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Degradation Potential of Bulk Versus Incrementally Applied and Indirect Composites: Color, Microhardness, and Surface Deterioration

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Clinical Relevance

Covering bulk fills with silorane-based composites might be a better practice for delaying surface degradation.

SUMMARY

This study investigated the color stability and microhardness of five composites exposed to four beverages with different pH values. Composite discs were produced (n=10); Filtek Z250 (3M ESPE) and Filtek P90 (3M ESPE) were applied in two layers (2 mm, 20 seconds), and Tetric N-Ceram Bulk Fill (TetricBF, Ivoclar Vivadent) and SonicFill (Kerr) were applied in bulk (4 mm) and then light cured (40 seconds, Ortholux-LED, 1600 mW/cm^2). Indirect composite Sinfony (3M ESPE) was applied in two layers (2 mm) and cured (Visio system, 3M ESPE). The specimens were polished and tested for color stability; ΔE was calculated using spectrophotometer readings. Vickers microhardness (50 g, dwell time=45 seconds) was assessed on the top and bottom surfaces at baseline, 40 days of storage, subsequent repolishing, and 60 days of immersion in distilled water (pH=7.0), Coca-Cola (pH=2.3), orange juice (pH=3.75), or anise (pH=8.5) using scanning electron microscopy (SEM). The materials had similar ΔE values (40 days, $p>0.05$), but TetricBF had a significantly greater ΔE than P90 or SF (40 days). The ΔE was less for P90

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and TetricBF than for Z250, SonicFill, and Sinfony (60 days). Repolishing and further immersion significantly affected the ΔE ($p<0.05$) except for P90. All composites had significantly different top vs bottom baseline microhardnesses. This was insignificant for the Z250/water, P90/orange juice (40 days), and Sinfony groups (40 and 60 days). Immersion produced variable time-dependent deterioration of microhardness in all groups. Multivariate repeated measures analysis of variance with *post hoc* Bonferroni tests were used to compare the results. ΔE and microhardness changes were significantly inversely correlated at 40 days, but this relationship was insignificant at 60 days (Pearson test). SEM showed degradation (40 days) that worsened (60 days). Bulk-fill composites differ regarding color-stability and top-to-bottom microhardness changes compared with those of other composites. P90 showed better surface degradation resistance. In conclusion, bulk-fill composites are not promising alternatives to incremental and indirect composites regarding biodegradation.

INTRODUCTION

Exposure to the oral environment, including a number of beverages, can induce different models of physical or chemical degradation in the structure of resin composite restorations, adversely affecting surface integrity and color stability.[1-9]

Many researchers have studied physical degradation evidenced by material loss or uptake as a result of sorption, extraction, dissolution, and mineralization, accompanied by physical changes, such as softening, stress cracking, and fatigue fracture. Less research has been conducted on the chemical degradation of resin composites. Bourbia[10] stated that chemical degradation might be oxidative, thermolytic, photolytic, radiolytic, or solvolytic in nature, of which solvolysis by hydrolytic degradation was the most frequently studied.

The chemistry, structural composition, and degree of conversion of composites play a major role in the material's mechanical, biological, and physical properties, including water sorption, hydrophilicity, and wear resistance, having consequences on clear surface integrity and color stability.[4] The degree of conversion of the resin matrix depends on the polymerization system and the curing condition. The layering technique for the placement of light-cured resin composites has been used to ensure uniform optimum properties at varying depths of the material. The depth of cure is a function of the material composition and the curing light parameters.[5,6,11,12] Measuring the microhardness at the top and bottom of the material is one of the methods used to assess the cure depth and hence the material performance.[2,4,6,11]

Color match and translucency, surface luster, roughness, and staining are among the criteria for clinically ranking and identifying whether repairs are possible vs whether the composite must be remade.[7,13] Extrinsic discoloration of the resin composite can be caused by the adsorption of colorants from food and beverages. Surface roughness due to wear and chemical degradation can adversely affect color stability and surface gloss, leading to extrinsic discoloration.[14] However, bulk discoloration is related to the resin matrix, filler particles, and photoinitiators.[1,2]

The extraoral high-intensity light curing of indirect composites is advocated in order to achieve a high degree of polymerization with optimum material properties, including a greater resistance to intrinsic discoloration.[1,15] Indirect resin composites are used to overcome the deficiencies of direct composites with different curing techniques and modifications in the resin matrix and inorganic filler content.[16]

Recently, bulk-fill composites have been introduced to improve the performance and shorten the clinical procedure of composite application. This group of materials allows for the placement of a 4-6-mm-thick increment relative to the conventional fill composite with a maximum increment thickness of 2 mm. These materials incorporate modifications in the resin matrix and photoinitiator chemistry, as well as filler particle technology, with a diversity of reports regarding their clinical performance, physical and mechanical properties, and degradation potential. In the study of bulk-fill composites, there is a high level of concern regarding the depth of the cure and the development of polymerization contraction stresses.[12,17]

The silorane resin matrix that polymerizes by ring opening has been employed to reduce polymerization contraction and contraction stresses without compromising the mechanical properties. Due to the hydrophobic siloxane backbone, silorane-based composites were reported to have lower water sorption than experimental ormocer- and methacrylate-based composites.[4-6,12]

The literature indicates that storage for 40 and 60 days is considered to be an extensive testing

period.[18,19] Because polishing is crucial in determining surface luster and color, aging by immersion in different beverages followed by repolishing has been considerably studied to relate the results to the influence of extrinsic vs intrinsic discoloration.[18,20] However, the resistance of the repolished surface to future degradation on further exposure to staining beverages present in the common diet has not been previously studied.

The objective of this study was to assess the influence of the immersion of composite discs in common beverages and repolishing followed by further immersion on the color stability, microhardness, and surface micromorphology. The effect was studied in bulk vs incremental dimethacrylate-based and silorane-based composites as well as in indirect polyfunctional-resin-matrix composites indicating their relative degradation potential. The null hypothesis was that bulk-fill composites behave similarly to incremental and indirect composites regarding their color, stability, and microhardness and that repolishing followed by further immersion does not improve color stability nor microhardness.

METHODS AND MATERIALS

Five composite materials were used for the fabrication of composite discs: Filtek Z250, coded as "Z250" (3M ESPE, St Paul, MN, USA), used as a control, and silorane-based composite Filtek P90, coded as "P90" (3M ESPE), were applied in two increments of 2 mm each.[21] Bulk-fill composites included Tetric N-Ceram Bulk Fill, coded as "TetricBF" (Ivoclar Vivadent, Schaan, Liechtenstein), and SonicFill (Kerr, Orange, CA, USA),[21] in addition to the indirect resin composite Sinfony (3M ESPE), as shown in Table 1. Custom-made molds of 7 mm in diameter and 4 mm in depth made from custom-made polyvinyl-siloxane impression material (Express STD Putty, 3M ESPE) were used in the preparation of 45 discs of each composite material with a total of 225 composite discs. An overview of the study outline is displayed in Figure 1.

For Z250 and P90, two subsequent horizontal increments of 2 mm each were inserted using a plastic instrument and light cured for 20 seconds using a high-intensity LED light-curing device of 1600 mW/cm^2 (Ortholux, 3M Unitek Orthodontic Products, Monrovia, CA, USA) with a wavelength of 430-480 nm and a peak at 455 ± 10 nm. A radiometer (Bluephase Meter, Ivoclar Vivadent) was used to read the power output of the curing unit immediately after each respective use. Light curing of the superficial layer was carried out after pressing the surface using a Mylar strip with a glass slide cover. The tip of the light guide was kept in contact with the glass slide (1.2-mm thickness) in order to maintain a constant distance from the specimen. The bulk-fill composites, TetricBF and SonicFill, were inserted in bulk (4 mm) and light cured for 40 seconds. Sinfony indirect composite was applied in two successive horizontal layers (2 mm), and each layer was preliminarily light cured for 15 seconds using an In-lab light curing device (Visio Alfa Light Curing Unit, 3M ESPE). A final 14-minute cure (Visio Beta Vario Light Curing Unit, 3M ESPE) was performed per the manufacturer's instructions.[15,16,22]

A V-shaped notch was cut at the edge of the top side of each sample with a diamond inverted cone to visually identify the top side. The specimens were stored in distilled water in darkness for 24 hours at 37°C.[17]

After 24 hours, the specimens were polished with 12.7-mm aluminum oxide discs (Sof-Lex, 3M ESPE) in order of decreasing grit size. Each size was used for 10 seconds at 10,000 rpm for coarse and medium discs and at 30,000 rpm for fine and superfine discs using an electric hand piece (K4, KaVo, Leutkirch, Germany).[23] The specimen was moistened with water before polishing with each disc. The polishing was performed by one investigator.

From each composite material, one specimen was randomly selected for scanning electron microscopy (SEM) at baseline. The remaining 44 specimens of each composite material were then divided into four groups (n=11) according to the immersion medium: distilled water (Al-Kawther Industries Co Ltd, Jeddah, Saudi Arabia), pH = 7.0; Coca-Cola (Coca-Cola Bottling Company of Saudi Arabia, Riyadh, Saudi Arabia), pH = 2.3; orange juice (Nada Fresh Orange Juice, Al-Khobar, Saudi Arabia), pH = 3.75; or anise (Aniseed, Alattar, Damascus, Syria), pH = 8.5. The anise was prepared by adding three teabags of anise to 100 mL of boiling water and soaking for five minutes, followed by cooling. The pH values of the immersion media were measured by a pH meter (Model 215, Denver Instrument, Bohemia, NY, USA) before immersion, and the media was replaced with fresh samples every 24 hours.

Before immersion in the respective media, 10 specimens of each group were tested for color and microhardness at baseline, while the eleventh specimen of each group was randomly selected only for SEM after 40 days of immersion. Color and

Table 1: *Materials Used in This Study and Their Composition*

Material	Classification	Composition	Batch Number	Manufacturer
Filtek Z250	Methacrylate-based microhybrid composite	Dimethacrylate (Bis-EMA)	N479326	3M ESPE
		Urethane dimethacrylate (UDMA)		
		Bisphenol A diglycidyl ether dimethacrylate (Bis-GMA)		
		Triethylene glycol dimethacrylate (TEGDMA)		
		Silica		
		Zirconia		
		Pigments		
		Camphorquinone initiator system		
Filtek P90	Silorane-based microhybrid composite	Silorane	N281586	3M ESPE
		Quartz		
		Yttrium fluoride		
		Pigments		
		Initiator system		
Tetric N-Ceram Bulk Fill	Nano-optimized resin composite (dimethacrylate)	Barium aluminum silicate glass filler of a mean particle size of 0.4-0.7 μm; ytterbium fluoride and mixed oxides of a mean particle size of 160- 200 nm; Ivocerin, a germanium-based initiator and a special shrinkage stress reliever	R65898	Ivoclar Vivadent
SonicFill	Sonic-activated resin composite (Bis-GMA, TEGDMA, Bis-EMA, SIMA)	83.5 wt% filler content	4252654	Kerr
Sinfony	Mixture of aliphatic and cycloaliphatic monomers with no TEGDMA or Bis-GMA	Borosilicate glass, quartz, silica (50-1 nm)	492345	3M ESPE
		45% by volume		
		Camphorquinone initiator		

Vickers microhardness measurements and SEM images were taken at baseline and 40 and 60 days after immersion in the media.

Color Stability

The color was measured by one investigator using a spectrophotometer (Color-Eye 7000A, Gretag Macbeth LLC, New Windsor, NY, USA) according to the Commission International de l'Eclairage L*a*b* color system against a white background. Color measurements were performed for each specimen twice, and the average was calculated and used for the analysis.[24] Measurements were performed at baseline (n=10 per study group), then the specimens were stored in the different immersion media for 40 days. After 40 days, each specimen was removed from the storage media, rinsed with distilled water, and dried with absorbent paper before the second color testing. Each specimen was repolished using the same polishing procedure previously described. Afterward, the specimens were stored in the respective media for 20 more days and before a final test.[18] The magnitude of the total color difference, which is represented by a single number (ΔE), was determined using the formula $\Delta E = [(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{1/2}$ after 40 and 60 days of immersion. Values of $\Delta E \geq 3.3$ were considered clinically unacceptable.[18]

Vickers Microhardness Testing

After the color measurements were taken, the tops and bottoms of the same specimens were measured for Vickers microhardness. The surfaces were dried and positioned under the indenter of a microhardness tester (Micromet 6040, Buehler, Lake Bluff, IL, USA), and a load of 50 g was applied through the indenter with a dwell time of 45 seconds.[24-26] After each measurement, the specimen was immersed in the respective medium in a separate and numbered vial. Microhardness values were measured at baseline, after 40 days before the repolishing procedure, and after 60 days of immersion. Color and microhardness measurements were recorded and analyzed using each respective specimen's own values at the different study levels.

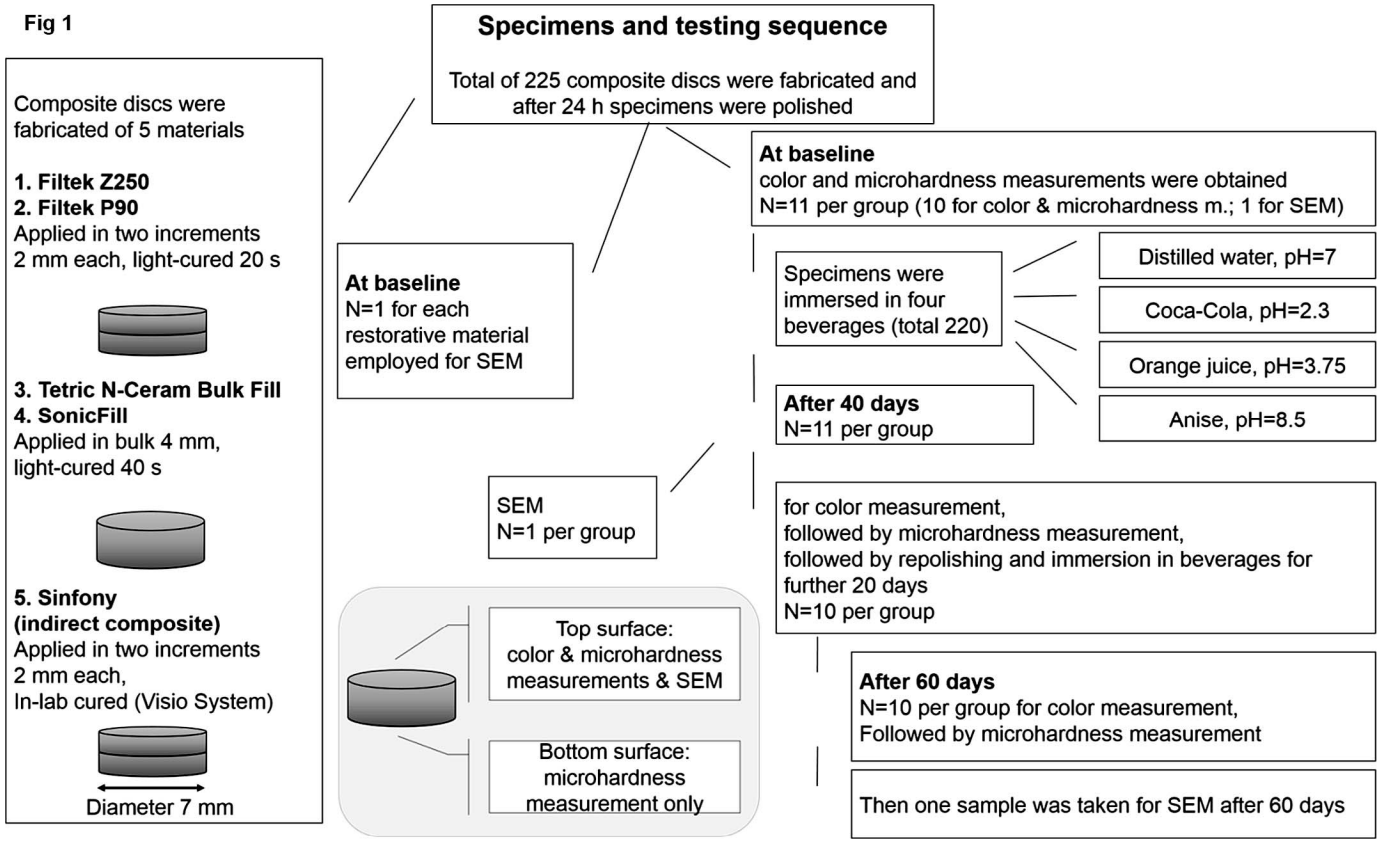


Figure 1. *Study overview.*

SEM

The specimens were examined using a scanning electron microscope (Inspect S50, FEI, Hillsboro, OR, USA) at 500× magnification to detect the surface microtopography of each material and changes due to storage in the immersion media.[27-29] At baseline, only one specimen of each restorative material was examined. After 40 days of immersion, the previously assigned specimen for SEM per each study group was examined and discarded and not used for microhardness or color-stability analysis. After 60 days of immersion, after the color and microhardness assessments, one specimen per group was randomly selected for SEM. Before scanning, the specimens were gold sputtered using the Q150R Rotary-Pumped Sputter Coater (Quorum Technologies, Ashford, UK).

Statistical Analysis

The ΔE color-stability results and the microhardness measurements were presented as mean and standard deviation values. Data were analyzed using the multivariate repeated measures analysis of variance (ANOVA) with the *post hoc* Bonferroni test for paired comparison of the materials and solutions in addition to the comparison of materials within each solution.[30-32] Moreover, the univariate ANOVA was used to compare the color-stability results ΔE of each material at 40 vs 60 days and for the comparison of top vs bottom microhardness values at each study interval.[11,32] The level of significance was 0.05 for all statistical analyses. The Pearson correlation test was performed to detect any correlation between color changes (ΔE) and microhardness changes (ΔD). Statistical data analysis was performed in IBM SPSS Statistics 22.0.

RESULTS

Color Stability

The mean values and standard deviations of the ΔE values and statistically significant differences between the materials within one beverage category and paired comparison of materials at 40 and 60 days are presented in Table 2. After 40 days of immersion in distilled water, Z250 was the only material to undergo clinically unacceptable changes

Table 2: *Color-Stability Results ΔE Mean Values and Standard Deviation (SD) of All Groups and Statistically Significant Differences Between Materials Within One Beverage Category and Paired Comparison*[a]

Solution	Material	Mean ± SD ΔE at 40 Days	Mean ± SD ΔE at 60 Days
Distilled water	Z250	3.9 ± 0.8 [a,d,1]	3.2 ± 0.8 [a,b,d,1]
	P90	2.6 ± 0.6 [b,c,d,1]	2.4 ± 0.9 [a,b,c,1]
	TetricBF	2.2 ± 0.6 [b,c,1]	1.7 ± 1.1 [b,c,1]
	SonicFill	3.1 ± 0.4 [a,b,d,1]	3.7 ± 0.2 [a,d,2]
	Sinfony	2.9 ± 0.7 [b,c,d,1]	2.6 ± 0.7 [a,b,c,1]
Coca-Cola	Z250	4.2 ± 2.6 [a,b,1]	4.5 ± 2.8 [a,b,1]
	P90	3.0 ± 0.6 [a,1]	2.6 ± 0.8 [a,1]
	TetricBF	6.0 ± 1.6 [b,1]	5.4 ± 1.9 [b,1]
	SonicFill	3.1 ± 0.7 [a,1]	3.4 ± 0.8 [a,b,1]
	Sinfony	5.3 ± 0.6 [b,1]	3.9 ± 0.9 [a,b,2]
Orange juice	Z250	3.8 ± 0.5 [a,b,1]	4.2 ± 0.5 [a,b,1]
	P90	4.1 ± 0.9 [a,b,c,1]	3.4 ± 1.6 [a,1]
	TetricBF	5.7 ± 1.6 [b,c,d,1]	5.0 ± 1.4 [a,b,1]
	SonicFill	4.2 ± 1.5 [a,b,c,1]	4.4 ± 1.2 [a,b,1]
	Sinfony	6.4 ± 1.3 [c,d,1]	5.9 ± 1.2 [b,1]
Anise	Z250	6.3 ± 2.3 [a,1]	9.9 ± 2.8 [a,2]
	P90	4.7 ± 1.1 [a,b,1]	4.3 ± 1.0 [b,d,1]
	TetricBF	4.8 ± 0.9 [a,b,1]	3.5 ± 1.4 [b,d,2]
	SonicFill	3.7 ± 1.2 [b,1]	7.0 ± 2.6 [c,d,2]
	Sinfony	5.5 ± 1.7 [a,b,1]	5.6 ± 1.2 [b,c,d,1]
Paired comparison of materials	Z250	4.5 ± 2.0 [a,1]	5.5 ± 3.3 [a,c,2]
	P90	3.6 ± 1.1 [b,1]	3.2 ± 1.3 [b,1]
	TetricBF	4.7 ± 2.0 [a,1]	3.9 ± 2.0 [b,c,2]
	SonicFill	3.5 ± 1.1 [b,1]	4.6 ± 2.0 [a,c,2]
	Sinfony	5.0 ± 1.7 [a,1]	4.5 ± 1.7 [a,c,2]

[a] *Significant differences between materials within one beverage category in each column are marked by different letters for the Bonferroni test at* $p < 0.05$*; significant differences between each material at 40 vs 60 days within one row are displayed by different numbers at* $p < 0.05$.

in color ($\Delta E > 3.3$). Immersion in Coca-Cola showed clinically unacceptable color changes for all composites except for P90 and SonicFill. When the test materials were stored in orange juice or anise, all showed clinically unacceptable color changes. After 60 days of immersion in distilled water, only SonicFill had an unacceptable color change, while storage in Coca-Cola resulted in acceptable color changes for P90 only. On storage in orange juice or anise, all materials showed clinically unacceptable color changes.

Paired comparisons showed variation of color changes in response to polishing and further immersion between materials with significant deterioration of Z250 and SonicFill and significant improvement of TetricBF and Sinfony. P90 was the only material with insignificant differences in color changes between 40 and 60 days and a clinically acceptable value of $\Delta E < 3.3$ after 60 days. The paired comparison of ΔE displayed that P90 had significantly less color change than TetricBF after 40 days and showed less color change than SonicFill after 60 days.

The results of color-stability paired comparisons based on beverages at 40 and 60 days are displayed in Table 3. Distilled water differed significantly from the other beverages ($p<0.05$), whereas the effect was insignificant between Coca-Cola, orange juice, and anise after 40 days of immersion. After 60 days, immersion in Coca-Cola and orange juice led to significantly greater color deterioration than water but significantly less than anise. Distilled water had the least deteriorating effect, while anise had the worst. For the color-stability results, there was a statistically significant interaction between the material and the solution ($p<0.001$) according to the repeated measures ANOVA.

Vickers Microhardness

Mean microhardness values and standard deviations of the top and bottom surfaces as well as paired

Table 3: *Results of Color-Stability Paired Comparisons Based on Beverages*[a]

Time Period	Beverage
At 40 days	Anise, orange juice, Coca-Cola > distilled water
At 60 days	Anise > orange juice, Coca-Cola > distilled water

[a] *> indicates clinical significance with the* post hoc *Bonferroni test at* $p<0.05$.

comparison at baseline and 40 and 60 days are presented in Table 4 in addition to the comparison of the top vs the bottom surface of each material at each study interval. There was a statistically significant deterioration in the top and bottom microhardness of all materials on aging regardless of the immersion medium. This was clear at 40 days with a significantly greater deterioration at 60 days. At baseline, the top surfaces showed a statistically significant higher microhardness than the bottom surfaces of all materials. This significant difference continued after 40 and 60 days in many groups, while in other groups it was only insignificant.

Comparisons of the top vs the bottom surfaces reveal that at baseline, the bottom surfaces of all specimens had statistically significant lower microhardness than the top surfaces ($p<0.05$). After 40 days of immersion, there was a deterioration in all of the top and bottom microhardness results. Significantly lower microhardness values of the bottom surfaces compared to the top surfaces were obtained in all materials except for Z250 in distilled water, P90 in orange juice, and Sinfony in all beverages. After repolishing and immersion for 20 more days, all materials demonstrated further microhardness deterioration of both surfaces. The microhardness of the bottom surfaces was significantly lower than that of the top surfaces in all groups except for Sinfony in all beverages.

The results of the microhardness paired comparisons based on materials are displayed in Table 5. Paired comparisons showed that at baseline, Z250 had the best statistically significant results compared to the other materials, while Sinfony had the worst. After 40 days, Z250 and P90 were not significantly different in microhardness, but both showed significantly greater microhardness compared to other materials. At 60 days, P90 showed significantly higher microhardness than other materials with least deterioration. According to the repeated measures ANOVA, there was a statistically significant interaction between the material and the solution ($p<0.001$) for the microhardness results.

SEM

The topographical analysis of specimens using the SEM images shows areas of degradation, material dislodgment, and erosion that varied in amount and extension between the materials. These degradation patterns were obvious after 40 days and progressed at 60 days of immersion in the different beverages, as shown in Figure 2. P90 underwent the least degradation.

Correlation Between (ΔE) and (ΔD)

After immersion in the beverages for 40 days, there was a strong inverse correlation between color change (ΔE) and microhardness change (ΔD) ($p=0.003$). However, after 60 days of immersion, there was a weak inverse correlation between ΔE and ΔD ($p=0.936$), as shown in Figure 3.

DISCUSSION

In vitro comparisons of the color stability and microhardness of materials have been used as a tool for predicting their degradability, especially when supported by microtopographic analysis at different levels of assessment.[1-4,11] Despite the ample amount of research indicating the clear effect of immersion in common beverages on the color-stability and surface properties of resin composite, few studies have compared the effect of variation in these properties relative to different composition and application techniques, especially for the new group of bulk-fill composites.[1-3,14,18]

Our study protocol lacked simulation of other factors, such as thermal cycling and the role of saliva and bacteria. The effect of repolishing and toothbrushing after immersion in different media on color stability was previously studied and was found to reduce staining and improve discoloration, as it removes the surface layer of pigments or absorbed stains of about 40 microns.[14,18,20,33] The effectiveness of repolishing in reducing stains and improving discoloration is related to the staining depth of the respective composite, its composition, degradability, and absorbability of water and stains.[33] Exposure of composites to beverages following repolishing is a normal consequence and addresses the reliability and longevity of improvement and can lead to a better understanding of composite degradation potential relative to the individual material composition.[24] Although immediate testing after repolishing is informative and might confirm previous findings on the effect of repolishing, it was felt that testing after further

Table 4: *Mean Values and Standard Deviation (SD) of the Vickers Microhardness of the Different Groups*[a]

Solution	Material	Mean ± SD Microhardness at Baseline			Mean ± SD Microhardness at 40 Days		
		Top	Bottom	Top vs Bottom	Top	Bottom	Top vs Bottom
Distilled water	Z250	109.8 ± 1.8[a]	89.9 ± 1.3[a]	+	81.4 ± 3.3[a]	78.3 ± 5.7[a]	–
	P90	97.2 ± 1.9[b]	83.8 ± 3.6[b]	+	89.3 ± 4.1[b]	83.1 ± 3.0[b]	+
	TetricBF	89.4 ± 2.5[c]	73.3 ± 3.7[c]	+	79.0 ± 3.3[a]	69.1 ± 2.2[c]	+
	SonicFill	96.1 ± 3.9[b]	84.3 ± 4.2[b]	+	88.7 ± 4.3[b]	81.6 ± 3.3[a,b]	+
	Sinfony	43.2 ± 5.1[d]	36.6 ± 4.9[d]	+	28.4 ± 3.9[c]	27.5 ± 4.7[d]	–
Coca-Cola	Z250	105.8 ± 2.5[f]	96.6 ± 1.9[f]	+	82.9 ± 2.2[f,h]	78.3 ± 4.7[f]	+
	P90	98.6 ± 3.0[g]	85.2 ± 5.2[g]	+	89.4 ± 4.6[g]	71.8 ± 3.6[g]	+
	TetricBF	87.1 ± 3.4[h]	74.2 ± 3.1[h]	+	83.0 ± 3.8[f,h]	71.8 ± 4.2[g]	+
	SonicFill	97.9 ± 4.5[g]	84.8 ± 3.9[g]	+	87.7 ± 3.4[f,g]	69.8 ± 5.0[g]	+
	Sinfony	44.8 ± 5.2[i]	38.8 ± 3.6[i]	+	27.6 ± 4.1[i]	24.6 ± 3.9[h]	–
Orange juice	Z250	112.1 ± 5.1[k]	89.6 ± 4.9[k]	+	94.3 ± 4.3[k]	75.6 ± 4.5[k]	+
	P90	95.1 ± 4.3[m]	88.9 ± 4.8[k]	+	85.7 ± 3.9[m]	85.9 ± 4.1[m]	–
	TetricBF	88.9 ± 2.7[n]	78.2 ± 3.3[m]	+	77.2 ± 3.5[n]	73.5 ± 2.3[k,n]	+
	SonicFill	94.8 ± 3.8[m]	81.5 ± 2.6[m]	+	89.3 ± 4.3[k,m]	69.8 ± 4.6[n]	+
	Sinfony	48.1 ± 3.6[p]	39.9 ± 2.9[n]	+	28.2 ± 4.0[p]	25.9 ± 4.0[p]	–
Anise	Z250	98.7 ± 4.2[q]	84.1 ± 4.4[q]	+	93.1 ± 4.1[q]	82.3 ± 4.9[q]	+
	P90	91.2 ± 5.2[r]	83.8 ± 4.7[q]	+	90.9 ± 5.2[q]	80.9 ± 3.7[q]	+
	TetricBF	87.9 ± 4.7[r]	74.3 ± 3.8[r]	+	82.1 ± 4.0[r]	71.3 ± 3.6[r]	+
	SonicFill	93.1 ± 3.4[q,r]	81.1 ± 4.4[q]	+	83.0 ± 3.5[r]	71.9 ± 4.2[r]	+
	Sinfony	47.9 ± 3.2[s]	39.7 ± 4.6[s]	+	26.1 ± 3.0[s]	24.7 ± 4.4[s]	–

[a] *Significant differences between materials within one beverage category in each column are marked by different letters for the Bonferroni test at* $p < 0.05$*; "+" denotes a statistically significant difference, while "–" stands for no difference at* $p < 0.05$ *in each material in one row at each study interval; significant differences between the study intervals within one material in one row of the paired comparison are displayed by different numbers for the Bonferroni test at* $p < 0.05$.

immersion following repolishing would provide a more descriptive image predicting the clinical reliability and material behavior.

Immersion times of 40 days followed by 20 more days were chosen according to Domingos and others,[34] who found that the most significant color changes take place at 60 days followed by 30 days of immersion. Moreover, the microhardness of materials immersed in coffee and Coca-Cola remained stable up to the seven-day measurement but decreased at the 30-day record and dropped more at the 60-day evaluation.[19]

The polishing technique employed in our study using Sof-Lex discs was found to provide the smoothest surface compared to other testing techniques.[20,35] Our light-curing procedure for bulk-fill composites for 40 seconds at high light intensity (1600 mW/cm^2) was chosen to obtain maximum color stability and greater microhardness.[10,36,37]

Table 5: *Results of Microhardness Paired Comparisons Based on Materials*[a]

Time Period	Material
At baseline	Z250 > P90, SonicFill > TetricBF > Sinfony
At 40 days	P90, Z250 > SonicFill > TetricBF > Sinfony
At 60 days	P90 > SonicFill, Z250 > TetricBF > Sinfony

[a] *> indicates clinical significance with the* post hoc *Bonferroni test at* ($p<0.05$).

Although restorations in the oral cavity are exposed to a variety of dietary colorants, as well as to a dynamic rather than a static exposure to beverages, most studies comprise exposing discs of the restorative material to one single coloring medium.[1] Our results showed variations in color stability among the test composites with varying levels of significance. Similar to our findings, Ren and others[1] observed that composites may have different affinities for diverse dietary colorants, possibly due to the difference in polarity between resin polymers and colorants. Composites of a resin matrix with similar polarity to yellow colorants may facilitate the absorption of the water, colored fluids, and yellow colorants into the organic phase of the material. Thus, modifying resin polymer polarities can improve resistance to staining.

Table 4: Extended.

Solution	Mean ± SD Microhardness at 60 Days			Mean ± SD Microhardness Paired Comparison (Top and Bottom)		
	Top	Bottom	Top vs Bottom	At Baseline	At 40 Days	At 60 Days
Distilled water	71.6 ± 4.5[a]	65.4 ± 3.4[a]	+	99.8 ± 10.3[1]	79.8 ± 4.8[2]	68.5 ± 5.0[3]
	84.5 ± 3.4[b]	78.9 ± 3.2[b]	+	90.5 ± 7.4[1]	86.6 ± 4.5[1]	81.7 ± 4.3[2]
	73.5 ± 3.7[a]	62.8 ± 4.2[a]	+	81.4 ± 8.8[1]	74.0 ± 5.7[2]	68.1 ± 6.7[3]
	81.5 ± 4.3[b]	74.7 ± 3.9[b]	+	90.2 ± 7.5[1]	85.1 ± 5.2[2]	78.1 ± 5.3[3]
	26.9 ± 4.0[c]	24.8 ± 2.9[c]	–	39.9 ± 5.9[1]	26.6 ± 4.1[2]	25.8 ± 3.6[2]
Coca-Cola	73.5 ± 4.8[f,g,i]	68.4 ± 5.5[f]	+	101.2 ± 5.2[1]	80.6 ± 4.3[2]	71.0 ± 5.7[3]
	79.3 ± 5.5[f,g]	67.1 ± 5.7[f,g]	+	91.9 ± 8.0[1]	80.6 ± 9.8[2]	73.2 ± 8.3[3]
	65.8 ± 5.3[h,i]	58.5 ± 5.2[h]	+	80.6 ± 7.3[1]	77.4 ± 6.9[1]	62.1 ± 6.3[2]
	67.2 ± 4.3[f,h,i]	61.3 ± 3.0[g,h]	+	91.4 ± 7.8[1]	78.8 ± 10.0[2]	64.3 ± 4.7[3]
	24.4 ± 4.2[j]	22.1 ± 3.4[i]	–	41.8 ± 4.8[1]	26.6 ± 4.2[2]	23.3 ± 3.9[2]
Orange juice	71.4 ± 3.6[k]	65.9 ± 3.9[k]	+	100.9 ± 12.5[1]	84.9 ± 10.5[2]	68.6 ± 4.6[3]
	83.2 ± 2.1[m]	74.4 ± 2.5[m]	+	92.0 ± 5.5[1]	85.8 ± 3.9[2]	78.8 ± 5.0[3]
	72.8 ± 2.9[k]	68.1 ± 3.3[k]	+	83.6 ± 6.2[1]	75.4 ± 3.4[2]	70.5 ± 3.9[3]
	80.8 ± 4.5[m]	66.8 ± 4.0[k]	+	88.2 ± 7.5[1]	79.6 ± 10.9[2]	73.8 ± 8.3[2]
	23.1 ± 3.3[n]	21.8 ± 3.1[n]	–	44.0 ± 5.3[1]	27.0 ± 4.1[2]	22.4 ± 3.2[3]
Anise	82.5 ± 5.0[q]	73.6 ± 5.4[q]	+	91.4 ± 8.5[1]	87.7 ± 7.0[1]	78.1 ± 6.8[2]
	84.1 ± 3.1[q]	70.7 ± 4.8[q,r]	+	87.5 ± 6.2[1]	85.9 ± 6.8[1]	77.4 ± 7.9[2]
	79.1 ± 4.2[q]	60.1 ± 3.9[s]	+	81.1 ± 8.1[1]	76.7 ± 6.6[1]	69.6 ± 10.5[2]
	79.6 ± 3.6[q]	67.1 ± 3.8[r]	+	87.1 ± 7.2[1]	77.5 ± 6.8[2]	73.4 ± 7.3[2]
	23.5 ± 4.9[r]	21.3 ± 5.1[t]	–	43.8 ± 5.7[1]	25.4 ± 3.7[2]	22.4 ± 5.0[2]

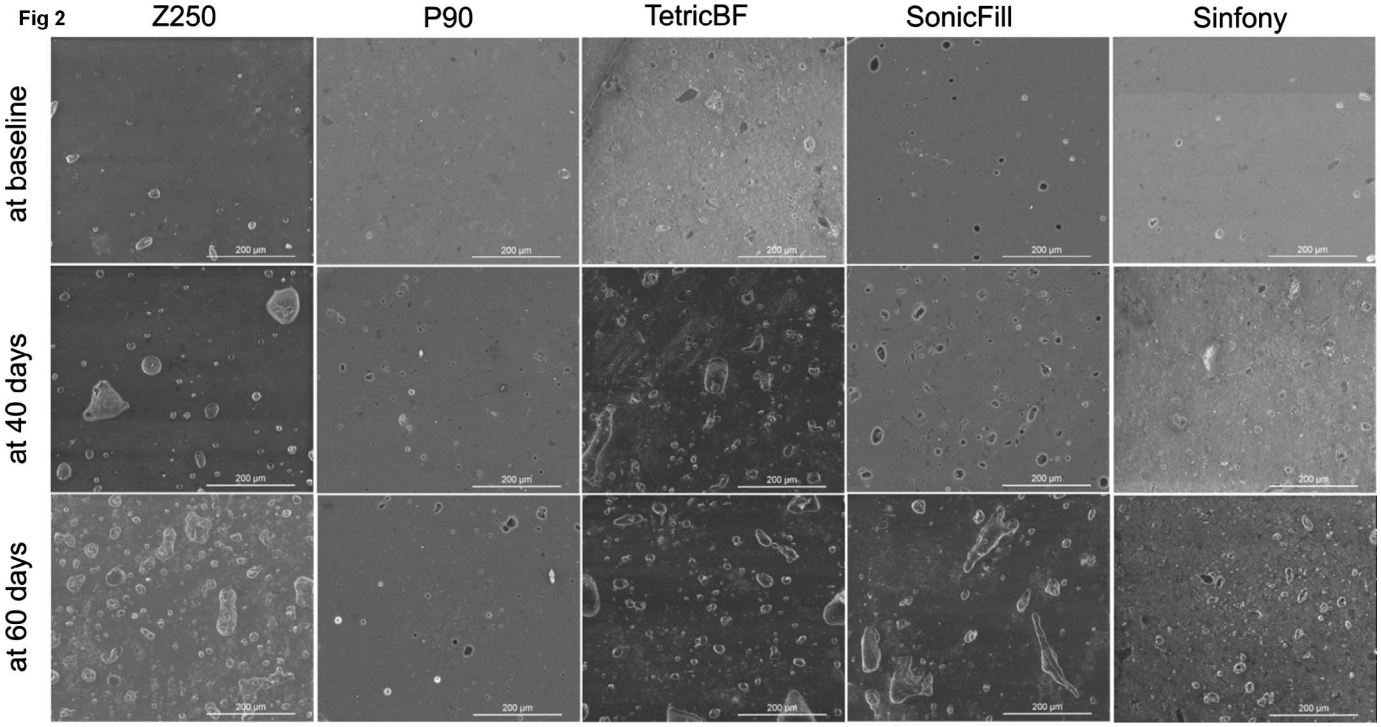
On immersion in water, most materials had clinically acceptable color changes ($\Delta E < 3.3$), which is similar to Schneider and others,[4] where 30 days of immersion in distilled water yielded clinically acceptable color changes and silorane- and ormocer-based materials did not produce higher color stability than the dimethacrylates. Additionally, silorane-based materials exhibited lower water solubility and lower hardness reductions than dimethacrylate-based composites, which is consistent with our microhardness results.

In this study, bulk-fill composites had similar color stability to Z250 after 40 days, which is similar to Tiba and others.[38] The better color stability of silorane-based P90 over TetricBF after 40 days and SonicFill after 60 days may confirm previous observations by Kang and others,[39] which may be related to the reduced hydrophilicity of silorane-based composites.[4]

Previous studies indicated that repolishing after staining reduces the ΔE value due to the removal of extrinsic stains.[18] However, our results indicate that repolishing followed by immersion had a varying effect on the color, possibly due to the variations in the organic matrix, polymer network density, filler loading, photoinitiator chemistry, and degree of conversion, which have a deciding influence on material properties, such as hydrophilicity, water sorption, adsorption, and dissolution potential and hence on the development of intrinsic or extrinsic staining.[1,4,36,39-43]

We analyzed the overall color change ΔE as an indication of degradation potential[44,45] since bulk-fill composites are more translucent and esthetically inferior than other composites.[43] However, analyzing the respective L*a*b values, which indicate the individual changes in lightness/darkness (L), red/green (a), and yellow/blue (b) scales, would have possibly provided more elaborative information about each composite. It might be more important to focus on the changes of surface reflectance of the material after staining than analyzing the color coordinate spaces to explain a range of staining effects on restorative materials.[46]

The effect of different immersion media compared to distilled water on color stability found in this study is similar to the findings of Kang and others[39] and Lepri and Palma-Dibb.[14] The significant deterioration of microhardness on the top and bottom surfaces after aging may be related to a gradual



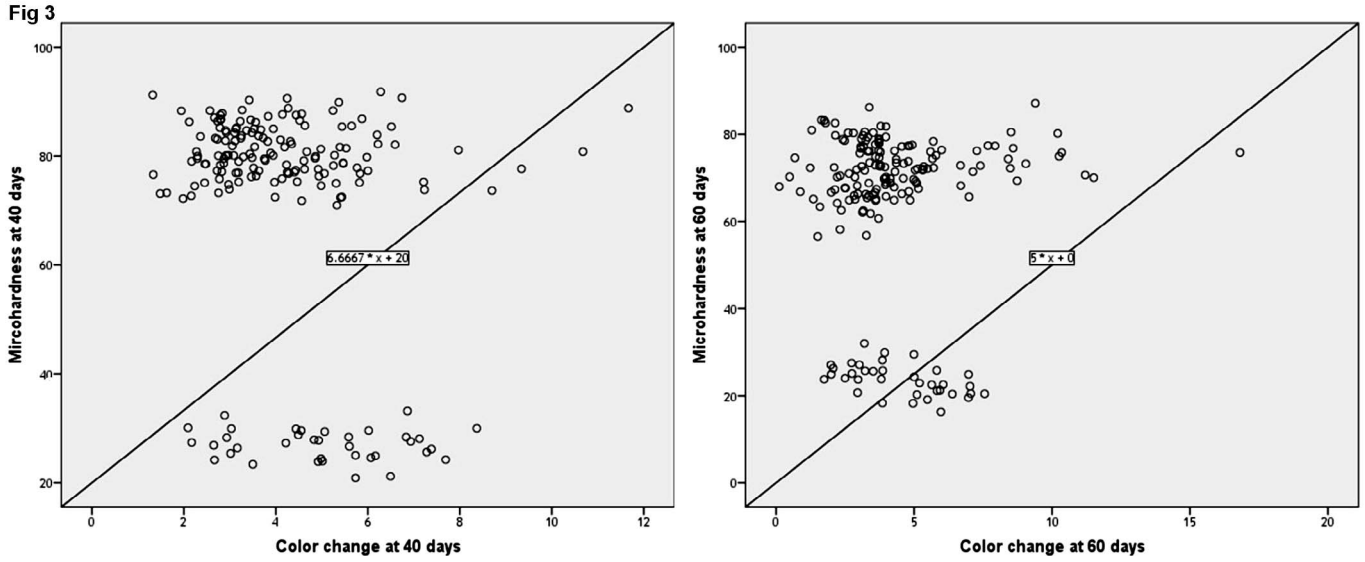


Figure 2. *SEM images (500×) of the various composites at baseline and 40 and 60 days of immersion in various beverages. The most prominent areas of degradation in the selected samples are presented.*
Figure 3. *The correlation between the ΔE and ΔD. After 40 days of immersion, there was a strong inverse correlation between color change (ΔE) and microhardness change (ΔD), while after 60 days of immersion, there was a weak inverse correlation between ΔE and ΔD.*

time-dependent degradation of the resin composite structure, which can be attributed to the hydrolysis of the ester groups in the resin matrix.[47] Further water sorption may decrease the durability of composites by expanding and plasticizing the organic matrix as well as by hydrolyzing the silane bonds between the fillers and the organic matrix.[18]

The presence of metallic ions such as zinc, barium, and strontium in fillers and radiopacifiers that are electropositive changes the silica network as they react with water, leading to charge balance changes. Therefore, hydroxyl ions increase and break the siloxane bonds inside the silica network, resulting in autocatalytic surface degradation, which might

explain the progression of surface softening with aging.[7,27,47,48]

The current study showed that at baseline, the bottom surface Vickers microhardness measures were significantly lower than those of the tops in all materials, similar to the findings of Jang and others[49] Our baseline results showed that the bottom microhardness exceeded 80% of that of the top surfaces in the SonicFill and TetricBF, which might be related to the use of the high-intensity LED curing unit. This is in line with the findings of Alshali and others.[50]

Bulk-fill materials incorporate modifications in the organic matrix and filler content, leading to higher translucency and reduced opacity with greater curing light transmission. Furthermore, they incorporate photoinitiators, such as Ivocerin in TetricBF, that yield more initiating radicals per molecule initiator than camphorquinone, leading to adequate cure depths at increased thicknesses.[37,45]

After 40 and 60 days, many groups maintained a significant difference between top and bottom microhardness values, indicating a consistent equivalent softening of the two surfaces. Others showed more deterioration in the top than the bottom surfaces. Our specimens were dropped to the floor of the beverage containers; thus, the top surface was more directly exposed to the beverage than the bottom. Nevertheless, the softening potential of a beverage is related to the compositional variations of the materials regarding the nature of the resin matrix and hydrophilicity, cross-linking density and the porosity of the network, the nature of the filler system, and the quality of the matrix/filler interface as well as the photoinitiators.[30,44,51,52] Furthermore, after light curing, dark polymerization, unpolymerized monomer conversion, and/or additional cross-linking reactions continue up to one week.[30,51,53] All of these factors vary between materials and influence the final respective material and specific surface degradation.[11,30,51,53] Therefore, it is recommended to suspend specimens rather than dropping them to the floor of the beverage container.[54]

Our results indicate that the initial proximity of the top surface to the curing light constitutes only one factor in the behavior of the material regarding surface microhardness on aging. Our findings support the multifactorial and complex nature of the degradation process.[4552] Although repolishing has removed a softened surface layer, yet further immersion produced a more detrimental effect on microhardness denoting the progressive nature of the degradation process extending deeper in the structure.[33,51,55]

SEM shows that the progressive deterioration in microhardness is related to the structural degradation of the material after immersion in different beverages, which progresses with aging.[27] This is similar to the findings of Martos and others,[48] who identified marked surface porosity and roughness in composite specimens following wet storage. The relatively superior degradation resistance pattern of P90 is similar to the findings of Schneider and others[4] and could be due to the lower hydrophilicity of the silorane-based resin matrix with a likely better resistance to hydrolytic degradation.[1,40] P90 seems to be more resistant to degradation, as observed by SEM and the microhardness as well as the color change results. Therefore, it is recommended to cover bulk-fill composites with silorane-based composite in order to delay surface degradation, which is in line with previous recommendations by Leprince and others[43] and Sunbul and others.[52]

The change in the Pearson correlation test between the ΔE and ΔD from strongly correlated at 40 days to weakly correlated at 60 days may indicate that repolishing followed by further immersion influences this correlation. This is in partial harmony with the findings of Soares-Geraldo and others,[24] who reported a significant positive correlation between the ΔE and ΔD and showed that not all color alterations were associated with surface degradation.

The findings of this study partially confirmed the null hypothesis that bulk-fill composites behave similarly to incremental and indirect composites regarding the effect of immersion in common beverages and repolishing followed by further immersion on color stability as well as microhardness. Future investigations should be directed toward combining oral environmental factors in addition to considering the depth of the individual influences of organic matrix, pigments, photoinitiator systems, and fillers in studying the degradability of this group of materials and the adverse consequences on their esthetic, physical, and mechanical qualities.[36]

CONCLUSIONS

Under the circumstances of the current investigation, it can be concluded that beverages of different pH values have a deteriorating effect on the color stability and microhardness of various formulations of resin composites. Immersion in beverages for 40 days produced variable deteriorations in color in

bulk-fill as well as incremental and indirect composites. Repolishing followed by immersion improved discoloration of TetricBF and Sinfony composites but deteriorated discoloration of Z250 and SonicFill composites. The effect of repolishing and further immersion is material dependent. Immersion in beverages produced progressive but variable patterns of deteriorations in the top and bottom microhardness of bulk-fill, incremental, and indirect resin composites. Degradability in terms of discoloration, deterioration in microhardness, and surface micromorphology is a complex multifactorial process influenced by material composition and application technique. Although color changes have a strong correlation with microhardness changes on immersion in beverages, repolishing followed by further immersion weakens such a correlation. P90 is more resistant to surface degradation than bulk-fill composites.

Conflict of Interest

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

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