Original Article / Özgün Araştırma

Evaluation of Color Stability and Surface Roughness of Bulk-Fill Resin Composites and Nanocomposites

Bulk Fill Rezin Kompozitlerin ve Nanokompozitlerin Yüzey Pürüzlülüğü ve Renk Stabilitesinin Değerlendirilmesi

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Abstract

Objective: The purpose of this study was to evaluate the color stability and surface roughness of four bulk-fill resin composites (SonicFill, Filtek Bulk Fill Flowable, X-tra fil, Filtek Bulk Fill Posterior) and three nanocomposites (G-aenial Universal Flo, Herculite XRV Ultra, Filtek Ultimate) after an aging simulation.

Materials and Methods: The upper surfaces of prepared composite discs were polished with Sof-Lex discs. The samples were subjected to a thermocycling process for 3000 cycles, then immersed in the prepared mixture solution for two weeks. Before and after the aging simulation, profilometer and spectrophotometer were used to measure surface roughness (Ra) and color of the composite discs. The color change (ΔE) of each material was calculated.

Results: The ΔE values showed a statistically significant difference among the studied materials ($p<0.001$). The Ra values of X-tra fil, Filtek Bulk Fill Flowable, SonicFill, and Filtek Bulk Fill Posterior were significantly increased by the aging process ($p<0.001$), while G-aenial Universal Flo, Filtek Ultimate, and Herculite XRV Ultra showed steady roughness ($p<0.001$).

Conclusion: Filtek Ultimate showed greater susceptibility to staining. Microhybrid X-tra fil and nanohybrid SonicFill with higher filler amounts revealed more surface deterioration.

Keywords
Color change, surface roughness, bulk fill composites, nanocomposites, spectrophotometer

Anahtar Kelimeler
Renk değişimi, yüzey pürüzlülüğü, bulk fill kompozit, nanokompozit, spektrofotometre

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Öz

Amaç: Bu çalışmanın amacı, yaşlandırma işleminden sonra dört adet bulk fill rezin kompozitin (SonicFill, Filtek Bulk Fill Flowable, X-tra fil, Filtek Bulk Fill Posterior) ve üç adet (G-aenial Universal Flo, Herculite XRV Ultra, Filtek Ultimate) nano-kompozitin yüzey pürüzlülüğü ve renk değişikliğinin değerlendirilmesidir.

Gereç ve Yöntemler: Kompozit disklerin üst yüzeyi Sof-Lex cila diskleri ile düzleştirildi. Örnekler 3000 termal siklusa maruz bırakıldı ve sonra örnekler hazırlanmış solüsyonda iki hafta bekletildi. Yaşlandırma işleminden önce ve sonra, örneklerin yüzey pürüzlülüğünü (Ra) ve rengini değerlendirmek için profilometre ve spektrofotometre kullanıldı. Materyallerin renk değişimi (ΔE) hesaplandı.

Bulgular: ΔE değerleri materyaller arasında önemli derecede farklılık gösterdi ($p<0{,}001$). Yaşlandırma işlemi, X-tra fil, Filtek Bulk Fill Flowable, SonicFill, and Filtek Bulk Fill Posterior kompozitlerin pürüzlülüğünü önemli derecede artırdı ($p<0{,}001$). Ancak, yaşlandırma işlemi G-aenial Universal Flo, Filtek Ultimate, and Herculite XRV Ultra kompozitlerinin yüzeyinde önemli bir değişiklik oluşturmadı ($p<0{,}001$).

Sonuç: Filtek Ultimate renklenmeye daha fazla duyarlılık gösterdi. Daha yüksek oranda doldurucu içeren Microhybrid X-tra fil ve nanohybrid SonicFill daha fazla yüzey bozulması gösterdi.

Introduction

Patients desire dental restorations that are successful long-term as well as functional and esthetically pleasing. Clinicians would like to perform high-quality restorations while minimizing the time taken to complete the procedure. Composite resin materials, which have the potential to fulfill the criteria for success for both patients and clinicians, have achieved wide popularity among dentists and are increasingly being used in direct techniques (1). Incremental filling techniques have been commonly used to reduce polymerization shrinkage stress and to optimize composite polymerization. However, this technique requires extended procedure time and implies the risk of incorporation of voids or contaminants between layers (2). Bulk fill resin composites have been developed as a new category of composite materials for which it is claimed that a single layer with a thickness of 4-5 mm can be applied because of the improved cure depth of the material.

The development of bulk-fill resin composites can reduce processing time during restoration, allowing use of an easy, quick one-increment technique in most clinical cases. However, composite resins have some limitations, including surface degradation, potential for marginal fractures, discoloration, polymerization shrinkage, and high plaque accumulation (3-5). The long-term performance of a composite resin depends on its properties, especially its resistance and durability in the oral cavity. Factors that vary from patient to patient, including masticatory forces, occlusal habits, dietary composition, fluctuations in temperature, bacterial products, and salivary enzymes, can impact this durability (6).

Over time, the material properties such as surface roughness, color stability, and brightness prove decisive with respect to achieving satisfactory results with restorative materials. Insufficient color is one of the most common reasons for the replacement of composite materials (7). The staining of composite materials can be extrinsic or intrinsic. External staining can be related to the adsorption or absorption of staining pigments from diet, plaque accumulation, and surface degradation. Intrinsic factors, which cause staining of the material in the absence of external influences, include the resin matrix ingredients, filler amount and size, and photoinitiator type (8). The resin matrix composition and filler particle properties may affect the mechanical characteristics as well as the surface roughness of composite resins (9). A smooth surface improves the longevity and the appearance of resin materials, whereas the rough surfaces of a restoration contribute to plaque accumulation, discoloration, gingival irritation, and recurrent caries (10).

Composite resin materials ideally would have high surface hardness and low surface roughness after the polishing procedure, with these properties maintained (along with the restoration's initial optical characteristics) in the mouth during long-term use (11,12). Currently, little is known with respect to the surface changes and the color stability of new dental composites during simulations of the oral conditions in which they are used. The purpose of this *in vitro* study was to investigate the color change and the surface roughness of bulk-fill composite materials and nanocomposites after aging.

Materials and Methods

Four bulk-fill composite materials: SonicFill (SF), X-tra fil (XF), Filtek Bulk Fill Posterior (FBP) and Filtek Bulk Fill Flowable (FBF), and three nanocomposites: Herculite XRV Ultra (HXU), G-aenial Universal Flo (GUF) and Filtek Ultimate (FU), were investigated in the present study. The manufacturer information for each of these composites is presented in Table 1. Ten samples of each composite were fabricated using a cylindrical stainless-steel mold (8 mm internal diameter, 2 mm thickness). The cylindrical mold was filled with one of the composite materials and manually pressed between two microscope slides covered with Mylar strips. All materials were cured for 20 s using a LED curing unit (VALO Cordless, Ultradent, USA) with an output power of 1000 mW/cm^2. The light output was checked using a radiometer (TREE, model TR-P004, China). After samples were gently removed, the top surfaces of samples were polished intermittently with Sof-Lex abrasive discs (coarse, medium, fine, superfine; 3M ESPE) for 15 s on a low-speed hand piece (10.000 rpm). The samples were stored in 37 °C distilled water for 24 h.

The samples were subjected to 3000 thermal cycles that were evenly split between water baths (dwell time, 25 s) at 5 °C / 55 °C. Then, the samples were incubated in the mixture prepared from five different beverages (at 37 °C for 14 days): coffee (Hisar Coffee, Turkey), tea (Doğuş Black Tea Bags, Turkey), grape juice (Tamek, Turkey), orange juice (Cappy, Turkey) and strawberry fruit punch (Dimes, Turkey). Tea or coffee solution was produced by adding 5 g tea or coffee to 500 mL distilled water and boiled for 5 minutes. The mixture solution was prepared by adding equal parts of each fruit beverage, tea solution, and coffee solution, then renewed every day. After the immersion process was complete, samples were washed under running water for 30 s.

Color measurements were made at baseline and after aging process on a white background with a spectrophotometer (VITA Easyshade Advance; Zahnfabrik, Bad Säckingen, Germany). Color readings were transformed into the Commission International de I'Eclairage (CIE L*a*b*) color system. The calibration of the spectrophotometer was performed before measurements of each material. ΔE values were calculated using the following formula:

$$\Delta E = \sqrt{\left\{\left(L_1^* - L_2^*\right)^2 + \left(a_1^* - a_2^*\right)^2 + \left(b_1^* - b_2^*\right)^2\right\}}$$

In this equation, the superscript “1” refers to the values after aging; the superscripts “2”, those before aging. The L* value indicates the lightness (white or black) of an object. Parameter a* indicates the red (+a) / green (-a) axis. Parameter b* indicates the yellow (+b) / blue (-b) axis.

A contact profilometer (MarSurf PS1; Mahr, Göttingen, Germany) was used to measure the surface roughness (Ra, nm) before and after the aging process. The calibration of the profilometer was performed before measurements of each material, with the cut-off value for surface roughness being 0.25 mm and the tracing length 5 mm. Three measurements were taken at different positions on the upper surface. The roughness value for each sample was accepted as the average of three measurements. Representative samples of each material were examined using scanning electron microscopy (SEM) [SEM (JSM-6610; JEOL, Peabody, Massachusetts, USA)] at 1.000× magnification.

Statistical Analysis

The data had a normal distribution. One-way analysis of variance test was used to evaluate the color change (ΔE) values. A two-way analysis of variance with Tukey's test was performed to assess the surface

Table 1. Compositions of materials studied

Material (Manufacturer)	Batch no./Shade (Type)	Composition	Filler loading
Filtek Bulk Fill Flowable (3M ESPE)	N616721/A2 (Bulk-fill)	Bis-GMA, UDMA, Bis-EMA, zirconia/silica (0.01-3.5 µm), ytterbium trifluoride (0.1-5.0 µm)	42.5 vol % 64.5 wt %
SonicFill (Kerr)	5192175/A2 (Bulk-fill)	Bis-GMA, TEGDMA, Bis-EMA, silicon dioxide, barium glass	83.5 wt %
X-tra fil (Voco)	1249480/Universal (Bulk-fill)	Bis-GMA, UDMA, TEGDMA, barium boron aluminum silica glass (0.05-10 µm)	70 vol % 86 wt %
Filtek Bulk Fill Posterior (3M ESPE)	N639224/A2 (Bulk-fill)	UDMA, DDDMA, AUDMA, Zirconia/silica (4-20 nm) cluster filler, ytterbium fluoride (100 nm)	58.4 vol % 76.5 wt %
G-aenial Universal Flo (GC Corp.)	1311082/A2 (Nanohybrid)	UDMA, TEGDMA, Bis-MEPP, Silicon dioxide, strontium glass (10-200 nm)	50 vol % 69 wt %
Herculite XRV Ultra (Kerr)	5127254/A2Enamel (Nanohybrid)	Bis-GMA, TEGDMA, barium glass filler (0.4 µm), silicon dioxide (0.02-0.05 µm)	78 wt %
Filtek Ultimate (3M ESPE)	N536547/A2Enamel (Nanofill)	Bis-GMA, TEGDMA, Bis-EMA, UDMA, silica/zirconia cluster filler (0.6-10 µm), zirconia particles (4-11 nm)	63.3 vol % 78.5 wt %

TEGDMA: Triethylene glycol dimethacrylate, DDDMA: Didode cyldimethylammonium bromide, UDMA: Urethane dimethacrylate, Bis-EMA: Bisphenol-A ethoxylated dimethacrylate, Bis-GMA: Bisphenol-A glycidyl dimethacrylate, Bis-MEPP: Bisphenol 4-methacryloxypolyethoxyphenyl propane roughness. The significance level was accepted at $p<0.05$.

Results

All CIE L*a*b* values and ΔE values are presented in Table 2. After aging, L* values for all materials decreased, while a* and b* values increased. The highest change in L* value was obtained for FU followed by FBP. The color change (ΔE) values were significantly different among the materials ($p<0.0001$). The highest variation in color change (ΔE) was obtained for FU, whereas the lowest variation was found for SF. GUF and HXU exhibited color changes that were statistically similar to those for SF ($p>0.05$).

The results obtained for surface roughness before and after aging are presented in Table 3. Baseline surface roughness values were significantly different among the materials ($p=0.000$). XF showed the highest surface roughness, but other materials had statistically similar surface roughness (Ra) values. The surface roughness for all composites increased after the aging procedure. This increase was statistically significant for XF ($p<0.001$), FBP ($p<0.001$), SF ($p<0.001$), and FBF ($p=0.002$), but not significant for HXU ($p=0.89$), FU ($p=0.14$), and GUF ($p=0.53$).

The SEM micrographs of test materials are presented in Figure 1. The resin matrix decomposition after aging was observed in the sample surfaces of FBF, FBP, SC, and XF. Irregular fillers were observed in the surfaces for SF and XF. No significant change was observed for GUF, FU, and HXU after aging. The surface of FU after aging showed scratch lines that were probably left by the mandrel of the polishing system.

Discussion

The esthetic properties and physical and mechanical characteristics of composite resins can alter when exposed to oral environment conditions. Thermocycling protocols have been suggested as efficient methods to simulate conditions of oral cavity. In previous studies, however, it has been reported that thermocycling and distilled water immersion caused

Table 3. Mean and standard deviation of surface roughness (Ra, nm)

Material	Surface roughness	
	Baseline	After aging
FBF	218.62±46.94^{aA}	558.14±105.88^{cB}
SF	242.71±51.01^{aA}	738.02±158.58^{dB}
XF	354.81±102.34^{bA}	902.34±194.97^{eB}
FBP	202.33±39.45^{aA}	645.48±195.45^{cB}
GUF	180.92±25.65^{aA}	200.42±73.57^{aA}
HXU	256.63±78.88^{aA}	249.11±185.72^{abA}
FU	245.92±49.39^{aA}	327.26±80.69^{bA}

The capital letters in the horizontal line, and the lower letters in the vertical line, indicate significant differences ($p<0.05$)

SF: SonicFill, FBF: Filtek Bulk Fill Flowable, XF: X-tra fil, FBP: Filtek Bulk Fill Posterior, HXU: Herculite XRV Ultra, FU: Filtek Ultimate, GUF: G-aenial Universal Flo

Table 2. Mean and standard deviation of color parameters for composite materials

		SF	FBF	XF	FBP	HXU	FU	GUF
Baseline	L*	64.94±1.42	64.62±1.37	62.70±1.72	63.04±2.07	70.41±1.25	70.55±1.48	67.57±1.57
	a*	-0.63±0.39	1.64±0.35	-0.42±0.28	-1.21±0.20	-1.14±0.38	-0.54±0.14	1.4±0.16
	b*	16.58±0.78	17.74±0.95	2.42±0.44	11.87±1.22	22.01±0.66	14.85±0.49	22.46±0.67
After staining	L*	59.28±1.26	54.58±1.20	57.5±1.18	50.09±2.10	63.36±2.50	54.92±1.20	60.32±1.53
	a*	2.20±0.82	4.95±0.71	1.98±0.88	3.04±0.48	3.43±1.11	3.37±0.64	3.53±0.58
	b*	19.4±0.89	19.63±1.01	10.46±0.58	15.83±1.82	23.45±0.64	18.04±0.70	24.86±1.13
	ΔE	6.63±2.01^{a}	12.51±1.87^{cd}	10.23±2.03^{bc}	13.98±2.65^{de}	8.34±2.03^{ab}	16.46±3.01^{e}	8.06±1.01^{ab}

In "ΔE values" horizontal line, different letters indicate statistically significant differences ($p<0.05$)

SF: SonicFill, FBF: Filtek Bulk Fill Flowable, XF: X-tra fil, FBP: Filtek Bulk Fill Posterior, HXU: Herculite XRV Ultra, FU: Filtek Ultimate, GUF: G-aenial Universal Flo no visually perceptible color change (ΔE <3.3) (8,13), while dark beverages caused clinically unacceptable staining in tooth-colored restorative materials (1). In the present study, the samples were immersed after the thermocycling procedure in the mixture prepared from beverages that contain common dietary colorants according to *in vitro* model proposed of Ren et al. (14), who suggested that storage of composite samples in a single colored fluid could not reflect the staining potential of human dietary habits.

The discoloration of a given resin-composite material is directly related to the hydrophilic properties of the resin material. Composites that can absorb more water also have a greater capacity to absorb other fluids with coloring agents (9). Glass filler particles cannot absorb water into the bulk of the composite resin, but only influence water adsorption on the material's surfaces (15). Based on the findings of this study, FU (nanofill composite) showed the highest staining and statistically similar performance to FBP (nanofill composite). Both materials have similar filler particles and aggregated zirconia-silica cluster filler, but the structure of resin monomers and filler loadings were different. In some studies, it has been shown that FU (or Filtek Supreme) was most prone to discoloration (16,17), which might be attributed to its less-than-ideal integration of the nano-aggregated cluster fillers that are loosely bound agglomerates of nano-sized particles. Nano-composites and their clusters have a much larger surface area per unit mass, which may cause staining when their interface is not perfectly silanized and integrated into the resin (18). FBF (microhybrid composite) with lowest filler content showed greater discoloration than the other nanohybrid composites (GUF, HXU, SF), which do not contain triethylene glycol dimethacrylate (TEGDMA) that promoted greater conversion of the resin matrix. Partial substitution of TEGDMA for urethane dimethacrylate (UDMA) comonomer in bisphenol-A glycidyl dimethacrylate (Bis-GMA)/TEGDMA has been reported to decrease water absorption and susceptibility to staining (19). XF, as a microhybrid composite, showed lower staining resistance than SF (nanohybrid composite). This finding could probably be attributed to bisphenol-A ethoxylated dimethacrylate (Bis-EMA), which is highly hydrophobic and has lower water sorption than UDMA (20). Numerous studies have investigated the color stability of nanocomposites and microhybrid composites, but these studies have reported conflicting results, potentially because of variations in the restorative materials tested. The color of SF (nanohybrid composite) was least affected among the tested materials, though this lower affect was not statistically different from that with GUF and HXU, which also did not exhibit a significant difference. GUF has lower filler content compared to SF and HXU, and contains UDMA monomer, which shows more resistance to discoloration than does Bis-GMA and lower water sorption than Bis-EMA (found in SF) (20). GUF contains the nano-sized filler particles, and its production with a new silane treatment has been revolutionary, improving hydrolytic stability and durability of the restorative material. The structure of silane used for the silanization has an influence on the solvent absorption and solubility of composite materials (21). The surface texture of resin-composite materials has a major influence on plaque accumulation, increasing the risk of secondary caries and gingival inflammation as well as susceptibility to discoloration of materials (22). Therefore, the extremely important finishing and polishing procedures are often essential for re-contouring and removal of overhangs. The final surface quality of the material depends on several factors that include filler size and shape, filler loading, surface hardness, polishing procedures, and the structure of

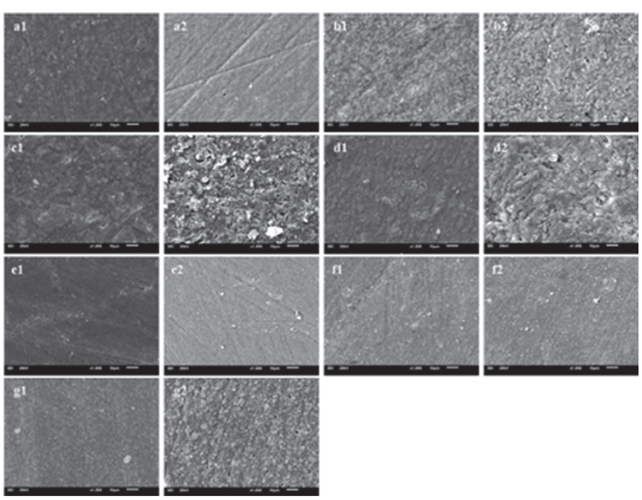

Figure 1. Representative scanning electron microscopy micrographs of the used materials: (a) Filtek Ultimate, (b) SonicFill, (c) X-tra fill, (d) Filtek Bulk Fill Posterior, (e) G-aenial Universal Flo, (f) Herculite XRV Ultra, and (g) Filtek Bulk Fill Flowable. Images were visible at baseline (1) and after aging (2)

resin-matrix (23,24). Traditionally, it is expected that composite resins with larger filler particles would have a higher surface roughness after polishing. Some investigations have reported that composite materials with smaller particles encourage higher gloss and lower surface roughness after polishing with several systems (25,26). In the current study, Sof-Lex discs were used for polishing, and the extent to which the used materials could be polished was significantly different. The highest Ra values were measured for XF (microhybrid), which may be attributed to XF having the largest filler particles. On the other hand, FBF, with the lowest filler amount as a microhybrid composite, showed similar surface roughness to other nanocomposites (SF, HXU, FU, GUF, FBP), none of which showed significant differences when compared to each of the others. A literature review evaluating surface characteristics of microhybrid and nanocomposites has reported that the surface structures of composite materials are dependent on both finishing/polishing system and restorative material used (27). During examination with SEM, the XF surface showed loosened filler particles and minor holes before aging, with defects forming due to the differential abrading effects on the large glass filler and resin matrix. The SF surface revealed surface defects formed with a relief polishing effect between filler particles and resin matrix. Polishing is complicated by the heterogeneous nature of composite materials with hard filler particles and a soft resin matrix (22). GUF, FU, and HXU showed scratch lines with Sof-Lex discs. The surface microstructures of GUF, FBF, HXU, and FU are very uniformly polished. The FBP surface presented minor surface irregularities. A disparity existed between SEM evaluation and the surface roughness tests for SF and FBP. SEM roughness is representative of local order rather than a global roughness obtained via profilometry. Therefore, the SEM technique showed a limited portion of the restoration surface and could neither represent a whole surface nor give the average surface roughness of material (28). The results of this study revealed that an aging procedure caused significant increases in surface roughness for the FBP, SF, FBF, and XF materials. These results are consistent with the findings of previous studies that supported the concept that beverages might lead to resin-matrix decomposition and wear of composite material (29,30). Many drinks and temperature changes can cause the decomposition of the structure of resin matrix and the removal of filler particles (31). According to SEM images, FBF showed surface erosion and fallout of the filler particles, which may be attributed to FBF having the lowest filler content and to specific FBF filler properties; these findings are consistent with those of Han et al. (29), who observed that flowable resins with lower filler content had a lower resistance to beverages. SF and XF surfaces had more profound surface erosion, whereas FBP showed slight erosion. GUF, HXU, and FU showed no significant surface degradation. GUF and HXU (nanohybrid) have smaller particles that are more homogeneous in size distribution, protecting the resin matrix against wear and erosion and increasing the durability of the material (26). This result supports the findings of da Costa et al. (25) and Turssi et al. (26), who reported that composite resins with smaller average filler particles showed a lower increase in surface roughness than did materials with larger filler particles. FBP and FU (nanofill) have the same filler particles, yet performed differently with respect to their surface roughness after the aging procedure. This result may be related to the chemical composition of resin matrix and filler loading. Also, the hydrolytic degradation of silane may lead over time to surface degradation on composite materials and affect the material's abrasion resistance (32). The microhybrid and nanohybrid materials -XF and SF, respectively-had the worst performance with respect to surface roughness than did the other tested materials after aging, likely due to the largest particles found in materials and their more heterogeneous distribution in filler size.

Conclusion

After aging simulation, the evaluated materials showed significantly different color stability. FU had the highest staining susceptibility. The surface roughness was significantly different (both before and after aging) among materials used. Surface roughness after aging increased significantly for FBP, SF, XF, and FBF. XF showed highest surface roughness.

Ethics

Ethics Committee Approval: It was not taken.

Informed Consent: It was not taken.

Peer-review: Externally and internally peer-reviewed.

Authorship Contributions

Surgical and Medical Practices: M.K., S.D., Concept: M.K., Design: M.K., Data Collection or Processing: M.K., S.D., Analysis or Interpretation: M.K., Literature Search: M.K., S.D., Writing: M.K.

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