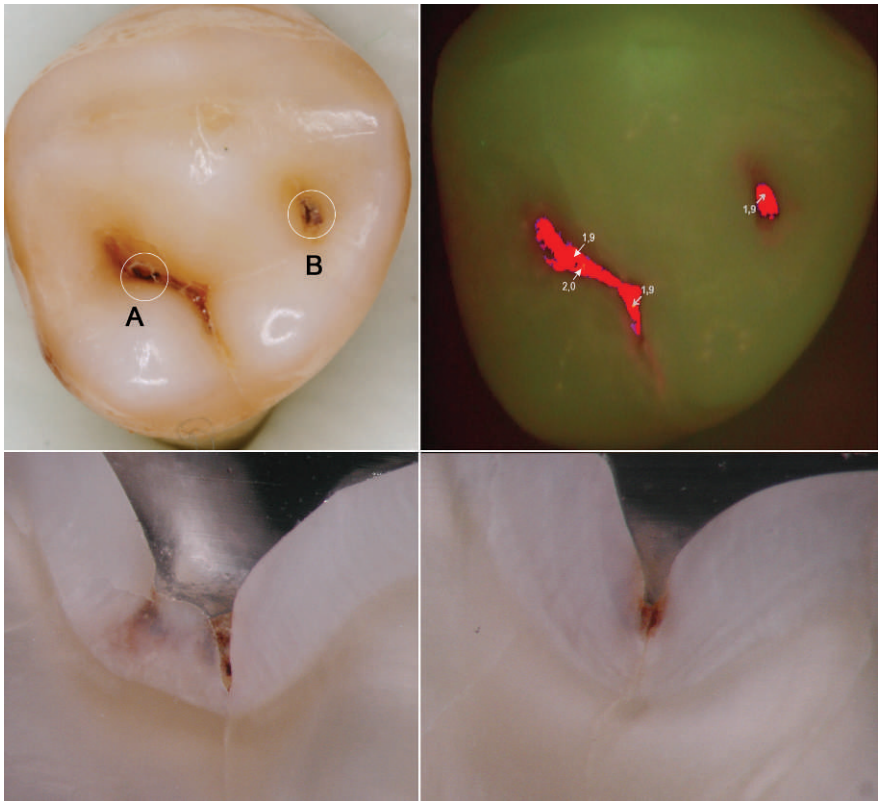


Figure 2. *(Upper left): Original photo image of a first lower premolar with two selected occlusal sites. (Upper right): Processed image of the surface for fluorescence analysis. (Lower left): Histological section of site A. (Lower right): Histological section of site B.*

Germany) at magnifications of 12.5× and 20× and photographed with a digital camera (Coolpix 990, Nikon Corp, Tokyo City, Japan). The depth of the lesions was evaluated microscopically and classified according to a five-grade caries scoring scale by the same single calibrated examiner (Table 1).

*Cutoff Limits*—To estimate the validity of the fluorescence devices, the original values obtained were transformed to a five-grade caries scoring scale as for all other methods. Cutoff points were calculated on the basis of the best agreement (Cohen's Kappa) between histological examination and measured values for each device (Table 1). For the VistaProof device, to determine the cutoffs that best agree with the lesion depth as found histologically, two different cutoffs for enamel and dentin involvement were used: the ones presently calculated (VPs) and those proposed by the manufacturer (VPm; Table 1).

### Statistical Analysis

The validity (sensitivity, specificity, and accuracy) of the VP and DP devices was calculated with the statistical package SPSS version 15 (SPCC Inc, Chicago, IL, USA) using the histological examination as the reference method (gold standard). Differences between the accuracy of the examination methods were detected using McNemar's test at a 0.05 significance level. Comparison of validity among all diagnostic methods was made by estimating the receiver operating characteristic (ROC) curves and the area under the curve (AUC) in MedCalc version 9.0.1.1 (MedCalc Software, Broestraat 52, Mariakerke, Belgium).

Two measurements with all methods, separated by a one-week period, were used to estimate the reliability (reproducibility) of the methods using the ICC.

## RESULTS

### Intraexaminer Reliability

Calibration of the examiner was achieved by blind repeated measurements of 25 sites from teeth not included in the sample. The level of agreement was very good to high for all diagnostic methods.

### Sample Distribution

The distribution of the 107 test sites for each diagnostic method according to their caries scoring

Table 2: *Distribution of the Examination Sites Into the Ekstrand 5-Caries Score Groups According to the Detection Methods*

| Caries Score | DV | IDV | DP | VPm | VPs | HIS |
|---|---|---|---|---|---|---|
| D0 | 23 | 27 | 35 | 5 | 29 | 24 |
| D1 | 31 | 35 | 29 | 50 | 18 | 37 |
| D2 | 31 | 31 | 20 | 48 | 13 | 26 |
| D3 | 22 | 14 | 23 | 4 | 47 | 20 |
| Total | 107 | 107 | 107 | 107 | 107 | 107 |

*Abbreviations: DV, direct visual; IDV, indirect visual; DP, DIAGNOdent Pen; HIS, histological examination; VPm, VistaProof using manufacturer's cutoffs; VPs, VistaProof using calculated cutoffs.*

is presented in Table 2. Regarding deep caries into dentin (D4), only two sites were detected histologically and thus were combined with the D3 lesions and are presented as D3.

### Validity Estimation

*Sensitivity, Specificity, and Accuracy*—Sensitivity, specificity, and accuracy for all diagnostic methods based on histological examination as the reference method are shown in Table 3 and are reported for enamel (D1, D2, D1+D2), for dentin (D3), and for all caries categories (D1+D2+D3).

Comparing the two fluorescence devices for enamel lesions (D1, D2, or D1+D2), VPs had slightly higher numerical accuracy than DP, but for dentin lesions (D3), the opposite was found. However, there was no statistically significant difference ($p>0.05$) between the accuracy of DP and VPs for both enamel and dentin lesions.

Among the different examination methods, the highest accuracy both for enamel and dentin caries was found with IDV. Comparing IDV to the fluorescence devices, IDV showed statistically significantly higher accuracy for dentin lesions (D3; $p<0.05$).

*ROC Analysis*—The AUC for the different examination methods at all caries levels was estimated, and data are shown in Table 4. Figures 3 and 4 show the ROC curves of all methods in detecting enamel (Figure 3) or dentin lesions (Figure 4). All diagnostic methods had a significantly smaller AUC than histological examination ($p<0.001$) at all caries categories. Comparing the other diagnostic methods,

Table 3: *Sensitivity, Specificity, and Accuracy of the Diagnostic Methods, Based on Histological Examination, as the Reference Method*

| Lesion Category | | DV | IDV | DR | VPm | VPs |
|---|---|---|---|---|---|---|
| D1 | sens | 0.459 | 0.514 | 0.432 | 0.568 | 0.351 |
| | spec | 0.800 | 0.771 | 0.814 | 0.586 | 0.929 |
| | accu | 0.682[a] | 0.682 [a] | 0.682 [a] | 0.579 [a] | 0.729 [a] |
| D2 | sens | 0.500 | 0.692 | 0.308 | 0.462 | 0.269 |
| | spec | 0.778 | 0.840 | 0.852 | 0.556 | 0.926 |
| | accu | 0.710 [a] | 0.804 [a] | 0.720 [a] | 0.553 [b] | 0.766 [a] |
| D1+D2 | sens | 0.730 | 0.825 | 0.540 | 0.905 | 0.429 |
| | spec | 0.636 | 0.682 | 0.659 | 0.068 | 0.909 |
| | accu | 0.692 [a] | 0.766 [a] | 0.589 [b] | 0.561 [b] | 0.626 [ab] |
| D3 | sens | 0.750 | 0.650 | 0.550 | 0.000 | 0.950 |
| | spec | 0.919 | 0.989 | 0.862 | 0.954 | 0.678 |
| | accu | 0.888 [ab] | 0.925 [a] | 0.804 [bc] | 0.776 [bc] | 0.729 [c] |
| D1+D2 +D3 | sens | 0.879 | 0.879 | 0.795 | 0.976 | 0.855 |
| | spec | 0.542 | 0.708 | 0.750 | 0.125 | 0.708 |
| | accu | 0.804 [a] | 0.841 [a] | 0.785 [a] | 0.785 [a] | 0.822 [a] |

*Abbreviations: DV, direct visual; IDV: indirect visual; DP: DIAGNOdent Pen; VPm: VistaProof using manufacturer's cutoffs; VPs: VistaProof using calculated cutoffs.*
*Note: Same superscript letters on accuracy indicate no statistically significant difference at $\alpha=0.05$, among accuracy values of the same line, based on MacNemar's test.*

IDV had the highest AUC for enamel lesions, and DV for dentin lesions, mostly but not always statistically significant with the other methods. Regarding the two fluorescence devices (DP and VPs), no statistically significant differences were found between their AUCs at all caries categories ($p>0.05$).

*Comparisons of VP Methods With Different Cutoff Limits*—The validity and the AUC of the VistaProof device based on the two different sets of cutoff limits, those given by the manufacturer (VPm) and those

Table 4: *Comparison of Receiver Operating Characteristic Curves of the Different Methods, Based on Histological Examination*

| Caries Score Diagnostic Methods | AUC | SE | 95% CI LB-UB | z Statistic | *p* Value |
|---|---|---|---|---|---|
| D1 lesion | | | | | |
| DV-HIS | 0.630[a] | 0.058 | 0.531-0.721 | 6.382 | <0.001 |
| IDV-HIS | 0.642[a] | 0.058 | 0.544-0.733 | 6.202 | <0.001 |
| DR-HIS | 0.623[a] | 0.058 | 0.524-0.715 | 6.473 | <0.001 |
| VPm-HIS | 0.577[a] | 0.059 | 0.477-0.672 | 7.174 | <0.001 |
| VPs-HIS | 0.640[a] | 0.058 | 0.541-0.730 | 6.237 | <0.001 |
| D2 lesion | | | | | |
| DV-HIS | 0.639[a] | 0.065 | 0.540-0.730 | 5.538 | <0.001 |
| IDV-HIS | 0.766[a] | 0.059 | 0.674-0.842 | 3.990 | <0.001 |
| DR-HIS | 0.580[b] | 0.066 | 0.480-0.675 | 6.358 | <0.001 |
| VPm-HIS | 0.509[b] | 0.066 | 0.410-0.607 | 7.497 | <0.001 |
| VPs-HIS | 0.598[b] | 0.066 | 0.498-0.691 | 6.101 | <0.001 |
| D1+D2 lesion | | | | | |
| DV-HIS | 0.683[a] | 0.051 | 0.586-0.770 | 6.206 | <0.001 |
| IDV-HIS | 0.754[a] | 0.046 | 0.661-0.832 | 5.344 | <0.001 |
| DR-HIS | 0.599[ab] | 0.055 | 0.500-0.693 | 7.291 | <0.001 |
| VPm-HIS | 0.514[b] | 0.057 | 0.415-0.611 | 8.528 | <0.001 |
| VPs-HIS | 0.669[a] | 0.052 | 0.571-0.757 | 6.386 | <0.001 |
| D3 lesion | | | | | |
| DV-HIS | 0.835[a] | 0.058 | 0.751-0.900 | 2.836 | 0.005 |
| IDV-HIS | 0.819[a] | 0.060 | 0.733-0.887 | 3.001 | 0.003 |
| DR-HIS | 0.706[ab] | 0.070 | 0.610-0.790 | 4.208 | 0.001 |
| VPm-HIS | 0.477[b] | 0.071 | 0.424-0.620 | 6.705 | 0.001 |
| VPs-HIS | 0.814[a] | 0.061 | 0.727-0.883 | 3.055 | 0.002 |

Table 4: Comparison of Receiver Operating Characteristic Curves of the Different Methods, Based on Histological Examination (cont.)

| Caries Score Diagnostic Methods | AUC | SE | 95% CI LB-UB | z Statistic | *p* Value |
|---|---|---|---|---|---|
| D1+2+3 | | | | | |
| DV-HIS | 0.711[a] | 0.055 | 0.615-0.794 | 5.293 | <0.001 |
| IDV-HIS | 0.794[a] | 0.046 | 0.705-0.866 | 4.531 | <0.001 |
| DR-HIS | 0.773[a] | 0.048 | 0.681-0.848 | 4.729 | <0.001 |
| VPm-HIS | 0.550[b] | 0.066 | 0.451-0.647 | 6.857 | <0.001 |
| VPs-HIS | 0.782[a] | 0.047 | 0.692-0.856 | 4.643 | <0.001 |

*Abbreviations: AUC, area under the curve; CI, confidence interval; DV, direct visual; IDV: indirect visual; DP: DIAGNOdent Pen; HIS, histological examination; LB, lower bound; SE, standard error; UB, upper bound; VPm: VistaProof using manufacturer's cutoffs; VPs: VistaProof using calculated cutoffs)*
*Note: The superscript letters above AUC values indicate comparisons among diagnostic methods (vertical values). Same superscript letters indicate no difference at $\alpha$=0.05 level of significance (p>0.05).*

calculated from the data of this study (VPs), are presented in Tables 3 and 4. The accuracy for VPs and VPm was not statistically significantly different for both enamel and dentin lesions. However, the AUC for VPs is statistically significantly greater than VPm for both enamel and dentin lesions. It should also be mentioned that VPm accuracy is misleading, as the VPm sensitivity for dentin lesions was zero. All of the above suggest that cutoffs calculated from the present data may be more accurate than the ones proposed by the manufacturer.

### Reliability-Reproducibility of Diagnostic Methods

Assuming low intraexaminer variation (as demonstrated by the calibration sample), the reproducibility of the diagnostic methods was estimated by the reliability coefficient (ICC) of the two sets of repeated

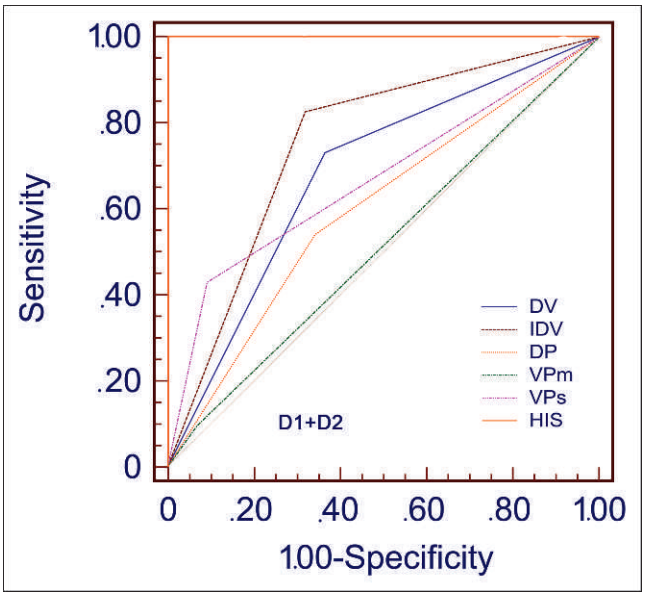


Figure 3. *Receiver operating characteristic curves of the examination methods for the detection of enamel (D1+D2) lesions.*

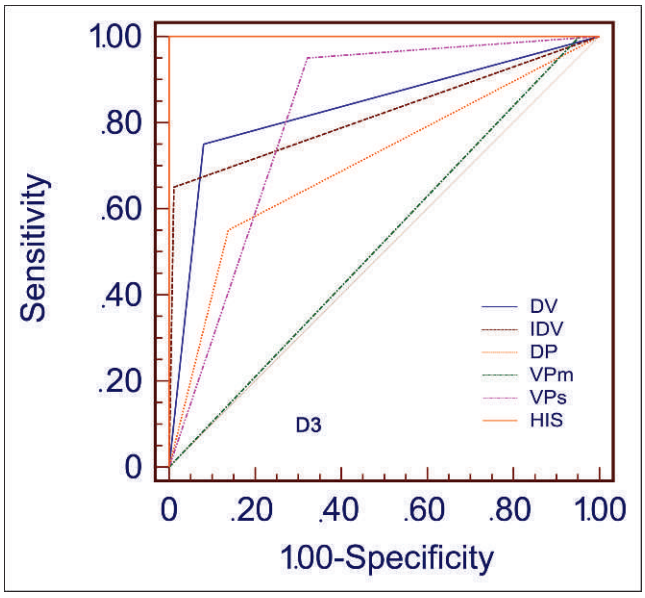


Figure 4. *Receiver operating characteristic curves of the examination methods for the detection of dentin (D3) lesions.*

Table 5: *ICCs of the Diagnostic Methods With the Lower and Upper Bounds of a 95% CI*

| Diagnostic Method | ICC* | 95% CI LB-UB |
|---|---|---|
| DV | 0.996[a] | 0,994-0,997 |
| IDV | 0.988[b] | 0.982-0.992 |
| DR | 0.982[b] | 0.971-0.988 |
| VPm | 0.887[c] | 0.828-0.925 |
| VPs | 0.937[c] | 0.905-0.958 |
| HIS | 0.975[b] | 0.963-0.983 |

*Abbreviations: CI, confidence interval; DV, direct visual; ICC, intraclass correlation coefficient; IDV: indirect visual; DP: DIAGNOdent Pen; HIS, histological examination; LB, lower band; UB, upper bound; VPm: VistaProof using manufacturer's cutoffs; VPs: VistaProof using calculated cutoffs.*
** Average measures of ICC estimated under an absolute agreement definition, and differences were based on nonoverlapping 95% CIs. ICCs with the same superscript letter are not significantly different (p>0.05).*

measurements and was found to be excellent for all examination methods (Table 5), with the most reliable being DV. Comparing the reliability coefficient of the two fluorescence devices, DP was statistically significantly more reliable than VPs.

## DISCUSSION

In this study, the two fluorescence devices were tested in permanent teeth to test, under *in vitro* conditions, their validity and reliability for occlusal caries detection, using histological examination as the gold standard. The accuracy of both devices was not statistically significantly different. DIAGNOdent Pen was, however, more reliable when compared with VistaProof, at a statistically significant level.

Regarding the design of the study, the visual caries scoring system used was based on the one proposed by Ekstrand and others[6] to allow for more comparisons with previous studies.

The selection of cutoff limits for both the DIAGNOdent Pen and VistaProof device for D1, D2, and D3 caries lesions was based on the best K-agreement of the devices' values with those from histological examination, a method previously used in caries studies.[32] The calculated cutoff limits of our study appear slightly higher than the ones used previously, mainly for lesions into dentin (D3), and may be attributed to differences in the cutoff selection method used, teeth storage medium, pit remnants, or drying time before each measurement.[11]

Cutoff limits for the DIAGNOdent Pen have been studied previously[32] and are close to the ones proposed by the manufacturer. For VistaProof, however, cutoffs have not been extensively studied, nor have the ones proposed by the manufacturer been tested.[11] Applying the manufacturer's cutoff limits in the present sample, no dentin lesions could be detected, suggesting that limits should be reevaluated, especially when detecting more advanced lesions. Discrepancies with the manufacturer's cutoffs have been also found by Rodrigues and others,[11] in which the calculated cutoffs for dentin lesions were smaller than the value of 2.0 proposed by the manufacturer. In our study, the cutoffs between D1-D2-D3 lesions have a narrow range, from 1.3 to 1.6 units, which makes proper discrimination between lesion categories difficult; thus, a more refined scale is needed for such discrimination. Until this is accomplished, its use would be more appropriate for discriminating D1 and D3 lesions. Cutoff limits found in this study by comparison to histological sections are thus specific to this sample and cannot be extrapolated to a clinical situation, indicating that further studies are needed to confirm or to reject the above limits. With cutoff limits differing widely between enamel and dentin lesions, clinical usage of fluorescence detection devices using a single compromise value may alter the calculated values and result in reduced accuracy. In clinical situations, this is even more difficult because of plaque presence, different bacteria metabolic by-products, and influence from the environment.

The results of the present study indicated no validity differences between the two devices and for all caries categories, since their accuracy values and AUCs were not statistically significant different. No difference in validity between the two devices was found in a previous study on permanent teeth,[11] although that study was based on only D3 lesions. Comparing all four methods, the present study shows DV/IDV having significantly better accuracy than DP for enamel lesions and DV/IDV having significantly better accuracy than VPs for dentin lesions. These results suggest that fluorescence methods for the detection of occlusal caries in permanent teeth were not superior to, and in certain cases worse than, visual methods. This may be attributed to the effect of stain inclusion in the lesions as Reis and others[33] suggested, resulting in higher measurement values and consequently in higher rates of false-positives with both devices.

Systematic reviews indicated variability in the accuracy of visual[34] and light fluorescence methods.[35] Comparisons of DIAGNOdent with the visual method[35] showed the former to be more specific and less sensitive for detecting occlusal caries. This is evident in our study and consistent with the newer device, DiagnoDent Pen (DP), which also showed greater specificity and lower sensitivity compared with DV. Results of the present study and the lack of homogeneity of evidence that is offered in the systematic reviews[35,36] stress the need for studies with generally accepted standardized methodologies and evaluation criteria.

The high accuracy of indirect visual examination found in this study was also found previously for primary teeth,[28] and this may have clinical implications. Visual diagnostic methods, such as the use of high-resolution intraoral cameras, might be promising for caries diagnosis in the future, especially with the use of a dedicated image analysis software.[31] However, more research is required before a definite conclusion is drawn on their use.

Findings of this study suggest that VP performs better for dentin (high sensitivity) than for enamel lesions (high specificity). This can be explained by the following: the red fluorescence, monitored by the device as caries, is related to a more advanced or deeper carious lesion since this fluorescence comes from microbial metabolic products,[37] expected to exist in greater amounts in larger or deeper cavities. Furthermore, the scattering of fluorescence light in early demineralized enamel areas cannot be quantified by the device so it has a lower performance for early enamel lesions. The red fluorescence has been suggested during demineralization to derive from exposed tooth matrix elements that have interacted with mutans streptococci and have unmasked fluorophores exhibiting strong fluorescence in red[38]. This probably means that the device is not sensitive enough to monitor this red fluorescence, which is scarce in early lesions.

The low sensitivity of VistaProof (VPs) for enamel lesions and its moderate specificity for dentin lesions imply that it can be combined with indirect visual examination to increase its diagnostic validity for the detection of occlusal caries in permanent teeth, since IDV has high sensitivity for enamel and high specificity for dentin lesions. Although the present results cannot be extrapolated to *in vivo* conditions, such a combination could be helpful in clinical situations. A similar combination of IDV with DIAGNOdent has worked well for primary teeth.[39]

DIAGNOdent Pen showed an overall higher specificity and lower sensitivity both for enamel and dentin lesions, suggesting that it might be more useful for the detection of healthy sites. Most of the validity values calculated in this study for DIAGNOdent Pen are within the range of previous studies, except for the sensitivity of enamel lesions, which was found to be much lower.[11,13,15,40] This may be attributed to parameters such as sample selection (eg, normal distribution of the selected teeth in the caries categories and exclusion of teeth with D4 lesions), storage techniques (eg, teeth stored in tap water and not frozen), specimen preparation techniques (eg, only rubber caps for cleaning), reference methods used, and differences in histological evaluation (no stains used).

Reliability of both devices was very good, according to their ICC values. The clinical significance of the above finding suggests that both devices could be used for long-term monitoring of the carious process. Furthermore, DIAGNOdent Pen had a significantly higher ICC value than VistaProof, suggesting that it is more reliable than VistaProof. This finding is in agreement with the study of Rodrigues and others,[11] in which reliability was reported for only D3 lesions.

Results of this study suggest that laser fluorescence, at least *in vitro*, is not superior to the visual methods for occlusal caries detection in permanent teeth. The two fluorescence devices may have different discrimination ability in clinical situations between intact, enamel, or dentin lesions. It is important to point out that although both fluorescence devices have advantages over the commonly used detection methods, they also have limitations. Confounding factors for laser/light fluorescence measurements, such as plaque, stains, and defects in tooth development, were eliminated in this study; however, clinical conditions could not be simulated, underlying the limitations of an *in vitro* study and the need for further research on the performance of the devices *in vivo*.

## CONCLUSIONS

1) There was no statistically significant difference in the accuracy and AUC between DIAGNOdent Pen and VistaProof for both enamel and dentin lesions.
2) The accuracy of direct and indirect vision was significantly higher than DIAGNODent Pen for enamel lesions (D1+D2) and than VistaProof for dentin lesions (D3).
3) Reliability (reproducibility) of both fluorescence devices was excellent, but the DIAGNOdent Pen

was higher than VistaProof. Direct vision presented the highest reliability among all methods and VistaProof the lowest.

4) VistaProof cutoffs proposed in this study (VPs) resulted in a better performance of the device for the detection of enamel and dentin lesions as compared with those proposed by the manufacturer (VPm).

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# Degree of Conversion of Simplified Contemporary Adhesive Systems as Influenced by Extended Air-Activated or Passive Solvent Volatilization Modes

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### Clinical Relevance

Active air-drying for 60 seconds to volatilize solvents can be necessary to increase the degree of conversion of some adhesive systems, which might be related to improved clinical performance.

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**SUMMARY**

**This study evaluated the effect of five methods of solvent volatilization on the degree of conversion (DC) of nine one-bottle adhesive systems using Fourier transform infrared/attenuated total reflectance (FTIR/ATR) analysis. Nine adhesives were tested: Adper Single**

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**Bond 2 (SB), Adper Easy One (EO), One Up Bond F Plus (OUP), One Coat Bond SL (OC), XP Bond (XP), Ambar (AM), Natural Bond (NB), GO, and Stae. The adhesive systems were applied to a zinc-selenide pellet and 1) cured without solvent volatilization, 2) left undisturbed for 10 seconds before curing, 3) left undisturbed for 60 seconds before curing, 4) air-dried with an air stream for 10 seconds before curing, and 5) air-dried with an air stream for 60 seconds before curing. FTIR/ATR spectra were obtained, and the DC was calculated by comparing the aliphatic bonds/reference peaks before and after light activation for 10 seconds (FlashLite 1401). The DC means of each material were analyzed by one-way analysis of variance and post hoc Tukey test ($p<0.05$). The DC of GO and Stae adhesive systems was not affected by the five evaporation conditions. Air-drying for 60 seconds before curing yielded the highest DC for SB, EO, and OC. Extended solvent volatilization time (60 seconds) either with or without air-drying before curing provided the highest DC for AM, NB, XP, and OUP. Thus, the monomer conversion of adhesive systems was material dependent. In general, the 60-second passive or active air-drying modes to volatilize solvents before curing enhanced the degree of conversion for the one-bottle simplified adhesive systems.**

## INTRODUCTION

The development of adhesive systems has completely changed the traditional concepts of dentistry. Today, adhesive systems are widely used in direct procedures for restoration of anterior and posterior cavities, fissure sealing, reattachment of fractured fragments, and corrections in tooth morphology and in indirect procedures involving cementation of root-canal posts and indirect ceramic and composite crowns.[1]

Based on the management of the smear layer substrate, contemporary adhesive systems are categorized as etch-and-rinse (ER) and self-etch (SE) systems. Both bonding strategies are also available in a full or simplified version. When the conditioning step is followed by a priming step and application of the adhesive resin, ER adhesives are available in three steps, or they are available in a two-step procedure when the primer and adhesive resin are joined into one application. Similarly, SE adhesives can employ two steps or a single one, depending on the way the acidic primer and bonding resin are provided by the manufacturer.[2] An immediate consequence of adhesive simplification is sacrifice of the universality of multistep adhesives,[3] and simplified systems are currently preferred by clinicians to perform adhesive procedures because of time savings.

Successful adhesion to hard tissues is a fundamental requirement prior to the placement of dental materials and is directly dependent on the quality of the hybrid layer. Hence, any approach to prolong the lifetime of adhesives might focus on improving the stability of the bonding interface of these biomaterials to tooth tissues.[4] Optimal monomer infiltration into the demineralized substrates and the achievement of a high degree of conversion (DC) are crucial factors in establishing long-lasting bonds.[5] A low DC of dental adhesives is associated with low bond strength values and mechanical properties, high monomer elution, increased permeability, and phase separation.[6-8] Moreover, reduced DC even accounts for the possible continuous etching of the tooth substrate due to suboptimally polymerized acidic monomer in self-etch adhesives.[9] Thus, obtaining a high DC of adhesive systems is a crucial factor in the long-term stability of the hybrid layer.

Simplified dental bonding agents are composed of a mixture of hydrophilic primers and hydrophobic adhesive resins dissolved in acetone, ethanol, water, or some combination of the solvents, which play an important role in the bond performance.[10] Although the presence of solvents makes the process of monomer infiltration easier, the remaining water and organic solvents can greatly inhibit the polymerization reaction and compromise the creation of a well-defined polymer matrix.[11] The use of air spray to accelerate solvent evaporation has been recommended by the manufacturers, and several techniques have been evaluated by researchers. It has been shown that the extended passive solvent volatilization that occurs when adhesive systems are left undisturbed for 60 seconds with or without posterior air-drying for 10 seconds before photo-activation may increase the DC of some commercially available adhesive systems.[12] However, little is known about the DC of contemporary adhesive systems with respect to whether an extended air-activated drying mode to volatilize the solvent should be performed before curing. Thus, this study aimed to evaluate the DC of commercially available adhesive systems when photo-activated after extended air-activated or passive methods of solvent volatilization. The null hypothesis was that there is

no difference in the DC of adhesive systems when photo-activated after different conditions of solvent evaporation.

## MATERIALS AND METHODS

### Experimental Design

Nine one-bottle commercially available adhesive systems with different solvents were tested: Adper Single Bond 2 (SB; 3M ESPE, St Paul, MN, USA), Adper Easy One (EO; 3M ESPE), One Up Bond F Plus (OUP; Tokuyama, Tokyo, Japan), One Coat Bond SL (OC; Coltène/Whaledent, Altsätten, Switzerland), XP Bond (XP; Dentsply/Caulk, Milford, DE, USA), Ambar (AM; FGM, Joinville, SC, Brazil), Natural Bond (NB; DFL, Rio de Janeiro, RJ, Brazil), GO (SDI, Victoria, Australia), and Stae (SDI). Moreover, five modes of solvent volatilization were performed before curing: 1) immediate cure without solvent volatilization, 2) passive solvent volatilization (left undisturbed) for 10 seconds, 3) passive solvent volatilization for 60 seconds (left undisturbed), 4) active solvent volatilization for 10 seconds (with air stream), and 5) active solvent volatilization for 60 seconds (with air stream). The composition, classification, manufacturers, and lot number of all adhesives systems tested are displayed in Table 1.

### DC Analysis

The DC was analyzed by Fourier transform infrared/attenuated total reflectance (Spectrum 100, PerkinElmer, Shelton, XX, USA) at 24°C under 64% relative humidity. One drop of each adhesive system (n=5) was applied to the surface of a zinc selenide pellet (PerkinElmer). Before curing for 10 seconds with an LED light (FlashLite 1401, Discus Dental, Culver City, CA, USA; irradiance at 1100 mW/cm$^2$) positioned 3 mm from the pellet surface, the solvent of each adhesive resin was volatilized in accordance with the aforementioned modes.

The absorption spectra of nonpolymerized and polymerized adhesive resins were obtained from the region between 4000 and 650 cm$^{-1}$ with 32 scans at 4 cm$^{-1}$. For adhesive systems containing aromatic vinyl bonds of bisphenol and aliphatic bonds of the methacrylate functional group (SB, EO, OC, OUP, AM, XP), the aliphatic carbon-to-carbon double-bond absorbance peak intensity (located at 1638 cm$^{-1}$) and that of the aromatic component (located at 1608 cm$^{-1}$; reference peak) were obtained. For Stae and GO, which do not present aromatic dimethacrylates, the intensity of the urethane reference peak (located at 1538 cm$^{-1}$) was obtained. For NB (a TEGDMA-based adhesive), the intensity of the carbonyl reference peak (located at 1716 cm$^{-1}$) was obtained. The DC (%) was calculated using the following equation: DC (%) = 100 × [1 − (R polymerized/R nonpolymerized)], where R represents the ratio between the absorbance peak at 1638 cm$^{-1}$ and 1608 cm$^{-1}$ (for SB, EO, OC, OUP, AM, and XP), 1638 cm$^{-1}$ and 1537 cm$^{-1}$ (for GO and Stae), and 1638 cm$^{-1}$ and 1716 cm$^{-1}$ (for NB).

The data were analyzed by one-way analysis of variance and post hoc Tukey test (only an intrabrand comparison was performed to compare the difference among the tested solvent volatilization modes for each adhesive resin). Statistical significance was established at $\alpha$=0.05.

## RESULTS

The means and standard deviations of the degree of conversion values are presented in Table 2. Only the application of air for 60 seconds yielded a statistically higher degree of conversion for SB, EO, and OC. Both extended times of solvent volatilization (60 seconds; active and passive methods) promoted statistically increased monomer conversion for AM, NB, XP, and OUP. On the other hand, the degree of conversion of GO and Stae was not influenced by the volatilization technique.

## DISCUSSION

The null hypothesis tested in this study was rejected because the degree of conversion of most adhesives was affected by different solvent volatilization methods. Air volatilization for 60 seconds provided the statistically highest monomer conversion for two ethanol-based (SB and EO) and one water-based (OC) adhesive system tested, whereas either air or passive volatilization methods for 60 seconds yielded the highest monomer conversion for other ethanol-based (AM, NB) and water-based (OUP) adhesive systems and for the tertiary-alcohol-based (XP) adhesive system. Nevertheless, none of the tested solvent volatilization techniques affected the monomer conversion of the acetone-based adhesive systems evaluated in the present investigation.

Solvent volatilization can facilitate the polymerization reaction because the distance among monomers is reduced, increasing the degree of conversion.[13] Ideally, solvents should be completely volatilized from the applied mixture prior to polymerization. However, it has been shown that solvents cannot be completely removed from adhesive systems.[14] As water/solvent volatilizes from the

Table 1: *Composition, Manufacturer, and Lots of the Adhesive Systems Used in This Study*

| Adhesive Systems | Composition (% by Weight) | Classification | Manufacturer | Lot No. |
|---|---|---|---|---|
| Adper Single Bond 2 | Ethyl alcohol (25-30), silane treated silica (nanofiller) (10-20), Bis-GMA (10-20), HEMA (5-10), glycerol 1,3-dimethacrylate (5-10), copolymer of acrylic and itaconic acids (5-10), water (<5), diurethane dimethacrylate (1-5) | Two-step E&R | 3M ESPE, St Paul, MN, USA | 8PT |
| Adper Easy One | Bis-GMA (15-25), HEMA (15-25), ethanol (10-15), water (10-15), phosphoric acid-6-methacryloxy-hexylesters (5-15), silane treated silica (8-12), 1,6-hexanediol dimethacrylate (5-10), copolymer of acrylic and itaconic acid (1-5), (dimethylamino)ethyl methacrylate (1-5), camphorquinone (1-3), 2,4,6-trimethylbenzoyldiphenylphosphine oxide (1-3) | One-step SE | 3M ESPE, St Paul, MN, USA | 84020 |
| One Up Bond F Plus | Agent A: Methacryloyloxyalkyl acid phosphate (30-60), MAC-10 (10-30), methyl methacrylate (10-20), Bisphenol A polyethoxy methacrylate (20-40)<br>Agent B: HEMA (30-60), methyl methacrylate (10-30), fluoroaminosilicate glass filler (10-15), borate catalyst (<5), purified water (5-20) | One-step SE | Tukoyama, Tokyo, Japan | 61184 |
| One Coat Bond SL | Methacrylate ([a]), polyalkenoat methacryliert ([a]), water ([a]) | Two-step E&R | Coltène/Whaledent, Altsätten, Switerland | 0173809 |
| XP Bond | Methacrylates (25-50), tert-butyl alcohol (10-25), acrylates (10-25) | Two-step E&R | Dentsply/Caulk, Milford, DE, USA | 17056CB |
| Ambar | UDMA (5-40), HEMA (5-40), methacrylate acidic monomers (1-20), methacrylate hydrophilic monomers (5-40), silanized silicon dioxide (<1), camphorquinone (<1), 4-EDAMB (<1), ethanol (<20) | Two-step E&R | FGM, Joinville, SC, Brazil | 161210 |
| Natural Bond | PMGDM ([a]), TEGDMA ([a]), HEMA ([a]), PHFA ([a]), camphorquinone ([a]), 4-EDAMB ([a]), butyl-hydroxytoluene ([a]), ethanol ([a]) | Two-step E&R | DFL, Rio de Janeiro, RJ, Brazil | 10121648 |
| GO | Acetone (30-50), acrylic monomer (30-50), balance ingredient (non-hazardous) (10-15) | One-step SE | SDI, Victoria, Australia | 164413 |
| Stae | Acetone (54), acrylic monomer (44), balance ingredient (non-hazardous) (2) | Two-step E&R | SDI, Victoria, Australia | 090301 |

*Abbreviations: Bis-GMA, bisphenol A-glycidyl methacrylate; E&R, etch-and-rinse; EDAMB, ethyl 4-dimethylaminobenzoate; HEMA, 2-hydroxyethyl methacrylate; MAC-10, 11-methacryloxy-1,1-undecanedicarboxylic acid; PHFA, potassium hexafluoroantimonate; PMGDM, pyromellitic glycerol dimethacrylate; SE, self-etching; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.*
*[a] Not provided by the manufacturer.*

Table 2: *Degree of Conversion Means (Standard Deviations) of Adhesive Systems According to Volatilization Conditions of Solvents*[a]

| Adhesive Systems | Immediate | 10-Second Passive | 60-Second Passive | 10-Second Active | 60-Second Active |
|---|---|---|---|---|---|
| Adper Single Bond 2 | 75.5 (0.3) C | 75.0 (0.6) C | 77.4 (0.7) B | 77.1 (0.7 ) B | 79.7 (0.2) A |
| Adper Easy One | 48.9 (0.4) B | 48.9 (0.2) B | 49.6 (0.4) B | 50.8 (0.1) B | 55.5 (1.5) A |
| One Up Bond F Plus | 89.9 (0.5) B | 89.5 (0.7) B | 94.6 (0.4) A | 90.4 (0.3) B | 95.7 (0.2) A |
| One Coat Bond SL | 77.5 (0.3) C | 78.5 (0.5) BC | 80.1 (1.0) AB | 79.5 (0.4) BC | 82.2 (0.9) A |
| XP Bond | 52.4 (0.3) B | 58.1 (0.7) B | 67.4 (0.8) A | 66.0 (0.5) AB | 68.8 (0.1) A |
| Ambar | 39.8 (1.7) C | 44.1 (2.0) BC | 57.6 (4.8) A | 50.4 (2.2) AB | 58.2 (3.0) A |
| Natural Bond | 24.3 (2.0) C | 28.9 (3.8) C | 51.6 (1.2) A | 42.6 (2.5) B | 50.8 (0.6) A |
| GO | 82.9 (0.5) A | 82.6 (0.6) A | 83.7 (0.2) A | 82.6 (0.8) A | 83.9 (0.3) A |
| Stae | 80.8 (0.1) A | 80.8 (0.1) A | 75.6 (0.9) A | 79.1 (0.1) A | 79.0 (0.4) A |

[a] *Means followed by different capital letters differ statistically by Tukey test ($p \leq 0.05$). No comparison among the products was performed.*

adhesive, monomer density is found to increase sharply, creating a monomer concentration gradient that acts as a barrier for further solvent evaporation, reducing the ability of water and solvents to volatilize from the adhesive.[15] Thus, clinicians should attempt to remove the highest amount of solvent to achieve an adequate monomer conversion. In fact, a low DC of adhesive systems is associated with low bond strength values and mechanical properties, high monomer elution, increased permeability, and phase separation.[6-8] Moreover, reduced DC even accounts for the possible continuous etching of the tooth substrate due to suboptimally polymerized acidic monomer in self-etch adhesives.[9]

Several factors have been related to the solvent retention in adhesive systems. Solvents with relatively low vapor pressure, such as water, when mixed with nonvolatile monomers, become less able to volatilize as monomer concentration increases.[14] On the other hand, acetone, with a relatively high vapor pressure of 184 mm Hg at 20°C, volatilizes much faster than ethanol or water, with vapor pressure of 43.9 and 17.5 mm Hg, respectively.[16] Also, the extent of solvent retention in polymer networks depends on the resin polarity. The resin polarity influences the number of hydrogen bonding sites and the attraction between the polymer and solvent.[17] The higher the formation of hydrogen bonds is between solvent and monomers, the more difficulty there will be in volatilizing the solvent. Although the solvent type is an essential factor, other ingredients in adhesive systems can influence solvent volatilization and, consequently, the monomer conversion. For these reasons, the different solvent volatilization methods provided statistically different degrees of conversion means for ethanol- and water-based adhesive systems with similar solvents but differing in their chemical components. For SB and EO, it is likely that a greater formation of hydrogen bonds was achieved so that only active air to evaporate the solvent would be sufficient to break them, increasing solvent volatilization and monomer conversion. This might have not occurred for AM, NB, and XP. For these materials, the degree of conversion of the samples whose solvent was volatilized using either active air for 60 seconds or no air application for 60 seconds was similar. Thus, even in the absence of active air, an extended passive method was probably enough to break the hydrogen bonds, increasing the solvent volatilization and degree of conversion means. This assumption also might be attributed to the water-based adhesive systems tested. While OC obtained the highest monomer conversion after only 60 seconds of air application, OUP already had the highest monomer conversion means after the extended passive method

to volatilize the solvent. Although further chemical analyses are necessary to confirm the aforementioned assumptions, the reported results justify the need to evaluate several commercially available adhesive systems in different conditions of solvent volatilization.

Both the acetone-based adhesive systems tested in this study (GO and Stae) presented similar composition, although the components were in different proportions. In addition to the high vapor pressure of acetone, its solubility for hydrogen bonding forces is 7 $(J/cm^3)^{1/2}$, compared with ethanol, for which it is 20 $(J/cm^3)^{1/2}$.[18] That is, the affinity of the carbonyl group of acetone to the hydrogen bond with itself or water or any functional group on monomers that are capable of hydrogen bond formation is only about one-third that of ethanol. This is why acetone is so volatile.[17] Thus, it is likely that the solvent might have been volatilized during the adhesive photoactivation, even without using a solvent volatilization technique. Moreover, the heat generated from the curing light also might have facilitated acetone volatilization, yielding statistically similar degrees of conversion for the samples tested in this study. On the other hand, it should be taken into account that no residual moisture from the wet bonding technique was mixed with adhesive solutions, which could completely alter the solvent retention and monomer conversion.[13,19] Thus, clinicians should be encouraged to volatilize the solvent even when using an acetone-based adhesive system, in particular by using more extended times than those recommended by the manufacturers.

## CONCLUSION

Therefore, the degree of conversion of the adhesive systems tested was material dependent. The ethanol- and water-based adhesive systems tested benefited from extended solvent volatilization time either with or without air application. The acetone-based adhesive systems tested were not influenced by solvent volatilization techniques.

### Conflict of Interest Declaration

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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# Shear Bond Strength of Different Repair Systems to Titanium After Water Aging

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### Clinical Relevance

Among the repair systems evaluated, those which use the tribochemical silica-coating procedure can be considered good options for repairing exposed titanium surfaces. Furthermore, the Cojet system's failure mode and stable behavior after water storage seems to indicate its use for a titanium surface repair technique.

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## SUMMARY

**This study evaluated the shear bond strength (SBS) and stability of commercially pure titanium (CP Ti)/repair material interfaces promoted by different repair systems. One hundred CP Ti cast discs were divided into five repair system groups: 1) Epricord (EP); 2) Bistite II DC (BT); 3) Cojet (CJ); 4) Scotchbond Multi-Purpose Plus (SB) (control group); and 5) Cojet Sand plus Scotchbond Multi-Purpose Plus (CJSB). The specimens were stored in distilled water for 24 hours at 37°C, thermal cycled (5000 cycles, 5°-55°C) and stored under the same conditions for either 24 hours or six months (n=10). SBS was tested and the data were analyzed by two-way analysis of variance (ANOVA) and Tukey test (α=.05). Failure mode was determined with a stereomicroscope (20×). The repair system, storage time, and their interaction significantly affected the SBS ($p<0.001$). At 24 hours, CJSB exhibited the highest SBS value, followed by CJ. At six months, these two groups had similar mean SBS ($p>0.05$) and higher means in comparison to the other groups. For both storage times, BT**

**presented the lowest SBS, while the EP and SB groups did not differ significantly from one another ($p>0.05$). There were no significant differences in SBS between the storage times for the groups EP and CJ ($p>0.05$). The groups BT, SB, and CJSB showed 100% adhesive failure, irrespective of storage time. The CJSB group showed the highest SBS at both storage times. At six months, the CJ group exhibited a similar SBS mean value when compared to the CJSB group. Water storage adversely affected the groups BT, SB (control group), and CJSB. Considering SBS values, stability, and the failure mode simultaneously, the CJ group showed the best CP Ti repair performance.**

## INTRODUCTION

Metal-ceramic tooth- or implant-supported prostheses are still widely used in oral rehabilitations.[1,2] Different metal compositions can be used to manufacture their frameworks, such as NiCr, NiCrTi, AgPd, CoCrMo, and Ti, with clinically satisfactory results. However, fracture or chipping of the ceramic veneer is a potential problem for these restorations,[3] with these occurrences reported as the second most likely cause for their replacement, after dental caries.[4] According to Libby and others,[5] failure resulting from porcelain fracture has been reported to range from 2.3% to 8.0%.

In certain clinical situations, a simple repair technique may reestablish the esthetics and function of a compromised restoration, avoiding the replacement of the fractured metal-ceramic prosthesis, which would increase the cost and time required.[6] Moreover, this procedure is not conservative, possibly increasing the risk of trauma to the tooth during removal of the restoration.[7]

In general, to repair fractured restorations, composite resins are employed. For this purpose, some commercially available composite resins have surface treatment protocols defined by their manufacturers.[8] The goal of these surface treatments is to provide both micromechanical retention and chemical bonding between the composite resin and the substrate.[9,10]

However, when complete deveneering of porcelain results in extensive metal exposure, the repair procedure is a potential clinical challenge,[11] especially in titanium frameworks. Despite its excellent biological and mechanical properties, when the oxide layer of the commercially pure titanium (CP Ti) surface is mechanically removed (by airborne-particle abrasion during a repair procedure), an unstable oxide layer is formed simply by contact with oxygen.[12] This oxide layer restricts the bonding of resin-based materials to titanium;[13] therefore, it is necessary to investigate the efficacy of different materials and their respective protocols on titanium surface repair.

Thus, the purpose of this *in vitro* study was to evaluate early adhesive bonding and stability of CP Ti/repair material interface provided by different repair systems. The null hypothesis to be tested was that all repair systems could provide statistically similar adhesive bonding and durability.

## MATERIALS AND METHODS

One hundred discs (9.0 mm wide and 3.0 mm thick) were cast in CP Ti Grade 2 (RMI Co, Niles, OH, USA) using an Ar-arc casting machine (EDG Equipamentos e Controles Ltda, São Carlos, SP, Brazil). The Rematitan Plus (Dentaurum JP Winkelstroeter KG, Ispringen, Germany) phosphate investment was used according to the manufacturer's instructions. The CP Ti discs were embedded in polyvinyl chloride (PVC) tubes (20.0 mm in diameter and 27.0 mm in length) containing polymethyl methacrylate (PMMA) autopolymerizing acrylic resin (Jet, Artigos Odontológicos Clássico, São Paulo, SP, Brazil). A polisher (Metaserv 2000, Buehler UK Ltd, Coventry, UK) was used to smooth all specimen bonding surfaces with silicon carbide sandpapers (120-, 220-, and 320-grit). The specimens were divided into five groups according to the repair systems (n=20): 1) Epricord (EP); 2) Bistite II DC (BT); 3) Cojet (CJ); 4) Scotchbond Multi-Purpose Plus (SB) (control group); and 5) Cojet Sand plus Scotchbond Multi-Purpose Plus (CJSB). Table 1 summarizes the sequence of materials and procedures used in each repair system group according to the manufacturer's specifications.

Airborne-particle abrasion was performed for 20 seconds with an air abrasion unit (Basic Classic, Renfert GmbH, Hilzingen, Germany), at 0.24 MPa air pressure. For this procedure, the specimens were mounted in a special holder, which allowed a 90° angle and a distance of 10 mm from the surface of the specimen to the blasting tip. All specimens were ultrasonically cleaned in distilled water for 10 minutes.

The bonding agents (metal primer, silane, and adhesive) were applied with a disposable brush in a single layer. The dual-cured resin cement Bistite II DC (paste-paste), used as opaque material, and

Table 1: *Sequence of Materials and Procedures Used in Each Repair System Group*

| Repair Systems (Manufacturer) | Sequence of Material Application |
|---|---|
| Epricord (EP) (Kuraray Co Ltd, Osaka, Japan) | . Airborne-particle abrasion with 50 μm $Al_2O_3$ particles |
| | . Alloy Primer (wait 60 s) |
| | . Epricord Opaque Primer (wait 60 s). Epricord Body Opaque |
| | . Epricord Dentin composite resin |
| Bistite II DC (BT) (Tokuyama Dental Corp, Tokyo, Japan) | . Airborne-particle abrasion with 50 μm $Al_2O_3$ particles |
| | . Bistite II DC resin cement |
| | . Estelite Σ composite resin |
| Cojet (CJ) (3M ESPE AG, Seefeld, Germany) | . Airborne-particle abrasion with 30 μm silica-modified $Al_2O_3$ particles (Cojet Sand) |
| | . Espe-Sil silane (wait 30 s) |
| | . Sinfony opaque |
| | . Visio-Bond adhesive |
| | . Z100 composite resin |
| Scotchbond Multi-Purpose Plus (control group) (SB) (3M ESPE, St Paul, MN, USA) | . Airborne-particle abrasion with 50 μm $Al_2O_3$ particles |
| | . Scotchbond phosphoric etchant by 15 s (rinse and dry) |
| | . Adper Scotchbond Multi-Purpose Plus adhesive |
| | . Masking Agent opaque |
| | . Z100 composite resin |
| Cojet Sand plus Scotchbond Multi-Purpose Plus (CJSB) (3M ESPE, St Paul, MN, USA) | . Airborne-particle abrasion with 30 μm silica-modified $Al_2O_3$ particles (Cojet Sand) |
| | . RelyX Ceramic Primer silane (wait 60 s) |
| | . Adper Scotchbond Multi-Purpose Plus adhesive |
| | . Masking Agent opaque |
| | . Z100 composite resin |

Sinfony opaque (powder-liquid) of Cojet were proportioned by weight and mixed according to the manufacturer's instructions. Opaque agents were applied to the treated CP Ti surfaces using a custom-made metal matrix (4.0 mm in diameter circular aperture and 0.3 mm in thickness), which was placed on the surface of the specimen by attaching a centralizing ring to the PVC tube.

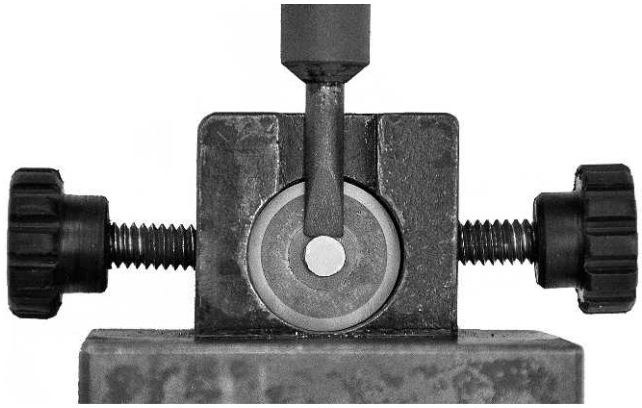

Figure 1. *Shear bond strength test apparatus.*

To apply the composite resins, a custom-made metal split matrix with a circular aperture (4.0-mm internal diameter and 2.0-mm thickness) was positioned on the surface of the specimen using the centralizing ring attached to the PVC tube. The fluid resins of the adhesive systems, Visio-Bond and Adper Scotchbond Multi-Purpose Plus, were light-cured for 20 seconds, while the opacifying agents and the dual-cured resin cement were light-cured for 40 seconds each. The composite resins were polymerized for 60 seconds (40 seconds with the matrix in place and 20 seconds after the metal matrix was removed). This procedure was performed with a visible light-curing unit (Curing Light XL3000, 3M ESPE, St Paul, MN, USA) and an intensity of approximately 550 mW/cm$^2$, which was assessed with the same radiometer (DMC Equipamentos Ltda, São Carlos, SP, Brazil) prior to each use.

After preparation, all specimens were stored in distilled water at 37°C for 24 hours before thermal cycling between 5°C and 55°C for 5000 cycles, with a 30-second dwell time. After thermal cycling, 10 specimens from each group were stored in distilled water at 37°C for 24 hours before the shear bond strengths were determined. The remaining 10 specimens in each group were stored under the same conditions for 6 months to evaluate the stability of the repair systems.

After water storage, using a mechanical testing machine (810 Material Test System, MTS Systems Corp, Eden Prairie, MN, USA), each specimen was placed in a metal apparatus and subjected to a shear bond strength (SBS) test (Figure 1). A knife-edge blade running at a 0.5 mm/min crosshead speed was used to direct a uniaxial compressive load to the specimen as closely as possible to the resin/metal interface until the materials' debonding was observed. The force (N) required to fracture the specimen was divided by the bonding surface area in order to obtain the shear bond strength values (MPa).

Table 2: *Two-way ANOVA*

| Source of Variation | SS | *df* | MS | F | *p* |
|---|---|---|---|---|---|
| Repair system | 2487.16 | 4 | 621.79 | 272.92 | <0.001 |
| Storage time | 183.90 | 1 | 183.90 | 80.72 | <0.001 |
| Repair system × storage time | 33.52 | 4 | 8.38 | 3.68 | <0.01 |
| Residual | 205.05 | 90 | 2.28 | | |
| Total | 2909.63 | 99 | | | |

Each specimen was examined under a stereomicroscope (M80, Leica Microsystems Ltd, Heerbrugg, Switzerland) at 20× magnification, and the digital images were captured and analyzed by imaging software (Leica Application Suite EZ, Leica Microsystems Ltd). A single calibrated observer recorded the failure mode as adhesive failure between titanium and resin, cohesive failure of the resin, or mixed failure (a combination of both). For this classification, the adhesive area was divided into quadrants,[14] and the predominant mode of failure was observed for each one. Failure was classified as adhesive or cohesive if either of these modes were predominate in three or more quadrants, and classified as mixed if two quadrants presented adhesive failure and the other two, cohesive failure.

The data were analyzed by two-way analysis of variance (ANOVA) and when there was a significant difference among the means, the Tukey (HSD) post-hoc test ($\alpha$=0.05) was applied.

## RESULTS

The results from the two-way ANOVA (Table 2) indicated that the repair system ($p<0.001$), storage time ($p<0.001$), and interaction between these variables significantly affected the SBS ($p<0.01$). Table 3 shows the mean SBS values (MPa), standard deviations for each group, and the statistical groupings identified with the Tukey HSD test.

Comparison of the repair systems showed that for 24 hours of storage time, CJSB exhibited the highest SBS value, followed by the CJ group. However, for a

Table 3: *Mean Shear Bond Strength Values (MPa), Standard Deviations (±) and Statistical Results**

| Repair Systems | Storage Times | |
|---|---|---|
| | 24 Hours | Six Months |
| EP | 13.0 ± 2.0 Ac | 11.2 ± 1.3 Ab |
| BT | 8.1 ± 1.2 Ad | 4.8 ± 0.4 Bc |
| CJ | 18.3 ± 2.2 Ab | 17.4 ± 1.9 Aa |
| SB (control group) | 13.4 ± 1.4 Ac | 10.1 ± 1.3 Bb |
| CJSB | 22.7 ± 1.4 Aa | 18.6 ± 1.2 Ba |

** Different uppercase letters indicate significant differences in row ($p<0.05$). Different lowercase letters indicate significant differences in columns ($p<0.05$).*
*Abbreviations: BT, Bistite II DC; CJ, Cojet; CJSB, Cojet Sand plus Scotchbond Multi-Purpose Plus; EP, Epricord; SB, Scotchbond Multi-Purpose Plus.*

storage time of six months, these two groups showed similar mean SBS values ($p>0.05$) and higher values than the other groups. For both storage times, BT presented the lowest SBS, while the EP and SB groups did not differ significantly from one another ($p>0.05$).

The storage time decreased the SBS mean values for the groups BT, SB, and CJSB.

Table 4 lists the predominant failure mode of the studied repair systems at 24 hours and six months of storage. Figures 2 and 3 illustrate the predominant modes of failure observed for each group at 24 hours and six months of storage, respectively.

## DISCUSSION

In the present study, the repair systems CJ, BT and EP were investigated. These systems, considering their different purposes, are also indicated for metal-ceramic prostheses repair. The group SB was used as a control, given that it is a widespread and largely available system in the literature. In the group CJSB, which was proposed to improve the efficacy of the SB group (control group), the steps of airborne-particle abrasion with 50 μm $Al_2O_3$ particles and acid etching (done in SB group) was replaced by silica-modified $Al_2O_3$ particles (Cojet Sand which was used in the Cojet group) followed by silane application (CJSB group).

At 24 hours and six months, the CJSB group exhibited the highest SBS values; however, at six months, the CJ group did not differ significantly from the CJSB group. In these groups, the metallic substrate was abraded with silica-modified $Al_2O_3$ particles (Cojet Sand) and then treated with silane (RelyX Ceramic Primer in the CJSB group and Espe-Sil in the CJ group). Thus, the Cojet Sand particles driven onto the CP Ti surface under pressure provided micromechanical retention and deposition of a silica layer, which causes the surface to be more chemically reactive to the silanes applied afterwards.[15,16] The results of the present study are in agreement with the literature, since this procedure, known as tribochemical silica-coating, has been highly effective for bonding resin-based materials to different substrates.[3,6,17] Lee and others[18] ob-

Table 4: *Percentage of Failure Modes of the Studied Repair Systems*

| Groups | 24 Hours | | | Six Months | | |
|---|---|---|---|---|---|---|
| | Adhesive | Cohesive[a] | Mixed[a] | Adhesive | Cohesive[a] | Mixed[a] |
| EP | 20 | 30 | 50 | 60 | 10 | 30 |
| BT | 100 | — | — | 100 | — | — |
| CJ | 30 | 60 | 10 | 30 | 60 | 10 |
| SB | 100 | — | — | 100 | — | — |
| CJSB | 100 | — | — | 100 | — | — |

*[a] All cohesive failures occurred in the opaque layer.*
*Abbreviations: BT, Bistite II DC; CJ, Cojet; CJSB, Cojet Sand plus Scotchbond Multi-Purpose Plus; EP, Epricord; SB, Scotchbond Multi-Purpose Plus.*

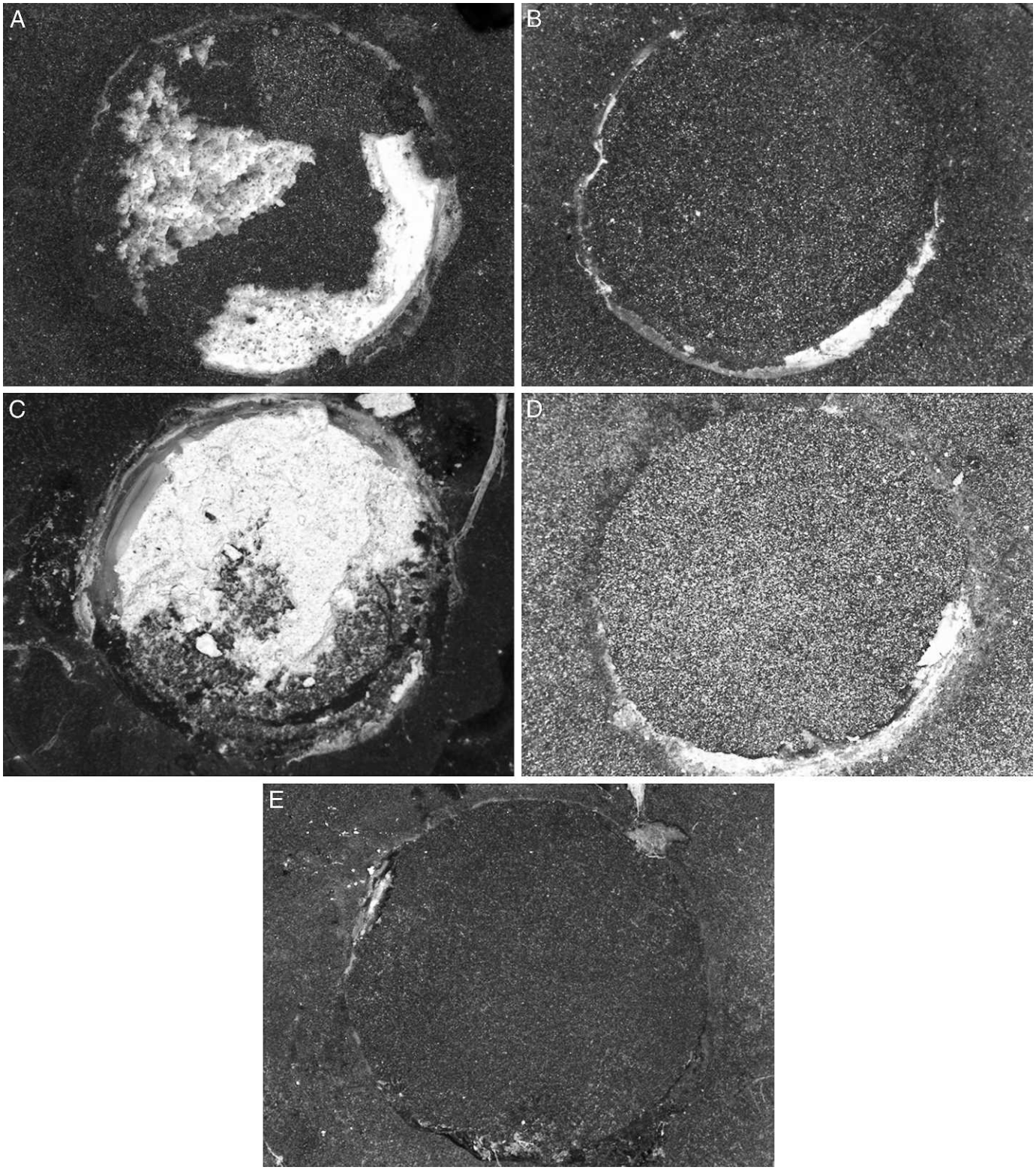


Figure 2. *Microscopic image (20×) of predominant modes of failure observed for each group at 24 hours. (A): Mixed failure - EP group. (B): Adhesive failure - BT group. (C): Cohesive failure - CJ group. (D): Adhesive failure - SB group. (E): Adhesive failure - CJSB group.*

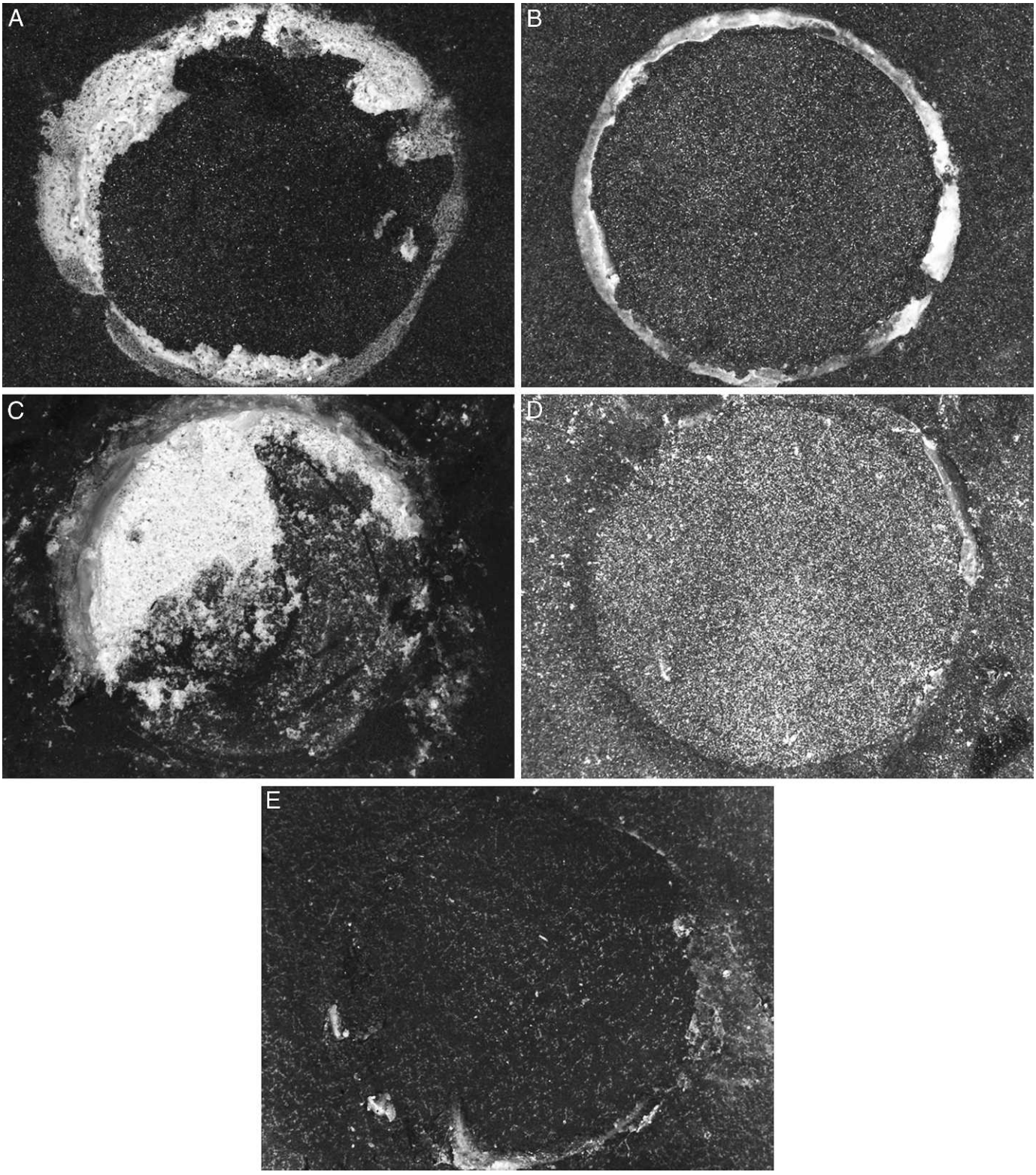


Figure 3. *Microscopic image (20×) of predominant modes of failure observed for each group at six months. (A): Adhesive failure - EP group. (B): Adhesive failure - BT group. (C): Cohesive failure - CJ group. (D): Adhesive failure - SB group. (E): Adhesive failure - CJSB group.*

served higher efficacy of a method used for silica-coating (Rocatec system; 3M ESPE AG) followed by silane Espe-Sil, when compared with airborne-particle abrasion with 250 μm $Al_2O_3$ particles on the bond strength at the Ti-6Al-4V alloy/composite resin interface. Santos and others[14] and Haneda and others[19] evaluated the efficacy of some repair systems used in the present study and found that abrasion with silica-modified $Al_2O_3$ particles followed by silane application promoted the highest SBS values at the interface resin-based material/NiCr alloy.

An important aspect that should be discussed is the difference of the predominant failure mode observed in the CJSB and CJ groups. The CJSB group presented 100% adhesive failure at 24 hours and at six months, while the CJ group exhibited a predominant cohesive failure of the opaque layer at both storage times. This failure mode indicates that the bond strength between the repair system and the CP Ti substrate in the CJ group was higher than the cohesive strength of the opacifying agent (Sinfony). It is important to highlight that the Masking Agent opaque used in the CJSB group is a single paste material and its organic phase is composed of triethylene glycol dimethacrylate (TEGDMA) and bisphenol A diglycidyl ether dimethacrylate (Bis-GMA) monomers, while the Sinfony opaque used in the CJ group is a powder-liquid material and is composed of methyl methacrylate (MMA). Özcan and Kumbuloglu[3] investigated the effect of composition, viscosity and thickness of four opaque agents on the bond strength of composite resin to CP Ti. According to these authors,[3] MMA-based opaque agent seems to adhere better to CP Ti than the one based on TEGDMA. This fact may possibly explain the continuation of SBS mean value at six months in the CJ group. On the other hand, the lower adhesion capacity of the opaque agent based on TEGDMA (CJSB group) may have been responsible for the decrease in SBS value after six months and for the predominance of adhesive failure, at both storage times. Moreover, the authors[3] also comment that MMA monomer is not sufficiently polymerized in the presence of oxygen. This fact may explain the predominance of cohesive failure presented by the CJ group.

The control (SB) and EP groups exhibited similar SBS values at 24 hours and six months. However, these groups provided significantly lower SBS mean values than those of the CJ and CJSB groups and higher values than the BT group. In the EP group, airborne-particle abrasion with $Al_2O_3$ particles was performed, followed by application of Alloy Primer and Epricord Opaque Primer. These metal primers contain the 10-methacryloyloxydecyl dihydrogen phosphate (MDP) adhesive monomer, which is able to establish chemical bonds to metal oxides of base metal surfaces and to copolymerize with the monomers of the resin-based materials. Thompson and others[15] comment that the bond strength promoted by airborne-particle abrasion with $Al_2O_3$ particles followed by application of phosphoric acid primers (MDP) is generally lower than that of a tribochemical silica-coating/silane association.

However, several studies[20-23] indicate that the MDP monomer is effective on the bond between titanium and resinous materials. This observation may explain the fact that at 24 hours the EP group showed 50% mixed failure and 30% cohesive failure in the opaque layer, indicating that the bond between opaque agent and CP Ti provided by both Alloy Primer and Epricord Opaque Primer was more effective than the mechanical strength of the opaque material. On the other hand, although for the EP group there was no significant difference between both storage times, the increase of adhesive failure from 20% to 60% may indicate that a possible hydrolysis of water degradable chemical bonds took place.

In contrast to the other groups, the control group (SB) is based only on micromechanical retention provided by airborne-particle abrasion with $Al_2O_3$ particles, as no material in this group is capable of establishing chemical bonds with the metal substrate. In this group, the adhesive was used after airborne-particle abrasion to increase the wettability of CP Ti by the Masking Agent opaque. Possibly, the absence of chemical bonds between repair material and CP Ti explains the 100% adhesive failure at both storage times and the significant decrease of SBS after six months. Santos and others[14] and Haneda and others[19] also verified, in NiCr alloy, no significant difference between Scotchbond Multi-Purpose Plus and Clearfil SE Bond (Kuraray Co Ltd), whose repair protocol is the same as that of Epricord, which was used in the present study.

Finally, the BT group showed the lowest mean SBS values at both storage times. In this group, the Bistite II DC resin cement is applied to the alumina abraded surface. This cement is used as an opaque material and contains the 11-methacryloyloxundecan 1,1-dicarboxylic acid (MAC-10) monomer, which like the MDP monomer, provides chemical bonds between the repair material and metallic oxides

present in base metal alloys. However, MAC-10 monomer seems to be less effective than MDP monomer, according to some authors.[20,24–27] Moreover, in this group, an intermediate bonding agent was not used, which could increase the metal surface wettability by the resin cement, which presents with an apparent high viscosity. These two factors may explain, for both storage times, the lower performance of this group (BT) when compared to the others, the high predominance of adhesive failure (100%) and, finally, the decrease of SBS after six months. These results were also observed by Santos and others[14] and Haneda and others.[19]

The results of the present study indicate the superiority of using the tribochemical silica-coating system followed by silane application in the production of metal-ceramic prosthesis repairs. The CJSB group proposed in the present study, despite undergoing a significant decrease in SBS at six months, was the only one that presented significantly higher SBS at both storage times. The CJ group, in which abrasion with silica-modified $Al_2O_3$ particles was also performed, exhibited SBS that was statistically similar to the SBS of the CJSB group only at six months. The CJ group did not show a decrease in SBS after water storage, indicating a higher stability in comparison to the CJSB group, which was also confirmed by the predominance of cohesive failure of opaque layer at both storage times.

Further studies to evaluate other factors that could exert a great influence on the bond strength of metal/repair system interfaces, such as long-term water storage and dynamic fatigue loading, as well as long-term clinical studies, should be conducted to establish the reliable behavior of these repair materials under clinical conditions.

## CONCLUSION

Within the limitations of this study, the results indicated that the silica-coated groups (CJ and CJSB) showed the highest SBS mean values at 24 hours and six months, while the BT group showed the lowest SBS. The EP and SB (control group) groups did not differ statistically at both storage times. The BT, SB (control group), and CJSB groups exhibited a decrease in SBS after six months, while the SBS values of the EP and CJ groups were not affected by water storage. Considering SBS values, stability, and failure mode, the CJ group showed the best performance in repairing CP Ti.

*(Accepted 14 October 2011)*

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# Composite Resin to Yttria Stabilized Tetragonal Zirconia Polycrystal Bonding: Comparison of Repair Methods

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### Clinical Relevance

Veneer chipping from yttria stabilized tetragonal zirconia polycrystal (Y-TZP) copings has become a common clinical concern. The present study presents information on the effect of different repair approaches on the bond strength of Y-TZP to a resin composite after aging. Among the assessed repair strategies, tribochemical silica coating provides the highest bond strength.

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## SUMMARY

**Purpose: The purpose of the current study was to evaluate different approaches for bonding composite to the surface of yttria stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics.**

**Methods: One hundred Y-TZP blocks were embedded in acrylic resin, had the free surface polished, and were randomly divided into 10 groups (n=10). The tested repair approaches included four surface treatments: tribochemical silica coating (TBS), methacryloxydecyldihidrogenphosphate (MDP)–containing primer/silane, sandblasting, and metal/zirconia primer. Alcohol cleaning was used as a "no treatment" control. Surface treatment was followed by the application (or lack thereof) of an MDP-containing resin cement liner. Subsequently, a composite resin was applied to the ceramic**

**surface using a cylindrical mold (4-mm diameter). After aging for 60 days in water storage, including 6000 thermal cycles, the specimens were submitted to a shear test. Analysis of variance and the Tukey test were used for statistical analyses (α=0.05).**

**Results: Surface treatment was a statistically significant factor ($F$=85.42; $p$<0.0001). The application of the MDP-containing liner had no effect on bond strength ($p$=0.1017). TBS was the only treatment that had a significantly positive effect on bond strength after aging.**

**Conclusion: Considering the evaluated approaches, TBS seems to be the best surface treatment for Y-TZP composite repairs. The use of an MDP-containing liner between the composite and Y-TZP surfaces is not effective.**

## INTRODUCTION

Yttria stabilized tetragonal zirconia polycrystals (Y-TZP) is a ceramic indicated for crowns and fixed partial denture frameworks as a result of its "high" toughness[1] and the good esthetic results it provides. One of the most common clinical complications involving ceramic restorations with Y-TZP frameworks is veneer chipping.[2] The immediate repair of this kind of failure is important to the well-being of the patient until the restoration can be replaced, if necessary. When the framework is exposed, effective bonding between composite resin and the Y-TZP framework is necessary.[3] However, the bonding of composite to Y-TZP has been shown to be a critical procedure, as indicated by the low bond strength values and reduced bond durability observed in *in vitro* tests.[4-6]

Surface conditioning (by mechanical or chemical methods) has been used to improve the bond strength of resin to the ceramic materials. Acid etching,[3,7-9] sandblasting (SAND),[8] the combination of both,[9] or silica coating,[8,10] followed by the application of silane produced high bond strength values of composite resin to feldspathic ceramics. The use of a resin cement containing methacryloxydecyldihydrogenphosphate (MDP),[4,11,12] SAND,[4,11] the combination of SAND and an MDP-containing primer/silane,[13] or tribochemical silica coating (TBS)[11,14-18] has been reported for bonding enhancement of composite resins to Y-TZP.

The investigation of different strategies for composite to Y-TZP bonding may yield some clinical direction when repair procedures are necessary. Therefore, the aim of this current study was to verify the effect of different repair approaches on the bond strength of Y-TZP to a resin composite. Different surface treatments—TBS, MDP-containing primer/silane system application (MDPS), SAND, metal/zirconia primer application (MZP), and no surface treatment (CRTL)—followed by the application (or lack thereof) of an MDP-containing resin cement liner application (RL), were evaluated. The hypotheses were as follows: 1) silica coating provides the highest bond strength values to Y-TZP among the evaluated surface treatments; 2) an MDP-based cement liner application increases bond strength values to Y-TZP.

## MATERIALS AND METHODS

### Y-TZP Block Preparation

One hundred 7.5 × 7.5 × 2.5–mm blocks of Y-TZP (In Ceram 2000 YZ cubes 40/15, Vita Zahnfabrik, Bad Säckingen, Germany) were cut with a diamond saw (#34570, Microdont, São Paulo, SP, Brazil) under water cooling in a customized machine. After sanding with #400 sandpaper the blocks were sintered in a VITA ZYRcomat furnace (Vita Zahnfabrik). After the recommended sintering cycle, the blocks presented with dimensions of approximately 5 × 5 × 2 mm as a result of the 20% to 25% sintering shrinkage.

The blocks were embedded in acrylic resin so that only the 5 × 5–mm surface was free for bonding. The exposed surface for bonding was then flattened using #400, #600, and #1200 sandpapers. Adhesive tape was placed around the Y-TZP surface of each specimen, leaving a circular 4-mm-diameter area exposed for bonding.

### Experimental Groups

The samples were randomly divided into 10 groups with 10 blocks of Y-TZP in each (n=10). Each group was submitted to one of the following approaches.

1. TBS-RL: Silica coating (TBS) + RL. The Y-TZP bonding area was submitted to tribochemical silica coating (CoJet system, 3M-ESPE, Saint Paul, MN, USA). Initially, the Y-TZP surfaces were air-abraded with 30-ìm–silica-coated alumina particles (COJET SAND, 3M-ESPE) using an intraoral air abrasion device at a pressure of 2.8 bar from a distance of 10 mm for 15 seconds. The conditioned surfaces were then coated with an MPS silane (ESPE Sil, 3M-ESPE) and left to dry at ambient conditions for five minutes. The Y-TZP surface then received a thin layer of dual

cure resin cement containing MDP (Panavia F, Kuraray, Ozaka, Japan). After cement polymerization, a divided cylindrical mold (4-mm diameter and 3-mm depth) was used for the incremental insertion and photocuring of a composite resin (Clearfil Majesty Esthetic, Kuraray) onto the ceramic surface.
2. TBS: The same procedures were used as for group 1, but there was no liner application.
3. MDPS-RL: MDPS + RL. The Y-TZP bonding surface was acid-etched with 37% phosphoric acid, washed, dried, and then had a mixture of Clearfil SE Primer (Kuraray) and Porcelain Bond Activator (Kuraray) applied on its surface. The resin cement liner and composite resin were applied as described for group 1.
4. MDPS: the same procedures were used as for group 3 (MDPS-RL), but with no liner application.
5. SAND-RL: SAND + RL. The Y-TZP bonding surface was submitted to sandblasting with 50-ìm alumina particles using an intraoral air abrasion device at a pressure of 2.8 bar from a distance of 10 mm for 15 seconds. The resin cement liner and composite resin were applied as described for group 1.
6. SAND: The same procedures were used as for group 5 (SAND-RL), but with no liner application.
7. MZP-RL: MZP + RL. An MZP (Ivoclar Vivadent, Schaan, Liechtenstein) was applied on the Y-TZP bonding surface. The resin cement liner and composite resin were applied as described for group 1.
8. MZP: The same procedures were used as for group 7 (MZP-RL), but with no liner application.
9. CRTL-RL: CRTL + RL. The Y-TZP bonding surface was rubbed with 96% isopropanol for 30 seconds. The resin cement liner and composite resin were applied as described for group 1.
10. CRTL: The same procedures were used as for group 9 (CRTL-RL), but with no liner application.

Table 1 shows how the groups were distributed and prepared, according to the different approaches for composite resin to Y-TZP bonding. The chemical descriptions for the materials used in the current study are presented in Table 2.

Table 1: *Experimental Groups Used in the Study*

| Group Description | Identification Code |
|---|---|
| Tribochemical silica coating; MDP-based cement liner | TBS-*RL* |
| Tribochemical silica coating | TBS |
| MDP-containing primer/activator system; MDP-containing cement liner | MDPS-*RL* |
| MDP-containing primer/activator system | MDPS |
| Sandblasting ($Al_2O_3$); MDP-containing cement liner | SAND-*RL* |
| Sandblasting ($Al_2O_3$) | SAND |
| Metal/zirconia primer; MDP-containing cement liner | MZP-*RL* |
| Metal/zirconia primer | MZP |
| No treatment (alcohol cleaning); MDP-containing cement liner | CRTL-*RL* |
| No treatment (alcohol cleaning) | CRTL |

### Water Storage and Thermal Cycling

The specimens were stored in distilled water at 37°C for 60 days. They were submitted to thermal cycling for approximately 103 hours (6000 cycles; 5°C and 55°C baths; 30 seconds each bath; two seconds of transition) while they were water-stored.

### Shear Testing

After storage and thermal cycling, the specimens were submitted to shear testing in the Universal testing machine, EMIC DL-1000 (EMIC, São José dos Pinhais, PR, Brazil). A knife-shaped indenter applied the load at a cross-head speed of 0.5 mm/min. A metal frame was used for holding each specimen to guarantee that the adhesive interface was parallel to the path of the knife and as near as possible to the long axis of the knife. The shear strength was recorded in $N/mm^2$ (MPa). The interfacial area (A) was 12.57 $mm^2$ ($A=\pi r^2$, where $\pi=3.1416$ and r=adhesive interfacial radius=2 mm).

### Failure Mode and Data Analysis

After testing, the specimens were analyzed in an optical microscope (Mitutoyo TM-505, Kanagawa, Japan), at 250× magnification to determine the predominant failure mode classification (adhesive = A; cohesive in ceramic = CC; cohesive in resin =

Table 2: *Chemical Composition and Use of the Materials Used in the Study*

| Brand Mark | Manufacturer | Chemical Components | Use |
|---|---|---|---|
| CoJet sand | 3M ESPE, Saint Paul, MN, USA | Aluminum oxide, amorphous silica | Blasting for silica coating |
| Metal/zirconia primer | Ivoclar Vivadent, Schaan, Liechtenstein | DMA, solvents, phosphonic acid acrylate, initiator and stabilizer | Primer |
| ESPE-Sil | 3M ESPE, Seefeld, Germany | MPS, ethanol, methyl ethyl ketone | Silane coupling agent |
| Clearfil SE Bond Primer | Kuraray Medical Inc, Okayama, Japan | MDP, HEMA, hydrophilic dimethacrylates, DL-camphorquinone, *N,N*-diethanol-*p*-toluidine, $H_2O$ | Self-etching primer |
| Clearfil Porcelain Bond Activator | Kuraray Medical Inc, Okayama, Japan | MPS, bisphenol-*a*-polyethoxy-dimethacrylate | Silane coupling agent |
| Aluminum oxide | — | Aluminum oxide | Sandblasting |
| Alcohol | Sigma Aldrich, Saint Louis, MO, USA | Isopropanol | Cleaning |
| Panavia F 2.0 | Kuraray Medical Inc, Okayama, Japan | Paste A: MDP, DMA, silanated silica, DL-camphorquinone, others<br>Paste B: DMA, silanated barium glass, sodium fluoride, others | Resin cement |

*Abbreviations: DMA, dimethacrylates; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyldihydrogenphosphate; MPS, 3-methacryloyloxypropyl trimethoxysilane.*

CR). The type of failure was confirmed under scanning electronic microscopy (SEM), using selected specimens.

Data were computed and submitted to analysis of variance (two-way ANOVA) and a 5% Tukey *post hoc* test.

## RESULTS

The two-way ANOVA of the bond strength data is summarized in Table 3. Mean values, standard deviations, failure modes (%), and statistical homogeneity are described in Table 4.

There were different effects due to surface treatments ($p<0.0001$). The application of a MDP-containing RL was not related to an improvement in bond strength ($p=0.1017$) (Table 3).

TBS coating (used in groups TBS and TBS-RL) was the most effective method among the tested approaches, resulting in statistically significant higher bond strengths of composite to Y-TZP after thermal cycling and storage. The MDPS group produced intermediate bond strengths, greater than those associated with the SAND, MZP and MZP-RL, and the CRTL and CRTL-RL groups (Figure 1; Table 4).

## DISCUSSION

The shear test is an acceptable alternative for bonding tests when other methods have little viability. In a pilot study, the attempt to produce bar specimens for microtensile bond testing resulted in de-bonding between the composite and Y-TZP during the cutting procedures, which was attributed to the difficulty in cutting the Y-TZP ceramic (excessive vibration, heating and damage to the adhesive interface) and the low bond strength values between the composite and Y-TZP.

The literature[4,6,11,14,15] shows that aging has an important role with regard to the longevity of composite to Y-TZP bonding. Therefore, water storage and thermal cycling were performed in the present study before the shear test in order to simulate oral conditions.

Table 3: *Sources of Variation and Two-Way Analysis of Variance (ANOVA) for the Bond Strength Results (MPa)*

| Sources of Variation | Degrees of Freedom | Sum of Squares | Mean Square | *F*-Value | *p*-Value |
|---|---|---|---|---|---|
| Resin liner | 1 | 17.06 | 17.06 | 2.73 | 0.1017 |
| Surface treatment | 4 | 2130.97 | 532.74 | 85.42 | 0.0000 |
| Resin liner × surface treatment | 4 | 100.84 | 25.21 | 4.04 | 0.0046 |
| Error | 90 | 561.31 | 6.2 | | |
| Total | 99 | 2810.18 | | | |

The use of no-treatment groups (CRTL and CRTL-RL) was necessary so that we had a baseline for the bond strength between an untreated Y-TZP surface and composite under the study conditions. Alcohol cleaning was performed to clean grease residues and other possible contaminants off the Y-TZP surface. "No treatment" resulted in null and very low (2.17-MPa) bond strengths (for the CRTL and CRTL-RL groups, respectively). Similar results had already been reported in earlier studies.[4-6] This indicates the necessity of procedures designed to improve the bond strength between composite resins and Y-TZP. This weak bond strength is attributed to the absence of mechanical interlocking and chemical bonding,[19,20] which can favor water penetration and de-bonding after a certain storage time.[21] However, in a clinical situation, in which the Y-TZP surface has some degree of roughness caused by machining, bonding might not be as weak as indicated in the current results, which were obtained using a polished Y-TZP surface.

MDP presents ester phosphate groups, which supposedly can bond directly to oxides of the ceramic surface and to the methacrylate groups of the composite matrix.[22] Some authors[4,11,12] report good bonding results to Y-TZP surfaces when using resin cements containing MDP. These good results are the reason for the inclusion of the RL and MDPS treatment options in the current study (prior to the application of the composite as a repairing material).

It was expected that the RL would allow both an improvement in bonding to Y-TZP and a chemical bond to the resin composite. However, RL did not have a significant effect on the composite/Y-TZP bonding in all groups. Table 4 shows that when comparing groups with the same treatment with and without RL, all pairs were statistically similar. Lüthy et al.[23] also found a small, but not significant, increase in the bond strength between an MDP-based dual resin cement and Y-TZP. Additionally, MDPS did not have a positive effect on the

Table 4: *Mean and Standard Deviation Values of Bond Strength (MPa) for the Tested Y-TZP–Composite Bonding Approaches, Failure Modes (%), and Homogeneous Groups*

| Surface Treatment | Bond Strength, MPa | Failure Mode, % | Homogeneous Groups[a] |
|---|---|---|---|
| TBS-RL | 13.97 ± 4.58 | 100 CR | A |
| TBS | 11.61 ± 2.12 | 90 CR; 10 A | A |
| MDPS-RL | 2.98 ± 1.32 | 100 A | BC |
| MDPS | 5.75 ± 4.13 | 80 CR; 20 A | B |
| SAND-RL | 3.03 ± 3.02 | 100 CR | BC |
| SAND | 0.65 ± 1.77 | 90 CR; 10 A | C |
| MZP-RL | 0 | 100 A | C |
| MZP | 0 | 100 A | C |
| CRTL-RL | 2.17 ± 2.41 | 100 A | C |
| CRTL | 0 | 100 A | C |

*Abbreviations: A, adhesive failure; CR, cohesive failure into the composite resin; CRTL, "no treatment" control; MDPS, methacryloxydecyldihidrogenphosphate (MDP)–containing primer/silane; MZP, metal/zirconia primer; RL, MDP-containing resin cement liner; SAND, sandblasting; TBS, tribochemical silica coating.*
*[a] Tukey ($p<0.05$): different online small-capital letters indicate statistical difference.*

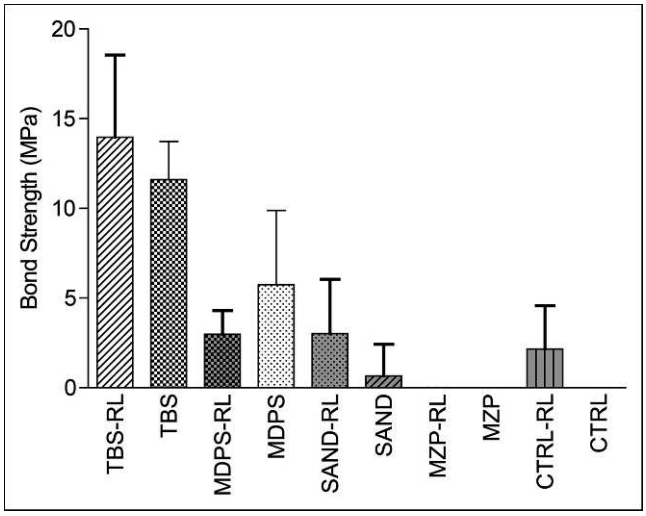


Figure 1. *Mean shear bond strength (MPa) and standard deviation (bars) of composite to Y-TZP for the different bonding approaches.*

composite/Y-TZP bonding. Similar findings were reported by Kern et al.,[24] for whom the application of an MDP-containing primer resulted in spontaneous failure between composite and Y-TZP during 150 days of storage. These findings seem to indicate that any possible reaction between MDP (from cement liner or primer/silane) and Y-TZP oxides is not enough to promote a significant increase in the bond strength. Therefore, the application of RL would only represent one more step in the repairing procedure, without a significant improvement in bonding.

TBS resulted in significantly higher bond strengths of composite to Y-TZP (13.97 MPa and 11.61 MPa for TBS-RL and TBS groups, respectively). Silica coating is a surface treatment in which aluminum-oxide particles modified with silica are blasted under pressure onto the ceramic surface, resulting in the embedding of silica particles on the ceramic surface.[25] The TBS system includes this air abrasion procedure associated with the application of a coupling agent (silane), which may result in chemical bonding between the silica-coated ceramic surface and the resin composite through cross-links with the methacrylate groups. Silane agents may also increase the ceramic surface energy, improving the wettability of the resin.[19,26,27] TBS has been reported to improve the bond strength of resin cements to alumina[25,28] and zirconia[18,25,29] ceramics. When using 90 days of water storage and thermal cycling, May et al.[14] and Passos et al.[15] verified stable and higher bond strength values between resin cements and Y-TZP when TBS was used, but lower or null bond strength values when no TBS was used. These findings substantiate the results found in the current study and indicate that TBS can be very useful for improving composite bonding to Y-TZP–exposed surfaces in cases that require an immediate repair of chipped restorations. The mechanisms likely associated with this enhancement of bonding are the fine surface roughness, enlargement of the bonding area,[26,30,31] and the chemical bond between the silica layer on the Y-TZP surface and the silane agent.[19,21,25,26,30-32] Figure 2c shows a micrograph of the Y-TZP surface after TBS treatment in which a fine complex of embedded silica particles were silanated and are available for micromechanical interlocking and chemical bonding to the composite.

Airborne alumina particle abrasion was not effective in improving the bond strength to Y-TZP (3.03 MPa and 0.65 MPa for SAND-RL and SAND groups, respectively). When comparing sandblasting to TBS for surface treatment of glass-infiltrated and polycrystalline ceramics, Bottino et al.[19] and Valandro et al.[33] found higher bond strength values when TBS was used. However, Wolfart et al.,[4] Oyagüe et al.,[11] Blatz et al.,[12] and Yoshida et al.[13] reported that SAND improved composite bonding to Y-TZP in short-term studies. Casucci et al.[34] showed that sandblasting with 125-μm particles (under 60 to 100 psi) did not alter the Y-TZP surface roughness. In the current study, the alumina particle size was 50 μm. Clearly, there were some topographic changes in the Y-TZP surface, as can be seen in Figure 2b. However, these changes were not significant enough to cause an increase on the bond strength after aging. Kern et al.[24] showed that the isolated treatment with alumina sandblasting (50-μm particle size) caused an immediate increase in bond strength between resin cement and Y-TZP; however, the bond strength decreased to 0 MPa after 150 days of storage. Sandblasting with the 50-μm alumina size seems to have an effect in the short term as a result of surface changes. However, this effect does not seem to be lasting. Lack of chemical bonding and possible water leakage in the adhesive interfaces could be involved.

MZP is a single-component primer that contains a phosphonic acid compound (phosphonic acid acrylate) as the active ingredient. According to the manufacturer's information, MZP establishes a chemical bond to oxidic surfaces, such as metal alloys or oxide ceramics (zirconium oxide, aluminum oxide) and methacrylate-based luting composites. In the current study, the eventual chemical bond was not enough to tolerate the storage/thermal cycling conditions, since all specimens treated with MZP

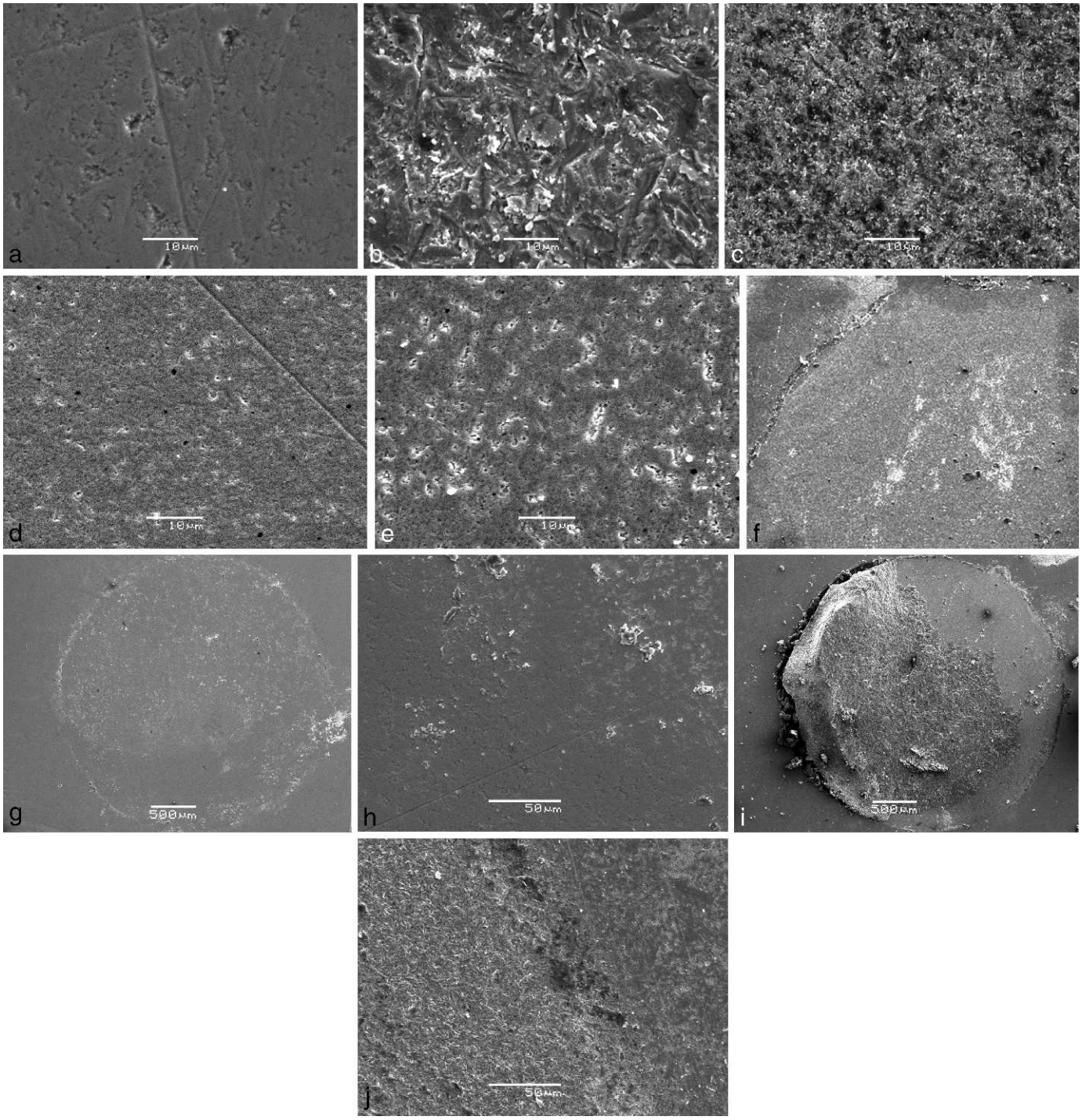


Figure 2. *SEM images: (a) polished Y-TZP surface; (b) sandblasted Y-TZP surface; (c) silica-coated Y-TZP surface; (d) MDPS-treated Y-TZP surface; (e) MZP-treated Y-TZP surface; (g, h) adhesive failure, MZP group; (i) resin cohesive failure, TBS-RL group; (j) resin cohesive failure (left side), TBS group. Dashed line indicates bond boundary.*

presented de-bonding before the shear test, producing null bond strength values for both the MZP and MZP-RL groups.

All surface treatments evaluated in this current study, with or without the application of an RL, with the exception of TBS, presented low or null values for bond strength after thermal cycling and 60-day water storage. These results confirm the weak bond between composite and Y-TZP and seem to indicate that tribochemical silica coating is the best strategy

for promoting bonding when a composite repair procedure is possible and indicated in order to restore the function of a chipped restoration and delay the exposure of the Y-TZP framework to water from the oral environment. In addition to the positive effect of TBS on the bond strength, this treatment seems to not affect the fatigue strength of Y-TZP.[35]

When looking at the failure types in Table 4, adhesive failures occurred mainly in the CRTL and MZP groups. These groups presented a high incidence of spontaneous de-bonding during the storage/thermal cycling period. The TBS, TBS-RL, MDPS, SAND, and SAND-RL groups presented cohesive failures in resin. It is supposed that the higher bond strength values between the Y-TZP surface and the resin liner or the repairing composite increased the possibility of cohesive failure occurrence, typical of a shear test configuration.

The first hypothesis was accepted, as the TBS groups provided the highest bond strengths for composite to Y-TZP. This treatment and MDPS had an effect that was different than that associated with the CRTL groups. However, the liner application had no significant effect on the bond strength between composite and Y-TZP. Therefore, the second hypothesis was rejected.

## CONCLUSIONS

Considering the current study conditions, the use of an MDP-containing cement liner between a composite and Y-TZP surface is not effective and not recommended. Tribochemical silica coating seems to offer the best strategy as a Y-TZP surface treatment for composite repairs when there is an exposure of this ceramic to the oral environment.

### Conflict of Interest Declaration

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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# Proximal Contact Tightness Between Direct-composite Additions in the Posterior Dentition: An *In Vitro* Investigation

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### Clinical Relevance

A novel three-step matrix technique for the application of posterior direct-composite additions was tested *in vitro*. It was proven that by using this technique the reconstruction of proximal contacts and the creation of well-contoured proximal surfaces between direct-composite additions are feasible in an *in vitro* setting. The preclinical testing of this novel technique is necessary to establish the work flow for clinical application and to acquire data for planning *in vivo* investigations.

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## SUMMARY

**Purpose: The aim of the study was to test whether a novel three-step matrix technique for posterior direct-composite additions creates sufficiently strong proximal contacts.**

**Materials and Methods: Contact tightness was measured between direct-composite additions and between original teeth on a model. Therefore, the frictional forces required to remove a straight, 0.05-mm-thick, metal matrix band inserted between adjacent teeth and held by a universal testing machine (Zwicki, Zwick GmbH, Ulm, Germany) were recorded. Measurements were taken at three time points to carry out reference analysis: at baseline, after removal of the maxillary right second premolar (tooth #15) to simulate a diastema, and after closure of the diastema by inserting two direct-composite additions with the three-step matrix**

**technique on the maxillary right first premolar (tooth #14) and first molar (tooth #16). Measurements were performed in the maxillary right (first) and left (second) quadrants to document sagittal displacement.**

**Results: The original contact tightness values were between 1.65 ± 0.88 N and 3.05 ± 0.60 N in the first quadrant and between 1.23 ± 0.51 N and 2.18 ± 0.43 N in the second quadrant. After removal of tooth 15, values decreased significantly in the first quadrant and insignificantly in the second. After reconstruction, the contact tightness between teeth 14 and 16 was significantly stronger (tighter) (3.20 ± 0.80 N) than the originally measured contact tightness between teeth 14 and 15 (2.86 ± 0.64 N) and teeth 15 and 16 (1.65 ± 0.88 N) ($p$=0.006 and 0.001, respectively).**

**Conclusions: Within the limitations of an *in vitro* investigation, this study has shown that by using a novel, three-step matrix technique, direct posterior composite additions can form sufficiently tight proximal contacts.**

## INTRODUCTION

Today, single missing teeth are generally replaced by conventional fixed prostheses or implants. In recent years, additional treatment options, such as direct-composite additions,[1-4] have been introduced whereby a diastema can be closed by adding composite resin on the proximal surface of one or both adjacent teeth. In addition, this technique facilitates correction of malformed or misaligned teeth.

In the course of steady advancement in the application of composite resin techniques, these restorations have become increasingly useful.[3] The development of specific matrix techniques for insertion of direct-composite additions to anterior teeth has greatly helped optimize functional and esthetic results. The proximal forming technique,[5] for example, involves the application of a translucent matrix band, which is fixed cervically and interproximally, helping to create a natural proximal tooth form. The clinical success of anterior direct-composite additions has been reported in three clinical studies[1,2,6] that offer promising long-term results.

The closing of diastemas in the posterior area was described by Vest as early as 1951.[7] In order to close a 4-mm diastema, Vest placed full crowns on the adjacent teeth, expanding them 2 mm into the gap. Staehle[3] refined the technique by substituting the crowns with direct-composite additions using a three-step matrix technique.[8] This novel concept (the step-by-step procedure is described below) included the successive application of straight circumferential matrices (AutoMatrix®, Dentsply DeTrey GmbH, Konstanz, Germany) and a sectional, contoured matrix (Palodent®, Dentsply DeTrey). The choice of matrix types has evolved from clinical practice over the years (see the example of a clinical case with a 7.5-year follow-up period; Figure 1). Standardizing this process permitted the fabrication of well-contoured composite additions without proximal resin overhangs or gaps between tooth and restoration. Furthermore, wedging, applying a separating ring, and additional separation by hand made the creation of strong contact tightness possible, allowing the patient to avoid subsequent food impaction or periodontal breakdown.[9,10]

Large composite additions must withstand occlusal loads on the marginal ridges, especially in cases where a wide diastema of several millimeters must be bridged, making support by a well-contoured proximal segment necessary. It has been shown[11] that on Class II restorations, marginal ridges can be loaded significantly higher when supported by convex, contoured proximal segments. However, to our knowledge, the size of such a resistant proximal segment remains undefined. Therefore, we aimed at forming the direct-composite additions to such an extent that the proximal contour was as convex as possible, leaving enough space for the interdental papilla and for interdental cleaning procedures.

Experimental evidence about the advantages of this novel technique—that is, creation of tight contacts and good proximal shape of the restorations—is still lacking. Therefore, the aim of this study was to test whether posterior direct-composite additions inserted with the three-step matrix technique can reestablish a proximal contact tightness similar to that measured between the model teeth of the intact KaVo model. Additionally, data on changes in contact tightness between adjacent and contralateral teeth at different time points were assessed to describe the effect of a simulated missing tooth and of a reestablished proximal contact on the displacement of teeth within the dental arch. Our hypothesis was that posterior direct-composite additions inserted with the three-step matrix technique could form well-contoured proximal contacts that are as tight as the original contacts on the model.

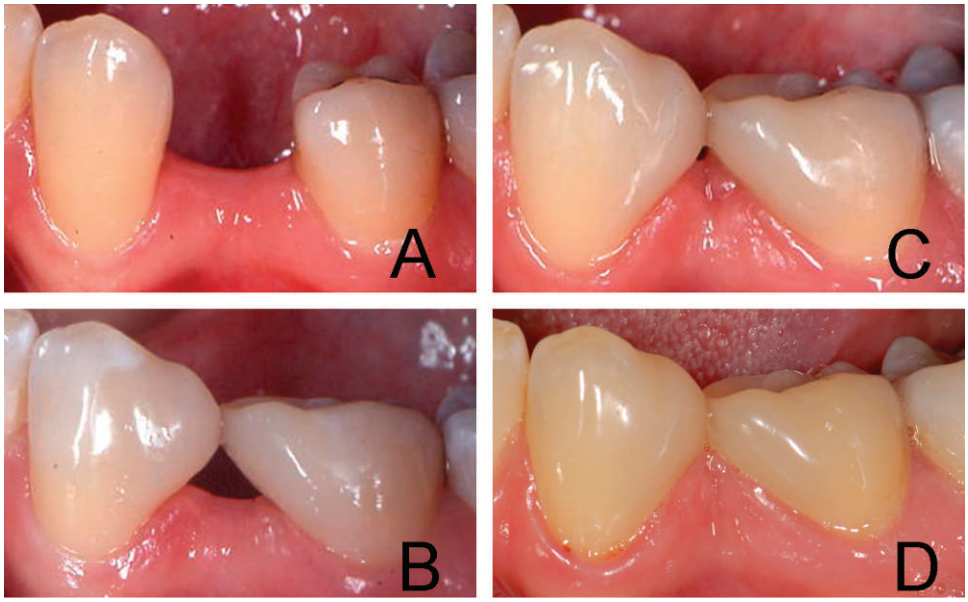


Figure 1. *Clinical case showing direct-composite additions on the mandibular left canine and second premolar. (A): Baseline situation; (B): clinical situation directly after insertion of the direct-composite additions; (C): clinical situation at recall appointment six months after treatment; and (D): 7.5 years after treatment.*

## MATERIALS AND METHODS

In order to standardize the restorative procedure and to simulate the clinical situation, an intraoral model for preclinical student exercise courses (KaVo Basic Model, KaVo Dental GmbH, Biberach, Germany) was used. In this setup, the artificial teeth are clicked into the model, and they display reproducible tooth mobility corresponding to a physiological range (internal data). For the experiments, the maxillary right first (tooth #14) and second (tooth #15) premolars and first molar (tooth #16) were selected. The definition of the terms "proximal contact" and "proximal contact tightness" can be found in Table 1. To measure the proximal contact tightness, the model was mounted in a custom-made, highly reproducible setup that allowed for standardized measurement of all contact areas of interest. Proximal contact tightness was measured using a universal testing machine (Zwicki, Zwick GmbH, Ulm, Germany). A straight metal matrix (0.05 mm thick; Hawe Tofflemire, KerrHawe, Bioggio, Switzerland) was inserted interdentally from the occlusal direction and pulled out by the machine (50 mm/min; Figure 2). To avoid false measurements or artifacts due to deformation or nonparallel removal, the matrix was positioned free of tension in the interproximal area 1 mm above the artificial gingiva. The tightness of the proximal contact was quantified as the maximum frictional force [$F_{max}$ (N); Figure 3] exerted during vertical removal of the matrix.[12]

Measurements were carried out in the first quadrant between tooth pairs 13/14, 14/15, 15/16,

Table 1: *Definition of Terms*

| Term | Definition |
|---|---|
| Proximal contact | The area where adjacent teeth touch each other |
| Proximal contact tightness | The maximum frictional force [$F_{max}$, (N)] exerted during vertical removal of a straight metal matrix |

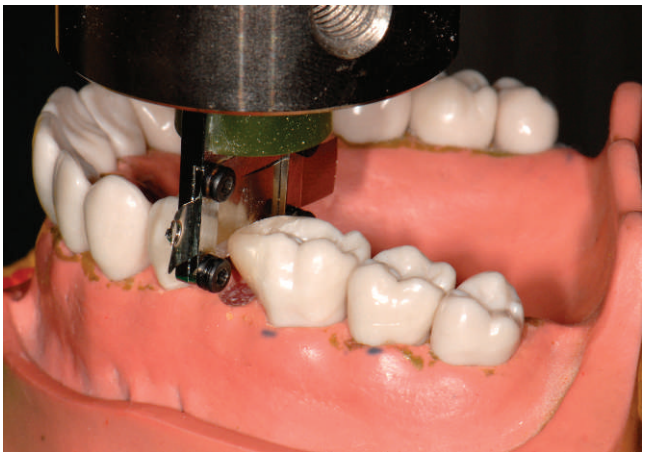

Figure 2. *Measuring contact tightness between direct-composite additions on teeth 14 and 16 with a universal testing machine (Zwicki, Zwick GmbH, Ulm, Germany).*

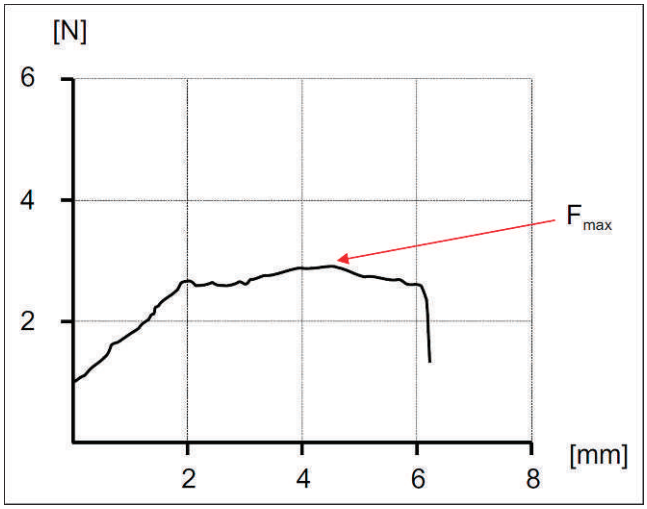


Figure 3. *Graph produced by the universal testing machine showing distance-force relation. The maximum force ($F_{max}$) is recorded as contact tightness.*

and 16/17; in the second quadrant, measurements were carried out between tooth pairs 23/24, 24/25, 25/26, and 26/27 (Figure 4). Baseline data were marked with the suffix "_1." Subsequently, tooth 15 was removed, and the measurements were carried out again. The data were labeled "_2" (Figure 5). After insertion of the direct-composite additions on teeth 14 and 16 following the three-step matrix technique (standardized protocol described by Staehle[13]; short description below), measurements of contact tightness were repeated. Final data were labeled "_3" (Figure 6).

Since no previous data on contact tightness of composite resin additions were available, this investigation was planned as a pilot study with a sample size of n = 30. Descriptive data analysis (precondition: normal distribution) was used to determine the arithmetic mean (Mean) and standard deviation (SD) of contact tightness at different locations and at the time points "_1," "_2," and "_3." Differences between contact tightness at corresponding locations were tested for significance with a paired *t*-test (two-sided significance; $p=0.05$).

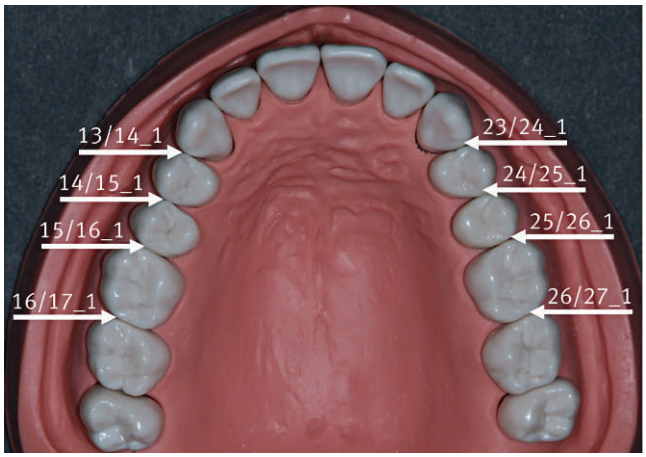


Figure 4. *Model with indicated baseline measurement points.*

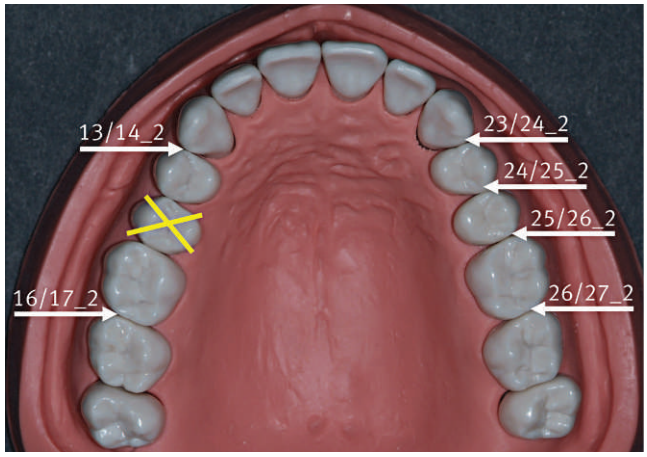


Figure 5. *Model with indicated measurement points after removal of tooth 15.*

### Three-Step Matrix Technique

*Prearrangement*—The prearrangement steps included the following:

- Application of rubber dam
- Proximal adhesive surfaces were cleaned, roughened, and prepared for treatment with an adhesive system (Optibond FL®, KerrHawe).

*First Step*—The first step comprised the following:

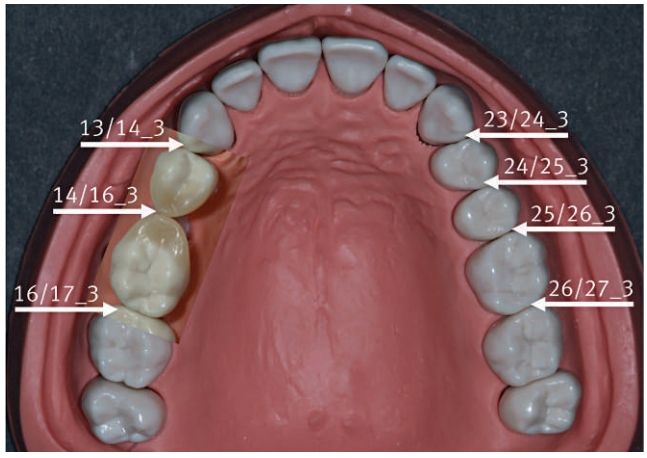


Figure 6. *Model with indicated measurement points after insertion of composite additions on teeth 14 and 16.*

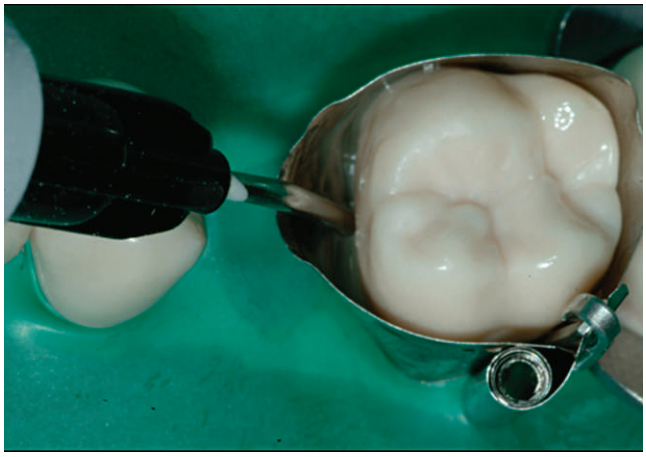

Figure 7. *Three-step matrix technique. First step: A straight circumferential matrix is fixed around tooth 16, "bulged out" toward tooth 14, and the first layer of flowable composite resin is applied.*

- A straight circumferential matrix (AutoMatrix®, Dentsply DeTrey) was applied on tooth 16 and "bulged out" toward tooth 14 using hand instruments. The cervical part of the restoration was built up with a layer of flowable composite resin (Figure 7) and a layer of viscous composite resin. With this technique, a gap-less transition from tooth to restoration was created.
- After removal of the matrix, the restoration was layered by hand with viscous composite resin (Figure 8) and polished.
- At this point, the interspace between teeth 14 and 16 had been reduced to half its original size (Figure 8).

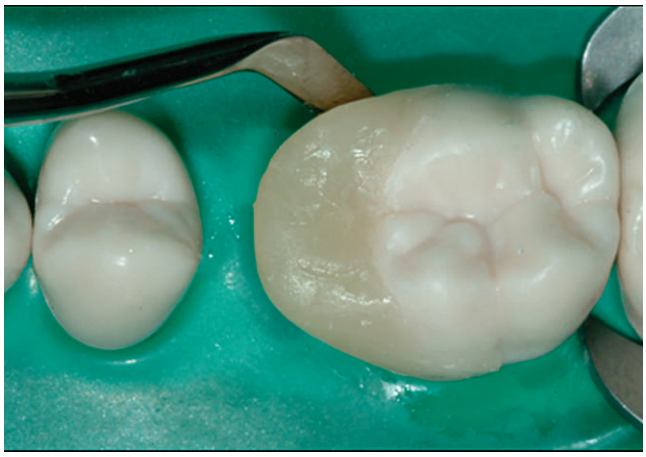

Figure 8. *Three-step matrix technique. First step (continued): Direct composite is layered using viscous resin, filling half the interspace between teeth 14 and 16.*

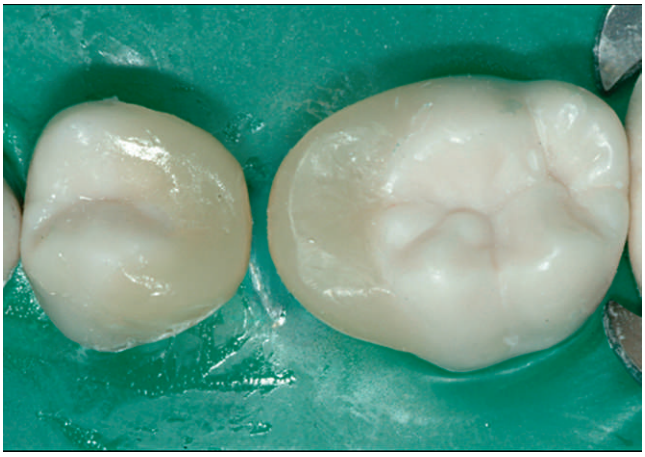

Figure 9. *Three-step matrix technique. Second step: Completion of this step reduces the interspace between teeth 14 and 16 to 0.5-1.0 mm.*

*Second Step*—The second step comprised the following:

- A straight circumferential matrix (AutoMatrix®) was applied on tooth 14 and bulged out toward tooth 16. The restoration was layered as described above.
- Here, two important comments must be made. First, at the end of the second step, an interspace of 0.5-1 mm was left between teeth 14 and 16 (Figure 9), and second, no polishing of the composite resin was done in order to maintain the oxygen inhibition layer for the next step.

*Third Step*—The third step comprised the following:

- To bridge the remaining interspace, a sectional, contoured matrix and an oval separating ring (Palodent®, Dentsply DeTrey) were applied on tooth 14. To ensure contact tightness, additional separating force was applied by twisting a hand instrument during light polymerization of the first layer of flowable composite resin (Figure 10, red arrow). Subsequently, viscous composite resin was applied and the restoration was completed.
- Final adjustment and polishing were done (Figure 11).

## RESULTS

Analysis of the collected data showed that teeth 16/17 and 25/26 were set in the model in such a way that they did not form regular contacts. Therefore, no valid measurements were possible and those variables were excluded from further analysis.

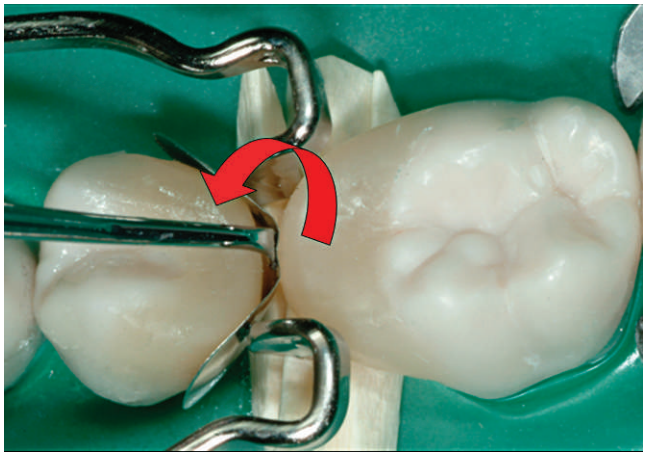

Figure 10. *Three-step matrix technique. Third step: A sectional contoured matrix, wedges, and an oval separating ring are applied on tooth 14. The remaining interspace between teeth 14 and 16 is filled with resin composite. During light polymerization, additional separating force is applied by twisting a hand instrument (red arrow).*

In the first quadrant, baseline measurements showed the highest proximal contact tightness between 13/14 (3.05 ± 0.60 N). The decrease in contact tightness after removal of tooth 15 and the increase after reconstruction of teeth 14 and 16 are shown in Figure 12 and Table 2. In the second quadrant, the baseline values were generally lower, decreasing slightly after removal of tooth 15 and reaching their original values again during final measurement (Table 2; Figure 13).

A paired *t*-test was performed to test for differences in contact tightness at corresponding proximal areas between baseline and final measurements. Values were checked for two-sided significance. Only the paired variables 14/15_1 and 14/16_3, as well as

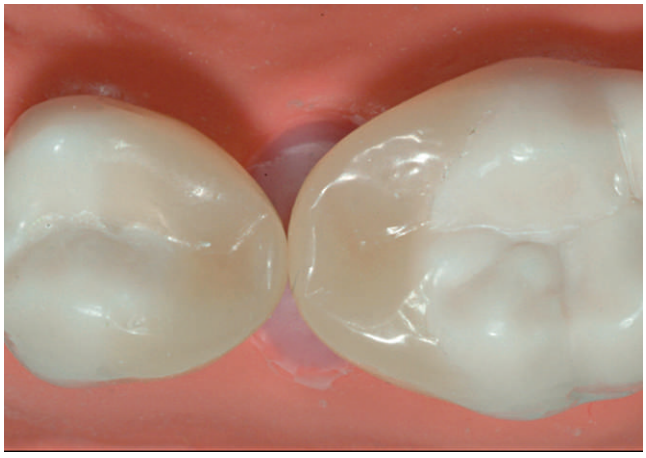

Figure 11. *Model situations before (top) and after (bottom) insertion of direct composite resin additions on teeth 14 and 16.*

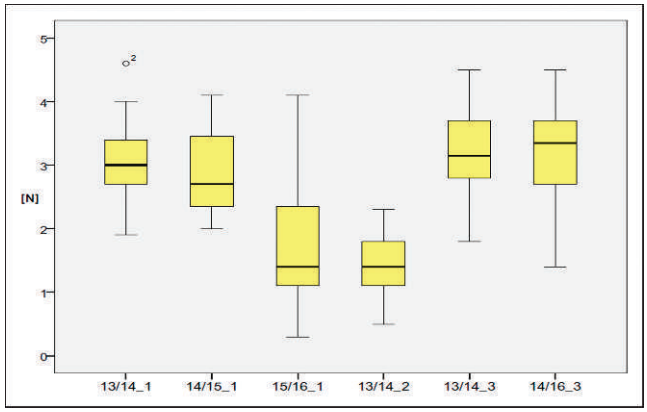


Figure 12. *Box plot of data from measurements in the first quadrant.*

15/16_1 and 14/16_3, yielded significant differences ($p$=0.006 and 0.001, respectively; Table 3).

## DISCUSSION

Direct-composite additions have become a significant tool in dentistry, especially for healthy teeth needing esthetic correction, for which a maximal tooth-saving approach is imperative.[1,2,6] Both patients and dentists can benefit from the advantages of this treatment option, which include the following: 1) tooth shape, color, and position can be corrected with an immediate restoration in a single treatment session; 2) the technique is either noninvasive or

Table 2: *Proximal Contact Tightness at Different Locations and Time Points (_1=Baseline; _2=After Removal of 15; _3=After Insertion of Composite Resin Additions on Teeth 14 and 16)*

| Location | Time of Measurement | | |
|---|---|---|---|
| | _1 | _2 | _3 |
| | Mean ± Standard Deviation, [N] | | |
| 13/14 | 3.05 ± 0.60 | 1.40 ± 0.47 | 3.20 ± 0.70 |
| 14/15 | 2.86 ± 0.64 | | |
| 15/16 | 1.65 ± 0.88 | | |
| 14/16 | | | 3.20 ± 0.80 |
| 23/24 | 1.23 ± 0.51 | 1.07 ± 0.42 | 1.23 ± 0.47 |
| 24/25 | 1.43 ± 0.45 | 1.34 ± 0.39 | 1.47 ± 0.33 |
| 26/27 | 2.18 ± 0.43 | 2.08 ± 0.42 | 2.16 ± 0.32 |

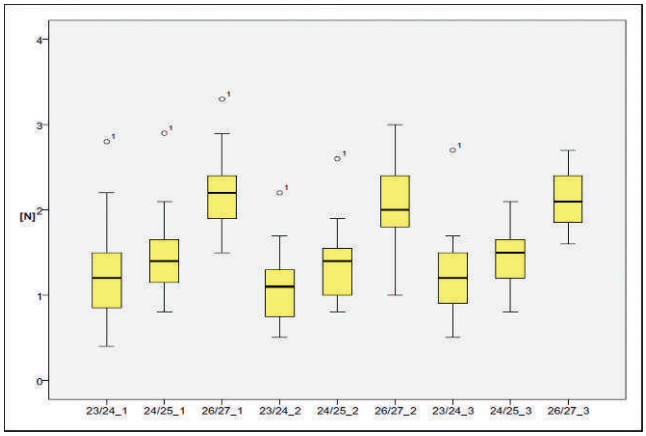


Figure 13. *Box plot of data from measurements in the second quadrant.*

| Table 3: *Results of Paired t-Test (Two-Sided Significance)* | |
|---|---|
| **Pair** | ***p*-Value** |
| 13/14_1–13/14_3 | 0.108 |
| 14/15_1–14/16_3 | 0.006 |
| 15/16_1–14/16_3 | 0.001 |
| 23/24_1–23/24_3 | 0.808 |
| 24/25_1–24/25_3 | 0.377 |
| 26/27_1–26/27_3 | 0.891 |

minimally invasive; 3) restorations can be removed and teeth returned to their original state, if necessary[14]; and 4) in case of minor failure, the restoration can be repaired with little effort, and in case of major failure (loss of restoration), other treatment options (implants, crowns, bridges) can be applied. Numerous case reports[3,15-18] related to direct-composite additions to close diastemas, recontour teeth, and change tooth color have presented excellent clinical results. Yet standardized fabrication procedures ensuring good esthetic, functional, and biological outcome—especially for the correction of posterior teeth—are still lacking.

Therefore, we have investigated, in an experimental setup, a novel, three-step matrix technique for posterior direct-composite additions in order to determine its potential for reconstructing well-contoured proximal surfaces and good proximal contacts. Our hypothesis—that the proximal contact between two adjacent composite resin additions bridging an interspace of a removed premolar can be made at least as tight as the original contact—has been proven. The newly created contact was even tighter than that of the original proximal contacts.

Unlike amalgam, composite resin is not condensable, making the reconstruction of good proximal contacts with this material complicated. The results of a questionnaire completed by German dental students showed that shaping the proximal contact areas creates the most problems during insertion of Class II resin restorations (86%) and that a low proximal contact is one of the most frequently reported failures (43%) of those restorations.[19] To overcome this problem, contoured sectional matrices and special devices such as separating rings were developed. An investigation[20] of their separation potential compared with that of wedges showed significantly more separation, allowing for tighter proximal contacts. Today, after the introduction of sectional contoured matrices and separating rings, the risk of failure due to weak proximal contacts and inferior proximal shape has declined.

However, posterior composite additions must bridge interspaces larger than that of conventional Class II restorations, further complicating reconstruction. Therefore, a novel matrix technique involving the successive application of three matrices, wedges, and a separating ring was developed. This technique enabled us to achieve very tight proximal contacts in the experimental setup. Compared with natural proximal contact tightness in patients investigated by Dorfer and others,[21] which lie in the range of 2.0 ± 1.6 N and 4.9 ± 1.9 N, the recorded values were still acceptable. Within certain limits, an increase in contact tightness can be influenced by the separation intensity applied by the dentist during the last step of the three-step matrix technique. Loomans and others[22] found that when contact tightness is increased after insertion of Class II resin restorations, it will decrease over time (as a result of wear of the proximal resin composite surfaces), whereas reduced contact tightness is not compensated. Since in clinical practice reconstruction of precisely the original contact tightness is difficult to achieve, a slight increase seems to be a useful approach.

The clinical case presented in Figure 1 displays two quite large direct-composite additions ("balconies") that bridge the gap. Here, the issue of whether cohesive strengths of these restorations are able to withstand occlusal loading might arise. It was shown[11] that convex shaping of Class II restorations

statistically significantly increases their load-bearing capacity. Therefore, the presented technique aims at creating convex-shaped proximal surfaces that provide sufficient support against cohesive failure. Furthermore, we recommend fabricating the direct-composite additions in such a way that the creation of novel occlusal contacts is avoided. The original contacts that are present before therapy should be maintained and the functional situation should not be altered. This approach protects the direct-composite additions from overstressing and failure.

The preclinical testing and the experiences with clinical application that we have gained over years helped us to define therapeutic indications. We recommend direct-composite additions in the posterior dentition when a gap of up to the width of one premolar (up to approximately 8 mm) has to be closed. Consequently, this treatment has become an alternative to implants or fixed dental prostheses. In our opinion there are no limitations concerning angulations of teeth or long clinical crowns. The mere addition of composite resin to teeth without any preparation does not compromise tooth integrity. An esthetic improvement is achieved, the functional situation is not altered, and we have observed that in several clinical cases the biological parameters have improved over the years (Figure 1). It is, however, crucial to reestablish sufficient proximal contacts to ensure sagittal support for the restored teeth. Otherwise, masticatory forces applied on the direct-composite additions could lead to a detrimental loading situation, comparable to that associated with cantilever fixed dental prostheses. When selecting patients it should be considered that the oral hygiene situation must be good. Patients must be trained in oral hygiene procedures and continuously supervised, and size-adjusted interdental brushes should be used. Then this kind of restorative procedure involves benefits for patients of all age groups who prefer noninvasive or minimally invasive treatment approaches. In comparison to other treatment alternatives, the procedure is very cost-effective since no laboratory work is necessary. As a result of the reported benefits, we suggest that practitioners expand the therapeutic indications and consider the three-step matrix technique (or a modification of it) for use in those clinical cases in which large class II cavities must be restored. In such a situation, a multiple-step matrix technique is reasonable to first reconstruct the cervical parts of the cavity and subsequently reestablish the proximal contacts.

As a second objective, the sagittal displacement of teeth in the model was investigated to determine the effect on the adjacent and contralateral contacts of removing tooth 15 and reconstructing teeth 14 and 16. The universal testing machine basically measured how much friction was present between adjacent proximal surfaces. When a gap (missing tooth) is present and the subsequent proximal contact is measured, the one tooth next to the gap can move aside, leading to a decrease in frictional force (= decrease in contact tightness). This phenomenon seems to continue throughout the dental arch, leading to a reduction in contact tightness even on the contralateral side. As expected, removal of tooth 15 led to a significant reduction in contact tightness in the first quadrant and to insignificant reduction in the second quadrant.[23] The reestablishment of a new tight proximal contact, however, did not lead to an increase in contact tightness at adjacent and contralateral proximal contacts. After insertion of the composite additions, all baseline tightness values were restored, illustrating that the contact tightness increase between teeth 14 and 16 had no detrimental influence on the adjacent or contralateral teeth.

Finally it should be emphasized that managing to reconstruct proximal contacts in clinical practice using the three-step matrix technique is a challenging and sophisticated job. The model situation that was used during the study facilitated the procedures significantly. Therefore, clinical results might not be as ideal as the results found within the study. Therefore, further investigations are necessary to test the feasibility of the technique in different clinical settings.

## CONCLUSIONS

Within the limitations of this *in vitro* investigation it was shown that posterior direct-composite additions inserted with the three-step matrix technique formed well-contoured proximal contacts that were at least as tight as the original contacts.

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# Effect of Ceramic Veneer Opacity and Exposure Time on the Polymerization Efficiency of Resin Cements

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### Clinical Relevance

Ceramic veneers can be used for the esthetic treatment of severely discolored teeth but must have high opacity if a satisfactory result is to be achieved. This study suggests that longer exposure to light- and dual-cured cement should be used to ensure greater polymerization efficiency.

## SUMMARY

**The objective of this study was to determine the degree of conversion (DC), hardness (H), and modulus of elasticity (E) of a dual-cured resin cement, a light-cured resin cement, and a flowable resin cured through opaque or translucent ceramic with different exposure times. RelyX ARC (dual), RelyX Veneer (light-cured), and Filtek Z350 Flow resin specimens 0.5 mm thick were cured for 40, 80, and 120 seconds through 1-mm thick translucent or opaque feldspathic ceramic disks (n=10). The specimens were stored at 37°C for 24 hours. Half of each specimen was used to test the DC and the other half to test H and E. The DC was determined in a Fourier transform infrared spectrometer in absorbance mode at peaks of 1638 $cm^{-1}$ and 1610 $cm^{-1}$. H and E were determined using nanoindentation with one loading cycle and a maximum load of 400 mN. The data were analyzed with three-way analysis of variance (ANOVA), the Games-Howell test, and the Pearson correlation test ($\alpha$=0.05). Statistically significant differences were found**

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**for all three factors (material, opacity, and exposure time), as well as interaction between them. The opaque ceramic resulted in lower DC, H, and E than the translucent ceramic for an exposure time of 40 seconds. An exposure time of 120 seconds resulted in a similar DC for all materials, irrespective of the opacity of the ceramic. Materials cured for 120 seconds had higher H and E than those cured for 40 seconds. The exposure time and opacity of the ceramic exerted an influence on the DC, H, and E of the materials evaluated.**

## INTRODUCTION

The use of porcelain veneers to change the color, shape, or position of anterior teeth in conservative restorations is becoming increasingly popular among today's dental practitioners. The success of this type of veneer is, to a large extent, determined by the strength and durability of the bond between the tooth surface, the luting composite, and the porcelain veneer.[1]

The materials used for luting ceramic restorations are composed of hybrid resins with a large amount of small filler particles (from 36% to 77% by weight) and a bisphenol A glycidyl dimethacrylate (Bis-GMA) matrix.[2] Commercially available cements for luting laminate veneers are usually activated by visible light or dual-cured depending on the opacity of the ceramic. The main advantages of light-cured cements are their greater color stability and working time compared with chemically cured and dual-cured systems.[3] Use of this type of cement makes it easier to remove excess material before light curing and reduces the time needed for finishing after the restorations have been luted.[1] In addition to their ease of use, light-cured resin cements for luting ceramic veneers have the further advantage that they do not use amine as a chemical initiator, which could cause the color of the material to change over time.[4] In order to take advantage of the properties of light-cured composite resins and their greater cost-effectiveness compared with resin cements, some dental practitioners have started using flowable resin composites to lute veneers. These were developed in 1996 and have the same particle size as hybrid composites but produce a mixture with lower viscosity and improved handling properties.[5] However, to date only one study has investigated the use of flowable resins as luting agents for laminate veneers.[6] Dual-cured resin cements are the most commonly used material for luting purposes. Various studies have shown that these have superior mechanical properties, such as flexural strength, modulus of elasticity, hardness, and degree of conversion, compared with cements that are exclusively light-cured or chemically activated.[7-10] Dual-cured cements have the advantage of additional chemical curing in deeper areas where light is subject to greater attenuation.[7,8,11] However, there is evidence that the chemical component of cure is lower than the light-curing component,[12] and little is known about the ability of dual-cured cements to polymerize when they are used in various clinical scenarios.[13]

While a number of studies have shown that various factors such as the type, thickness, and color of the ceramic[14-16] as well as the type of curing light, curing mode, and light intensity[14,17,18] can affect the polymerization of resin cements, there are fewer studies in the literature into the effects of the opacity of ceramic restorations on the properties of resin cements.[19,20] Nevertheless, esthetic treatment of severely darkened teeth requires the use of more opaque ceramics, which can attenuate the curing light before it reaches the luting agent.

The aim of this study was therefore to investigate the influence of exposure time and the opacity of a feldspathic ceramic on the polymerization efficiency of a dual-cured resin cement, light-cured resin cement, and flowable composite using Fourier transform infrared (FTIR) spectroscopy and nano-indentation. The hypotheses investigated in the study were 1) that exposure time and the opacity of the ceramic would not affect the polymerization efficiency of the luting cements and 2) that the polymerization efficiency of the flowable composite would be similar to that of the dual-cured and light-cured cements.

## MATERIALS AND METHODS

In order to simulate the ceramic veneers, Noritake EX-3 feldspathic porcelain discs (Noritake Dental Supply Co Limited, Tokyo, Japan) 10 mm in diameter and 1.0 mm in thickness were made with an opaque ceramic (O) in shade OBA3 and a translucent ceramic (T) in shade BA3.

Three types of materials were used: a dual-cured resin cement (RelyX ARC, 3M-ESPE, St. Paul, MN, USA), a light-cured resin cement (RelyX Veneer, 3M-ESPE), and a flowable composite (Filtek Z350 Flow, 3M-ESPE), all in shade A3. Table 1 shows the materials with their respective manufacturers, type, composition, and filler content. The resin cements were handled in accordance with the manufacturers' instructions for luting ceramic veneers.

Table 1: *Materials Used in the Study*

| Material | Manufacturer | Type | Composition | Filler |
|---|---|---|---|---|
| RelyX ARC | 3M ESPE, St Paul, MN, USA | Dual-cured resin cement | Bis-GMA, TEGDMA, zirconia/silica filler, pigments, benzoyl peroxide, amine, and photoinitiator | 67.5 wt% |
| RelyX Veneer | 3M ESPE, St Paul, MN, USA | Light-cured resin cement | Bis-GMA, TEGDMA, zirconia/silica filler | 66.0 wt% |
| Filtek Z350 Flow | 3M ESPE, St Paul, MN, USA | Flowable composite | Bis-GMA, Bis-EMA, TEGDMA, zirconia/silica filler | 65.0 wt% |

*Abbreviations: Bis-EMA, bisphenol A ethoxylated dimethacrylate; Bis-GMA, bisphenol A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.*

The specimens were produced using a polytetrafluoroethylene (Teflon) mold 8 mm in diameter and 0.5 mm in thickness. The mold was placed on a glass plate with black adhesive paper to reduce the amount of light reflected from the benchtop surface onto the specimens. After the material had been inserted, a Mylar strip was positioned on top of it to ensure a smooth surface. A glass slide was pressed onto the strip for 15 seconds with a pair of locking pliers to allow the material to flow out and so keep a standard specimen thickness. The glass slide was then removed and the ceramic disc positioned over the assembly. Half of the specimens were light-cured through the opaque ceramic disc and the other half through the translucent ceramic disc; the purpose of both discs was to simulate ceramic veneers. Light curing was carried out with a conventional halogen curing light (Optilux 501, Demetron Corp, Orange, CA, USA) with an irradiance of 550 mW/cm$^2$ for 40, 80, and 120 seconds. Ten specimens were prepared for each of the experimental conditions.

Immediately after light curing, the specimens were stored in lightproof containers in a high relative humidity at 37°C for 24 hours to avoid additional exposure to light. Each specimen was cut down the middle with a diamond disc to form two halves; one half was used to determine the degree of conversion, and the other was used for nanoindentation tests.

The degree of conversion was measured using a FTIR spectrometer (Spectrum 100, Perkin-Elmer Corp, Norwalk, CT, USA) with an attenuated total reflectance accessory. The spectra were recorded with 20 scans at a resolution of 2 cm$^{-1}$ in the 1500 to 1800 cm$^{-1}$ band. Monomer conversion for all the materials was calculated using the standard method for monitoring the change in the ratios of the aliphatic to aromatic C = C absorption peaks at 1636 cm$^{-1}$ and 1610 cm$^{-1}$, respectively, in the uncured and cured forms according to the following equation:

$$GC = 100 \times 1 - \frac{C{=}C\,1636\,C / C{=}C\,1610\,C}{C{=}C\,1636\,U / C{=}C\,1610\,U}$$

where $C$ is the absorption peak of the cured material and $U$ the absorption peak of the uncured material.

The data were recorded in a spreadsheet (Excel 4.0, Microsoft Corp, Redmond, WA, USA) and used to calculate the descriptive statistics.

The nanoindentation tests were carried out on the other halves of the discs with an XP nanoindenter (MTS Systems Corporation, Oakridge, OK, USA) using a matrix with nine indentations, one loading cycle, a maximum load of 400 mN, and a Berkovich tip. The hardness and moduli of elasticity of the materials were determined from the curves of the applied load vs penetration depth at the surface of the sample according to the method described by Oliver and Pharr.[21]

Statistical analysis was performed with the SPSS 15.0 statistics program (SPSS Inc, Chicago, IL, USA) using three-way ANOVA (material, opacity, and exposure time), the Tukey test, and the Pearson correlation test with a significance level of 5%.

## RESULTS

Degree of conversion (DC), hardness (H), and modulus of elasticity (E) were found to differ significantly with material, opacity of the ceramic, and curing time, and there was significant interaction between material and opacity ($p<0.05$). The Pearson correlation test revealed a strong correlation between DC and H ($r^2=0.83$) and between DC and E ($r^2=0.82$) and a very strong correlation between H and E ($r^2=0.97$).

Table 2: *Mean Values (SD) of the Degree of Conversion, Hardness, and Modulus of Elasticity of the Materials Evaluated Using Different Exposure Times*

| | Material | Ceramic | 40 s (SD) | | 80 s (SD) | | 120 s (SD) | |
|---|---|---|---|---|---|---|---|---|
| **Degree of conversion, %** | RelyX ARC | Translucent | 73.21 | (1.88) | 74.93 | (1.15) | 76.06 | (1.85) |
| | | Opaque | 71.70 | (1.42) | 73.16 | (2.18) | 76.69 | (2.85) |
| | RelyX Veneer | Translucent | 69.16 | (2.37) | 71.29 | (2.07) | 73.05 | (2.40) |
| | | Opaque | 64.39 | (2.20) | 68.04 | (1.41) | 68.99 | (1.79) |
| | Filtek Z350 Flow | Translucent | 63.91 | (1.74) | 66.70 | (1.33) | 68.25 | (1.28) |
| | | Opaque | 63.86 | (0.91) | 65.25 | (0.95) | 68.36 | (0.93) |
| **Hardness, GPa** | RelyX ARC | Translucent | 0.47 | (0.02) | 0.49 | (0.01) | 0.50 | (0.01) |
| | | Opaque | 0.43 | (0.02) | 0.48 | (0.01) | 0.51 | (0.03) |
| | RelyX Veneer | Translucent | 0.37 | (0.02) | 0.40 | (0.02) | 0.43 | (0.02) |
| | | Opaque | 0.36 | (0.01) | 0.39 | (0.01) | 0.41 | (0.01) |
| | Filtek Z350 Flow | Translucent | 0.37 | (0.01) | 0.40 | (0.01) | 0.43 | (0.01) |
| | | Opaque | 0.31 | (0.02) | 0.37 | (0.01) | 0.40 | (0.01) |
| **Modulus of elasticity, GPa** | RelyX ARC | Translucent | 9.72 | (0.22) | 10.00 | (0.15) | 10.24 | (0.18) |
| | | Opaque | 8.99 | (0.48) | 9.75 | (0.26) | 10.21 | (0.39) |
| | RelyX Veneer | Translucent | 7.86 | (0.37) | 8.20 | (0.50) | 8.61 | (0.35) |
| | | Opaque | 7.73 | (0.22) | 8.26 | (0.12) | 8.50 | (0.15) |
| | Filtek Z350 Flow | Translucent | 7.79 | (0.14) | 8.42 | (0.17) | 8.52 | (0.15) |
| | | Opaque | 6.83 | (0.35) | 7.82 | (0.16) | 8.17 | (0.15) |

Table 2 and Figures 1 to 3 show the mean and standard deviations of the degree of conversion, hardness, and modulus of elasticity for the luting agents investigated.

## Degree of Conversion

When the variable ceramic opacity alone was considered, the DC was significantly higher with the translucent ceramic ($p<0.05$). Considering only the exposure time, the DC increased significantly when this variable was increased from 40 to 120 seconds. When exposure time and ceramic opacity were considered together, an exposure time of 120 seconds resulted in a statistically higher DC irrespective of the opacity of the ceramic, and the DC was not significantly different from that obtained with an exposure time of 80 seconds and a translucent ceramic ($p>0.05$). The opacity of the ceramic did not have a statistically significant influence ($p>0.05$) on the DC of the dual resin cement or the flowable composite.

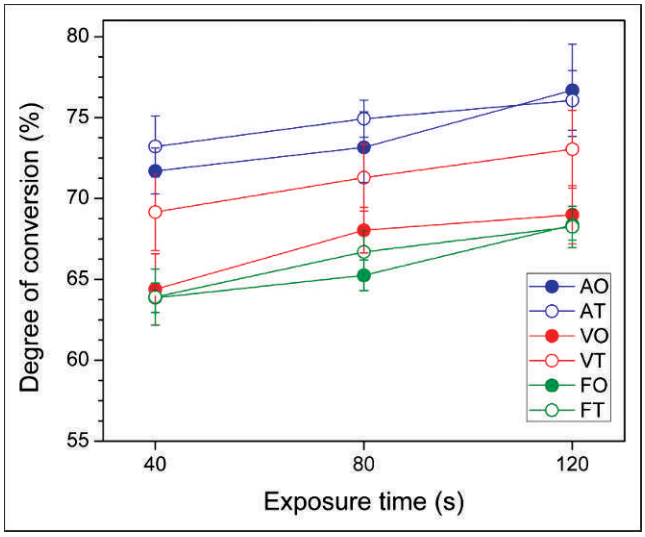


Figure 1. *Mean and standard deviations of the degree of conversion of the materials evaluated as a function of exposure time.*

When all of the variables were considered, the dual resin cement cured for 120 seconds had higher mean values of DC than all other materials, irrespective of the opacity of the ceramic ($p<0.05$). The lowest mean values of DC were observed for the flowable composite cured for less than 80 seconds through the opaque or translucent ceramic, and for the light-cured cement cured for 40 seconds through the opaque ceramic.

## Hardness

As observed for the DC, when only the opacity of the ceramic was considered, the hardness of the materials evaluated was significantly higher for the translucent ceramic and increased with increasing exposure time ($p<0.05$). When exposure time and the opacity of the ceramic were considered together, an exposure time of 120 seconds resulted in statistically higher hardness for both opacities. These mean values were not significantly different from those observed for an exposure time of 80 seconds in association with a translucent ceramic ($p>0.05$). The opaque ceramic exhibited lower hardness when an exposure time of 40 seconds was used. When the materials and the opacity of the ceramic were considered, the hardness of both light-cured materials, RelyX Veneer and Filtek Z350 Flow, was found to be affected by the opacity of the ceramic ($p<0.05$). For the variables material and exposure time, hardness increased with time, and higher mean values were observed with the dual resin cement for all exposure times evaluated.

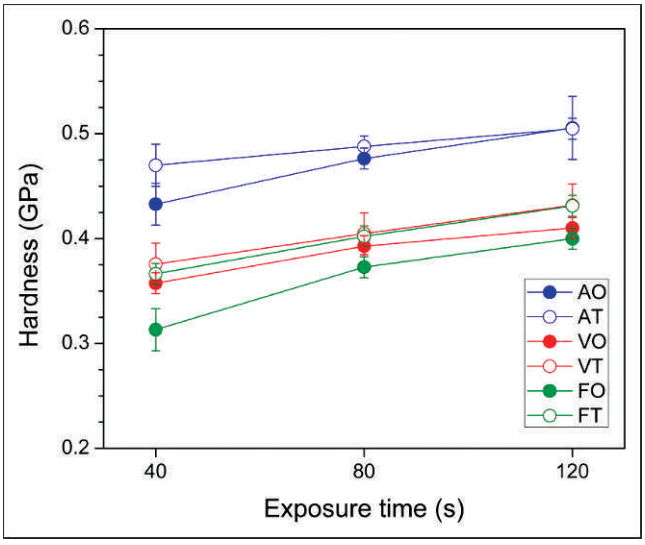


Figure 2. *Mean and standard deviations of the hardness of the materials evaluated as a function of exposure time.*

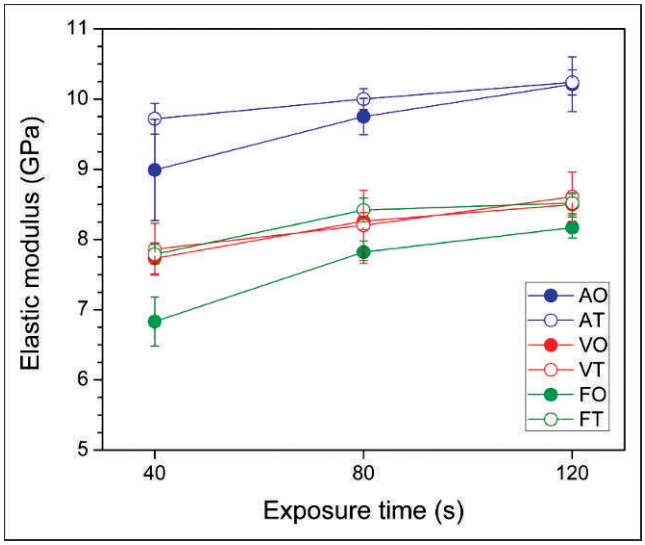


Figure 3. *Mean and standard deviations of the modulus of elasticity of the materials evaluated as a function of exposure time.*

When all variables were considered, significantly higher hardness values were achieved with the dual-cured resin cement than with the other materials, with the exception of an exposure time of 40 seconds with an opaque ceramic ($p<0.05$). The lowest mean hardness recorded was that observed for the flowable composite light-cured through opaque ceramic for 40 seconds ($p<0.05$).

## Modulus of Elasticity

There was a statistically significant increase in the modulus of elasticity both when the translucent ceramic was used and when the exposure time was

increased from 40 seconds to 120 seconds ($p<0.05$). When the opacity of the ceramic and the exposure time were considered together, an exposure time of 40 seconds in association with an opaque ceramic resulted in an E value significantly lower than that for all other conditions ($p<0.05$). When the materials and the opacity of the ceramic were considered, only the E of the flowable composite Filtek Z350 Flow was found to be affected ($p<0.05$). For the variables material and exposure time, higher mean E values were observed with the dual resin cement for all exposure times evaluated, with statistically significant differences compared with the E values for the light-cured resin cement and the flowable composite for all exposure times ($p<0.05$).

As with the hardness, the highest mean E value was observed for the dual resin cement for all conditions, with the exception of the group cured for 40 seconds through opaque ceramic, which yielded similar results to those for the light-cured resin cement cured for 120 seconds through a translucent ceramic. The lowest mean E value corresponded to that observed for the flowable composite light-cured for 40 seconds using an opaque ceramic and was statistically different from the E values for all other groups evaluated in this study ($p<0.05$).

## DISCUSSION

The present study evaluated the polymerization efficiency of resin cements testing the degree of conversion, hardness, and modulus of elasticity using ceramic discs with different opacities and exposure times of light curing. The purpose in using a ceramic disc was to simulate a clinical situation, as luting agents are used underneath a restoration, leading to some attenuation of the light.[16,22-25] With this in mind, opaque and translucent feldspathic ceramic discs were used here to simulate two different clinical situations, respectively: 1) severely discolored teeth that require the use of a more opaque ceramic to mask the darkened tooth structure; and 2) teeth whose color is unaltered or only slightly altered, for which a translucent ceramic can be used.

When visible light reaches the restorative material, part of it is transmitted through the material, part is absorbed, and part is reflected at the surface. The greater the transmittance of the indirect restorative material used as a spacer, the greater the irradiance that will reach the resin cement and the greater the degree of conversion.[26] A previous study reported better polymerization in light-cured and dual-cured cements when a translucent ceramic was placed between the cement and the light source.[22] In recent studies,[27,28] less translucent ceramics were shown to result in lower hardness in resin cements than more translucent ones when the same curing mode was used. Here, the increase in the degree of conversion, hardness, and modulus of elasticity of all materials evaluated was greater when a translucent rather than an opaque ceramic was used.

The amount of light that reaches dual-cured and light-cured cements is an extremely important factor in ensuring effective polymerization. Dual-cured resin cements need to be light-cured to ensure satisfactory polymerization, although they have a chemical activator to complete the reaction.[9] Studies of the mechanical properties of resin cements cured in different modes have shown that dual-cured cements are superior to light-cured and chemically activated ones.[7,8,18,19] The dual-cured resin cement investigated in this study had better performance than the light-cured materials, particularly when exposed to light for 120 seconds.

The irradiance of the light that reaches the cement is drastically reduced when the light is transmitted across a ceramic restoration because of the effects of absorption, reflection, or transmission.[28] Longer exposure times are therefore recommended to counteract these effects.[12] The results reported here support this recommendation, as all the materials cured for 120 seconds exhibited greater polymerization efficiency than those cured for 40 seconds, a finding observed for all three properties investigated.

Incomplete polymerization of resin materials results in a low degree of conversion and large amounts of residual monomers, which can adversely affect mechanical properties and increase water sorption and solubility.[29-31] The degree of conversion of a resin material depends on factors such as the chemical structure of the monomers, the curing conditions, including light intensity and photoinitiator concentration, as well as the ambient conditions, such as atmosphere and temperature.[32] Furthermore, it has been reported that inadequate conversion as a result of low light intensity during cement curing can adversely affect the clinical performance of the restoration.[17]

Traditionally, the degree of conversion of dental materials has been determined by either direct methods, such as FTIR or Raman spectroscopy, or indirect methods, such as microhardness testing. More recently, instrumented indentation testing has

become very popular in a variety of areas, primarily because it allows the mechanical behavior of materials to be characterized on a nanoscale, a process known as nanoindentation.[33] This type of testing measures the hardness and modulus of elasticity of the material directly from indentation load and displacement measurements. Another advantage of the method is that it eliminates operator-induced error as there is no need to measure the indentation area with the aid of an image.[21]

In the present study a strong correlation was observed between the degree of conversion and nanohardness of the materials evaluated. This finding is in agreement with other studies that demonstrated a positive correlation between the degree of conversion and microhardness of composite resins.[34-37] However, although both methods can be used to determine the extent of cure in resin-based materials, care should be exercised when interpreting such findings as each method is sensitive to different variables.[34] For example, the type and amount of filler particles are variables that can affect the mechanical properties of resin-based materials.[38] The materials investigated in the current study are produced by the same manufacturer and have the same type of filler particles (silica and zirconia) and very similar filler loads by weight (65.0% to 67.5%). However, the composition of the organic matrix of the flowable composite differs from that of the other materials as it contains bisphenol A ethoxylated dimethacrylate (Bis-EMA) as well as Bis-GMA and triethylene glycol dimethacrylate (TEGDMA). A recent study[39] of experimental resin cements showed that replacement of TEGDMA/Bis-GMA by various quantities of Bis-EMA does not necessarily imply significant improvements in particular mechanical properties.

The concept of energy density was adopted here since the irradiance was fixed in 550 mW/cm$^2$ with different exposure times. Other studies investigating the effects of energy density on the properties of resin cements found that the flexural strength,[40] degree of conversion,[17,40,41] and hardness[8,41-43] of dual-cured and light-cured cements were strongly dependent on this factor.

The first hypothesis in this study was rejected since both the opacity of the ceramic and exposure time affected the properties investigated. The second hypothesis was partially rejected as the flowable composite proved to be inferior to the dual-cured cement, although some of the properties of the former were similar to those of the light-cured cement. The findings indicate that flowable composites should not be used as the material of choice when cementation of an opaque ceramic veneer is indicated in a clinical setting.

Although curing times of 40 seconds are recommended by most manufacturers of luting agents, a longer exposure time should be considered to ensure sufficient conversion in cases involving more opaque ceramic restorations. In addition, dual-cured cements should be considered luting agents of choice when the esthetics are not a concern, as the presence of a chemical activator minimizes the effects of light attenuation caused by opaque ceramics.

## CONCLUSIONS

Within the limitations of this *in vitro* study, it can be concluded that:

- the opacity of the ceramic and the exposure time affected the degree of conversion, hardness, and modulus of elasticity of the luting materials evaluated;
- the dual-cured resin cement can be recommended for cementation of ceramic veneers since it demonstrated better polymerization efficiency than the light-cured cement and flowable composite; and
- when a light-cured material or an opaque ceramic are used, the minimal exposure time for polymerization should be 80 seconds.

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# Microleakage Resistance of Minimally Invasive Class I Flowable Composite Restorations

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### Clinical Relevance

Despite recent developments in dentin-bonding systems, flowable composite resins, and restorative procedures, microleakage resistance of the restoration–tooth surface interface remains problematic. In this *in vitro* study on minimally invasive Class I restorations, the flowable composites used, with their manufacturers' bonding systems, all produced more microleakage than a conventional microhybrid composite control. Microbubbles were found within many of the flowable composite restorations; these might result in undue restoration pitting or degradation.

## SUMMARY

**Minimally invasive flowable composite Class I restorations are widely used. However, flowable composites are characterized by low filler contents, modified resin formulations, low moduli of elasticity, low viscosity, generally poor mechanical properties, and decreased long-term stability. The purpose of this study was to compare the microleakage resistance of a wide variety of flowable composites used with their manufacturers' recommended bonding systems to that of a long-used and widely studied microhybrid composite when placed as minimally invasive occlusal restorations. Molar teeth were prepared in a standardized manner, restored, artificially aged, stained, sectioned, evaluated, and analyzed. Microleakage varied substantially, by a whole order of magnitude, among the material groups tested. The control group, a conventional microhybrid composite material, leaked significantly less than all the flowable composite groups. Microleakage varied very slightly among measurement site locations. Tiny microscopic bubbles were seen within many of the flowable composite specimens, as were a few voids.**

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## INTRODUCTION

Although fluoride treatment can successfully reduce smooth surface caries, occlusal pits and fissures remain at risk.[1-3] Early diagnosis combined with conservative interceptive adhesive treatment of occlusal caries may preserve the integrity of the remaining tooth and increase its longevity.[4,5] Minimally invasive flowable composite Class I restorations have gained widespread use because of their pleasing esthetics, the belief that they require less removal of sound tooth structure than for amalgam restorations, and the perception that adhesion to enamel will reduce the risk of leakage and secondary caries.[6,7]

However, polymerization shrinkage of resinous composites can result in loss of adhesion and microgap formation.[8] Loss of adhesion and microgap formation may allow microleakage of bacteria and their toxic products; these contribute to postoperative tooth sensitivity, development of secondary caries, pulpal disease, marginal staining, and restoration failure.[9-13]

Techniques such as incremental buildup and soft-start light polymerization have been used with the aim of improving the depth of cure, reducing polymerization stress, and minimizing its unwanted developed effects.[14,15] However, other studies have not found significant differences between bulk and incremental insertion in terms of magnitude and distribution of stress at the bonded interface.[16,17]

Highly filled composites, containing proportionally less resin, likely undergo less shrinkage. However, these materials have higher elastic moduli and a lower propensity for stress relaxation, as well as are thought to be more difficult to place in conservative tooth preparations. Alternatively, it has been suggested that flowable composites with less filler and lower viscosities might be easier to place, especially in inaccessible areas, and might reduce the effects of polymerization shrinkage through increased stress relaxation.[18-21] It has been suggested that the ease of flowable composite placement facilitates superior adaptation, but supportive data are lacking. Since their introduction in the mid-1990s, flowable composites have become widely used for a broad range of restorative applications such as liners, bases, build-ups, bulk restorative materials, or sealants. Flowable composites generally contain lower filler concentrations and modified resin formulations. They are characterized by low moduli of elasticity, low viscosity, generally poor mechanical properties, and decreased long-term stability.[22] Flowable resins may manifest superior notched-beam fracture toughness values in comparison to conventional microhybrid composites[23,24]; however, such findings must be tempered by their tendency toward greater deformation. Some studies have suggested that flowable composites may protect bonding agents from the effects of polymerization stress of the restorative material, thus reducing the microleakage.[18,19] However, other studies have found no significant reduction of marginal microleakage when flowable composite linings were used.[13,25]

The microleakage resistance of composites for conservative Class I restorations has rarely been evaluated. Therefore, the purpose of this study was to compare the microleakage resistance of flowable composites to that of a long-used and widely studied microhybrid composite, used with their manufacturers' recommended bonding agents, when placed as minimally invasive occlusal restorations.

## METHODS AND MATERIALS

### Tooth Preparation

One hundred freshly extracted human molars were stored in water at room temperature. These were mostly third molars with an even distribution of upper and lower teeth. The roots of the teeth were cleaned using periodontal curettes and then mounted in acrylic resin blocks, leaving the crowns exposed. A fissurotomy bur (Micro STF, SS White, Lakewood, NJ, USA), with flutes 0.6 mm in diameter and 1.5 mm in height, was used to make shallow preparations within the enamel of the central fossae of the molars. These minimally invasive Class I restorations were 3 mm in length from mesial to distal, 0.6 mm in width, and 1.5 mm in depth. To aid in standardizing the preparations, the handpiece (Tradition Midwest, Dentsply, York PA, USA) was mounted in a surveyor parallometer system (Ney products, Dentsply). Prepared teeth were arbitrarily assigned to one of 10 material groups (n=10) to ensure that upper and lower teeth, or larger and smaller teeth, were evenly distributed.

### Restoration

Ten different flowable resin composite–bonding agent groups were included (Table 1). These included representative products from a wide range of manufacturers. Nine of these groups contained a flowable composite; one control group contained a widely used microhybrid composite as a control. Each restorative material was paired with an adhesive from, and specifically recommended for

| Table 1: *Flowable Composites, Their Bonding Agents (Group Abbreviations), and Manufacturers* | |
|---|---|
| (R2-OB) | Revolution 2; OptiBond SOLO; SDS Kerr, Orange, CA, USA |
| (VF-TQ) | Virtuoso Flowable; Tenure Quick w/FL; Den-Mat, Santa Maria, CA, USA |
| (UF-UB) | UniFil FLOW; UniFil Bond; GC America, Alsip, IL, USA |
| (HM-EX) | Heliomolar Flow; Excite; Ivoclar Vivadent, Amherst, NY, USA |
| (AF-OS) | Aelite Flo LV; One Step PLUS; Bisco, Schaumburg, IL, USA |
| (FF-SB) | Filtek Flow; Single Bond; 3M ESPE, St Paul, MN, USA |
| (PF-PQ) | Permaflo; PQ1; Ultradent Products, South Jordan, UT, USA |
| (FI-B1) | Flow-it ALC; Bond 1; PENTRON Clinical Technologies, Wallingford, CT, USA |
| (GD-GB) | Gradia Direct Flo; G Bond; GC America, Alsip, IL, USA |
| (HX-OB) | Herculite XRV; OptiBond SOLO; SDS Kerr, Orange, CA, USA |

this purpose by, the same manufacturer. Conditioners, dentin bonding agents, and the flowable composite resins were applied following their manufacturer's instructions using syringes. The control material was delivered from a unidose tip directly into the preparation using a unidose gun/dispenser. The preparations were filled to the occlusal cavosurface margins. After the excess composite resin was removed with a composite metal spatula, the composites were light cured for 40 seconds at 800 mW per square centimeter (Spectrum 800, Denstsply), finished, and polished using a composite polishing kit (Diacomp, Brasseler, Savannah, GA, USA) with water spray.

### Microleakage Assessment

After restoration, the teeth were stored in water at 37°C for 14 days to ensure hydration of the resinous restorations. Next, the restorations were then artificially aged by thermal cycling from 5°C to 50°C for 1000 cycles, with dwell and travel times of 20 seconds. The process of thermocycling causes differential contraction of restoration and tooth, which stresses their interface. After artificial aging, the entire surface of each tooth was coated with two layers of a clear nail polish, with the exception of 1 mm around the circumference of the restoration margins, to prevent leakage through other avenues. The restored teeth were then submerged in a 50% solution of silver nitrate for 60 minutes, rinsed with water, placed in photo developer (D76, Eastman Kodak, Rochester, NY), and exposed under a 150-W floodlamp for 30 minutes to ensure reduction and immobilization of the silver nitrate stain. The specimens were then embedded in slow-setting, low-viscosity, clear epoxy resin (Hapex 1200A/1226, Hastings Plastics, Santa Monica, CA, USA). The specimens were sectioned faciolingually through reference marks, scribed on the midfacial and midlingual aspects of the teeth using a wide diamond blade in a slow-speed saw with copious aqueous irrigation (Isomet, Buehler, Evanston, IL, USA). This provided four tooth-restoration interfaces for measurement of stain penetration on each specimen: mesiofacial (MF), mesiolingual (ML), distolingual (DL), and distofacial (DF). The sectioned samples were then re-exposed to the 150-W floodlamp for 5 minutes to ensure that all of the silver nitrate stain would turn black and be fixed.

Microleakage was recorded as a continuous parametric variable as the distance of stain penetration in millimeters, in a plane parallel to the long axis of the tooth, measured at 30× magnification using a toolmakers microscope (Unitron, Commack, NY, USA) and digital positioners calibrated to an accuracy of 0.1 μm (Boeckeler Instruments, AZ, USA; Figure 1).

### Analysis

Mean microleakage means and associated standard deviations were calculated for each material group and plotted. Two-way analysis of variance was performed to evaluate the main effects of material type, measurement site, and their interaction ($p<0.05$). The Tukey multiple comparisons test was then computed to determine which materials differed from one another ($p<0.05$).

## RESULTS

Microleakage varied substantially, by a whole order of magnitude, among the material groups tested (Table 2; Figure 2; $p<0.0001$). Material type accounted for almost all of the variation produced in

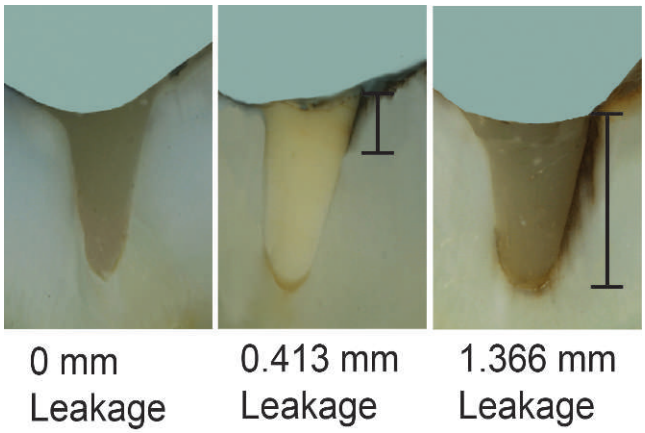


Figure 1. *Macro photographs of representative sectioned tooth specimens. Microleakage was recorded as the distance of stain penetration in millimeters, in a plane parallel to the long axis of the tooth, from the restoration margin to its most apical extent using a toolmaker's microscope and digital positioners. Microbubbles can be visualized as white spots within the bodies of all of these sectioned restorations, especially the one on the right.*

this experimental model (Table 2). Microleakage also varied among measurement site locations (Table 2; Figure 3; $p=0.005$). However, material type and measurement site did not interact to influence microleakage; that is, their effects were simply summative, and materials were equally affected by the measurement site locations (Table 2; $p=0.1$).

Almost all of the variation was attributable to the material choice (Table 2). Among the material groups, multiple comparisons testing showed that the control group, a conventional microhybrid composite with its associated bonding agent (HX-OB), leaked significantly less than all nine of the flowable composites tested. Many statistically significant differences in microleakage resistance were discerned among the nine flowable composites (Figure 2). Material choice had an enormous impact on the microleakage resistance of minimally invasive composite restorations.

Measurement site had a statistically significant, but very small, effect (Table 2; Figure 3). Multiple comparisons testing revealed that the ML/P site leaked significantly less than MF and DF there was no difference in leakage among DL,DF, and MF sites. This experimental model attributed a difference in microleakage to tooth morphology.

Defects or bubbles were seen within the bulk restorative materials in 56% of the 200 sections (Figure 1). Mostly, these bubbles were very small, approximately 50 to 100 μm in diameter, but three larger voids were seen. They often appeared as white

Table 2: *Two-Way Analysis of Variance for the Main Effects of Material Group and Measurement Site and Their Interaction* ($p<0.05$).

| Source of Variation | Sum of Squares | Degrees of Freedom | F-Ratio | *p* Value |
|---|---|---|---|---|
| Material | 39.9 | 9 | 865 | <0.0001 |
| Site | 0.1 | 3 | 4 | 0.005 |
| Interaction | 0.2 | 27 | 1 | 0.1 |
| Residual | 1 | 200 | | |
| Total | 41 | 239 | | |

spots on the sectioned specimens because they tended to be filled with sectioning debris. The defects had no influence on interfacial leakage. Only groups HX-OB and UF-UB were entirely without bubbles or voids. Groups R2-OB and FI-B1 had six specimens with bubbles or voids, group FF-SB had five specimens with bubbles or voids, groups AF-SB and PF-PQ had four specimens with bubbles or voids, groups VF-TQ and GD-GB had three specimens with bubbles or voids, and group HM-EX had two specimens with bubbles or voids. Because of the unexpected identification of tiny bubbles and larger voids, the flowable composites that had manifested bubbles and voids on sectioning were expressed directly onto glass slabs, polymerized, sectioned, and examined as above. For all of these materials, tiny bubbles were revealed, but larger voids were absent.

## DISCUSSION

Despite a long search for materials and techniques to ensure adhesion to tooth structure so as to minimize leakage, the interface between restoration and tooth remains problematic.[26] Advances continue, but limitations persist at macro, micro, and nano levels.[27,28] In the present *in vitro* study, the inorganic compound silver nitrate was selected because it has been accepted as a suitable method for measuring both microleakage and nanoleakage.[29,30] The silver ion is very small (0.059-nm diameter) when compared with the size of a typical bacteria (0.5-1.0 μm).[31] This small size and high reactivity makes silver nitrate an appropriate agent to detect the nanoporosities.[32]

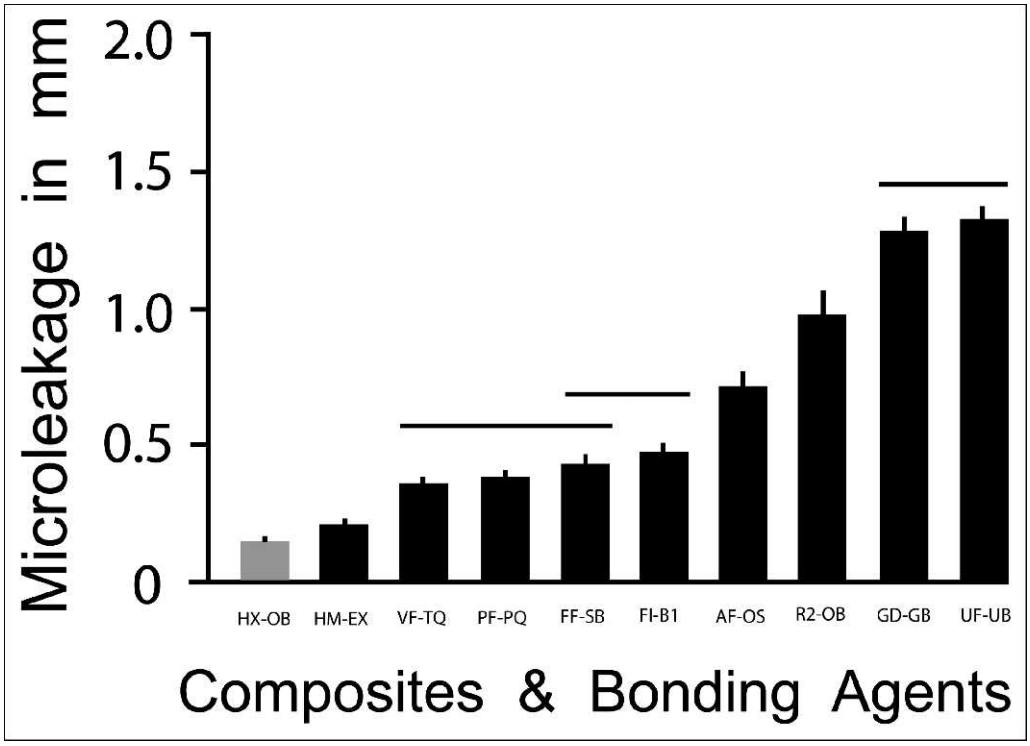


Figure 2. *Bar graph of microleakage of composite and bonding agent groups in millimeters. Means and standard deviations are displayed. Flowable composite groups are illustrated by black bars; the control microhybrid composite group is illustrated by a gray bar. Statistically similar groups are linked by horizontal lines (*$p > 0.05$*).*

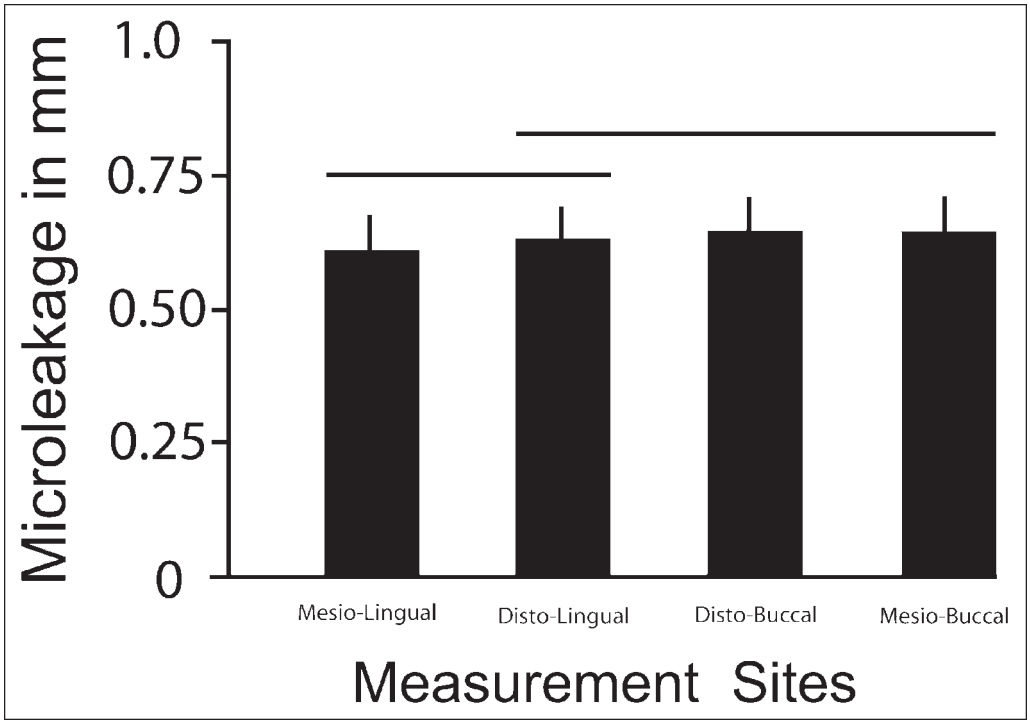


Figure 3. *Bar graph of microleakage of measurement site locations in millimeters. Means and standard deviations are displayed. Statistically similar groups are linked by horizontal lines (*$p > 0.05$*).*

Multiple factors may influence the microleakage resistance of minimally invasive composite restorations. These likely include polymerization shrinkage, resistance to deformation or elastic modulus, cavity configuration, the amounts of exposed enamel and dentin, and the restorative procedures themselves.[8,33-36] It has been reported that marginal gaps increase as cavity designs change from a V-shaped to box-shaped configurations.[37] The term *cavity configuration factor* (C-factor) has been used to describe differences in cavity design.[38] A high C-factor indicates a high ratio of bonded to unbonded tooth/composite surfaces, corresponding to high stress levels and increased probability of separation of the composite from the wall of the tooth preparation. This effect is caused by a reduction in unbonded surfaces, which restricts the composite's ability to flow to relieve polymerization stress. Hence, shallow cavities tend to have lower C-factors, but deep or narrow cavities tend to have higher C-factors. In this study of minimally invasive fissure preparations, the cavities were relatively shallow but proportionally very narrow. In contrast, a fissure sealant would tend to be shallower and much wider with a much more favorable configuration, whereas a conventional deeper and bulkier restoration would have a less favorable configuration.

Conflicting data on the microleakage and caries resistance of bonded flowable composites in comparison to those of conventional fissure sealants have been reported.[39,40] This may be explained by the wide variation in filler content and other properties of commercially available flowable composites.[22,24,41] This current study identified substantial differences, sixfold, in microleakage resistance among nine commercially available bonded flowable composites (Table 2; Figure 2).

In this study, the entire preparation was in enamel and had a shallow and narrow standardized conservative outline form. Because bonding to enamel is known to be very predictable, similar performances might have been expected. However, some of the differences found may be ascribed to the use of self-etching primers. The two test groups with significantly more microleakage than all others used self-etching primers. Self-etching adhesives are more effective on ground enamel than on intact enamel because self-etching materials do not create an enamel-etching pattern as well defined as those produced by a separate step 37% phosphoric acid etching.[42] Pertinently, the same bonding agent was used both with the conventional microhybrid control material (HX-OB) and with a flowable composite made by the same manufacturer (R2-OB); the flowable composite recorded five times more microleakage than the conventional microhybrid (Figure 2). It is possible that the conservative cavity preparations, without undercuts, used in this current study tended to be cut along or obliquely to rods, rather than across enamel rods, exacerbating the lesser etching ability of the self-etching adhesives.

Recently, preheating composite resin with appropriate devices such as Calset (AdDent Inc, Danbury, CT, USA) has been advocated as a method to reduce paste viscosity, to improve internal adaptation and marginal adaptation, and to shorten curing times.[43-45] Preliminary studies have demonstrated improved flowability and handling characteristics of preheated resins without alteration of their physical properties.[46] A strong positive correlation between temperature and monomer conversion has also been demonstrated *in vitro*.[47,48] However, *in vitro* testing of adaptation and microleakage resistance of preheated resins has produced mixed results.[49,50]

Identification of tiny bubbles in flowable composites that were expressed directly onto glass slabs and polymerized suggests that they were pre-existing as a result of manufacturing methods. The authors believed that the three larger voids they identified were related to the technical difficulties in placing flowable composite into narrow minimally invasive restorations. Although it is widely believed that flowable composite resins are easy to apply without voids, our results and those of others suggest that porosities remain.[25,51] Flowable composites inherently contain proportionally more resin and less filler than conventional composites. This reduces their viscosity and enhances their flowability. However, this makes them more difficult to pack into cavity preparations and increases the technical difficulty of removing microbubbles during the manufacturing process.

Anusavice, long ago, discussed criteria for the selection of restorative materials: properties vs technique sensitivity.[52] He identified the viscosity of composite resins as one of many factors influencing technique sensitivity. Both viscosity and void concentration influence rheology, or flow. However, study of the influence of rheology on technique sensitivity and clinical performance still remains in its infancy. It is also important to note that operator preference is quite a different matter than technique sensitivity or material performance.

Clinical preparation designs for minimally invasive restorations differ widely. The preparation

design used in this study, created using a standard fissurotomy bur, was extremely conservative, but it was also relatively narrow and deep, having an unfavorable C-factor. Another approach was taken by Mertz-Fairhurst and others in a landmark study on ultraconservative cariostatic sealed restorations.[53] They used 45° to 60° enamel bevels at least 1 mm wide but did not excavate carious dentin. This cavity conformation likely attained a highly favorable C-factor. An autocured highly-filled hybrid composite was placed and shaped, then covered with bonded fissure sealants. These restorations, placed directly over carious dentin in frankly cavitated lesions, arrested carious progress for 10 years, despite frequent loss of marginal seal. That data indicated that shallow sealed hybrid composite restorations are capable of conserving tooth structure, preventing recurrent caries, and extending restoration survival. That data also indirectly suggested that both the use of flowable composites and narrow deep fissurotomy preparations must be critically examined, or at least compared with other preparation designs.

The authors recognize that the generally disappointing results of this current study cannot necessarily be extrapolated to clinical performance. However, the authors advocate that minimally invasive Class I composite restorations be restored using long-used and well-studied conventional microhybrid composites until flowable composites have been validated in long-term controlled clinical trials.

## CONCLUSIONS

Microleakage varied substantially, by a whole order of magnitude, among the material groups tested. The control group, a conventional microhybrid composite material used with its manufacturer's recommended bonding agent, leaked significantly less than a wide variety of flowable composites used with their manufacturers' recommended bonding agents. Microleakage varied very slightly among measurement site locations. Tiny microbubbles were seen within many of the flowable composite specimens, as were a few voids.

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# Effect of Composite Insertion Technique on Cuspal Deflection Using an *In Vitro* Simulation Model

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### Clinical Relevance

All insertion techniques using composite materials caused measurable cusp deflection during polymerization, with little difference between different incremental techniques. The silorane-based composite produced significantly less cuspal movement.


## SUMMARY

**Objective: The objective of this study was to investigate, by simulation, the effect of conventional composite resin insertion techniques on cuspal deflection using bonded typodont artificial teeth. The deflection produced by a new low-shrinkage composite was also determined.**

**Materials and Methods: Sixty standardized MOD preparations on ivorine maxillary premolars were prepared: group A at 4 mm depth and group B at 6 mm depth. Each group was further subdivided according to composite insertion technique (n=6), as follows: 1) bulk insertion, 2) horizontal increments, 3) tangential increments, and 4) a modified tangential technique. Preparations were microetched, acid-cleaned, and bonded with adhesive resin to provide micromechanical attachment before restoration with a conventional composite (Spectrum TPH$^3$, Dentsply). Two additional subgroups at 4 mm and 6 mm depth (n=6) were restored in bulk using low-shrinkage composite (Filtek LS, 3M/ESPE). All groups received the same total photo-polymerization time. Cuspal deflection was measured during the restorative procedure using two Linear Variable Differential Transformers attached to a data acquisition system.**

**Results: The average cuspal deflections for group A were 1) 40.17 ± 1.18 μm, 2) 25.80 ± 4.98 μm, 3) 28.27 ± 5.12 μm, and 4) 27.33 ± 2.42**


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**μm. The deflections in group B were 1) 38.82 ± 3.64 μm, 2) 50.39 ± 9.17 μm, 3) 55.62 ± 8.16 μm, and 4) 49.61 ± 8.01 μm. Cuspal flexure for the low-shrinkage composite was 11.14 ± 1.67 μm (group A: 4 mm depth) and 16.53 ± 2.79 μm (group B: 6 mm depth).**

**Conclusions: All insertion techniques using conventional composite caused cuspal deformation. In general, deeper preparations showed increased cuspal deflection—except in the case of bulk insertion, which was likely affected by decreased depth of cure. Cuspal movement using low-shrinkage composite was significantly reduced.**

## INTRODUCTION

In spite of the increased use of resin composite materials in various procedures in dentistry, bulk contraction or polymerization shrinkage remains a major contributor to the clinical drawbacks associated with these materials.[1,2]

Polymerization stresses generated by polymerization shrinkage may compromise the bond integrity,[3] leading to concerns such as microleakage, postoperative sensitivity, and ultimately secondary caries.[1-8] If the composite-tooth bond remains intact, stresses transferred to tooth structure may result in cuspal flexure, enamel fracture, or fractured cusps.[4,7,9-13]

All methacrylate-based composite materials undergo polymerization shrinkage upon curing, with a reported range of 2-5%.[4] Optimizing particle sizes, maximizing filler content, and minimizing the concentration of 'diluent' monomers in the resin formulation are steps taken by the manufacturers to reduce the degree of polymerization shrinkage.[2]

In addition, material development by incorporation of ring-opening monomers has resulted in formulation of a new class of silorane composites with significantly lower volumetric shrinkage (less than 1%).[2] However, conventional composites are still widely used in practice, and polymerization shrinkage remains a clinical concern.

Incremental insertion techniques are recommended to reduce the undesirable effects of polymerization shrinkage by maximizing the ratio of unbonded to bonded surfaces (C-factor).[12] The unbonded surface purportedly allows for unhindered "flow" of composite monomers and permits stress relief along this surface. Incremental insertion techniques can also reduce the effects of polymerization shrinkage by reducing the bulk of composite cured with each layer, and it is generally recognized that the overall size and cavity configuration influence the resulting shrinkage stress and the degree of cuspal deflection.[12-16] Many different incremental insertion techniques are recommended; however, the evidence used to define the most appropriate technique is inconclusive, and many questions remain. Research to measure the degree of cuspal deflection with different materials and/or techniques *in vitro* has inherent limitations. The use of extracted teeth can be problematic as a result of their size, shape, and biological differences.[16] In addition, the modulus of elasticity varies between teeth, which can affect the degree of flexure and the interpretation of results. The use of artificial materials, such as aluminum blocks, has been suggested[16] to avoid these biological problems; however, the specimens used do not provide morphological similarity to teeth.

The aims of this study were to use an *in vitro* simulation model 1) to determine the effect on cuspal deflection of different incremental insertion techniques and 2) to determine the effect of a new proprietary low-shrinkage resin composite on cuspal deflection.

## MATERIALS AND METHODS

### Specimen Preparation

Sixty stylized, MOD preparations were prepared in maxillary second bicuspid ivorine teeth (Kilgore International, Coldwater, MI, USA). Cavities were prepared using a uniform cavity design with standardized measurements and were facilitated by a single operator. The width of the prepared cavities was two-thirds of the intercuspal distance (4 mm) at two variable cavity depths, 4 mm and 6 mm. The cavity depth was gauged from the tip of the buccal cusp to the pulpal floor. Buccal and lingual walls were prepared parallel without occlusal convergence. The stylized, slot MOD preparation, prepared without proximal boxes, was utilized in order to minimize preparation variation. The depth of 4 mm provided an intermediate overall MOD depth instead of the combination of a shallower occlusal portion with deeper proximal boxes. The 6mm depth preparations were included to simulate endodontically treated teeth that are deeper because of the necessity for occlusal access into the pulp.

All cavity preparations were air-abraded using 50-μm aluminum-oxide powder for 60 seconds to create cavity wall microroughness in order to simulate a bonded restoration. To ensure an adequate strength of micromechanical attachment between the com-

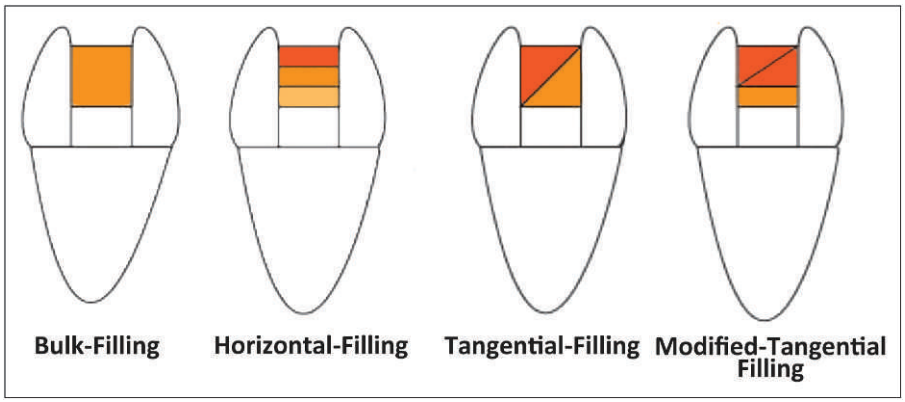


Figure 1. *Schematic diagram illustrating the different composite placement techniques.*

posite and ivorine teeth, a pilot study using standard microtensile testing was carried out. A group of ivorine teeth were sectioned, air-abraded, and bonded to both methacrylate and silorane-based resin composite using identical bonding agents and procedures as for the cuspal deflection study described below. Standard microtensile serial sectioning and bond strength testing (Bisco, Schaumburg, IL, USA) were performed. Means were calculated and were considered adequate to ensure attachment of restorations to the cavity walls.

Prepared teeth were divided into two main groups of 30 specimens each: group A (4 mm depth) and group B (6 mm depth). Each group was further subdivided into five subgroups (n=6) according to the composite and the insertion technique used. Four subgroups were all restored using a conventional hybrid composite, Spectrum TPH$^3$ (Dentsply, Caulk, Milford, DE,USA), and were categorized according to their insertion technique, as follows: 1) bulk insertion, 2) horizontal increments, 3) tangential increments, and 4) a modified tangential technique (Figure 1). The fifth subgroup of both groups was restored using a proprietary low-shrinkage (silorane-based) composite (Filtek LS, 3M ESPE, St Paul, MN, USA) inserted in bulk.

Bonding for all subgroups restored with Spectrum TPH$^3$ was carried out using Scotchbond Multi-Purpose Adhesive (3M ESPE), omitting the dentin primer step. The low-shrinkage composite restorations were bonded using the corresponding silorane adhesive, following the manufacturer's recommendations. Restorations were inserted and photopolymerized using a Demetron LC halogen light curing unit (Sybron Dental Specialist, Orange, CA, USA) with an intensity of 400 mW/cm$^2$. All subgroups received the same total curing time of 80 seconds, as defined in Table 1, and in all cases the light cure was directed from the occlusal surface. Light source was held as close as possible to the occlusal surface, avoiding any contact with the tooth to eliminate any effect on the Linear Variable Differential Transformers (LDTVs).

### Cuspal Deflection Measurements

Cuspal deflection was measured continuously throughout the restorative placement procedure using two LVDTs (AX/1/S, Omega, Stamford, CT,

Table 1: *Photo-Polymerization Scheme Followed for the Different Subgroups*

| Cavity Depth | Bulk Insertion | Horizontal Increments | Tangential Increments | Modified Tangential Increments |
|---|---|---|---|---|
| Group A (4 mm) | 80 seconds | 1 mm + 1.5 mm + 1.5 mm | Two equal increments | 1 mm + two equal increments |
| | | 20 × 20 × 40 = 80 s | 40 × 40 = 80 s | 20 × 30 × 30 = 80 s |
| Group B (6 mm) | 80 seconds | 2 mm + 2 mm + 2 mm | 2 × 2 mm increments | 2 mm + two equal increments |
| | | 20 × 20 × 40 = 80 s | 40 × 40 = 80 s | 20 × 30 × 30 = 80 s |

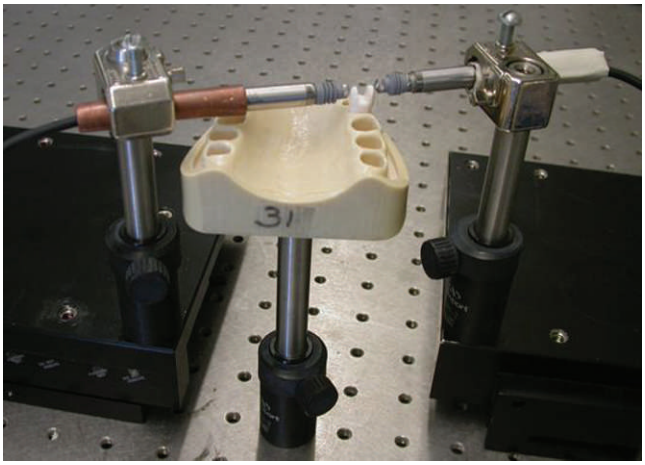

Figure 2. *LVDTs touching the buccal and lingual cusps (the mechanism allowed LVDTs to be constantly in contact with the tooth).*

USA) with a sensitivity of 2/2.03 mV/V. These were attached to an Instron machine DAX V7.0 data acquisition system at room temperature and mounted on a stabilized table. The LVDTs were placed such that they touched the buccal and lingual surfaces of the mounted ivorine tooth throughout the test (Figures 2 and 3). Cuspal deflection was recorded from the start of the restorative procedure until the deflection became a continuous plateau. The combined extent of buccal and lingual cuspal deflection was calculated and statistically analyzed using univariate analysis of variance with post hoc Tukey's test ($p<0.05$). If detachment from the bonded interface was identified from an abnormal pattern of deflection, the sample was rejected.

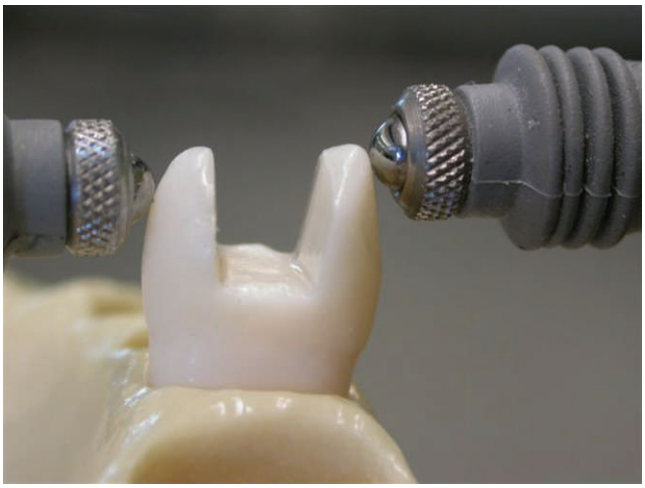

Figure 3. *LVDTs connected to data acquisition system, in which the entire setup was assembled on a stabilized table to minimize noise.*

Table 2: *Means ± Standard Deviations of Combined Buccal and Lingual Cuspal Deflection of all Subgroups Restored with Conventional Composite*[a]

| Insertion Technique | Group A (4 mm Depth) | Group B (6 mm Depth) |
|---|---|---|
| Bulk placement | 40.17 ± 1.18 Aa | 38.82 ± 3.64 Aa |
| Horizontal increments | 25.80 ± 4.98 Bb | 50.39 ± 9.17 Cb |
| Tangential increments | 28.27 ± 5.12 Bb | 55.62 ± 8.16 Cb |
| Modified tangential increments | 27.33 ± 2.42 Bb | 49.61 ± 8.01 Cb |

[a] *Means followed by different on-line small capital letters in the same row and lowercase letters in the same column are significantly different at* $p < 0.05$.

## RESULTS

In a pilot study, used to assess the strength of the composite to ivorine attachment, the mean microtensile bond strengths of conventional hybrid and low-shrinkage composite materials to air-abraded ivorine "tooth" surfaces were 27.56 and 28.33 MPa, respectively, ensuring adequate attachment with which to measure cuspal deflection.

All insertion techniques using conventional composite caused measurable cuspal movement. Table 2 shows the results for the combined palatal and lingual cusp deflection of all experimental groups restored with conventional composite. Table 3 provides the results for the combined palatal and lingual cusp deflection for those groups restored in bulk using proprietary low-shrinkage material. Group B (6 mm deep) generally revealed higher cusp flexure when compared to group A (4 mm deep); however, bulk insertion at both 4 mm and 6 mm produced essentially similar cusp movement (Figure

Table 3: *Means ± Standard Deviations of Combined Buccal and Lingual Cuspal Deflection of Subgroups Restored with Low-Shrinkage Composite*[a]

| Insertion Technique | Group A (4 mm Depth) | Group B (6 mm Depth) |
|---|---|---|
| Bulk-placement | 11.14 ± 1.67 A | 16.53 ± 2.79 B |

[a] *Means followed by different on-line small capital letters are significantly different at* $p < 0.05$.

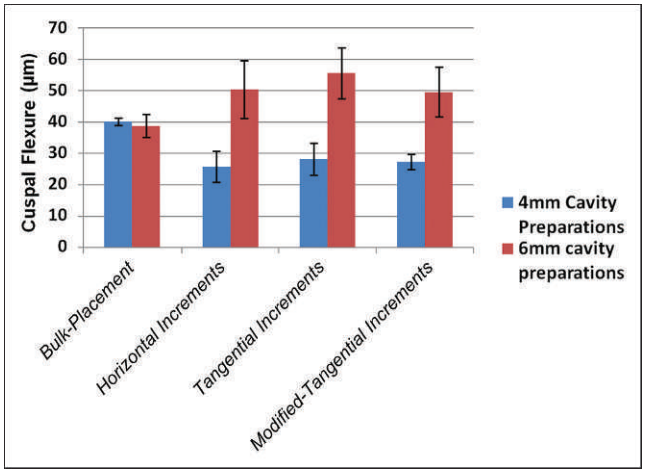


Figure 4. *Bar chart representing means and standard deviations (SDs) of combined buccal and lingual cuspal deflection of all subgroups restored with conventional composite.*

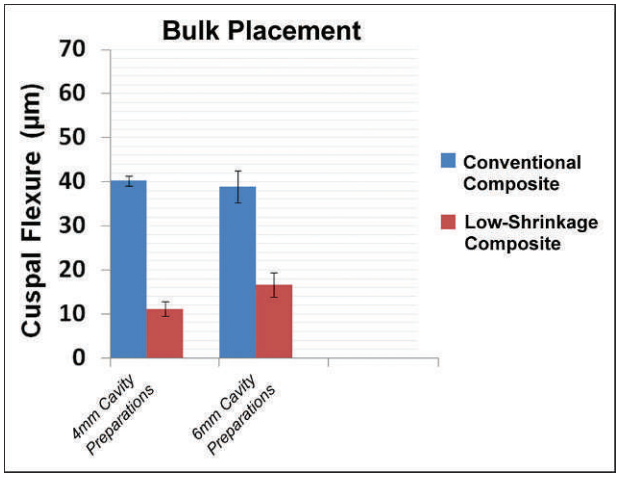


Figure 5. *Bar chart comparing means and standard deviations (SDs) of combined buccal and lingual cuspal deflection of subgroups restored with conventional and low-shrinkage composites placed only using bulk technique.*

4). At 4 mm depth, bulk placement produced greater cusp flexure than with any of the incremental insertion techniques. All incremental techniques used in 6 mm depth preparations were significantly higher than those for 4 mm depth. Overall, there were no statistically significant differences among the different incremental placement techniques at each preparation depth (Table 2).

Figure 5 illustrates the difference in cuspal flexure between conventional hybrid composite and low-shrinkage material when bulk placement was used. The low-shrinkage material demonstrated the lowest cuspal flexure of all experimental groups.

## DISCUSSION

It was possible to simulate the cuspal flexure resulting from intracoronal composite restorative procedures by using a micromechanical approach to achieve composite attachment to artificial ivorine plastic teeth.

All insertion techniques using composite resin produced measurable cuspal movement, which could be accurately and continuously recorded during the photo-polymerization process using LVDTs and a data acquisition system on a stable platform. An LVDT is a type of electrical transformer used for measuring linear displacement, and two LVDTs were placed such that they were touching the outer and upper buccal and lingual surfaces of the tooth. The stability of the setting was essential since any slight motion was easily detected by the data acquisition setting. Micromechanical attachment of composite to ivorine polymer was provided through use of air-abrasion. Preliminary microtensile bond strength measurements using this method showed that appropriate bond strengths could be obtained, as indicated by the results recorded in the pilot study. Detachment of composite from the internal preparation walls of the ivorine tooth during polymerization occurred only on two occasions and was obvious as an abrupt halt in movement on the continuous plot recording. Such samples were not included in the results. This experimental simulation system was therefore successful in mimicking the microscopic cusp deformation caused by polymerization shrinkage of composite, without the inherent difficulties involved in the use of extracted teeth. Natural teeth come in many different anatomical shapes and sizes, cannot be standardized, and are difficult to procure. Even a standardized cavity preparation on natural teeth would result in differing cavity wall thicknesses from tooth to tooth, which would affect the resulting cusp movement. The subsequent high standard deviations achieved with natural teeth often preclude determination of significant differences between materials or techniques.

That the use of artificial ivorine teeth was an effective method of simulating cusp movements in natural teeth is shown by the comparable range of cusp movements recorded in the published literature. According to a review by Versluis and others,[8] the overall reported range for studies using natural teeth was 16-45 µm, and the results of these authors' finite element analysis resulted in intercuspal changes of between 25.5 and 45.5 µm for MOD restorations of different sizes. Smaller deflections (15-23 µm) have been reported[11] for natural teeth

when smaller dimension cavities were utilized. Use of an experimental silorane low-shrinkage composite in natural bicuspids resulted in a cuspal deflection of 6 μm using a slightly smaller preparation size and a greater (8) number of increments than were used in the current study. [4] These comparisons provide some validation of the ivorine model used.

Ivorine teeth do not, however, replicate the combined properties of dentin and enamel inherent in natural tooth structure; therefore, the use of artificial replacements cannot provide absolute values of expected intraoral cuspal movement, nor do they permit subsequent assessment of marginal microleakage in the restored teeth. The simulation does allow comparison of different restorative materials, preparation sizes, and/or insertion techniques by providing a standardized tooth model. Such comparisons are more clinically realistic than the use of metal or plastic blocks as a result of the anatomical similarity to real teeth.[16]

All insertion techniques using conventional composite caused measurable cusp movement, and, in general, deeper preparations showed significantly higher cuspal deflection. The use of incremental insertion reduced the overall amount of flexure over bulk insertion at standard cavity depth (4 mm); however, there were no significant differences among the different incremental insertion techniques used. It is generally recognized that incremental insertion techniques can reduce the negative effects of polymerization shrinkage by reducing the bulk of composite cured with each layer. Increasing the ratio of unbonded to bonded surfaces has also been suggested to reduce the curing shrinkage by allowing unhindered "flow" in the unbonded surface layer. In this study differences between horizontal and tangential incremental techniques were not apparent, refuting the increased efficacy of tangential increments.

Deeper preparations were clearly more vulnerable to cuspal movement, which almost doubled between 4 mm and 6 mm depths of preparation, despite the use of increments. This is in general agreement with the mathematical theory discussed by Hood,[17] who stated that doubling the cavity depth increases the deflection by a factor of eight, hence the significantly greater risk of fracture for endodontically treated posterior teeth, which may have a cavity depth many times greater than a vital tooth as a result of the access opening. Parenthetically, in this study, the amount of cuspal flexure observed with bulk filling was essentially the same for the 6 mm cavity depth as it was for the 4 mm depth. It is hypothesized that this effect was due to light attenuation preventing the deepest layers of the restoration from full polymerization, which was carried out from the occlusal surface. In essence it is possible that the polymerization light was curing the composite to full cure only for the first 4 mm of depth, thus negating any major differences between 4 mm and 6 mm depths. In contrast, the use of increments allowed full access of each increment to the light, and full cure was effected, resulting in the development of greater contraction shrinkage over the full depth of the cavity preparation. The effect on cusp movement was therefore more significant.

The development of novel low-shrinkage, resin-based composites offers a potential reduction in polymerization shrinkage stresses generated at the tooth/restoration interface compared with current conventional methacrylate composites. The proprietary low-shrinkage silorane material used in this study showed significantly lower cuspal flexure, in accordance with the manufacturer's claim. A reduction in cuspal deflection, as well as a decrease in restoration microleakage, for a similar experimental silorane material has also been reported.[4] With lower polymerization shrinkage, increased marginal integrity,[2] and decreased microleakage,[4] silorane composites may provide potential for decreased marginal staining, decreased postoperative sensitivity, and greater restoration longevity. However, caution is advised with respect to attributing greater clinical success on the basis of lower shrinkage alone. A recent study[18] indicated that low volumetric shrinkage does not necessarily correspond to low polymerization stress development, particularly if the material has a high flexural modulus. Many factors determine restoration success and longevity; therefore, true outcome data will be dependent on appropriate clinical trials.

Shrinkage during curing of resin composite restorative materials can cause significant problems in adhesive dentistry, such as debonding of the restoration-tooth interface, microleakage, marginal staining, and postoperative sensitivity. The use of incremental insertion is recognized as one method of reducing these negative effects. This study confirms the advisability of incremental insertion but was unable to demonstrate the superiority of one particular incremental technique. The amplified negative effect of increased cavity depth was apparent despite the use of incremental insertion. Furthermore, the potential for a proprietary silorane composite material to significantly reduce cusp deflection during polymerization was demonstrated.

## CONCLUSIONS

Under the conditions of this study:

- All insertion techniques using conventional composite caused measurable cusp movement.
- In general, deeper preparations showed significantly higher cusp deflection.
- There were no significant differences in cusp deformation among different incremental insertion techniques.

Cusp flexure using low-shrinkage composite was significantly lower than that caused by use of conventional composite.

### Conflict of Interest Declaration

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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# Caries-preventive Activity of Fluoride-containing Resin-based Desensitizers

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### Clinical Relevance

Resin-based desensitizing agents may serve as persistent mechanical barriers and prevent the development of root caries.


## SUMMARY

**Objective: The purpose of this study was to evaluate the effects of different desensitizing agents on the prevention of root caries when applied to root surfaces.**

**Materials and Methods: Thirty human roots were sectioned into quarters with a 3 × 4 mm window. A desensitizer (VX, Clinpro™ XT Varnish; SP, Seal & Protect®; or PB, Clearfil™ Protect Bond) was applied to three of the quarters in each window. Teeth were stored separately in water for one day, 30 days, or 60 days. The remaining quarter, without the application of desensitizer, served as a control. After storage in water, all specimens were subjected to pH cycling. Scanning electron microscopy was used to observe the demineralization bands created on the subsurface layer. The weight percentages of fluorine (F), silica, and calcium (Ca) were determined using electron probe microanalysis to quantify the elemental distributions in the root dentin. The concentrations of F released during a pH cycling were measured.**

**Results: For the control group, the average lesion depth was 18.92 ± 5.42 μm, and the average Ca loss was 15.66% ± 6.80% in the superficial layer and 30.44% ± 9.61% in the subsurface layer. No Ca loss occurred in the desensitizer-treated groups. All desensitizing agents remained intact for at least 60 days. F levels were increased in the hybrid layer but not in the subhybrid area. Outward release of F diminished with time.**

**Conclusion: The F-containing resin-based desensitizers protected exposed root surfaces**


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**from demineralization. F liberated from the desensitizers was detected only at minimal levels.**

## INTRODUCTION

Exposed root dentin often contributes to the clinical signs of dentin hypersensitivity.[1] Dentin hypersensitivity can be treated by covering the exposed tubules, thereby preventing the transmission of pain-causing stimuli to pulpal nerve fibers. Denuded root surfaces have a large proportion of organic material, which makes these surfaces more soluble than enamel in acidic environments, resulting in increased susceptibility to caries development.[2,3] Fluoride-containing gels or varnishes are good choices for treatment of exposed roots with dentin hypersensitivity. Specifically, the creation of a calcium fluoride ($CaF_2$) barrier on root surfaces not only provides a fluorine (F) reservoir but also blocks the patent tubules. To maintain a formidable barrier composed of calcium (Ca) and F, topical F needs to be reapplied to the root surface on a regular basis.[4] For this reason, patient compliance is essential for successful outcomes using this treatment modality.[5]

F-containing resin-based desensitizers were introduced recently as an adjunctive option for the prevention of root caries. The rationale for the use of F-containing desensitizers for the prevention of caries development is that these types of desensitizers enhance the chemical resistance of dentin to mineral dissolution. Additionally, the polymerized resin layer of these desensitizers is expected to provide a physical shield for a prolonged period of time.[6] In this study, three commercial desensitizers were applied to the root dentin, and the effectiveness of these desensitizers as caries-protective agents was examined under conditions of mild acidic challenge. Scanning electron microscopy (SEM) was used to examine the extent of dentin demineralization. The levels of F, silica (Si), and Ca content were measured in the cross-sectioned surfaces of dentin that had been treated with desensitizers using electron probe microanalysis (EPMA). In order to compare the F-releasing abilities of the desensitizers, F concentrations were also assessed after aging at one day, 30 days, and 60 days.

The aims of this study were 1) to determine whether the root dentin treated with F-containing resin-based desensitizers consistently resists acidic challenge and 2) to quantify the amount of the F ions released from these desensitizers in two directions: outward liberation and inward penetration.

## MATERIALS AND METHODS

### Specimen Preparation

This study was approved by the Institutional Review Board of the Seoul National University Dental Hospital. Thirty human lower premolars extracted during orthodontic treatment were used within six months of extraction. The teeth were disinfected in 0.5% chloramine-T for a week and stored in distilled water at 4°C. The teeth were inspected to ensure that they were free of fractures or other defects. Cementum was removed using polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA) under microscopy. The crowns of these teeth were removed at the cementoenamel junction using a low-speed diamond saw (Isomet™ , Buehler Ltd, Lake Bluff, IL, USA), and the apical roots were also removed leaving 5-mm-long root segments. The segments were sectioned mesiodistally and buccolingually into four parts (Figure 1). Each of the root quarters was covered with a thin layer of acid-resistant nail varnish, with the exception of a 3 × 4 mm window on the outer root surface. Three of these window surfaces were coated using one of the following materials: Clinpro™ XT Varnish (3M ESPE; designated the VX group), Seal & Protect® (Dentsply, Konstanz, Germany; designated the SP group), or Clearfil™ Protect Bond (Kuraray, Okayama, Japan; designated the PB group). Additionally, one control window surface was left free of desensitizer (CC group: no treatment) (Table 1).

Following light curing, each of the 10 specimens in the three experimental groups was stored in distilled water for one day, 30 days, or 60 days, respectively. The 10 control specimens were stored in distilled water for one day. Specimens were then immersed in 10 mL of demineralizing solution (1.5 mM $CaCl_2$, 0.9 mM $KH_2PO_4$, and 50 mM acetate buffer, pH 4.8) for three hours, followed by immersion in 10 mL of remineralizing solution (1.5 mM $CaCl_2$, 0.9 mM $KH_2PO_4$, and 20 mM 4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid, pH 7.0) for 20 hours. During a five-day cycle, each specimen in solution was placed in a shaking incubator (SI-600R, JEIO TECH, Korea) at 37°C with agitation. Solutions were changed every day and stored at 4°C.

### SEM/EPMA Analysis

Specimens were embedded in epoxy resin (Epofix, Struers, Glasgow, UK) and horizontally cross-sectioned along the midline. The exposed cut surfaces were serially polished with 1200, 2400, and 4000 grit

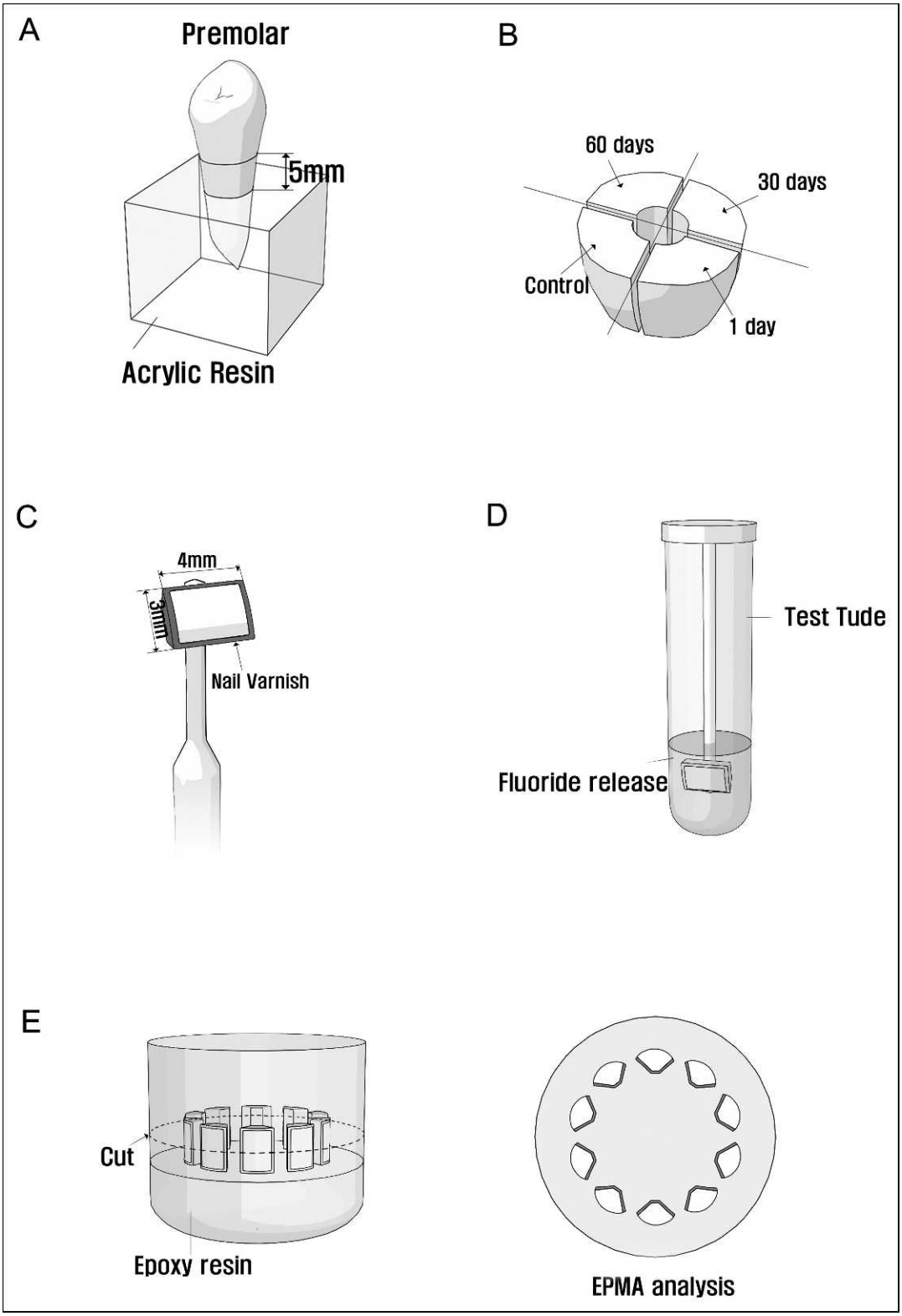


Figure 1. *Schematic diagrams of specimen preparation. (A) A human lower premolar was sectioned to produce a 5-mm-long root segment. (B) Each quarter segment was divided into one control and three treated groups. (C) Each quarter was painted with nail varnish, leaving a 3 × 4–mm window on the root surface. (D) After treatment, each specimen was immersed in pH cycling solution. (E) After cycling, specimens were embedded in epoxy resin and cross-sectioned at the midline for EPMA analysis.*

Table 1: *Materials Used in the Study*

| Product | Component | | Application |
|---|---|---|---|
| Group VX: Clinpro™ XT Varnish (3M ESPE, St Paul, MN, USA) | Copolymer of acrylic and itacoic acids, water, HEMA, silane-treated glass, silane-treated silica, Bis-GMA | | Etch for 15 seconds with 35% phosphoric acid (3M™ ESPE™ Scotchbond™ Etchant). Rinse for 15 seconds. Mix paste/liquid components together rapidly (10–15 seconds). Apply a thin layer (1/2 mm or less) of the mixed material to the tooth surface. Light-cure for 20 seconds. |
| Group SP: Seal & Protect® (Dentsply, Konstanz, Germany) | Di- and trimethacrylate resins, PENTA, functionalized amorphous silica, photoinitiators, butylated hydroxytoluene, cetylamine hydrofluoride, triclosan, acetone | | Apply Seal & Protect to the dentin surface and leave it undisturbed for 20 seconds. Remove excess solvent by gentle air-blowing. Light-cure for 10 seconds. Apply a second layer of Seal & Protect and remove excess solvent from the second layer by gentle air-blowing. Light-cure for 10 seconds. |
| Group PB: Clearfil™ Protect Bond (Kuraray, Okayama, Japan) | PRIMER | MDP, MDPB, HEMA, hydrophilic dimethacrylate, water | Apply PRIMER to the dentin surface and leave it in place for 20 seconds. Remove excess solvent by gentle air-blowing. Apply BOND and create a uniform bond film using a gentle air flow. Light-cure for 10 seconds. |
| | BOND | MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, DL-camphorquinone, N, N-diethanol-p-toluidine, silanated colloidal silica, surface-treated sodium fluoride | |

*Abbreviations: Bis-GMA, bis-phenol A diglycidylmethacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MDPB, 12-methacryloyloxydodecylpyridinium bromide; PENTA, dipentaerythritol penta acrylate monophosphate.*

aluminum oxide ($Al_2O_3$) abrasive papers, followed by 1 μm and 0.25 μm diamond and 0.1 μm and 0.05 μm alumina polishing suspensions (Struers, Copenhagen, Denmark). The specimens were ultrasonically cleaned in deionized water for 10 minutes, dried for 72 hours in a desiccator, and then sputter-coated with carbon. Demineralization bands on the cross-sectioned surfaces were identified at a magnification of 600× using the phase contrast of backscattered electron imaging mode of SEM (JEOL JSM-6610LV, JEOL, Akishima, Japan). To identify variations in the amounts of the specified elements from the outer dentin to the inner dentin, two scans were performed perpendicular to the outer surface at 0.3 μm pixel intervals. The observation areas (superficial layer, demineralized layer, sound dentin, desensitizer layer, and hybrid layer) were determined according to changes in Ca, F, and Si content using EPMA (JEOL JXA-8100, JEOL). The operating conditions for both the image and elemental analyses were 15 kV of accelerating voltage and 50 nA of beam current. Measurements along the scan were averaged into a single value for each area. The F content was expressed as the percentage of weight relative to the total weight of a standard material where the measurement was taken (Table 2). A fluorapatite crystal (3.38% F) was used as a standard comparison for F.

Table 2: *The Mean Weight Percentage (Standard Deviation, SD) of Fluoride in the Desensitizer Layer*[a]

| Aging Period, d | N | Group | | |
|---|---|---|---|---|
| | | VX Desensitizer Layer | SP Desensitizer Layer | PB Desensitizer Layer |
| 1 | 20 | 11.94 (2.39) A | 1.01 (0.45) A | 1.32 (0.25) A |
| 30 | 20 | 11.88 (2.74) A | 0.67 (0.13) B | 1.23 (0.13) A |
| 60 | 20 | 10.77 (2.63) A | 0.56 (0.13) B | 1.30 (0.13) A |

*Abbreviations: PB, Clearfil™ Protect Bond; SP, Seal & Protect®; VX, Clinpro™ XT Varnish.*
[a] *Online small-capital letters denote values that are not significantly different from one another in each column ($p<0.05$).*

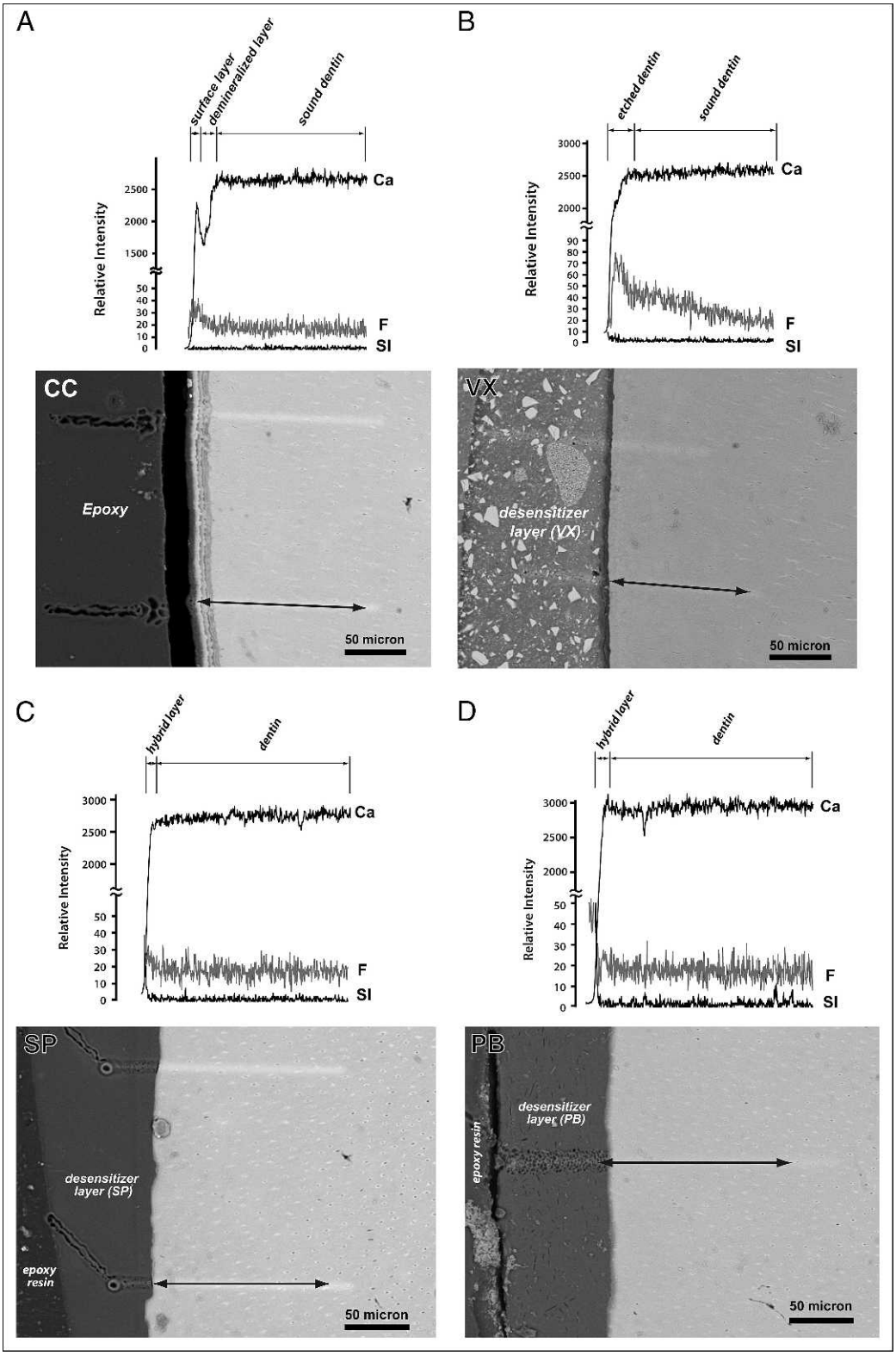


Figure 2. *SEM images and EPMA graphs were compared for each experimental group at 60 days of aging (600× magnification). (A) CC group. Elemental analysis along the scan line (dark arrow) illustrated the differentiated elemental composition (Ca, F, Si) of root dentin. Repeated demineralization bands were produced by a pH cycling series. Note that Ca level decreased in the superficial layer, followed by a sharp decline in the subsurface demineralized layer. F was detected at minimal amounts throughout the sound dentin, while no detectable Si was observed. (B) VX group. In a thickly built-up desensitizer layer, RMGI displayed various sizes of fillers embedded in matrix. No demineralization bands were observed in*

### Fluoride Concentration Measurement

A fluoride-specific ion electrode connected to a digital pH/millivolt meter (Accumet© Research Model AR 25, Thermo Fisher Scientific, Beverly, MA, USA) was calibrated using F reference solutions (1.0 and 10.0 ppm). The demineralization solution (4 mL) was mixed with total ionic strength adjustment buffer (TISAB II; 4 mL; Thermo Fisher Scientific) to obtain a constant background ionic strength. The mixed solution was placed over a nonheating magnetic stirrer, and readings were taken after a five-minute immersion period. The temperature of the solution was maintained at 22°C. Sample readings were obtained in μg F/mL (ppm F).

### Statistical Analysis

The normality and homogeneity of the samples were tested using the Kolmogorov-Smirnov test. The weight percentages of F in the desensitizer layer and the hybrid layer were compared among all groups using two-way analysis of variance (ANOVA) and the Tukey *post hoc* test. The F released during demineralization cycling was compared among groups using repeated-measures ANOVA with mixed model. A $p$-value of 0.05 was selected as the threshold for statistical significance. Analyses were performed using SAS 9.1.3 (SAS Institute, Cary, NC, USA).

## RESULTS

SEM revealed the formation of demineralization bands under the root surfaces in the control group (CC group) (Figure 2A). The width of the surface layer was 4.92 ± 0.92 μm, and that of the subsurface demineralized layer was 13.97 ± 4.44 μm. In the desensitizer-treated groups (VX, SP, and PB), no demineralized layers were observed at any stage of aging (one day, 30 days, or 60 days). Additionally, the desensitizer layer covering the dentin surface remained intact even at 60 days.

Line analyses revealed the individual distributions of Ca, F, and Si, and it was possible to distinguish the desensitizer layer from the dentin (Figure 2B-D). In the CC group, the average Ca loss was 15.66% ± 6.80% in the superficial layer and 30.44% ± 9.61% in the subsurface layer. In the experimental groups (VX, SP, and PB), Ca levels were maintained from the surface layer to a depth of 200 μm without any discernable changes. Si levels were elevated in the desensitizer layer of each of the three experimental groups. However, these levels abruptly dropped to near zero at the dentin interface. In the desensitizer layer, F levels were significantly higher in the VX group ($p<0.001$). Ca levels started to rise at the dentin interface and reached a plateau at the inner dentin layer. In the SP and PB groups, there were transitional areas or hybrid layers in which a decrease in Si levels and an increase in Ca levels overlapped (Figure 2C,D). Unlike the SP and PB groups, in the VX group no Si was detected in the etched dentin layer. However, a significantly higher level of F was detected in the etched dentin area of the VX group than in the hybrid layers of the SP and PB groups ($p<0.001$). F levels were significantly different in the desensitizer layers of the SP group (between one day and 30 days; Table 2). The hybrid layers showed a significant change in F levels in the CL group (between one day and 30 days; Table 3). The mean widths of the etched dentin layer of the VX group and the hybrid layers of the SP and PB groups are listed in Table 4.

Table 5 summarizes the concentrations of F released during each demineralizing cycle. F release was the highest on the first day and significantly decreased throughout the duration of cycling ($p<0.001$; Figure 3). There was also a significant difference in F release between the one-day and 30-day time points for each of the experimental groups (VX, $p=0.022$; SP, $p=0.036$; and PB, $p<0.001$). The VX group released significantly more F than did the SP and PB groups at one day and 30 days ($p<0.001$), and the SP and PB groups did not differ significantly at either time point. The level of released F was not obtained from the SP and PB groups at 60 days.

## DISCUSSION

Fluoride has been shown[7] to be one of the materials used to decrease the permeability of dentin, possibly by precipitation of insoluble calcium fluoride within the tubules. The fluoride in the desensitizers examined in this study was expected to result in

←
*dentin. Ca levels started rising in the etched dentin layer, leveling off in the sound dentin. F and Si in the desensitizer layer were detected at elevated levels but were not depicted in the above graph. In the etched dentin, only F was maintained at an increased level compared to the sound dentin. (C) SP group. The desensitizing layer was protecting the dentin surface well, without signs of demineralization. Ca started from the zero level, abruptly increased, and leveled off in the sound dentin area. In contrast, Si started decreasing at the beginning of the hybrid layer and disappeared in the sound dentin. F showed a similar pattern to Si in the hybrid layer. (D) PB group. A well-maintained desensitizer layer covered the dentin surface. No demineralization occurred. Ca, F, and Si showed the same pattern as in the SP group.*

Table 3: *The Mean Weight Percentage (Standard Deviation, SD) of Fluoride in the Hybrid Layer*[a]

| Aging Period, d | N | Group | | |
|---|---|---|---|---|
| | | VX Etched Dentin Layer[b] | SP Hybrid Layer | PB Hybrid Layer |
| 1 | 20 | 1.94 (0.51) A | 0.88 (0.49) A | 0.90 (0.18) A |
| 30 | 20 | 2.14 (0.88) A | 0.73 (0.55) A | 0.66 (0.23) B |
| 60 | 20 | 2.00 (0.68) A | 0.65 (0.38) A | 0.67 (0.31) B |

*Abbreviations: PB, Clearfil™ Protect Bond; SP, Seal & Protect®; VX, Clinpro™ XT Varnish.*
*[a] Online small-capital letters denote values that are not significantly different from one another in each column ($p<0.05$).*
*[b] No hybrid layer was determined in the VX group. Instead, the etched dentin layer in the VX group was compared with the hybrid layers in the SP and PB groups.*

Table 4: *The Mean Width (Standard Deviation, SD) of the Superficial Layer and the Demineralized Layer in the Control Group (CC) and of the Hybrid Layer in the Experimental Groups (VX, SP, and PB) ($\mu m$)*

| N | Group | | | | |
|---|---|---|---|---|---|
| | CC | | VX | SP | PB |
| | Superficial Layer | Demineralized Layer | Etched Dentin Layer | Hybrid Layer | Hybrid Layer |
| 60 | 4.92 (0.92) | 13.97 (4.44) | 16.63 (1.91) | 4.24 (1.12) | 4.41 (1.07) |

*Abbreviations: CC, no treatment; PB, Clearfil™ Protect Bond; SP, Seal & Protect®; VX, Clinpro™ XT Varnish.*

tubule obturation and caries inhibition. In addition, a resinous coating of the desensitizers is capable of blocking the acid transportation into the root dentin.[4] The first aim of our study was to determine whether the application of the desensitizers to the root surfaces is protective against the effects of exposure to an acidic environment. We subjected freshly exposed root dentin to a mild acidic challenge and simulated a previously low- to moderate-caries risk group that became newly susceptible to the disease as a result of recently uncovered root surfaces. The use of pH cycling produced multiple demineralization bands underneath a relatively intact superficial layer, implying that a series of dynamically balanced reactions had occurred (Figure 2). The desensitizer-coated dentin showed no signs of demineralization, indicating that the coating material provided a complete seal against the effects of acidic challenge. The acid-resistant protection remained for 60 days in all three desensitizer groups. VX, a resin modified glass ionomer (RMGI)-based desensitizer, had a thick consistency and produced thicker layers than did SP, a desensitizer based on one-step self-etch resin adhesive, and PB, a two-step self-etch resin adhesive. Many *in vitro* analyses of artificial caries have been performed under conditions of severe acidic challenge (immersion in strong acid without the use of a remineralizing cycle).[8–10] When the polymerized resin barrier is thick, less soluble, and water stable, it is able to prevent acid penetration.[9] Clinically, it is rare to use only adhesive resin without consecutive build-up of composite resin and to directly expose adhesive resin-applied dentin to an aqueous environment. We wondered whether a desensitizer, composed largely of solvent and hydrophilic monomers, would resist hydrolytic degradation. SEM images showed that all three desensitizers remained intact with a minimum thickness of 40–50 μm. Additionally, relatively mild pH cycling did not result in disintegration of the surface coating layer during the 60 days of water storage. The mechanical impact induced by toothbrush abrasion on resin-adhesive bonded root surfaces should be considered in future studies.

Another purpose of our study was to quantify the amount of F released from the solidified desensitizer layer. The manufacturers of the three desensitizers claim that their supplementation with F has the benefit of caries prevention. However, the resin-based desensitizers used in this study may not contain as much F as conventional F varnishes, which often contain 5% sodium fluoride (22,600 ppm F).[4] Furthermore, the way in which sodium F in CL and amine F in SP dissociate from the photopolymerized resin composite and dissolve in water is unclear. If the F were to dissolve, surface wash-off processes could be induced by water sorption of hydrophilic monomers. In this case, the resin matrix may provide an aqueous environment to facilitate the transport of F ions.[11] In RMGI, an acid-base reaction enhances the leaching of F ions to form a polysalt matrix, enabling the release of more F than is released from resin adhesives with F-incorporated filler particles.[12] However, even RMGI has a limited span of rapid F release, with a significant decrease within an initial period.[13] The amount of F released

Table 5: *The Mean Daily Fluoride Release (Standard Deviation, SD) During a Demineralization Cycle at One Day and 30 Days ($10^{-1}$ ppm)*

| Aging Period, d | pH Cycle | Group | | |
|---|---|---|---|---|
| | | VX | SP | PB |
| 1 | Day 1 | 3.78 (1.45) | 0.64 (0.04) | 0.83 (0.02) |
| | Day 2 | 3.18 (1.23) | 0.53 (0.01) | 0.65 (0.03) |
| | Day 3 | 2.73 (0.89) | 0.50 (0.01) | 0.64 (0.04) |
| | Day 4 | 2.42 (0.61) | 0.50 (0.00) | 0.57 (0.03) |
| | Day 5 | 2.31 (0.48) | 0.49 (0.00) | 0.54 (0.01) |
| 30 | Day 1 | 3.31 (0.44) | 0.61 (0.05) | 0.62 (0.01) |
| | Day 2 | 2.07 (0.59) | 0.49 (0.01) | 0.52 (0.01) |
| | Day 3 | 1.15 (0.11) | 0.48 (0.01) | 0.51 (0.01) |
| | Day 4 | 1.10 (0.10) | 0.47 (0.01) | 0.51 (0.01) |
| | Day 5 | 1.06 (0.09) | 0.48 (0.15) | 0.49 (0.01) |

*Abbreviations: PB, Clearfil™ Protect Bond; SP, Seal & Protect®; VX, Clinpro™ XT Varnish.*

in *in vitro* models is largely dependent on the volume of the material used, the size of the exposed surface, the duration of immersion, and the pH of the solution.[14,15] Each quarter root of human premolar only had a 3 × 4–mm surface area exposed and allocated to each of four groups. Furthermore, the demineralization solutions were changed at each cycle, which is more similar to a clinical situation than to cumulative collection. The short duration (three hours) of the demineralizing cycle allowed a very small amount of F to be leached, and levels were below levels of detection for the SP and PB groups at 60 days. Previous studies have often used cumulative concentrations of F in order to obtain the viable data for statistical analysis. This is especially true when low-concentration F-containing resin adhesives or resin composites have been investigated.[12,13,16-18] *In vitro* caries models have indicated that materials containing relatively small quantities of F should be capable of releasing, absorbing, and re-releasing over a long period in order to be effective in caries inhibition.[19] Based on the result of this study, the resin-based desensitizers did not seem to release sufficient levels of F to bring about a clinically remarkable impact.

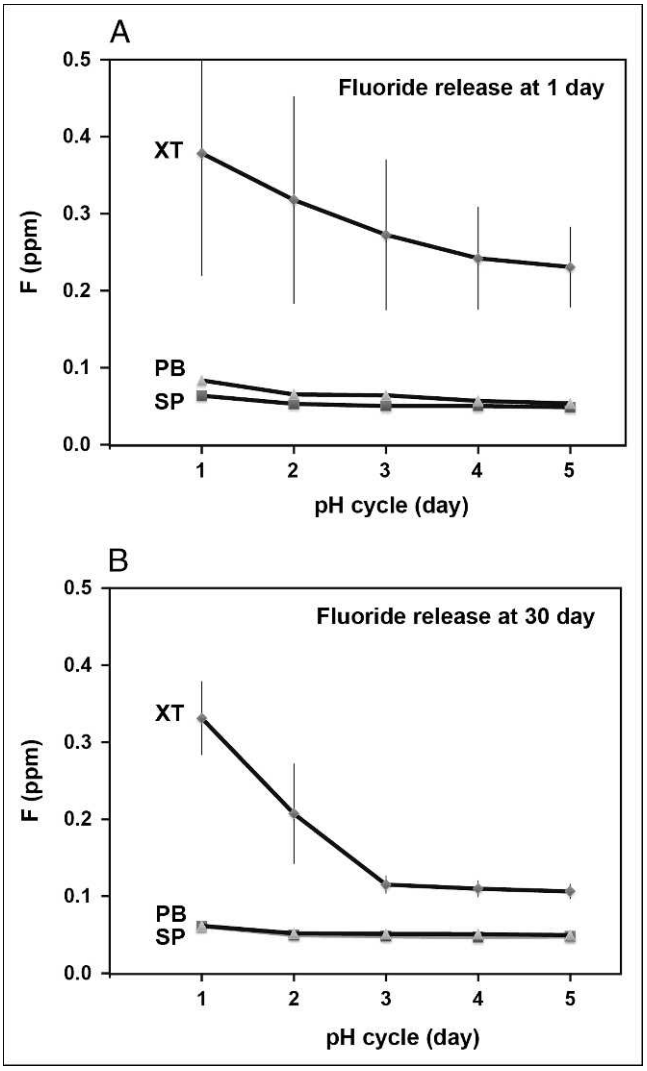


Figure 3. *Concentration of F released during a demineralizing cycle was measured at one day and 30 days. VX released significantly more fluoride than did SP and PB at each cycle at both time points ($p<0.001$).*

Freshly exposed dentin surfaces after the grinding of cementum were used in this study and were made to resemble cavity walls prepared for restoration. Few studies have quantified the depth and the intensity of F penetrated into dentin in terms of the inward migration of F from resin-based materials. Using energy dispersive spectroscopy (EDS) and wavelength dispersive spectroscopy WDS, Ferracane and others[20] determined that F moved from the filler particles through the adhesive matrix and then down a concentration gradient into the hybrid layer and dentin. Additionally, the migration of F was observed only when marginal leakage was produced

and when water was available to dissolve F ions. More recently, Han and others[8] used EPMA to show that the F density in the interfacial area is increased by the diffusion of pre-reacted glass ionomer fillers. However, this was the case only with a new form of adhesive resin, but not with conventional F-containing adhesive resin. When F ions can be supplemented from the bottom of the hybrid layer, the acid-resistant zone may build up to prevent secondary caries formation and lesion progression.[21] Previous studies[3,9,13,16] have examined the association between the extent of secondary lesions produced in root dentin and the availability of F in the adhesive resin. In the case of a self-etch adhesive system, it was suggested that the spaces created by acidic monomers underneath the hybrid layer may be filled by dissolved calcium and phosphate ions and that F ions may migrate from the hybrid layer.[11] This F concentration at the base of hybrid layer may then continue to diffuse into the deeper dentin over time. We detected significant changes of F in the desensitizer layer of the SP group and in the hybrid layer of CL over time (Tables 2 and 3). The time-related observations were not performed with identical specimens, since specimens needed to be sectioned and subjected to the elemental analysis. Instead, each quarter segment produced from a single root was compared in order to decrease individual substrate variation. Newly dispensed materials were also used for every application, and the manufacturer's instructions were carefully followed in order to produce a uniform mixture of coating on each specimen. No elevated levels of F were detected in the subhybrid layers, and the diffused F ions, if any, may not have been sufficient in number to be identified by EPMA. The root surface was etched with 37% phosphoric acid prior to application of VX in order to facilitate F incorporation.[22] As dentin permeability increased, the effective surface area for F ion absorption and ionic exchange was expected to be enhanced. The phosphoric acid–etched dentin area was wider (16.63 ± 0.78 μm) than the hybrid layer in two of the groups (SP, 4.24 ± 1.12 μm; CL, 4.41 ± 1.07 μm). Si was not detected in this partially Ca-depleted area, while F was present at elevated levels throughout the area. The amount of F in the etched dentin area was higher than in the hybrid layer for the SP and CL groups ($p<0.001$). This F-infiltrated interface may serve as a front against acidic challenge.

Occlusion of the exposed dentinal tubules *via* wear-resistant surface coating is a key mechanism for the prevention of dentin hypersensitivity.[3] When resin-based desensitizing agents are micromechanically attached to dentin, they build hermetic barriers against acid penetration. Even non–F-containing resin adhesives were shown to successfully protect root dentin surfaces from caries development.[9,10,23] Furthermore, the thinly applied desensitizer layer would likely be minimally influenced by its own polymerization shrinkage, thereby retaining bonding integrity better than composite-filled cavities. Another mechanical impact related to *in vivo* simulation may be mechanical abrasion mimicking daily brushing practices, which might result in wear-off of the resin coatings. However, when root surfaces are exposed as a result of gingival recession, some areas are not easily accessible by a toothbrush. Elderly patients who have difficulties maintaining adequate oral hygiene are particularly prone to rapid root caries initiation.[24] Thus, the susceptible area with plaque retention may benefit from root surface sealants such as resin-based desensitizers as a form of intensive preventive care.

## CONCLUSIONS

Within the limitations of this *in vitro* study, F-containing resin-based desensitizers were shown to be effective in sealing dentinal root surfaces for at least 60 days. The release of F from the desensitizers dropped over time, and the inward diffusion of F into the deep dentin layer was below detectable levels. The physical barrier provided by the polymerized resin matrix remained during a 60-day period.

### Conflict of Interest Declaration

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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# Cervical Margin Integrity of Class II Resin Composite Restorations in Laser- and Bur-Prepared Cavities Using Three Different Adhesive Systems

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### Clinical Relevance

Bur-prepared cavities represented less interfacial gap width than laser-prepared cavities. A self-etching adhesive system showed the least interfacial gap compared to etch-and-rinse adhesives and performed similarly in bur- and laser-prepared cavities.

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## SUMMARY

**One of the challenges in durability of posterior tooth-colored restorative materials is polymerization shrinkage, which results in gap formation between the restoration and tooth structure. The aim of the present study was to investigate marginal adaptation of Class II composite restorations using a self-etching and two etch-and-rinse adhesive systems in cavities prepared either with bur or Er,-Cr:YSGG laser. A total of 45 extracted sound human premolars were selected. In each tooth, mesial and distal Class II cavities were prepared either by a diamond bur or by Er,-Cr:YSGG laser with the margins 1 mm apical to the cemento-enamel junction. Then the teeth were randomly divided into three groups of 15 each, according to the type of the adhesive system used (Single Bond, Single Bond 2,**

**and Adper Easy One adhesive systems). Subsequent to restoring the teeth, the specimens were subjected to thermal cycling between 5 ± 2°C and 55 ± 2°C for 500 cycles and were then cut longitudinally into two halves using a diamond disk. Marginal adaptation was evaluated using a stereomicroscope, and the values for gap widths were obtained in micrometers. Data were analyzed using two-factor analysis of variance and *post hoc* tests. There were statistically significant differences in mean marginal gap widths between the adhesive type and preparation groups ($p<0.05$). The interfacial gap width in bur-prepared cavities was significantly less than that in laser-prepared cavities, and the lowest gap width was observed in Adper Easy One regardless of the type of the preparation.**

## INTRODUCTION

Adhesive dentistry combined with tooth-colored restorative materials plays a significant role in minimally invasive dentistry.[1] Since their development, several improvements have been made to the physical and mechanical properties of resin composites and have permitted their successful use in posterior restorations.[2] However, one of the challenges in durability of tooth-colored restorative materials is polymerization shrinkage, which results in stress and gap formation between the restoration and tooth structure.[3] Bacteria and fluids present at this interface compromise the durability of the restoration. This is more prominent in the cervical margins of proximal boxes.[4] In addition to polymerization shrinkage, surface characteristics of prepared enamel and dentin can influence shrinkage stress and gap formation at the tooth-resin composite interface.[5] Surface characteristics of teeth are different, depending on the preparation procedure.[6] In light of minimal-invasive dentistry, laser technology has been widely used as an alternative to the conventional use of diamond burs.[7] The ability of laser to remove enamel and dentin was found comparable to that achieved with the conventional dental drills. It has been reported that laser-irradiated surfaces are rough, clean, and free of debris with most of the dentinal tubules visible and wide open. These characteristics would favor the adhesion procedure.[6] Moreover, some studies have reported that the dentin layers that have undergone a beam of pulsed erbium lasers might resist the removal of minerals by the acid-etch technique and, as a result, can prevent the penetration of resin tags into the intertubular dentin, which might give rise to an incomplete hybridization with the resultant low bond strength.[8,9]

The quality of the margins of composite fillings is a frequently discussed topic in relation to the use of a laser for dental hard tissue preparation.[10] In addition, the interaction of lasers with newly developed dental materials is not fully understood.[6] To date, little information is available about the quality of margins of composite fillings in cavities prepared with Er,Cr:YSGG laser using gap analysis. Therefore, the aim of this comparative study was to investigate the marginal adaptation of Class II composite restorations in cavities prepared with bur or Er,Cr:YSGG laser, using a self-etching and two etch-and-rinse adhesive systems.

## METHODS AND MATERIALS

Forty-five human premolars were selected from extracted teeth that met the inclusion criteria (sound, without cracks and wear facets). The selected teeth were stored in 0.5% chloramine T solution at 4°C and used within three months.

In each tooth, mesial and distal standard Class II cavities were prepared either by cylindrical diamond burs (SS White Burs, Inc, Lakewood, NJ, USA) or by the Er,Cr:YSGG laser. The axial wall depth and the buccolingual dimension of the cavities were 1.5 mm and 2 mm, respectively (Figure 1). The cervical margins of Class II cavities were placed 1 mm apical to the cemento-enamel junction (CEJ). The Er,Cr:YSGG laser used was the Waterlase (Biolase Europe GmbH, Floss, Germany). The system emits light with a wavelength of 2870 nm. The pulse frequency is a constant 20 Hz, and the pulse length is 140 μs. A power of 6 W (300 mJ) was used for enamel preparation, and 5 W power was used (250 mJ) for dentin preparation.[10] The working distance

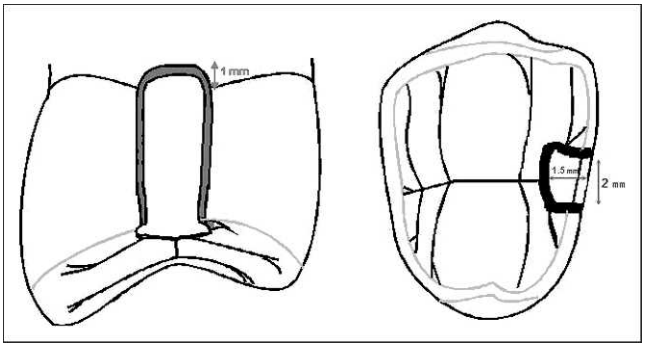


Figure 1. *Schematic representation showing Class II cavity dimensions.*

Table 1: *Chemical Composition and Application Mode of Adhesive Systems Used*

| Adhesive system | Composition | Application mode |
|---|---|---|
| Single Bond; two-step etch and rinse | Etchant: 37% phosphoric acid gel<br>Adhesive: HEMA, Bis-GMA, ethanol, water, polyalkenoic acid copolymer | Apply the etchant for 15 s; rinse for 10 s; apply two coats of adhesive; gently air-dry the surface for 5 s and light cure for 10 s. |
| Single Bond 2; two-step etch and rinse | Etchant: 37% phosphoric acid gel<br>Adhesive: HEMA, BisGMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids, silica nanoparticles | Apply the etchant for 15 s; rinse for 10 s; apply adhesive for 15 s; gently air dry the surface for 5 s and light cure for 10 s. |
| Adper Easy One; one-step self-etch | HEMA, Bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate methacrylate functionalized polyalkenoic acid (Vitrebond™ copolymer), finely dispersed bonded silica filler with 7-nm primary particle size, ethanol, water, initiators based on camphorquinone, stabilizers | Apply adhesive to tooth surface for a total of 20 s; dry the adhesive for 5 s and light cure for 10 s. |

*Abbreviations: HEMA, hydroxyethyl methacrylate; BisGMA, bisphenol-glycidyl methacrylate.*

was 0.5 mm while the air and water pressures for tooth preparation were 65% and 55%, respectively.

Then the teeth were divided into three groups of 15 each according to the type of the adhesive system used. In the first group, Single Bond adhesive system (3M ESPE, Dental Products, St. Paul, MN, USA) was used according to the manufacturer's instructions (Table 1). Then Filtek Supreme (3M ESPE), a nanohybrid composite, was filled into the cavities incrementally using Tofflemire matrix, and each layer was cured using Australis 7 light-curing unit (Ivoclar Vivadent, Schaan, Liechtenstein) at a light intensity of 500 mW/cm$^2$ for 40 seconds. Subsequent to the matrix removal, postcuring was carried out for 60 seconds at an intensity of 700 mW/cm$^2$.

In the second and third groups, Single Bond 2 (3M ESPE) and Adper Easy One (3M ESPE) adhesive systems were used according to the manufacturer's instructions (Table 1). The restoration procedure was the same as that in the first group.

The specimens were subjected to thermal cycling between 5 ± 2°C and 55 ± 2°C for 500 cycles with a dwell time of 20 seconds per bath.[10] Then the teeth were stored in distilled water for 24 hours and cut longitudinally (mesiodistally) into two halves using a diamond disk (Diamant GmbH, D&Z, Berlin, Germany). Marginal adaptation evaluation was performed using a stereomicroscope (Olympus SZX9, Tokyo, Japan). The selected areas were photographed with a digital imaging system (Olympus, DP12-BSW, version 01.03) and then the images were transferred to a computer for measuring the gap.

Interfacial gap width was measured with Olysia software (Olympus soft imaging system (SIS), Build 0831), which measured the marginal gaps at three locations (the mean values in Table 2). Gap width measurement was performed by determining two points on each gap vector (restoration-side vector) and (root-side vector) and by measuring the distance between them. The previously mentioned procedure was carried out at three locations that are specified in Figure 2 (outer part, middle part, and inner part of the cervical margin). The mean values in Table 3 have been obtained by averaging the three measurements for each specimen. The values for gap width were obtained in micrometers. One-way analysis of variance (ANOVA) was used to compare mean gap width values at three locations of the cervical margin, and two-by-two comparisons were performed using Tukey HSD test. For each treatment group, the mean values of marginal gaps at the three locations were calculated. Two-factor ANOVA was used to determine the effect of the type of adhesive and preparation on gap width. Pairwise comparisons of the study groups were performed using Tukey HSD and Mann-Whitney U-tests. Significance level was defined at $\alpha = 0.05$ for comparison of the groups.

Table 2: *Mean Marginal Gap Width (μm) ± SEM at Three Locations of the Cervical Margin in Study Groups*

| | Type of preparation | | | | | |
|---|---|---|---|---|---|---|
| | Bur | | | Laser | | |
| | Outer | Middle | Inner | Outer | Middle | Inner |
| Single Bond | 0.11 ± 0.01 | 0.08 ± 0.008 | 0.06 ± 0.008 | 0.21 ± 0.02 | 0.19 ± 0.01 | 0.14 ± 0.01 |
| Single Bond 2 | 0.22 ± 0.02 | 0.09 ± 0.01 | 0.02 ± 0.01 | 0.28 ± 0.03 | 0.21 ± 0.03 | 0.10 ± 0.04 |
| Adper Easy One | 0.09 ± 0.01 | 0.05 ± 0.01 | 0.01 ± 0.01 | 0.08 ± 0.01 | 0.07 ± 0.01 | 0.05 ± 0.01 |
| *p*-value[a] | 0.001 | | | 0.001 | | |

[a] *Results of one-way analysis of variance for different locations of all adhesives. Bur group: all locations (p<0.017); laser group: inner part-middle part (p=0.04), inner part-outer part (p=0.001), middle part-outer part (p=0.4).*

## RESULTS

The mean values of marginal gap width ± standard error of the mean (SEM) for the study groups at three locations of the cervical margin (outer part, middle part, and inner part) are shown in Table 2. In all the groups, the highest gap width was recorded in the outer part of the cervical margin, while the lowest gap width was observed in the inner part of the cervical margin. The results of one-way ANOVA revealed that there were statistically significant differences in gap width among the three locations of the cervical margin in laser- and bur-prepared cavities ($p$=0.001). In two-by-two comparisons, there were statistically significant differences in gap width in the bur-prepared group between all parts of the cervical margin ($p$<0.017), whereas in the laser-prepared group, there were statistically significant differences in gap width between the inner part and outer part and between the inner part and middle part of the cervical margin ($p$=0.001 and $p$=0.04, respectively); however, the differences in gap width between the middle part and outer part were not statistically significant ($p$=0.4).

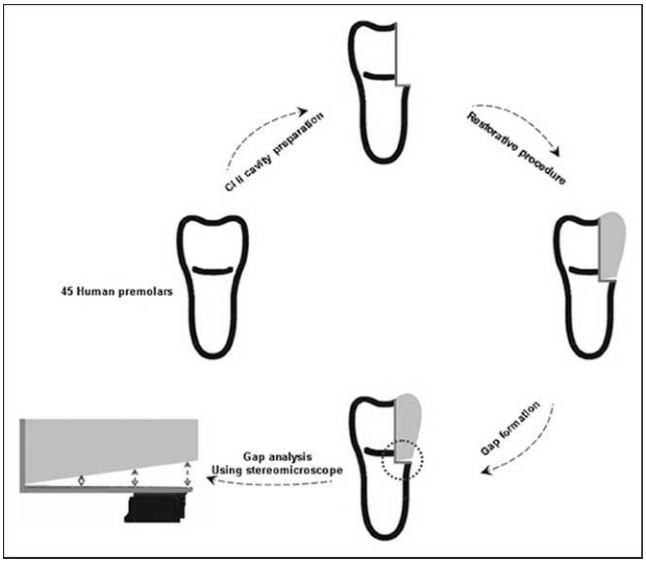


Figure 2. *The schematic representation showing the method to evaluate marginal adaptation. Gap width was measured at three locations (outer part, middle part, and inner part) of the cervical margin.*

The mean values of marginal gap width ± SEM for each treatment group are represented in Table 3. The results of two-factor ANOVA showed that there were statistically significant differences in mean marginal gap widths between the adhesive groups ($p$<0.001) and preparation groups ($p$<0.001). In addition, interaction effects between the type of the adhesive and cavity preparation were statistically significant ($p$=0.006).

The results of Mann-Whitney U-test showed that there were statistically significant differences in the mean marginal gap widths between the laser and bur preparation in Single Bond 2 ($p$=0.042) and Single Bond ($p$<0.001), but the differences were not statistically significant in Adper Easy One ($p$=0.39). Pairwise comparison with the *post hoc* Tukey test in the bur-prepared samples showed statistically significant differences between Single Bond 2 and Adper Easy One ($p$=0.02). There were no significant differences between Single Bond and Single Bond 2 ($p$=0.98) and Single Bond and Adper Easy one ($p$=0.55). In addition, pairwise comparison with the *post hoc* Tukey test in the laser-prepared samples

Table 3: *Mean Marginal Gap Width (μm) ± SEM in Study Groups*

| Type of adhesive | Type of preparation | | |
|---|---|---|---|
| | Bur | Laser | *p*-value[a] |
| Single Bond | 0.08 ± 0.007 (0.4-0.14) | 0.18 ± 0.01 (0.07-0.31) | <0.001 |
| Single Bond 2 | 0.11 ± 0.02 (0-0.3) | 0.20 ± 0.03 (0-0.6) | 0.042 |
| Adper Easy One | 0.05 ± 0.01 (0-0.14) | 0.07 ± 0.009 (0-0.14) | 0.39 |
| *p*-value[b] | <0.001 | <0.001 | |

[a] *Results of Mann-Whitney U-test.*
[b] *Results of two-factor analysis of variance. Bur group: Single Bond-Single Bond 2 (p=0.98), Single Bond-Adper Easy One (p=0.55), Single Bond 2-Adper Easy One (p=0.02); laser group: Single Bond-Single Bond 2 (p=0.81), Single Bond-Adper Easy One (p=0.007), Single Bond 2-Adper Easy One (p=0.01). Figures inside parentheses are the maximum and minimum values.*

showed statistically significant differences between Adper Easy One and Single Bond 2 ($p$=0.01) and between Adper Easy One and Single Bond ($p$=0.007), while the differences between Single Bond and Single Bond 2 were not significant ($p$=0.81).

## DISCUSSION

The longevity of a resin composite restoration depends on several factors, including sealing of the cavity-composite interface. From this viewpoint, investigations related to the gap formation mechanism and factors related to this phenomenon are crucial to improving the clinical longevity of resin composite restorations.[11] If there is insufficient bonding to the dental hard tissue, polymerization shrinkage can result in a gap between the filling material and the cavity wall.[12] In addition, marginal adaptation of composite resins is influenced by a variety of other factors, including the cavity size, the angle at which enamel prisms and dentin tubules are cut based on their location, the procedure in which dental hard tissues are conditioned, the layering protocol, and the polymerization technique used.[13]

Invasion of marginal gaps by bacteria would be expected to be in the range of 0.5-1.0 μm or larger. Smaller gaps may not allow the bacterial penetration but may allow the diffusion of toxins and other bacterial products that could be harmful to the tooth.[10] In the current study, the majority of the gap sizes were less than 0.25 μm; therefore, extensive bacterial penetration would not be expected. However, toxins and other bacterial products could diffuse through the smaller gaps.[10] It has been reported that bacterial products that diffuse toward the pulp can activate the immune system, provide chemotactic stimuli and cytokine production, and produce pain and pulpal inflammation.[14] To overcome such a discrepancy at the margin, various clinical protocols have been proposed, including the control of curing light irradiance (such as soft-start or pulse-delay techniques), use of a cavity liner with a low modulus of elasticity (such as a flowable resin liner), use of incremental techniques, and the use of composite resins with low polymerization shrinkage rates (such as silorane-based composite resins). Nevertheless, no single method has been totally efficacious in counteracting the effects of polymerization shrinkage.[15]

In all the groups, the gap width decreased from the outer part of the cervical margin to the inner part which represents a V-shaped gap formation in composite restorations on root surfaces. It has been reported that this phenomenon occurs because polymerization shrinkage forces are greater than the initial bond strength of composite to root dentin.[16]

The results of the present study showed that the mean interfacial gap width in bur-prepared cavities is significantly less than that in the laser-prepared cavities regardless of the type of adhesive resin used, which might be attributed to different dentinal surface characteristics in laser- and bur-prepared cavities. It has been demonstrated that surfaces irradiated by Er:YAG and Er,Cr:YSGG lasers have a characteristic rough, clean, and debris-free surface with most of the dentinal tubules visible and wide open.[6] Although Ceballos and others[17] and Aoki and others[18] demonstrated that adhesion to lased dentin would be explained by the mechanical retention

provided by resin tag formation and the infiltration of adhesive resin into the irregularities in lased, mineralized dentin, the main mechanism of bonding to dentin surface relies directly on the infiltration of hydrophilic monomers to the exposed dentin collagen web.[19] Therefore, bonding depends on the exposure and integrity of the collagen fiber network. The potential impact of the Er:YAG and Er,Cr:YSGG lasers on the collagen network has not been completely elucidated. It is known that laser irradiation is able to develop micro-structural alterations as well as micro-rupture of collagen fibers.[6] If the collagen structure collapses or is altered, the penetration of primers and monomers will be incomplete.[20] Similarly, Benazzato and Stefani[21] reported that Er:YAG laser dentin ablation plus air/water spray denatures dentinal collagen fibers in deep regions of dentin and structurally modifies dentinal collagen in the intertubular area. Moreover, De Munck and others[22] reported that cavities prepared by laser appear less receptive to adhesive procedures than conventional bur cavities. Ceballos and others[19] have reported that there exists a 3-4-μm dentin subsurface where denatured collagen fibrils are fused and cross-banding is lost in the Er:YAG-irradiated dentin. Other studies[23,24] have also shown that laser irradiation can negatively influence the dentin/adhesive system interface, hampering the hybrid layer formation. Laser irradiation of the dentinal substrate not only can result in consequences on collagen fibers but also can influence the quality of the mineral content of this substrate. Laser effectively influences the acid resistance of the dentin, as demonstrated by Schein and others[25] in Er:YAG lased dentin. Therefore, it seems that the quality of the hybrid layer is not satisfactory in laser-ablated dentin.

Another important finding in the present study was the lowest gap width observed in both laser- and bur-prepared cavities when using the self-etching system (Adper Easy One). In addition, in this adhesive system there were no statistically significant differences in gap widths between laser and bur preparations. Different adhesive systems can interact differently with lased surfaces.[26] Adper Easy One, a one-bottle self-etching adhesive formulation, includes a carefully balanced combination of phosphoric acid esters, water, and methacrylates in order to optimize stability. In addition, it contains bonded nanosilica fillers. The etching and subsequent penetration of resin monomers into the demineralized dentin is carried out as one step with self-etching Adper Easy One Adhesive. A major benefit of this procedure for dentin bonding is that the etching depth and the depth of penetration of the adhesive are identical.[27] Because of lower acidity, there are smaller openings at the end of dentinal tubules that are etched. This leads to better infiltration and proper coverage of demineralized dentin.[9]

Cardoso et al.[26] reported that the bonding efficacy of adhesives to laser-irradiated dental tissue depends not only on the structural substrate alterations induced by the laser but also on the characteristics of the adhesive employed. Adper Easy One contains nanofiller particles in its composition**.** It has been demonstrated that the collagen fibril network mostly filters out the nanofillers, holding them at the hybrid layer surface, thus acting as an intermediate shock absorber.[28,29] Therefore, the bond strength can be preserved, and the marginal gap and microleakage might be reduced.

Even though Single Bond 2, an etch-and-rinse adhesive system, contains filler particles in its chemical composition, it resulted in the highest gap width in both laser- and bur-prepared cavities in the current study. It seems that the interaction of adhesives with the substrate and their bonding mechanism can affect the gap formation more than the filler content.

However, the results of a previous study on flat dentin surfaces showed that etch-and-rinse adhesive systems produce better interfacial adaptation in dentin either in bur- or laser-prepared cavities.[27] Furthermore, in a study carried out by Yazici and others,[30] the Er,Cr:YSGG laser-prepared cavities exhibited the same amount of microleakage as that with bur-prepared cavities with etch-and-rinse and single-step self-etch adhesive systems in Class V composite resin restorations. In another study, no significant differences were observed in the cementum microleakage of bur- and laser-prepared Class V cavities in nanorestorative materials.[31] The differences in the results might be attributed to differences in preparation techniques of the samples, cavity configurations, restorative materials, and adhesive systems.

Considering the effect of acidity of self-etch systems and hybrid layer thickness formed by these adhesives on interfacial dentin gap formation,[27] it is suggested that the effect of hybrid layer thickness and pH of different self-etch adhesive systems on marginal gap formation in laser-prepared cavities be investigated.

Evaluation of marginal adaptation provides a more reliable prognosis about the efficacy of adhe-

sive systems in composite resin restorations compared to bond strength tests.[13] Therefore, in the present study, gap analysis was used to compare the marginal integrity of different adhesive systems in laser- and bur-prepared cavities. Several studies have reported a positive correlation between gap size and secondary caries; however, some other studies have not been able to correlate the results of gap analysis with the clinical performance of restorative materials. As a result, there is no conclusive evidence supporting this relationship or vice versa.[13,32,33] Furthermore, in clinical circumstances, patient-related factors, including caries activity and oral hygiene status, can have an influence on the quality of marginal adaptation and seal.[13] Therefore, long-term clinical studies are warranted to evaluate the clinical outcome of laser-prepared cavities with different adhesive systems.

## CONCLUSIONS

Within the limitations of the present study, it can be concluded that the gap width in the samples prepared by bur is less than those prepared by laser and that the gap width is adhesive system dependent. The self-etching system revealed less gap width in bur- and laser-prepared cavities.

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## Clinical Technique/Case Report

# Minimally Invasive Intervention in a Case of a Noncarious Lesion and Severe Loss of Tooth Structure

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BP Saldini • AL Stefanello Busato

### Clinical Relevance

Minimally invasive interventions play an important role considering human life expectancy and the evolution of restorative materials. Lower amounts of tooth structure removal will result in a stronger tooth and will positively influence patient satisfaction.

### SUMMARY

**The present article describes a minimally invasive technique used for the restoration of loss of tooth structure caused by erosion of intrinsic etiology. First, the cause of erosion was treated and controlled. Subsequently, taking into consideration patient characteristics, especially a young age, a more conservative technique was chosen for dental rehabilitation with the use of composite resin. The advantages and disadvantages of the technique employed are discussed.**

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### INTRODUCTION

On a daily basis, clinical observation has shown that an increasing number of patients have been affected by dental erosion. In this scenario, the early diagnosis of lesions, the study of etiologic factors, and the discussion of possible treatment approaches stand out as primary goals to be fulfilled.

Erosion is defined as the progressive and irreversible loss of tooth structure, resulting from chemical processes that do not involve bacterial action.[1-3] The process starts with demineralization of enamel

surface layers and may evolve to loss of relevant amounts of tooth structure. Dental tissue demineralization is caused by the frequent and long-lasting contact between acids and the tooth surface.[4]

The acids that cause dental erosion may originate from extrinsic or intrinsic sources. Intrinsic factors have been associated with several eating disorders, such as bulimia and anorexia, with systemic abnormalities, such as gastric reflux[5,6] and a decreased salivary flow. In particular, dental erosion affecting the posterior teeth are an important finding in the diagnosis of gastroesophageal reflux disease. Among extrinsic factors, it is possible to mention working for manufacturers of corrosive agents, such as acids used in the production of batteries, aerosols, and acidic foods and beverages.[1,5,7]

The onset and progression of erosion has a multifactorial etiology and is modulated by chemical, biological, and behavioral factors.[2] Biological factors, such as saliva, biofilm, tooth structure, and position, are all related with the pathogenesis of dental erosion. In particular, saliva is considered to be the most important biological parameter involved in dental erosion, as it promotes the dilution and clearance of erosive substances from the mouth, neutralizes acids, controls the passage of fluids, and enhances the remineralization process by providing calcium, phosphate, and fluoride to eroded areas.[8] Behavioral factors may include the frequent consumption of soft drinks or acid energy drinks and the practice of sports activities that reduce salivary flow and consequently affect the protection of tooth structures.

The most common characteristic of erosion is the opaque appearance of enamel, with a wedge-shaped, smooth surface. Lesions are usually large, shallow, with no clear-cut angles.[3] On the palatal surface, lesions resemble shoulder preparations with grooves.[9] More advanced disease stages include the development of concavities in enamel, in which the width clearly exceeds the depth, surrounded by a wall of intact enamel along the gingival margin. In more severe cases, the whole occlusal morphology of the tooth disappears. In sum, then, some typical signs of erosion include a smooth, silky-glazed, or "ground glass" appearance; a wall of intact enamel along the gingival margin; change in tooth color; and cupping and grooving on occlusal surfaces.[2]

The morphology and severity of defects may vary substantially, depending on the predominant etiologic factor.[10] Patients with eating disorders such as bulimia may present wear or chipping of the incisal edges, open bite, and loss of occlusal vertical dimension due to occlusal wear in posterior teeth.[11]

Erosion should be distinguished from attrition and abrasion; the latter usually produce lesions with a smooth and shiny appearance. However, the clinical differentiation of noncarious lesions is often complex. Moreover, different types of tooth wear lesions may occur simultaneously.[12]

The sequelae of dental erosion in more severe cases may include compensatory tooth eruption, diastema formation, changes in occlusal vertical dimension,[5,6] muscle pain resulting from occlusal instability, and temporomandibular joint dysfunction.[6]

The present article describes the case of a young patient presenting with erosion of intrinsic etiology submitted to a minimally invasive treatment approach.

## DESCRIPTION OF THE TECHNIQUE

A 26-year-old female patient sought treatment for dental esthetic complaints, referring specifically to chipping of maxillary central incisors. Clinical examination revealed extensive erosion of the occlusal surfaces of maxillary and mandibular posterior teeth, on the palatal surface of maxillary teeth, and on the lingual surface of premolars and canines (Figure 1).

The suspected cause of erosion in our patient was a systemic condition. The patient was referred to a physician, and a diagnosis of bulimia was established. Dental rehabilitation was initiated after completion of the medical treatment for bulimia.

Using cast models and an articulator, it was possible to identify approximately 2 mm of total loss of tooth structure, equally distributed in maxillary and mandibular areas (but sometimes more intense in one arch than in the other). This procedure was decisive to determine the amount of material needed to restore each tooth.

Restorative treatment was conducted with composite resins despite the fact that direct restorations have a shorter life span when submitted to extreme conditions. However, the decision was based on patient age, with an aim to preserve tooth structure, and on physician information that the systemic condition was under control.

Following anesthesia and complete isolation of the operating field, low-speed diamond burs were used to create roughened surfaces in preparation for application of the adhesive system. The restorative treatment phase followed the conventional adhesive protocol: enamel was acid etched for 30 seconds and

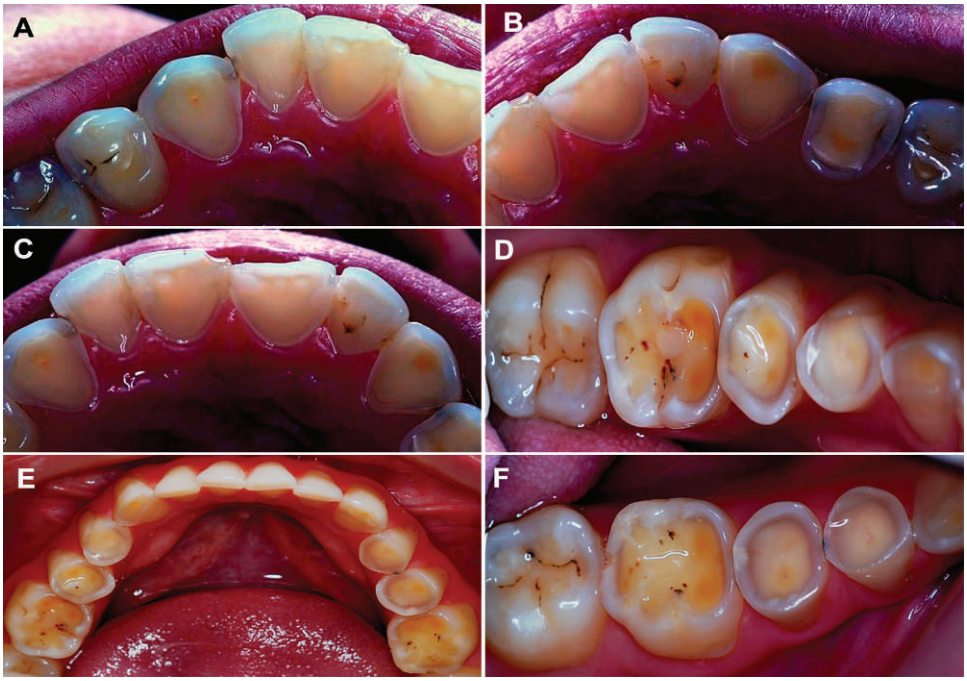


Figure 1. *Baseline photograph: (a): First quadrant, palatal view. (b): Second quadrant, palatal view. (c): anterior teeth, palatal view. (d): Third quadrant, occlusal view. (e): Occlusal view. (f): Fourth quadrant, occlusal view*

dentin for 15 seconds, followed by thorough rinsing and gentle drying. Subsequently, the adhesive system was applied according to the manufacturer's instructions: 1) application of one layer of primer, 2) drying, 3) application of adhesive layer, and 4) light curing for 20 seconds (Figure 2).

Composite resin was applied using approximately 2-mm-thick layers and the incremental technique.

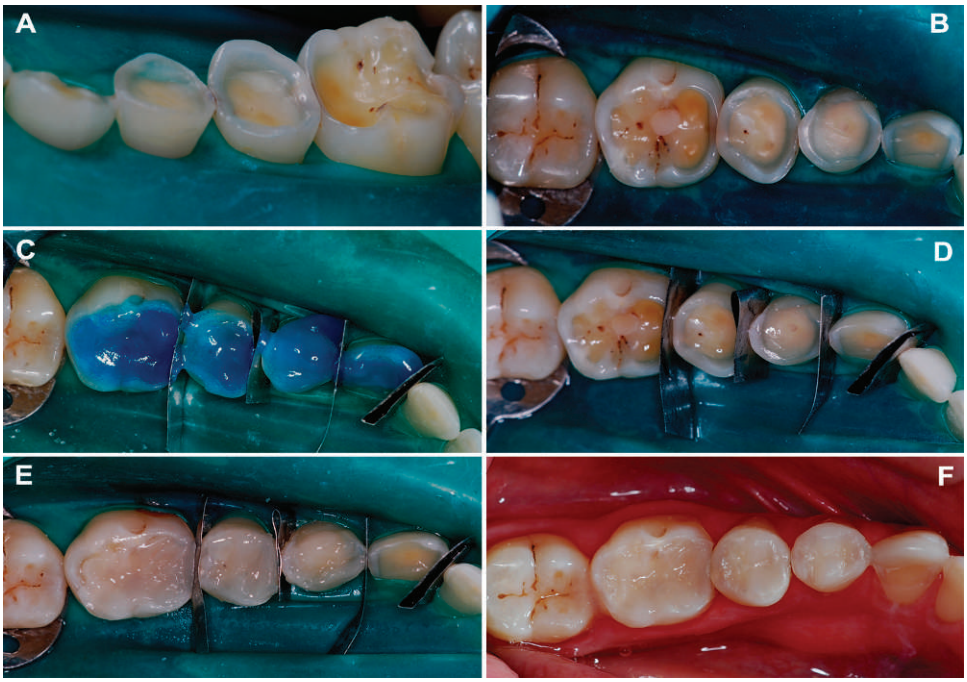


Figure 2. *(a): Rubber dam isolation. (b): View following isolation. (c): Acid etching in preparation for application of adhesive system. (d): Application of adhesive system. (e): Finishing and polishing. (f): Final view*

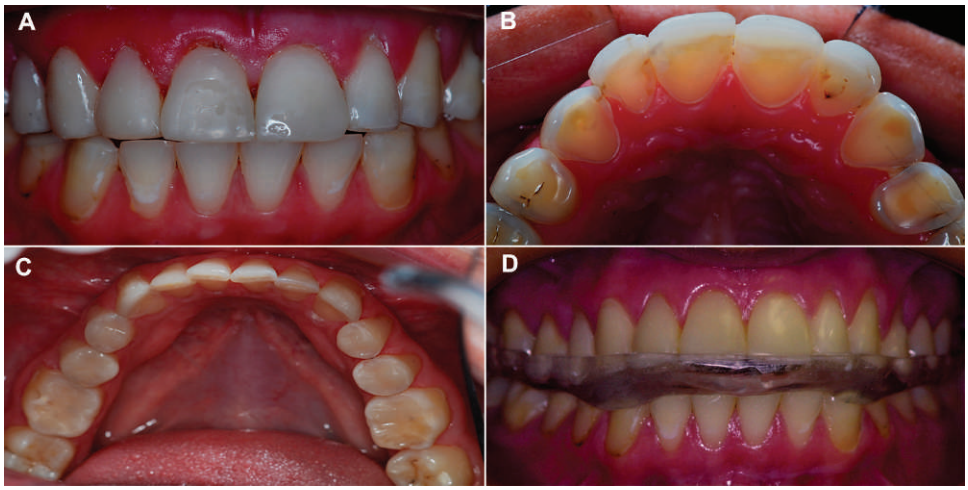


Figure 3. *Posttreatment photograph: (a): Front view. (b): Palatal view. (c): Occlusal view. (d): Final view of patient wearing a Michigan-type occlusal splint*

Each layer was light cured for 40 seconds. This procedure was repeated until each tooth was entirely restored. Rubber dam isolation was a key factor for successful adhesion.

In order to ensure functional stability, treatment sessions were planned so that teeth were restored in pairs: two teeth on the right side and two on the left, thus allowing bilateral occlusal contacts to be obtained at the end of each session. This system was adopted to improve patient comfort throughout treatment.

Areas submitted to increased masticatory forces, such as the occlusal surface of molars and the palatal surface of canines, received a composite resin specifically indicated for posterior restorations undergoing heavy loads.

Finishing and polishing were performed using multiblade burs, Sof-Lex polishing discs, and silicone tips, according to the surface under treatment.

On completion of the restorative treatment, a Michigan-type occlusal splint was fabricated to protect composite resin restorations. This decision was based on information provided by the patient referring to tooth clenching at some parts of the day (eg, while watching television, reading, or driving). The patient was instructed to wear the occlusal splint every night and also during the day whenever possible (Figure 3).

## LIST OF MATERIALS USED

- Filtek Z350, A2 shade (3M ESPE Dental Products, St Paul, MN, USA)
- Phosphoric acid 35% (3M ESPE)
- Scotchbond Multipurpose Adhesive System (3M ESPE)
- Sof-Lex Pop-On polishing discs (3M ESPE)
- Silicone tips (Cosmedent, Chicago, IL, USA)
- Filtek P60, A3 shade (3M ESPE)

## POTENTIAL PROBLEMS

Even with the recent advances observed in the field of restorative materials, composite resins still have limited indications in cases requiring extensive restoration of occlusal surfaces. However, such limitation becomes more evident when thinner layers of material are used.

Treatment of eroded areas with composite resins has to be fully performed in office and demands a high level of technical expertise, knowledge of occlusion and restorative techniques, and a certain degree of manual dexterity. Recovery of occlusal vertical dimension requires an adaptation period, as the loss of dimension was gradual and lasted for an unknown period of time. Control of the systemic cause of erosion is mandatory, and any change in patient general health status may have direct effects on the lifespan of restorations.

## SUMMARY OF ADVANTAGES AND DISADVANTAGES

Noncarious loss of tooth structure in young patients poses a dilemma for dental practitioners. If, on the one hand, indirect restorations using ceramic, both as onlays and as full crowns, have a longer life span, on the other hand they demand extensive reduction

of tooth structure to an extent that exceeds the wear caused by the disease itself over a long period of time. Moreover, other aspects have to be taken into consideration when deciding to perform indirect restorations (eg, the potential need for endodontic treatment and the financial status of patients). In this sense, composite resins are a promising alternative therapeutic approach that allows preserving tooth structure and simplifying treatment procedures. Conversely, the shorter life span of resin when compared with ceramic restorations is indeed a limitation of the technique. Decisions should be made according to the peculiarities of each case, focusing on either preservation of the patient's teeth (which usually seems to be more relevant) or a longer life span of restorations. In addition, the use of composite resins at an initial stage does not invalidate the performance of more complex restorations in the future, partially or totally replacing more conservative restorations.

## CONCLUSION

Restoration of areas of dental erosion with composite resins is a simple, minimally invasive technique that allows patients to achieve esthetic rehabilitation and to recover stomatognathic function and balance and should therefore be considered a reliable therapeutic option.

Long-term satisfactory results in the treatment of dental erosion requires follow-up monitoring of the systemic condition and regular clinical follow-up with photographs of the areas affected, study models, and clinical assessment of the restoration conditions.

### Conflict of Interest Declaration

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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## Departments

# Faculty Positions

**The University of Michigan School of Dentistry**

Full-time Faculty Position in Oral & Maxillofacial Surgery/Hospital Dentistry. Full description available at http://umjobs.org/ ID# 67054. The University of Michigan is an EEO/AA employer.

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