RESEARCH ARTICLE

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Relationship Between Surface Roughness and Colour Stability of Alkasite, Dual-Cure and Bulk-Fill Composites

Alkasit, Dual-Cure ve Bulk-Fill Kompozitlerin Yüzey Pürüzlülüğü ile Renk Stabilitesi Arasındaki İlişki

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ABSTRACT

Objectives: This study aims to investigate the surface roughness of four different restorative materials on polished and non-polished surfaces and assess its impact on color stability.

Materials and Methods: For the study, we utilized four distinct restorative materials: an alkasite, a nanohybrid composite resin, a dual-cure bulk-fill composite resin, and a posterior bulk-fill composite resin. For each composite resin, polished and non-polished samples were prepared. Following immersion in a coffee solution for a duration of 28 days, the color changes of all samples were assessed with a portable spectrophotometer at days 1, 7, 14, 21 and 28. Surface roughness (Ra) was evaluated using a conventional profilometer. Additionally, surface analyses were conducted for one sample from each group utilizing Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM). Statistical analyses were performed using the Kruskal-Wallis test, the Mann-Whitney U test, Bonferroni-corrected ANOVA and Pearson correlation analysis.

Results: Upon examining the ΔE values at the conclusion of the 28-day period, significant differences among the groups were observed. Cention N exhibited the greatest color change, while Fill-Up demonstrated the least. Notably, the surface roughness values differed significantly among the groups ($p<0.05$). Within the polished groups, Cention N exhibited the highest Ra value, while Filtek One Bulk Fill presented the lowest Ra value. In the samples finished with Mylar strip, Fill-Up showed the highest Ra value and Z 550 showed the lowest Ra value. No linear correlation was identified between the surface roughness and the discoloration of the composite resins.

Conclusion: When using acid neutralising ion-releasing alkasite composite, their disadvantage in terms of colouration and surface roughness should be taken into account.

Keywords: *Composite resin, Discoloration, Surface properties*

ÖZET

Amaç: Bu araştırma, dört farklı restoratif materyalin polisajlı ve polisajsız yüzeylerde pürüzlülüğünü incelemeyi ve bu pürüzlülüğün renk stabilitesi üzerindeki etkilerini değerlendirmeyi amaçlamaktadır.

Gereç ve Yöntemler: Çalışma için bir alkasit, bir nanohibrit kompozit rezin, bir dualcure bulk-fill kompozit rezin ve bir posterior bulk-fill kompozit rezin dahil olmak üzere dört farklı restoratif materyal kullanıldı. Her bir kompozit rezin için polisajlı ve polisajsız örnekler hazırlandı. Tüm örnekler 28 gün boyunca kahve çözeltisinde bekletildikten sonra, renk değişimi değerleri taşınabilir spektrofotometre ile 1, 7, 14, 21 ve 28. günlerde ölçüldü. Yüzey pürüzlülüğünü değerlendirmek için geleneksel bir profilometre kullanıldı (Ra). Yüzey analizleri, her gruptan bir örnek için Atomik Kuvvet Mikroskobu ve Tarama Elektron Mikroskobu kullanılarak yapıldı. İstatistiksel analizler, Kruskal-Wallis testi, Mann-Whitney U testi, Bonferroni düzeltmeli ANOVA ve Pearson korelasyon analizi kullanılarak gerçekleştirilmiştir.

Bulgular: 28. günün sonunda ΔE değerleri incelendiğinde gruplar arasındaki fark anlamlı bulundu ($p<0.05$). Cention N en büyük renk değişimini, Fill-Up ise en az renk değişimini gösterdi. Yüzey pürüzlülüğü değerleri gruplar arasında önemli ölçüde farklıydı ($p<0.05$). Polisaj uygulanmış gruplarda, Cention N en yüksek Ra değerini, Filtek One Bulk Fill ise en düşük Ra değerini gösterdi. Mylar bant ile bitirilen örneklerde ise Fill-Up en yüksek Ra değerini, FZ ise en düşük Ra değerini gösterdi. Kompozit rezinlerin yüzey pürüzlülüğü ile renk değişimi arasında doğrudan bir ilişki bulunmamıştır.

Sonuç: Asidleri nötralize edici iyon salan alkasit kompozitler kullanıldığında, renklenme ve yüzey pürüzlülüğü dezavantajları dikkate alınmalıdır.

Anahtar Kelimeler: *Kompozit rezin, Renklenme, Yüzey özellikleri*

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Introduction

Currently, various types of composite resins are available which are biocompatible, mechanically robust, offering easy and rapid application and high polishability as well as improved aesthetic features.[1,2] Although aesthetic expectations of patients have been fulfilled owing to technical developments in the composite resin structure, restorative dental materials have a number of drawbacks including the requirement for layered placement, bonding failure, risk of gap formation and extended restoration time. In an effort to mitigate these drawbacks, various strategies have been employed, such as incorporating new monomers, advancing filler technology, enhancing translucency, and altering photoinitiator systems.[3] Thanks to these innovations, manufacturers have introduced composite materials that allow the placement of composites up to 4-5 mm thick in a single step, replacing the current technique, in order to simplify the composite restoration procedure and save time.[4] Bulk-fill composites have higher polymerization depths and translucency compared to conventional composite resins.[5]

Dual-cure bulk-fill composites that combine both chemical and light-cure technology have been designed to improve polymerization of bulk-fill composites that can be placed in thick layers. Dual polymerization can eliminate the limitations of light-curing and the need for layered placement.[6] Furthermore, dual-cure resin restorations produce a deeper polymerization and a higher degree of conversion due to the continued reaction after photoactivation.[7] Although composite resins are stable materials, they are associated with a number of shortcomings that limit their use including microleakage caused by polymerization shrinkage/incomplete polymerization, adhesive applications that require extreme technical precision, and cytotoxic effects of degradation in the dynamic oral environment. In an attempt to resolve these problems, researchers have worked to develop composite materials that can provide chemical adhesion with dental hard tissues as well as stimulate remineralization and recently, Alkasite-based dual-cure bulk-fill composite materials have been formulated.[8] Alkaline fillers increasing the release of acid-neutralizing ions are used in these composites.[9,10] Diverse properties imparted to the composite materials may lead to variations in the surface structure of composite resins and in their responses to aging factors. Discoloration is one of the most visible reflections of aging factors and can occur for various reasons.[11] The reasons are related to the characteristics of the composite encompassing resin matrix structure, matrix-particle interface and particle size and volume. Extrinsic causes of discoloration are poor oral hygiene, food and beverages consumed, smoking and occupational factors.[12,13]

Apart from color change, surface roughness is another important parameter for assessing restorative materials.[14,15] It has been claimed that a smooth restoration surface reduces plaque accumulation and delays discoloration.[11] However, there are mixed results from the literature for this claim.[16-18] This study aimed to examine the surface roughness and coloration of four structurally different composite materials to assess the impact of surface roughness on color stability Accordingly, the study tested three hypotheses. The first null hypothesis ($H0_1$) was that there would be no difference among composite materials in terms of discoloration. The second null hypothesis ($H0_2$) was there would be no difference among composite materials in terms of roughness. The third null hypothesis ($H0_3$) was that there is no linear relationship between discoloration and surface roughness.

Materials and Methods

Ethical approval for the study was secured from the Sivas Cumhuriyet University Ethics Committee for Non-Interventional Clinical Studies (Ethical approval no: 2019-08/12). Four different composite resins were used in this in vitro study including an alkasite (Cention N (CN) Ivoclar Vivadent, Schaan, Liechtenstein), a nanohybrid composite resin (Filtek Z550 (FZ), 3M ESPE, St. Paul, MN, USA), a dual-cure bulk-fill composite resin (Fill-Up (FU), Coltene Whaledent, CH), and a posterior bulk-fill composite resin (Filtek One Bulk Fill (FOB), 3M ESPE, St. Paul, MN, USA) (Table 1).

Table 1. Restorative materials used in the study

Material	Type	Content	Manufacturer	Particle Ratio (w/v)
Cention N	Alkasite	Liquid: Dimethacrylates (UDMA, Aromatic aliphatic UDMA), initiators, stabilizers Powder: Calcium-barium-aluminum fluorosilicate glass, calcium fluorosilicate glass, isofillers, ytterbium trifluoride, initiators and pigments (particle size 0.1-7 µm)	Ivoclar Vivadent, Schaan, Liechtenstein	78.4%/5.6%
Filtek Z550	Nanohybrid Composite Resin	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA, Zirconia/ Silica (0,1-10 µm), Modified silica particles (20 nm)	3M ESPE, St. Paul, MN, USA	81.8%/67.8%
Fill-Up	Flowable Bulk-fill	TMPTMA, UDMA, Bis- GMA, TEGDMA, Dental glass, methacrylate, amorphous silica, zinc oxide (particle size 2 µm)	COLTENE, Whaledent, CH	65%/49%
Filtek One Bulk Fill	Bulk-fill	AUDMA, UDMA, AFM, diurethane-DMA, (1,12-dodecane-DMA), Ytterbium Fluoride, EDMAB non-agglomerated/non-aggregated silica (20 nm) non-agglomerated/non-aggregated zirconia (4-11 nm) non-aggregated zirconia /silica cluster filler (20 nm silica and 4-11 nm zirconia particles) agglomerated 100 nm particles	3MM ESPE, St Paul, MN, USA	76.5%/58.5%

Preparation of Samples

Teflon molds (8 mm in diameter, 4 mm in depth) were used to prepare samples in standard sizes. Teflon molds separated by Mylar strip were placed on a glass coverslip. After packing the composite resins into the molds, Mylar strip and glass coverslip were placed again sequentially. Then, polymerization was performed using a LED Curing Light (Elipar DeepCure-S, 3M ESPE, St. Paul, MN, ABD) with a wavelength of 430 nm–480 nm and light intensity of 1470 mW/cm^2 in accordance with the manufacturer's guidelines. While bulk fill composites were inserted at a 4 mm thickness in a single step, the conventional composite was placed in 2 mm increments by light-curing. A total of 40 specimens were prepared from each restorative material, 20 specimens (polished; non-polished) to evaluate the colour change and 20 specimens (polished; non-polished) to evaluate the surface roughness (n=10). For polished groups surface standardization was achieved using 1000-grit silicon carbide sandpaper. Then, samples were polished using gray, green and pink-colored Astropol (Ivoclar Vivadent, Schaan, Lihtenştayn) rubbers respectively. Each rubber was applied to one surface of the samples for 30 seconds with the help of a micromotor at an average pressure, 10,000 rpm using light rotational motion and water to avoid heat generation and groove formation. All samples were washed under distilled water for 1 minute and kept in an oven at

37°C for 24 hours. Samples finished with Mylar strip were stored at 37°C for 24 hours without any surface treatment. Randomly one sample from the samples whose surface roughness was evaluated was used for SEM and AFM analyses.

Assessment of Color Changes

The coffee solution was prepared by mixing 3.6 g of coffee (Nescafe Gold Classic, Nestle, Turkey) with 300 ml of boiled distilled water for 10 minutes. After storing the samples in distilled water for 24 hours, the initial color measurements were taken and recorded as baseline values. Subsequently, over a 28-day period, the samples were kept in an oven (FN 400, Nüve, Turkey) at 37°C for 3 hours a day in the coffee solution and for the remaining 21 hours in distilled water. During this period, the coffee solution was freshly prepared and renewed daily. Before taking color measurements at all observation times (days 1, 7, 14, 21, and 28), the samples were rinsed under running tap water for 10 seconds and dried with drying paper.

A portable spectrophotometer, Vita Easyshade Advance (Vita Zahnfabrik, Bad Sackingen, Germany), was used for color measurements. Measurements were conducted by placing the measuring tip of the device on the center of the sample, at the same time of the day and at the same place. Each sample underwent three repeated measurements, and the average values were documented as L0, a0, and b0*. The device was recalibrated after every three measurements." The following formula was used to calculate ΔE values between two measurements according to the CIELAB color system: ΔE*= [(L1*-L0*)2+(a0*-a1*)2+(b0*-b1*)2]½

Surface Roughness Measurement and SEM and AFM Examination

Surface roughness measurements of the samples were performed using a profilometer (Mitutoyo Surftest/ SJ-301, Tokyo, Japan). Each sample was placed on the profilometer platform ensuring a contact angle of 90º with the reader tip. The surface evaluation length of the surface profilometer was set at 4 mm and the surface cut-off length at 0.25 mm. The profilometer was recalibrated before and after measurements for each group. Measurements were taken from three points on each sample and the average surface roughness (Ra) was calculated from the arithmetic mean of three readings.

Following surface roughness measurements, one sample from each group was evaluated under AFM (Park System, XE-100 E, Korea) and SEM device (Tescan MIRA3, Czech Republic).

Statistical Analysis

The study data were analyzed using the SPSS statistical software program (22.0 Version, Armonk, NY: IBM Corp). The normality of data distribution was assessed using the Kolmogorov-Smirnov test. The ΔE values of the groups were compared using the Kruskal-Wallis and Mann-Whitney U tests, while the surface roughness values of the groups were analyzed using ANOVA with Bonferroni correction. Pearson correlation analysis was performed to examine the relationship between color change and surface roughness values. The Type I error rate was set at 0.05.

Results

Color Change

The results for color changes were expressed as ΔE1 for color change on Day 1, ΔE2 for color change on Day 7, ΔE3 for color change on Day 14, ΔE4 for color change on Day 21 and ΔE5 for color change on Day 28. Looking at the ΔE values at the end of 28 days, CN exhibited the greatest color change, while FU demonstrated the least (Table 2). Pairwise comparison of color change values from examination days 1, 7, 14, 21 and 28 among polished FOB, FZ, FU and CN restorative materials demonstrated a statistically significant difference among the composites at these timepoints ($p<0.05$) (Table 2).

Table 2. Comparison of ΔE values by examination days for polished Filtek One Bulk Fill, Z550, Fill-Up and Cention N composite samples immersed in coffee

	N	Mean	Median	Min.	Max.	Result
ΔE1 Filtek One Bulk-Fill	10	0.91 ± 0.29[a]	0.90	4.56	0.97	KW=13.60
Z550	10	1.20 ± 0.84[a]	0.97	0.97	3.44	P=0.004*
Fill-Up	10	1.06 ± 0.38[a]	1.03	0.57	1.65	
Cention N	10	3.73 ± 1.44[b]	3.88	1.58	6.82	
ΔE2 Filtek One Bulk-Fill	10	2.07 ± 0.52[a]	1.97	1.20	2.82	KW=20.80
Z550	10	2.11 ± 0.78[a]	2.14	0.98	3.67	P=0.000*
Fill-Up	10	1.47 ± 0.41[a]	1.62	0.82	2.02	
Cention N	10	7.77 ± 3.23[b]	7.80	3.80	14.71	
ΔE3 Filtek One Bulk-Fill	10	5.53 ± 0.82[a]	5.77	3.86	6.49	KW=32.80
Z550	10	7.50 ± 0.69[b]	7.50	5.92	8.51	P=0.000*
Fill-Up	10	2.12 ± 1.03[c]	1.92	0.73	4.71	
Cention N	10	14.55 ± 4.08[d]	14.22	8.75	20.55	
ΔE4 Filtek One Bulk-Fill	10	5.44 ± 1.28[a]	5.09	3.71	7.85	KW=32.80
Z550	10	8.63 ± 0.92[b]	8.54	7.33	10.35	P=0.000*
Fill-Up	10	2.54 ± 0.72[c]	2.55	1.45	4.24	
Cention N	10	15.17 ± 5.62[d]	15.41	2.98	23.05	
ΔE5 Filtek One Bulk-Fill	10	6.71 ± 1.05[a]	6.74	5.05	8.38	KW=40.00
Z550	10	10.44 ± 0.87[b]	10.40	9.55	12.50	P=0.000*
Fill-Up	10	3.66 ± 1.57[c]	3.34	1.87	6.83	
Cention N	10	20.08 ± 4.56[d]	20.59	12.46	25.99	

*Kruskal-Wallis test and Mann-Whitney U test: Different letters indicate statistically significant difference between the groups on the same day.

When the color measurements of the composite groups at days 1 and 7 were compared in pairs, the differences were found to be significant for FOB versus CN, FZ versus CN and FU versus CN ($p<0.05$) but the differences between other groups were non-significant ($p>0.05$). Significant differences in color change values were observed in pairwise comparisons among all groups on days 14, 21, and 28($p<0.05$). For the non-polished composite samples (those finished with Mylar strip), the difference in color change values was significant at days 7, 14, 21 and 28 when compared in pairs ($p<0.05$) (Table 3). On days 7, 14, 21 and 28, the difference was significant for the comparison between FU and FOB, between FU and FZ and between FU and CN N ($p<0.05$), with no substantial difference among other groups ($p>0.05$).

Table 3. Comparison of ΔE values by examination days for non-polished (finished with Mylar strip) composite groups immersed in coffee

	N	Mean	Median	Min.	Max.	Result
ΔE1 Filtek One Bulk-Fill	10	2.23 ± 0.96[a]	2.37	0.67	4.07	KW=2.40
Z550	10	2.34 ± 1.22[a]	2.28	0.84	4.98	P=0.494
Fill-Up	10	1.56 ± 0.70[a]	1.47	0.76	2.55	
Cention N	10	10.25 ± 5.66[a]	8.70	1.27	19.58	
ΔE2 Filtek One Bulk-Fill	10	5.21 ± 2.04[a]	5.03	2.86	9.82	KW=8.80
Z550	10	5.49 ± 2.43[a]	4.79	2.09	9.17	P=0.032*
Fill-Up	10	2.58 ± 0.69[b]	2.51	1.64	3.94	
Cention N	10	24.09 ± 9.21[a]	20.47	14.07	42.91	
ΔE3 Filtek One Bulk-Fill	10	8.87 ± 2.98[a]	8.86	3.65	15.32	KW=12.80
Z550	10	12.42 ± 2.19[a]	12.05	9.28	17.04	P=0.005*
Fill-Up	10	4.47 ± 0.754[b]	4.32	3.45	5.76	
Cention N	10	32.70 ± 7.38[a]	31.55	21.84	43.64	
ΔE4 Filtek One Bulk-Fill	10	10.94 ± 3,40[a]	9.49	7.98	18.45	KW=16.80
Z550	10	13.91 ± 2.19[a]	13.57	10.75	17.49	P=0.001*
Fill-Up	10	6.06 ± 1.15[b]	5.95	4.53	8.18	

Cention N	10	36.68±10.07[a]	35.21	24.01	57.54	
ΔE5 Filtek One Bulk-Fill	10	13.25 ± 4.40[a]	11.94	7.75	21.02	KW=13.60
Z550	10	15.69 ± 1.90[a]	15.19	12.98	19.30	P=0.004*
Fill-Up	10	7.40 ± 1.51[b]	7.21	5.59	10.28	
Cention N	10	42.50±11.35[a]	42.10	25.51	63.06	

*Kruskal-Wallis test and Mann-Whitney U test: Different letters indicate statistically significant difference between the groups on the same day.

Table 4. Surface roughness (Ra) values of polished and unpolished (Finished with Mylar strip) samples

		N	Mean	Median	Min.	Max.	Result
Polished	Filtek One Bulk-Fill	10	0.21 ±0.11[a]	0.18	0.12	0.52	P=0.001*
	Z550	10	0.23 ±0.10[ab]	0.21	0.12	0.51	
	Fill-Up	10	0.44 ±0.23[b]	0.19	0.13	0.84	
	Cention N	10	2.00 ±1.13[b]	0.38	0.16	3.86	
Non-polished (Mylar strip)	Filtek One Bulk-fill	10	0.34 ±0.24[cd]	0.24	0.14	0.93	P=0.000*
	Z550	10	0.14 ±0.02[c]	0.14	0.11	0.20	
	Fill-Up	10	0.76 ±0.73[d]	0.38	0.15	2.08	
	Cention N	10	0.48 ±0.18[d]	0.45	0.26	0.93	

* Analysis of Variance, Bonferroni Correction: Different letters indicate a statistically significant difference between the groups. The difference among the polished composite groups is indicated with the letters a and b and the difference among non-polished groups is indicated with the letters c and d.

Evaluation of surface roughness values for polished and non-polished samples of each restorative material showed a non-significant difference (p>0.05) except for FZ (p=0.006). Greater Ra values were found for polished FZ samples (Table 5).

Table 5. Surface roughness (Ra) values of polished and non-polished samples of the restorative materials

		N	Mean	Med.	Min.	Max.	Result
Filtek One Bulk Fill	Polished	10	0.21 ± 0.11[a]	0.18	0.12	0.52	P=0.139
	Mylar strip	10	0.34 ± 0.24[a]	0.24	0.14	0.93	
Z550	Polished	10	0.23 ± 0.10[a]	0.21	0.12	0.51	P=0.006*
	Mylar strip	10	0.14 ± 0.02[b]	0.14	0.11	0.20	
Fill-Up	Polished	10	0.29 ± 0.23[a]	0.19	0.13	0.84	P=0.096
	Mylar strip	10	0.76 ± 0.73[a]	0.38	0.15	2.08	
Cention N	Polished	10	0.85 ± 1.13[a]	0.38	0.16	3.86	P=0.762
	Mylar strip	10	0.48 ± 0.18[a]	0.45	0.26	0.93	

* Analysis of Variance, Bonferroni Correction: Different letters indicate a statistically significant difference between polished and non-polished subgroups of each composite group.

Pearson correlation test did not show a correlation between the surface roughness values and color change values of polished and non-polished groups (p>0.05) (Table 6).

Table 6. Correlation between surface roughness values and color change values of polished and finished with Mylar strip composite resins

Composite	Correlation	N	Mean	Result
Filtek One Bulk Fill	ΔE	10	6.71 ± 1.05	P=0.623
	Polished ΔRa	10	0.17 ± 0.02	
Z550	ΔE	10	10.44 ± 0.87	P=0.643
	Polished ΔRa	10	0.20 ± 0.08	
Fill-Up	ΔE	10	3.66 ± 1.57	P=0.120
	Polished ΔRa	10	0.42 ± 0.56	
Cention N	ΔE	10	0.08 ± 4.56	P=0.225
	Polished ΔRa	10	2.17 ± 4.39	
Filtek One Bulk Fill (Mylar strip)	ΔE	10	13.25 ± 4.40	P=.820
	Non-polished ΔRa	10	0.19 ± 0.06	
Z550 (Mylar strip)	ΔE	10	15.69 ± 1.90	P=.496
	Non-polished ΔRa	10	0.55 ± 0.67	
Fill-Up (Mylar strip)	ΔE	10	7.40 ± 1.51	P=.532
	Non-polished ΔRa	10	0.42 ± 0.12	
Cention N (Mylar strip)	ΔE	10	42.50 ±11.35	P=.991
	Non-polished ΔRa	10	0.68 ± 0.37	

* Pearson correlation test did not show a correlation

On AFM analysis, Average of Ra values (nm) obtained from three separate 10×10 µm scanning areas (Polished-Mylar Strip); FOB 19.228 – 18.348 nm, FZ 32.580 - 27,530 nm, FU 57,272 - 100,730 nm, CN 68,715 - 138,015 (Figure 2; A, B, C, D, E, F, G, H). For CN (Figure; G,H), deep grooves and irregularities were observed on the surface of non-polished groups and medium level surface irregularities were seen in polished groups. SEM images were in line with profilometer and AFM findings (Figure 1; A, B, C, D).

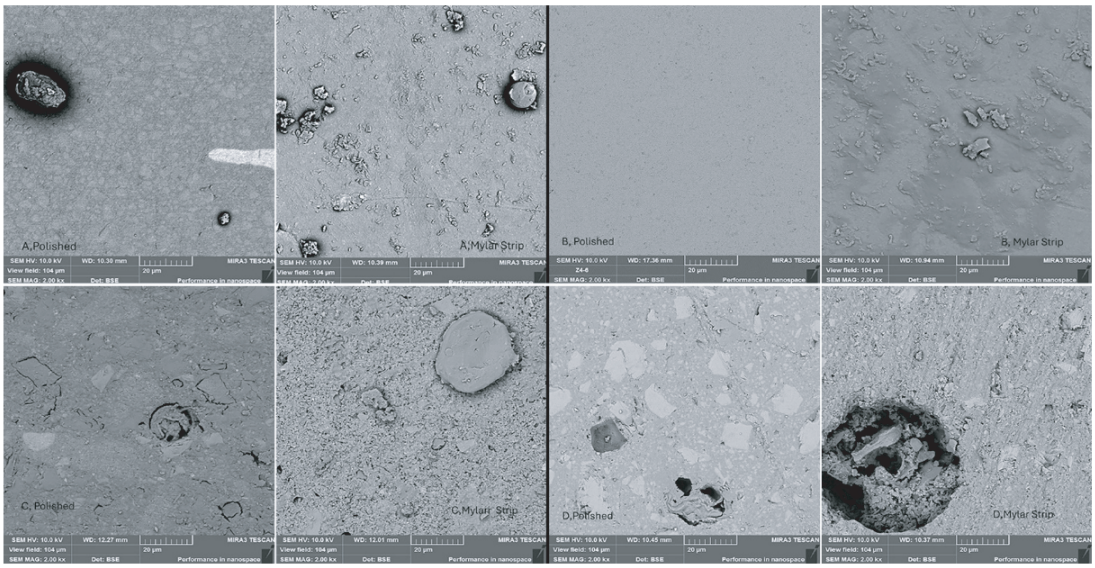

Figure 1, A: Filtek One Bulk-Fill polished and Mylar strip; B: Z550 polished and Mylar strip; C: Cention N polished and Mylar strip; D: Fill-Up polished and Mylar strip

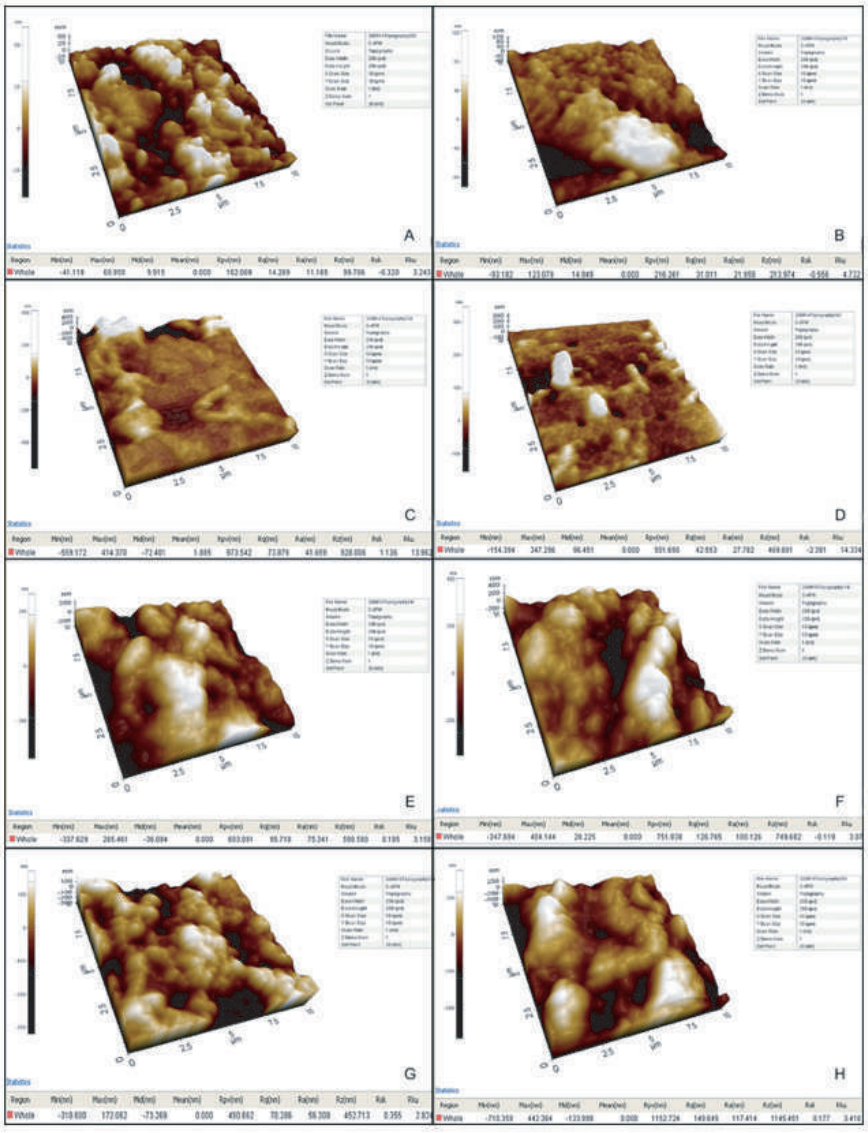

Figure2, A: Filtek One Bulk-Fill polished; B: Filtek One Bulk-Fill Mylar strip; C: Z 550 polished; D: Z 550 Mylar strip; E: Fill-UP polished; F: Fill-Up Mylar strip; G: Cention N polished; H: Cention N Mylar strip

Discussion

Discoloration that occurs in composite resin restorations over time is one of the primary causes for replacing dental restorations.[19] Many factors can affect the process of discoloration in composite materials, including resin matrix structure, incomplete polymerization, water absorption, foods and drinks, oral hygiene and surface roughness.[20]

Water absorption predominantly takes place directly within the resin matrix. In contrast, filler particles cannot absorb water but adsorb water on their surfaces. This causes more water absorption, resulting in a lower adhesion between the composite resin matrix and filler particles.[21] Water absorption by the resin matrix may lead to expansion and plasticization of the resin composition, hydrolysis of silane and microcrack formation. Penetration of coloring agents between the filler and matrix may cause discoloration.[22] Coffee contains water-soluble dyes, tannic acid, caffeine, phenolic acid and chlorogenic acid. The low polarity of the composite resin leads to increased adsorption and dye penetration by coffee pigments. Thus, coffee stains cannot be removed easily by brushing or polishing.[23]

There is no consensus on whether the resin structure or surface roughness accounts for discoloration of composite resins.[24,25] In our study, staining of composite resins, surface roughness and their correlation were investigated to find an answer to this question. Based on our findings, the first null hypothesis of our study "There would be no difference among composite materials in terms of discoloration" was rejected. According to the color changes of the polished and Mylar strip-finished groups immersed in the coffee solution, the materials were ranked in the greatest order and the lowest staining for all days: CN > FZ > FOB > FU.

CN was the only material that exceeded the clinically acceptable limit of 3.3 in ΔE values on Day 1[21]. It has been reported that the greatest color change observed in CN may be explained by the fact that 12-40% of the final matrix may show monomer leakage and also, fluoride release from the material may account for discoloration.[26,27] Günülol et al.[28] reported that a fluoride-releasing composite showed significantly higher water absorption and color change (ΔE) values compared to other materials. They stated that fluoride release from a restorative material depends on water diffusion capacity, which can result in degradation of the material's chemical structure and matrix bonds and matrix bonds of the material and the release of residual monomers. In a study by Park et al.[29] evaluating polymerization of fluoride-containing composites, it was suggested that dissociation of filler particles due to fluoride release may create gaps on the composite surface which may then cause a reduction in surface microhardness.

In line with our findings, some studies have reported a high level of staining for CN.[26,30] Amalavathy et al. suggested that this may be related to acidity of beverages or ion exchange activity on the sample surface.[31] Tannic acid found in coffee has phenolic hydroxy structures and the polyphenolic end groups of tannic acid are highly favorable for hydrogen bonding. [27] The highest ΔE value observed with CN can be ascribed to the formation of hydrogen bonds between tannic acid and the fluorosilicate compounds in CN.[32]

François et al.[30] reported a significant degradation in highly reactive calcium fluorosilicate glass fillers under acidic conditions due to acid attack. We think that the organic acids in coffee may cause degradation of filler glass particles[33, 34] and over time, cracks may occur at the resin-filler interface, weakening the material and further increasing surface roughness and discoloration.[35-37]

In our study, CN, FZ and FOB were the materials with the greatest color alteration. Analysis of the polished samples immersed in coffee solution showed a significant difference among the materials at day 14, which was also observed at other timepoints. At day 28, average ΔE values were 10.40 for FZ and 6.71 for FOB., Although the statistical analysis revealed no significant differences among the non-polished samples, FZ showed greater ΔE values than FOB at all examination days. Bis-GMA and TEGDMA monomers found in the resin matrix

are hydrophilic, whereas UDMA is hydrophobic. Therefore, the UDMA monomer is more resistant to color changes. Bis-GMA has a higher water absorption capacity compared to UDMA and Bis-EMA.[38] We posit that the monomer structure of FZ contributed to these observed findings.

In their study examining the long-term water absorption and solubility of composite resins with different structures, Alshabib et al.[39] reported that FOB showed low water absorption, which was attributed to UDMA found in the material.

In our study, FU, a medium-viscosity dual-cure bulk-fill composite showed the lowest ΔE values. It was also the only material that did not exceed the clinically acceptable ΔE limit at day 21. In contrast to our findings, the greatest staining was found in the FU group in a study by Freitas et al.[40] evaluating bulk-fill (Filtek Bulk Posteior, FU) and microhybrid (Filtek Z250) composites immersed in coffee solution after polishing; however, they did not observe a difference in discoloration among FU, Filtek Z250 and Filtek Bulk Fill Posterior with one of the polishing protocols they used.[18]

In a study by Monterubbianesi et al.[41] investigating the degree of monomer conversion of bulk-fill composites, they applied FU in one increment at a thickness of 4 mm, followed by light-curing with Elipar S10. Measurements taken from the bottom surface of the material after 24 hours showed that the extent of monomer conversion was 94.71% for this material. The high color stability of FU as observed in our study may be connected to the high extent of monomer conversion.

Surface roughness one of the key factors affecting the success of a restoration.[42,43] Surface roughness of the resin materials is affected by the type of monomers in their composition, the size and shape of fillers, the quality of adhesion to matrix and the depth of polymerization.[44] Based on our findings, the second null hypothesis of the study, "There would be no difference among composite materials in terms of roughness" was rejected. The rank order of surface roughness was CN > FU> FZ> FOB for the polished groups, whereas it was FU > CN> FOB> FZ for the non-polished groups.

The least surface roughness (Ra) values were observed in FOB and FZ. Consistently, low surface roughness was reported for FZ[45] and FOB[46,47] in three separate studies. However, the observation of the lowest Ra values in polished samples of FOB in contrast to non-polished samples of FZ in our study is noteworthy. These two composite resins have a similar inorganic structure but FZ is a nanohybrid material with a particle size ranging from 0.1 to 10 μm (mean 0.02 μm).[48] FOB contains 100 nm agglomerate ytterbium trifluoride to enhance the contrast of X-rays.[49] In addition to nanomer structures, FOB contains nanoclusters which are composed of loosely bound, nano-scale inorganic fillers and can be abraded without breaking off from the surface during polishing.[41,44] On the other hand, for FZ, larger particles may have detached from the surface during polishing and increased the Ra value. This may explain the small, non-significant difference in terms of surface roughness. Although both composite resins displayed a homogenous and smooth surface on SEM and AFM (Figure1;A,B. Figure 2;A,B,D,E) images, there were also scratches from the polishing process.

Greater surface roughness observed on both polished and non-polished composite surfaces of FU compared to the two a forementioned composites may be related to the larger particle size of fillers in FU. In order to achieve application of the composites in 4 mm thick layers, fillers with large particle size were used by increasing translucency.[50] Reduced size and increased volume of the particles result in less interparticle spacing, which protects the resin matrix during the polishing process and makes it difficult for the filler to detach from the surface.[51] FU (49%) has a lower filler volume compared to FZ and FOB (67.8% and 58.5% respectively) as well as larger filler particle size and irregular particle shape, all contributing to its higher roughness values.[52] On SEM images (Figure 1;D), traces of polishing rubbers and gaps created by the glass particles breaking off from the surface during polishing were observed.

AFM images (Figure2; E, F) showed deep slits and surface irregularities. FU cannot be applied as easily as light-cured composites and hardens without obtaining a perfectly smooth surface. We consider that the high surface roughness of FU is also associated with the difficulty of manipulation. While polishing after polymerization reduced the surface roughness of FU, it could not reach the Ra values of FOB and FZ. Paolone et al.[53] investigated surface roughness of four distinct bulk fill composites finished with different polishing systems and found that FU had the greatest surface roughness in each polishing group. The authors suggested that high Ra values of FU may be related to its large particle size (2 μm) and low filler content (65% by weight, 49% by volume).

CN was the other material showing a higher surface roughness in our study. Studies by Naz et al.[54] and Setty et al.[55] support our findings. This may have resulted from the large and irregularly shaped fillers of CN (0.1 to 7 μm) and the difficulty of inserting it into the cavity. On SEM images (Figure 1; C), non-polished samples of this composite showed an uneven surface appearance and small pits were observed in polished samples due to the particles detached from the surface.

With our findings, the third null hypothesis of our study "There is no linear relationship between discoloration and surface roughness" was accepted. Lu et al.[17] stated that surface roughness may not always be positively correlated with staining, and that surface roughness values below 0.1 μm (100 nm) have no effect on color stability. Öztürk et al.[25] did not find a correlation between color stability and 3D surface topography analysis and suggested that this may be related to the use of a different methodology and equipment.

Conclusions

In this in vitro study, based on the findings of high discoloration and roughness observed in alkasite samples, we conclude that these materials cannot be considered as viable alternatives to nano-filled composites. Considering the superior color stability of dual-cure composites, they are recommended for effective polymerization, particularly in the restoration of cavities where light penetration is challenging. However, the limited shade options and difficulties in cavity application are noted as significant disadvantages of these materials. In our study, no correlation was found between the surface roughness and discoloration of composite resins. The differences observed among the ΔE values of the materials may be attributed more to the composition of the resins rather than surface roughness.

Ethical Approval

Ethical approval for the study was obtained from the Sivas Cumhuriyet University Ethics Committee for Non-Interventional Clinical Studies. (Ethical approval no: 2019-08/12).

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Conflict of Interest Statement

The authors declare no conflicts of interest

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