Cumhuriyet Dental Journal, 26(3): 310-320, 2023
DOI: https://doi.org/10.7126/cumudj.1342922

Cumhuriyet Dental Journal

| cdj.cumhuriyet.edu.tr | Founded: 1998 | Available online, ISSN: 1302-5805 e-ISSN: 2146-2852 | Publisher: Sivas Cumhuriyet Üniversitesi

The Effect of Food-Simulating Liquids and Thermal Aging on Surface Roughness and Color Stability of Bulk-Fill and Conventional Composites

Vahti Kılıç[1-a*], Feridun Hürmüzlü[2-b], Alper Kaptan[3-c]

[1] Department of Restorative Dentistry, Faculty of Dentistry, Erciyes University, Kayseri, Türkiye.
[2] Department of Restorative Dentistry, Faculty of Dentistry, Lokman Hekim University, Ankara, Türkiye.
[3] Department of Restorative Dentistry, Faculty of Dentistry, Cumhuriyet University, Sivas, Türkiye.
*Corresponding author

Research Article

History

Received: 14/08/2023
Accepted: 06/09/2023

ABSTRACT

Objectives: The aim of this study was to evaluate the effect of food-simulating liquids (FSLs) and thermal aging on the surface roughness and color stability of bulk-fill and conventional composites.

Materials and Methods: A total of 320 disc-shaped samples were prepared, with 40 samples from each of 4 different bulk-fill composites (Filtek Bulk Fill, X-tra fil, Beautifil Bulk Restorative, and Estelite Bulk Fill Flow), and 4 conventional nano-filled composite resins (Filtek Z550, CeramX SphereTEC one, Admira, and Kalore). The prepared samples were randomly divided into subgroups for exposure to FSLs (ethanol, heptane, citric acid) and thermal cycling (TC) (n=10 per subgroup) for 28 days. Initial profilometric surface roughness measurements (Ra_0) of all samples and AFM and SEM analyses of selected samples were followed by exposure to FSLs and TC. After completion of aging protocols, measurements and analyses were repeated to obtain the Ra_1 (post-treatment surface roughness), and change in surface roughness (ΔRa_{1-0}) was then calculated. Subsequently, initial color measurement of the samples was conducted using a spectrophotometer, followed by immersion of the samples in a coffee solution for 24 hours. Color measurements were repeated, and color change (ΔE) was calculated. Two-way repeated measures ANOVA was used to compare Ra_0 and Ra_1 values and one-way ANOVA for comparing ΔRa and ΔE values. Post-hoc Tukey tests were employed for pairwise comparisons. The significance level was set at α=.05.

Results: While the surface roughness of bulk-fill composites was affected by the protocols applied ($p<0.05$), most of the conventional composites generally remained unaffected. Bulk-fill composites exhibited greater ΔRa and ΔE values. The highest ΔRa and ΔE values were observed in the Beautifil Bulk Restorative group, with the greatest discoloration seen after immersion in citric acid.

Conclusions: Thermal cycling and immersion in FSLs affect surface roughness and color stability of composite resins depending on the content and structure of the composites.

Key words: Bulk-Fill Composites, Food-Simulating Liquids, Thermal Cycling, Surface Roughness, Color Stability.

Gıdaları Taklit Eden Sıvıların Ve Termal Yaşlandırmanın Bulk-fill Ve Konvansiyonel Kompozitlerin Yüzey Pürüzlülüğü Ve Renk Stabilitesi Üzerine Etkisi

ÖZ

Amaç: Bu çalışmanın amacı gıdaları taklit eden sıvılar ve termal yaşlandırmanın bulk-fill ve konvansiyonel kompozitlerin yüzey pürüzlülüğü ve renk stabilitesi üzerine etkisini değerlendirmektir.

Gereç ve Yöntemler: 4 farklı bulk-fill kompozit (Filtek Bulk Fill, X-tra fil, Beautifil Bulk Restorative ve Estelite Bulk Fill Flow) ve dört konvansiyonel nano dolduruculu kompozit rezin (Filtek Z550, CeramX SphereTEC one, Admira ve Kalore) kullanılarak disk şeklinde her birinden 40 toplam 320 adet örnek hazırlandı. Hazırlanan örnekler gıdaları taklit eden sıvılarda (etanol, heptan, sitrik asit) bekletilmek ve termal siklus uygulaması için rastgele 4 alt gruba ayrıldı (n=10). Gruplardaki örneklerin ilk profilometrik yüzey pürüzlülük ölçümü (Ra_0) ve seçilen örneklerin AFM ve SEM işlemi sonrası gıdaları taklit eden sıvılarda 28 gün bekletme ve termal siklus uygulamasına geçildi. İlgili işlemler sonrası ölçüm ve analizler tekrarlanarak (Ra_1) değerleri elde edilerek pürüzlülük değişimi (ΔRa_{1-0}) hesaplandı. Daha sonra örneklerin ilk renk ölçümü spektrofotometre ile yapılarak 24 saat kahve solüsyonunda bekletildi ve sonrasında renk ölçümü tekrarlandı ve renk değişimi (ΔE) hesaplandı. Ra_0- Ra_1 değerlerinin karşılaştırılması için two-way repeated measures ANOVA, ΔRa ve ΔE değerlerinin karşılaştırılması için one-way ANOVA, ikili karşılaştırmalar için post-hoc Tukey testi kullanıldı (α=,05).

Bulgular: Bulk-fill kompozitlerin yüzey pürüzlülükleri uygulanan protokollerden etkilenirken ($p<0,05$) konvansiyonel kompozitlerde grupların çoğu etkilenmemiştir. Bulk-fill kompozitler daha yüksek ΔRa ve ΔE değerleri göstermiştir. En yüksek ΔRa ve ΔE Beautifil Bulk Restorative grubunda görülürken en fazla değişim ise sitrik asitte bekletme sonrası görülmüştür.

Sonuçlar: Gıdaları taklit eden sıvılarda bekletme ve termal siklus uygulaması, kompozit rezinlerin içeriği ve yapısına göre yüzey pürüzlülüğü ve renk stabilitesi üzerine etkilidir.

Anahtar Kelimeler: Bulk-Fill Kompozitler, Gıdaları Taklit Eden Sıvılar, Termal Siklus, Yüzey Pürüzlülüğü, Renk Stabilitesi.

[a] dt.vahtikilic@gmail.com https://orcid.org/0000-0003-4653-3729
[b] hurmuzluf@gmail.com https://orcid.org/0000-0003-2812-1835
[c] alperkapdan@gmail.com https://orcid.org/0000-0001-5773-8522

How to Cite: Kılıç V, Hürmüzlü F, Kaptan A. (2023) The Effect of Food-Simulating Liquids and Thermal Aging on Surface Roughness and Color Stability of Bulk-Fill and Conventional Composites, Cumhuriyet Dental Journal, 26(3):310-320.

Introduction

In recent years, the use of resin-based composite (RBC) materials in dental restorations has increased due to their ability to meet aesthetic expectations and technological advances in matrix, filler structure, and content.[1] Nano-filled composite resins, which are among the most commonly preferred composites today, are routinely used in the restoration of both posterior and anterior teeth in the clinical setting. Bulk-fill composites that can be inserted into cavities in a single layer with a thickness of 4-5 mm have been introduced as an alternative to these conventional composite resin materials, placed in 2 mm increments using layering techniques.[2] This innovation has overcome shortcomings of layering techniques, such as interlayer contamination, air entrapment, and weak interlayer bonding, which require technical precision and extended time for the restoration of large and deep cavities.[3,4] Bulk-fill composites are available in two different types: flowable (low-viscosity) and sculptable (high-viscosity). Studies comparing the mechanical properties of bulk-fill and conventional composites have yielded contradictory results.[2,5,6]

Over time, RBC restorations in the oral cavity are exposed to various factors such as the forces generated during chewing and the moisture introduced by saliva, as well as chemical compounds present in food and beverages, and thermal effects due to the varying temperatures of the food ingested.[7] RBC materials are susceptible to changes caused by chemicals found in food and beverages, leading to alterations in surface structure and physical properties. Consequently, increased surface roughness can occur over time, resulting in the loss of material's aesthetic qualities and aging.[8,9] The increase in surface roughness resulting from chemical degradation can lead to issues such as discoloration of restorations, increased plaque accumulation, irritation of soft tissues, and the development of recurrent caries.[10,11]

In order to assess the impact of chemical compounds present in the oral environment on dental composites, food simulating liquids (FSLs) have been established by the Food and Drug Administration (FDA).[12] Thermal aging and aging using FSLs are commonly employed methods for the artificial aging of composite resins.[13,14] Thermal aging is intended to simulate temperature fluctuations within the oral cavity. For this purpose, ethanol and citric acid can be used to simulate certain alcoholic beverages, soft drinks, vegetables, fruits, mouthwashes, candies, and syrups, while heptane is used to simulate butter, fatty meats, and vegetable oils.[15,16] It has been observed that the chemical composition and physical-mechanical properties of the composites are affected by the use of these solutions, with the matrix being more affected than the filler component.[17] In various studies, color changes and color stability of composites after exposure to FSLs and thermal aging have been examined, showing differential results based on the materials and solutions used.[18,19]

Profilometers are among the most commonly used devices to evaluate the surface roughness of dental composites. Profilometers provide quantitative measurements of surface roughness, typically expressed as Ra, for the examined samples. In addition to profilometric measurements, scanning electron microscopy (SEM) and atomic force microscopy (AFM) can be used to analyze surface roughness and characteristics.[20] AFM has the ability to provide detailed three-dimensional topography of the examined surface at a nanometric level. Thus, AFM is regarded as a reliable method for investigating the surface properties of dental materials following various procedures.[21]

In the literature, there are studies reporting the effects of FSLs on the surface and physical properties of bulk-fill composites.[14,22,23] Unlike previous studies, the present study aimed to investigate the effects of both FSLs and thermal aging on surface properties and color stability using conventional and bulk-fill composites with different content and characteristics, as well as to compare these properties among composites. The null hypotheses tested in our study were that FSLs and thermal aging would not have an impact on the surface roughness and color stability of the composite materials used, and that the surface roughness and color stability of the composites would not change according to the aging protocol.

Material and Methods

In our study, a total of 320 samples were prepared using four different bulk-fill composite resins (Filtek Bulk Fill, X-tra fil, Beautifil Bulk Restorative, and Estelite Bulk Fill Flow) and four conventional nano-filled composite resins (Filtek Z550, CeramX SphereTEC one, Admira, and Kalore), with 40 samples from each composite. Detailed information about the composites used is provided in Table 1. Teflon discs with a width of 8 mm and thickness of 2 mm were used for sample preparation. After placing the composites onto the discs, they were polymerized for 20 seconds using a LED light-curing device (Elipar DeepCure-S, 3M ESPE, St. Paul, MN, USA) with an output of 1470 mw/cm^2. At this step, the light intensity of the device was periodically monitored using a radiometer (Radiometer, Peng Lim Enterprise Co., Ltd., Taiwan). Following polymerization, the sample surfaces were sanded using 1000-grit sandpaper to achieve standardization. Subsequently, polishing was carried out using fine and extra-fine steps of a polishing system (Optidisc, Kerr Corporation, Orange, CA, USA) in a standard manner. Next, the samples were rinsed with water-air spray to remove residues from the surface and then dried. Finally, the samples prepared from each composite were divided into four subgroups (n=10 per subgroup) based on the solution in which they will be immersed (aging method).

Surface Roughness, SEM and AFM Analyses

Surface roughness and color measurements were conducted for all samples, and one sample from each

group was selected for SEM and AFM analyses. A contact-type profilometer (Surftest SJ-301, Mitutoyo, Japan) was used to measure the surface roughness of the samples, setting the cut-off length (λc) at 0.8 mm, tracing length at 4 mm, and stylus speed at 0.25 mm/s. Calibration of the profilometer was checked before starting the measurements and between the samples. Three measurements were obtained from different points on the surface of the samples, and the average of the measurements was used to determine the initial surface roughness value (Ra_0) for each sample, expressed as Ra.

One sample from each group was used for SEM and AFM analyses in this study. As the analysis would be repeated after the aging procedure following the initial SEM analysis, the samples were not coated. To enhance conductivity, the samples were wrapped with carbon tape to ensure no direct contact with the surface. The samples were then placed on the platform of the SEM device (Jeol Jsm 6510, Jeol Ltd., Tokyo, Japan). Representative areas were chosen for obtaining baseline SEM images at 1000x magnification under 5 kV electric current. Subsequently, for AFM analysis, the samples were placed in the AFM device (XE-100, Park Systems, South Korea), and images were acquired from a 20x20 µm area at a scanning rate of 1.0 Hz and a resolution of 512x512 pixels. The images from the AFM analysis were examined using the XEI Data Analysis Program, 1.6 Version (PSIA Inc., USA), and 3D topographic images were obtained. For each sample, the root mean square roughness (Sq) and average roughness (Sa) values were noted.

Aging with FSLs and Thermal Cycling

After completing the initial surface roughness, SEM, and AFM analyses, the samples underwent aging with three different FSLs and thermal cycling. For the first group consisting of each composite resin, thermal cycling (TC) was applied to the samples for a total of 5000 cycles of increasing temperatures from 5°C to 55°C (±2°C), with transfer times of 5 seconds and dwell times of 30 seconds. The second group of samples was immersed in a n-heptane solution (Tekkim Chemical Ind., Istanbul, Turkey) at 37°C for 28 days. The third group of samples was immersed in a 10% (w/v) citric acid solution (ChemBio Laboratory Research, Istanbul, Turkey) at 37°C for 28 days. The fourth group of samples was stored in a solution of 75% ethanol and water (Teksoll, Tekkim Chemical Ind., Istanbul, Turkey) at 37°C for 28 days. After completion of the immersion and thermal cycling steps, the samples were removed, rinsed, and dried. Surface roughness measurements were conducted as previously mentioned to determine the final surface roughness values (Ra_1). The change in surface roughness ($\Delta Ra_{1-0} = Ra_1 - Ra_0$) was calculated by subtracting the initial surface roughness values (Ra0) from the final Ra values (Ra1). Additionally, SEM and AFM analyses were repeated for one sample from each group.

Table 1. Type, chemical composition and manufacturer details of the composite resins used in the study.

Composite resins (Abbreviation)	Type	Contents	Filler particle size and ratio (%Wt/Vol)	Manufacturer
Filtek Bulk Fill (FBF)	High-viscosity bulk-fill	Bis-GMA, Bis-EMA, UDMA, Zirconia, Silica, Ytterbium trifluoride	0.004- 0.1 µm 76.5 / 58.4	3M ESPE, St. Paul, MN, USA
X-tra fil (XF)	High-viscosity bulk-fill	Bis-GMA, UDMA, TEGDMA Inorganic fillers in a methacrylate matrix	2 – 3 µm 86 / 70.1	Voco, Cuxhaven, Germany
Beautifil-Bulk Restorative (BF)	High-viscosity giomer based bulk-fill	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, S-PRG based on F-Br-Al- Si glass	No Data 87 / 74.5	Shofu Inc. , Kyoto, Japan
Estelite Bulk Fill Flow (EB)	Flowable bulk-fill composite	Bis-GMA, Bis-MPEPP, TEGDMA, SiO2, and ZrO2 fillers	avg 0.2 µm 70 - 56	Tokuyama, Japan
Filtek Z550 (Z550)	Nanohybrid	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA, Zirconia, Silica	0.02 - 3 µm 81.8 - 67.8	3M ESPE, St. Paul, MN, USA
Ceram.X SphereTEC One (CX)	Nanoceramic	Methacrylate modified polysiloxane, dimethacrylate, Barium-aluminum borosilicate glass, functional prepolymerized silicon dioxide	0.1 - 1.5 µm 77 / 55	Dentsply, Milford, USA
Admira (AD)	Ormocer	Bis-GMA, UDMA, Organic modified ceramic, silica	0.04-0.7 µm 79 - 56	Voco, Cuxhaven, GERMANY
Kalore (KAL)	Nanohybrid	UDMA, DX-511 (UDMA), Bis-EMA lanthanide fluoride, strontium glass, barium glass, fluoroalumina silicate glass, silicon dioxide	0.4–0.7 µm 82 - 69	GC Corporation, Tokyo, JAPAN

* Bis-GMA: Bisphenol A-diglycidyl methacrylate; Bis-EMA: ethoxylated bisphenol A glycol dimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; Bis-MPEPP: 2,2-bis(4-methacryloxypolyethoxyphenyl) propane; S-PRG: Surface pre-reacted glass-ionomer; PEGDMA: polyethylene glycol dimethacrylate;

Evaluation of Color Stability

To assess the color stability of the samples after aging, a contact-type spectrophotometer (Vita Easyshade® V, VITA Zahnfabrik GmbH & Co. KG, Germany) was used for color measurements. Color measurements of the samples were conducted on a white background under standard conditions. For each sample, color measurements were obtained three times consecutively from the center of the samples using the CIEL*ab* system. The average of three measurements was noted as the initial color values of L_0*, a_0* and b_0*.

Subsequently, the samples from each group were immersed in a coffee solution [Nescafé Classic 2 g (Nestlé, Switzerland) - 200 ml boiling water] for 24 hours, which corresponds to one month of coffee consumption [24]. After 24 hours, the samples were removed from the solution, rinsed with distilled water, dried using absorbent paper, and then subjected to the second round of color measurement. The color measurements were repeated three times as previously mentioned, and the average values were recorded as the final color values as L_1*, a_1* and b_1*.

The initial (L_0*, a_0*, b_0*) and final (L_1*, a_1*, b_1*) color values were used to calculate the color change (ΔE_{1-0}) for the samples using the formula;

$$\Delta E_{1-0} = [(L_1^* - L_0^*)^2 + (a_1^* - a_0^*)^2 + (b_1^* - b_0^*)^2]^{1/2}.$$

Statistical Analysis

Statistical analysis of the study data was performed using IBM SPSS Ver. 22.0 (Statistical Package for Social Sciences; IBM Corp., Armonk, NY) software. The normality of data distribution was checked using Kolmogorov-Smirnov and Shapiro-Wilk tests, while the homogeneity of the data was assessed using the Levene test. The results of these tests showed that the data exhibited normal distribution and homogeneity. For the comparison of initial and post-aging surface roughness (Ra_0/ Ra_1) values, a two-way repeated measures ANOVA was used, and Tukey's test was employed for comparisons among the Ra_1 values. One-way ANOVA was used to compare the data for color change (ΔE) and change in surface roughness (ΔRa), and Tukey's test was used to compare the differences among the groups. The statistical analysis of all data was conducted at a significance level of $p < 0.05$ with a 95% confidence interval.

Results

Changes in Surface Roughness Following Aging with FSLs and Thermal Cycling

Pre-aging (initial) and post-aging surface roughness values with standard deviations are presented in Table 2. After thermal aging and immersion in FSLs, the average surface roughness values of bulk-fill composite resins were found to be significantly different compared to the initial surface roughness values in all groups. In conventional composites, significant differences in Ra values were observed only in the groups where the CX composite resin was immersed in heptane and the AD composite resin was immersed in citric acid, while no significant differences were found in Ra values in other groups after aging.

Following thermal aging and immersion in FSLs, the BF composite resin group aged with citric acid showed the highest Ra value, while the lowest Ra value was observed in the KAL group subjected to TC. Except for the BF composite resin group, the final surface roughness values (Ra_1) were statistically similar among all composite resins. In the BF group, similar Ra_1 values were obtained after TC and heptane application, while the Ra_1 values observed after citric acid and ethanol applications were different. For the purpose of comparing the effects of TC and FSLs applications on surface roughness more accurately, surface roughness change (ΔRa) values were used for statistical analysis and comparisons were made based on these values. The ΔRa values and standard deviation values after TC and FSLs applications are displayed in Table 3, and the distribution by groups is shown in Figure 1. Considering the ΔRa values of the composite resins subjected to aging protocols, the highest ΔRa value was observed in the BF composite resin group after exposure to citric acid, while the lowest ΔRa value was found in the KAL composite resin group aged in heptane solution. The ΔRa values of bulk-fill composite resins were higher than those of conventional composites. Overall, all applications resulted in greater surface roughness change (ΔRa) in the BF composite resin compared to other composite resins. Except for the BF composite resin, all other bulk-fill composites showed similar surface roughness changes depending on the protocol applied after aging with TC and FSLs, while BF had higher ΔRa values compared to those of other bulk-fill composites. All protocols resulted in statistically similar ΔRa values in the FBF composite resin ($p=0.395$). For conventional composites, similar ΔRa values were observed after TC and ethanol immersion, while the KAL composite resin showed lower ΔRa values than other conventional composites after exposure to citric acid and heptane. Among other bulk-fill composite resins, the highest ΔRa values were obtained after ethanol application, whereas in the BF composite resin group, citric acid resulted in the highest ΔRa values.

Effects on Color Stability

The average color change (ΔE) and standard deviation values of the samples stained in coffee solution after TC and FSLs applications are presented in Table 4, and the distribution by groups is shown in Figure 2. Following immersion in coffee solution after aging with TC or FSLs, the ΔE values were significantly different depending on the aging protocol in all groups except for the FBF and XF composite resin groups ($p<0.001$). For the FBF and XF composite resin groups, the ΔE values resulting from staining solution after FSLs and TC applications showed no significant difference, indicating that they had no effect on color change ($p=0.094$ and 0.092).

The highest ΔE value was observed in the BF composite resin group after citric acid application, while the KAL composite resin group aged with TC showed the least color

change. After all applications, the highest ΔE values were observed in the BF composite resin in all groups, and the lowest ΔE values were observed in the KAL composite resin in all groups. For the BF composite resin, the ΔE values of the samples subjected to TC, heptane, and ethanol after staining were similar, while the color change of the samples treated with citric acid was statistically different. For the EB composite resin, the ΔE values of the groups exposed to TC and heptane were similar to those of the groups subjected to ethanol and citric acid. For the FZ550 composite resin, the ΔE values of the groups subjected to heptane and ethanol were comparable. For the CX composite resin, staining after heptane and citric acid treatment resulted in a similar color change which was greater than the color change caused by other applications. Compared to other conventional composites, the AD composite resin exhibited the greatest color change after exposure to staining solution following all aging protocols.

For the KAL composite resin, the ΔE values of the groups subjected to TC and heptane were similar to those of the groups treated with ethanol and citric acid. EB, FZ550, CX and KAL showed similar color changes after immersion in the staining solution following TC. Although KAL and XF composites exhibited similar color changes compared to FBF, EB, FZ550, CX following staining after immersion in citric acid, their color changes were different from each other. After all applications, BF exhibited significantly different color changes compared to other composites.

Table 2. Average surface roughness values (Ra_0/ Ra_1) and standard deviations before and after aging protocols

Composite Resins	Aging Types	Ra_0	Ra_1
Filtek Bulk Fill	Thermal Cycling	0.199 ± 0.012 ^{A}	0.222 ± 0.006 ^{B,a}
	Heptane	0.202 ± 0.008 ^{A}	0.221 ± 0.010 ^{B,a}
	Citric Acid	0.203 ± 0.012 ^{A}	0.226 ± 0.007 ^{B,a}
	Ethanol	0.201 ± 0.012 ^{A}	0.228 ± 0.009 ^{B,a}
			p=0.755
X-tra fil	Thermal Cycling	0.332 ± 0.015 ^{A}	0.351 ± 0.017 ^{B,a}
	Heptane	0.340 ± 0.014 ^{A}	0.362 ± 0.015 ^{B,a}
	Citric Acid	0.339 ± 0.016 ^{A}	0.368 ± 0.017 ^{B,a}
	Ethanol	0.336 ± 0.018 ^{A}	0.366 ± 0.019 ^{B,a}
			p=0.372
Beautifil-Bulk Restorative	Thermal Cycling	0.252 ± 0.013 ^{A}	0.314 ± 0.013 ^{B,a}
	Heptane	0.254 ± 0.013 ^{A}	0.313 ± 0.009 ^{B,a}
	Citric Acid	0.252 ± 0.011 ^{A}	0.428 ± 0.016 ^{B,b}
	Ethanol	0.254 ± 0.011 ^{A}	0.345 ± 0.010 ^{B,c}
			p=0.001
Estelite Bulk Fill Flow	Thermal Cycling	0.170 ± 0.012 ^{A}	0.190 ± 0.013 ^{B,a}
	Heptane	0.176 ± 0.013 ^{A}	0.194 ± 0.012 ^{B,a}
	Citric Acid	0.171 ± 0.011 ^{A}	0.193 ± 0.010 ^{B,a}
	Ethanol	0.180 ± 0.012 ^{A}	0.205 ± 0.012 ^{B,a}
			p=0.101
Filtek Z550	Thermal Cycling	0.178 ± 0.022 ^{A}	0.186 ± 0.022 ^{A,a}
	Heptane	0.183 ± 0.027 ^{A}	0.196 ± 0.024 ^{A,a}
	Citric Acid	0.176 ± 0.024 ^{A}	0.192 ± 0.029 ^{A,a}
	Ethanol	0.173 ± 0.022 ^{A}	0.185 ± 0.020 ^{A,a}
			p=0.400
Ceram.X SphereTEC	Thermal Cycling	0.212 ± 0.008 ^{A}	0.223 ± 0.005 ^{A,a}
	Heptane	0.208 ± 0.012 ^{A}	0.232 ± 0.015 ^{B,a}
	Citric Acid	0.220 ± 0.008 ^{A}	0.237 ± 0.011 ^{A,a}
	Ethanol	0.211 ± 0.014 ^{A}	0.227 ± 0.014 ^{A,a}
			p=0.124
Admira	Thermal Cycling	0.220 ± 0.013 ^{A}	0.231 ± 0.013 ^{A,a}
	Heptane	0.218 ± 0.012 ^{A}	0.232 ± 0.013 ^{A,a}
	Citric Acid	0.222 ± 0.019 ^{A}	0.241 ± 0.018 ^{B,a}
	Ethanol	0.217 ± 0.023 ^{A}	0.230 ± 0.024 ^{A,a}
			p=0.745
Kalore	Thermal Cycling	0.128 ± 0.012 ^{A}	0.135 ± 0.013 ^{A,a}
	Heptane	0.134 ± 0.013 ^{A}	0.139 ± 0.012 ^{A,a}
	Citric Acid	0.133 ± 0.011 ^{A}	0.141 ± 0.010 ^{A,a}
	Ethanol	0.137 ± 0.012 ^{A}	0.149 ± 0.012 ^{A,a}
			p=0.193

* Superscript uppercase letters indicate differences between Ra_0 and Ra_1 values in the same row. Superscript lowercase letters and p values indicate differences among the aging protocols in the same composite group.

SEM and AFM Analyses

Representative SEM and AFM images acquired for the purposes of observing any changes on the sample surfaces are provided in Figures 3-6. AFM analyses showed similar Sa values compared to those obtained with the profilometer. The surface topography and Sa values varied depending on the aging method and the composites used. Among the composite resins, the most irregular surface topographies were observed in the groups where BF composite resin was used. The BF composite resin stored in citric acid showed greater Sa values.

The results of SEM analysis were consistent with those of the AFM analysis. The increased susceptibility of the BF composite resin to FSLs was evident in the SEM images, where greater surface irregularities were observed. There were no significant changes on the surfaces of conventional composites.

Table 3. Average change in surface roughness values (ΔRa) and standard deviations following aging protocols

Composite Resins	Thermal Cycling	Heptane	Citric Acid	Ethanol	p values
Filtek Bulk Fill	0.023 ± 0.009 ^{A,a}	0.020 ± 0.005 ^{A,a}	0.023 ± 0.007^{A,a}	0.026 ± 0.006^{A,a}	p=0.395
X-tra fil	0.019 ± 0.005 ^{A,a}	0.022 ± 0.005^{A,ab}	0.028 ± 0.003^{A,b}	0.030 ± 0.008^{A,b}	p<0.001
Beautifil-Bulk Restorative	0.062 ± 0.002 ^{B,a}	0.060 ± 0.008^{B,a}	0.176 ± 0.005^{B,b}	0.091 ± 0.002^{B,c}	p<0.001
Estelite Bulk Fill Flow	0.020 ± 0.002 ^{A,ab}	0.018 ± 0.003^{A,a}	0.022 ± 0.002^{A,bc}	0.025 ± 0.003^{A,c}	p<0.001
Filtek Z550	0.008 ± 0.003 ^{C,a}	0.013 ± 0.007^{C,a}	0.016 ± 0.006^{C,b}	0.013 ± 0.005^{C,ab}	p<0.001
Ceram.X SphereTEC	0.011 ± 0.006 ^{C,a}	0.024 ± 0.009^{A,b}	0.017 ± 0.006^{C,ab}	0.016 ± 0.005^{C,ab}	p<0.001
Admira	0.011 ± 0.003 ^{C,a}	0.014 ± 0.005^{C,b}	0.019 ± 0.003^{C,c}	0.012 ± 0.003^{C,ab}	p<0.001
Kalore	0.007 ± 0.002 ^{C,ab}	0.005 ± 0.002^{D,a}	0.007 ± 0.002^{D,b}	0.011 ± 0.002^{C,b}	p<0.001
	p<0.001	p<0.001	p<0.001	p<0.001	

* Superscript uppercase letters indicate differences between the rows in the same column (composite resins). Superscript lowercase letters indicate differences between the columns in the same row (aging protocols).

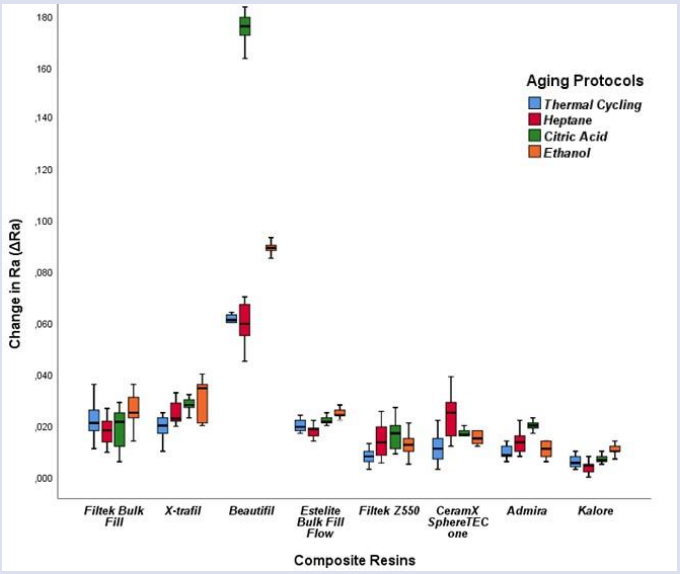

Figure 1: Comparison of change in surface roughness values (ΔRa) among composite resins after different aging protocols.

Table 4. Average color change (ΔE_{1-0}) values and standard deviations after immersion in staining solution, presented by aging protocols

Composite Resins	Thermal Cycling	Heptane	Citric Acid	Ethanol	p values
Filtek Bulk Fill	1.989 ± 0.318 ^{A,a}	2.072 ± 0.082 ^{ADE,a}	1.939 ± 0.167 ^{AB,a}	2.155 ± 0.147 ^{AB,a}	p=0.094
X-tra fil	2.328 ± 0.234 ^{B,a}	2.199 ± 0.084 ^{AF,a}	2.179 ± 0.183 ^{B,a}	2.333 ± 0.146 ^{A,a}	p=0.092
Beautifil-Bulk Restorative	3.643 ± 0.243 ^{C,a}	3.950 ± 0.273 ^{C,a}	4.548 ± 0.313 ^{C,b}	3.863 ± 0.206 ^{C,a}	p<0.001
Estelite Bulk Fill Flow	1.590 ± 0.216 ^{D,a}	1.729 ± 0.155 ^{DG,a}	2.141 ± 0.124 ^{AB,b}	1.971 ± 0.170 ^{BD,b}	p<0.001
Filtek Z550	1.625 ± 0.058 ^{D,a}	1.887 ± 0.199 ^{DE,b}	2.161 ± 0.097 ^{AB,c}	1.845 ± 0.097 ^{D,b}	p<0.001
Ceram.X SphereTEC	1.593 ± 0.067 ^{D,a}	2.074 ± 0.133 ^{E,b}	2.162 ± 0.099 ^{AB,b}	1.862 ± 0.094 ^{D,c}	p<0.001
Admira	2.838 ± 0.128 ^{E,a}	2.365 ± 0.155 ^{F,b}	2.509 ± 0.200 ^{C,b}	2.830 ± 0.213 ^{E,a}	p<0.001
Kalore	1.390 ± 0.216 ^{D,a}	1.529 ± 0.159 ^{G,a}	1.921 ± 0.125 ^{A,b}	1.771 ± 0.170 ^{D,b}	p<0.001
	p<0.001	p<0.001	p<0.001	p<0.001	

* Superscript uppercase letters refer to the differences between the rows in the same column (composite resins). Superscript lowercase letters refer to the differences between the columns in the same row (protocols).

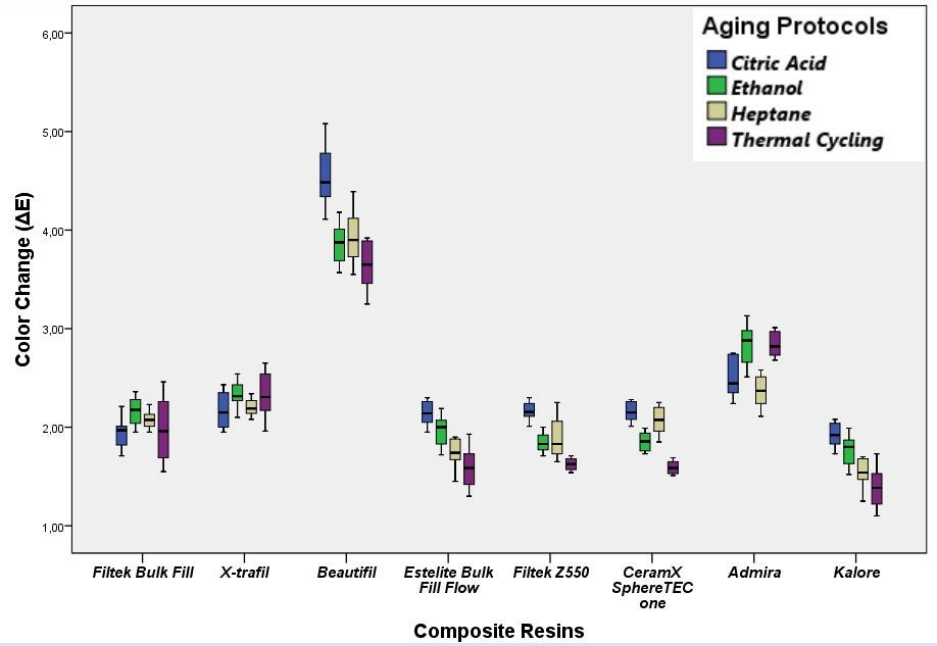

Figure 2: Comparison of color change (ΔE) among composite resins after different aging protocols

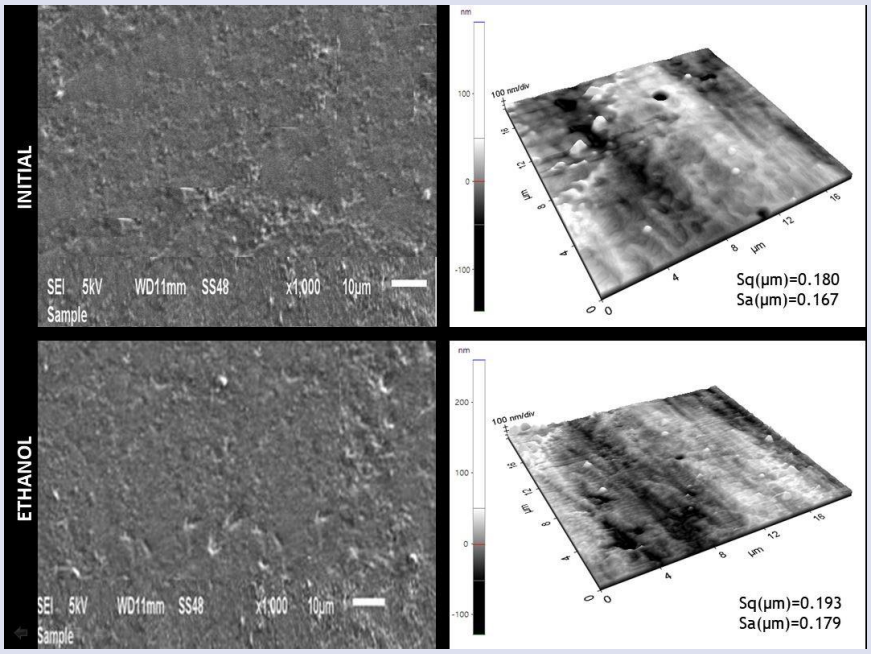

Figure 3: SEM and AFM images of FZ550 composite resin initial and after immersion in ethanol solution.

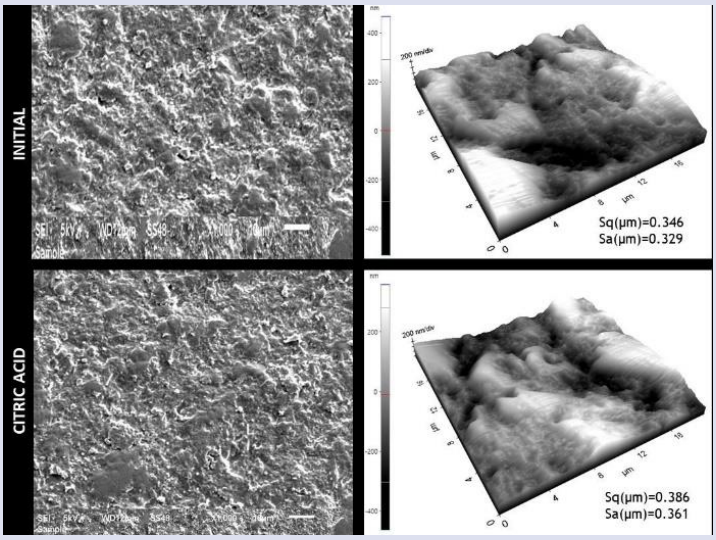

Figure 4: SEM and AFM images of XF composite resin initial and after immersion in citric acid.

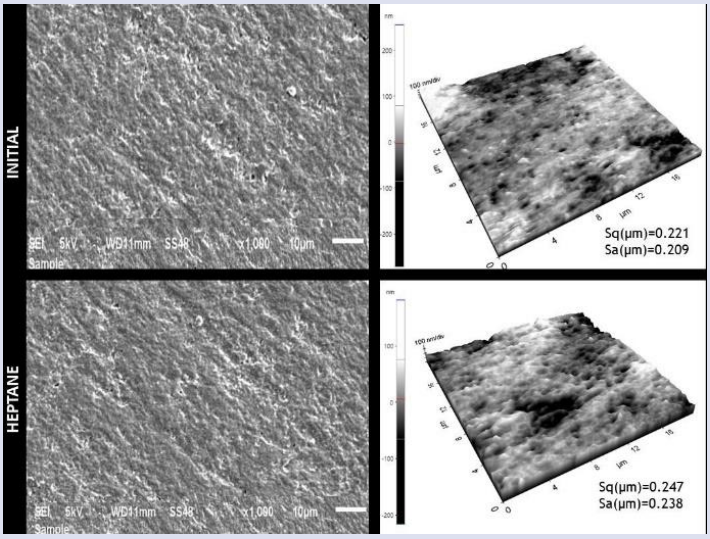

Figure 5: SEM and AFM images of AD composite resin initial and after immersion in heptane.

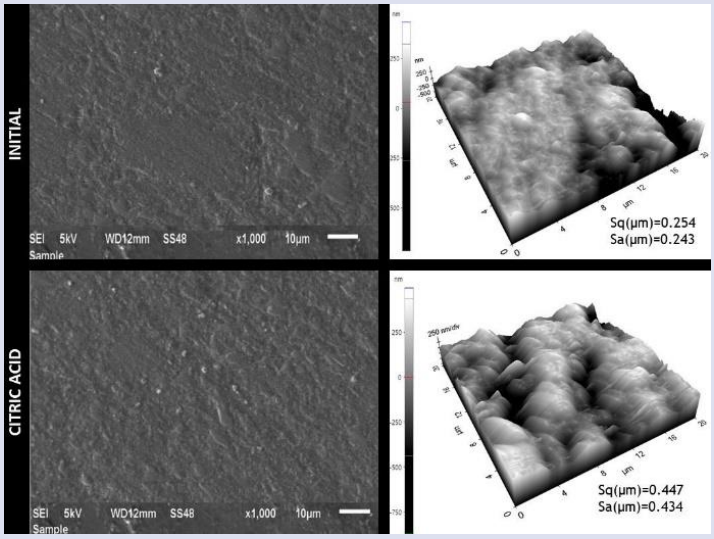

Figure 6: SEM and AFM images of BF composite resin initial and after immersion in citric acid.

Discussion

In this study comparatively evaluating the effects of thermal cycling and immersion in food-simulating liquids on the surface properties and color stability of bulk-fill and conventional composite resins, the protocols applied affected surface roughness and color stability of the bulk-fill composite resins, while for conventional composites, surface roughness was unchanged in all groups except for two groups. Moreover, changes in surface roughness varied depending on the protocol and composite used. The color stability of the stained composites also differed among the composite groups based on the protocol applied after thermal cycling and immersion in food-simulating liquids. Therefore, both null hypotheses were rejected.

Although improvements have been achieved in the materials used for dental restorations in modern dental practice, the longevity of dental composites in the oral environment remains a concern for clinicians. Dental composites can undergo changes over time in the oral cavity, such as discoloration, water absorption, dissolution, microleakage, increased roughness, and wear. Such changes are influenced by the type and ratio of fillers, as well as the content and monomers that make up the resin matrix.[25] Studies have reported that FSLs and TC which are commonly used to simulate the oral environment, lead to degradation, monomer release, dissolution, increased surface roughness, discoloration, reduced hardness, and accelerated aging of composite resins.[25, 26] These alterations have been attributed to the deterioration of the polymer matrix of the composite and the interface between resin and filler, as well as the loss of inorganic filler particles.[27, 28] In this study, the FSLs used, significantly increased surface roughness and discoloration particularly in bulk-fill composites, with variations observed among different composite resins. This discrepancy could stem from the structural differences among composite resins, as mentioned earlier.

The use of bulk-fill composites by clinicians is rapidly increasing due to the advantages they offer. Changes in the composition of bulk-fill composites have been designed to enhance polymerization depth compared to conventional composites. One of these changes includes increasing the size of filler particles. In bulk-fill composites, the aim of increasing the filler particle size is to enhance translucency to enable single-layer polymerization. The size, shape, and quantity of the filler particles and their relationship with the resin matrix are the key determinants of surface morphology.[29] The increase in filler particle size in bulk-fill composites leads to an increase in surface roughness, which also results in increased spaces between fillers, i.e., the resin matrix.[30, 31] Furthermore, irregularities resulting from the detachment of small particles from the surface are less pronounced. In our study, the greater increase in surface roughness and discoloration after FSLs and TC observed in the bulk-fill composites, especially those with larger particle sizes, compared to conventional composites might be attributed to the larger particle size and the greater ratio of resin matrix between the particles.

Giomers are a new generation of glass ionomer hybrid materials that release fluoride due to the surface pre-reacted glass ionomer (SPRG) particles embedded in their resin matrix. In our study, the highest ΔRa and ΔE values were observed in the BF group following aging protocols. Similar findings have been reported in other studies, where the greatest increase in surface roughness after FSL was found in giomer-containing materials, and particularly the high ΔRa and ΔE values observed in the groups exposed to citric acid were attributed to the potential degradation of SPRG particles in the presence of citric acid.[32,33] Looking at other composites, different aging protocols led to similar ΔRa values, and the variation in BF composite depending on the applied protocols supports the potential sensitivity of SPRG to citric acid. While there is no specific data on particle size for BF composite in the literature, it is believed that the void resulting from the degradation of SPRG particles might contribute to increased surface roughness and discoloration.[33] In a study by Cabadag and Gonulol, similar to our study findings, the surface roughness values of the BF increased compared to the initial values, but contrastingly, no significant difference was observed between the initial and final surface measurements of other bulk-fill composites; this discrepancy might be explained by variations in the duration of FSLs exposure in our study.[32]

Deterioration and discoloration of the composite restorations over time still represent their major disadvantages. Especially in today's world, where patients have higher aesthetic expectations, discoloration is one of the main factors affecting the replacement of composite restorations. The color stability of composite resins depends on various factors such as the resin matrix structure, water absorption, filler particle structure and size, and the matrix-filler relationship.[34] In our study, the higher ΔE values observed in bulk-fill composites indicating discoloration could be attributed to these factors. Additionally, the higher ΔE value of the AD composite compared to other conventional composite resins might be due to the presence of different types of fillers and their bonding to the resin matrix.[35] The better color stability of the KAL composite resin compared to other composites could be related to the hydrophilic properties of the UDMA and DX-511 monomers in its content, resulting in lower water absorption compared to Bis-GMA[36]. Degradation occurring at the filler particle-resin matrix interface and increased surface roughness due to mechanical and chemical deterioration can enhance discoloration.[33] It has been reported that the solutions used as FSLs, especially ethanol, reduce the surface hardness of composite resins and induce degradation at the filler particle-resin matrix interface, leading to the formation of microcracks.[7] In a study, the average ΔE value for the participants to notice color

changes was reported to be 1.8.[37] In our study, except for a few groups, color changes exceeding this value were observed in most groups.

As with any in vitro study, this study has a number of limitations. Restorations within the oral cavity are subjected to various conditions such as different temperatures of food and beverages, chewing forces, other mechanical factors, and the erosive effects of organic acids produced by bacteria. The inability to replicate combinations of these conditions in vitro is a limitation of this study. With advances in the characteristics and content of restorative materials, further research would be needed to fully assess the physical and chemical stabilization of these materials under various conditions.

Conclusions

Based on the data obtained from this study, it can be concluded that the food-simulating liquids and thermal aging have an impact on the surface roughness and color stability of the bulk-fill composites, while the surface roughness of conventional composites is less affected compared to bulk-fill composites. The findings of this study also suggest that the protocol applied and the composite structure affect the extent of surface roughness and color change. Moreover, the content of the products that people take with the diet is effective on the physical and chemical properties of the restorative materials used.

Acknowledgements

This study is supported by the Scientific Research Project Fund of Sivas Cumhuriyet University under the project number Diş-222.

Conflicts of Interest Statement

The authors declare that there is no conflict of interest regarding the publication of this article.

References

1. Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G. Progress in dimethacrylate-based dental composite technology and curing efficiency. Dent Mater. 2013;29(2):139-156.
2. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, Leloup G. Physico-mechanical characteristics of commercially available bulk-fill composites. J Dent. 2014;42(8):993-1000.
3. Abbas G, Fleming GJ, Harrington E, Shortall AC, Burke FJ. Cuspal movement and microleakage in premolar teeth restored with a packable composite cured in bulk or in increments. J Dent. 2003;31(6):437-444.
4. Benetti AR, Havndrup-Pedersen C, Honore D, Pedersen MK, Pallesen U. Bulk-fill resin composites: polymerization contraction, depth of cure, and gap formation. Oper Dent. 2015;40(2):190-200.
5. Ilie N, Bucuta S, Draenert M. Bulk-fill Resin-based Composites: An In Vitro Assessment of Their Mechanical Performance. Operative Dentistry. 2013;38(6):618-625.
6. El Gezawi M, Kaisarly D, Al-Saleh H, ArRejaie A, Al-Harbi F, Kunzelmann KH. Degradation Potential of Bulk Versus Incrementally Applied and Indirect Composites: Color, Microhardness, and Surface Deterioration. Oper Dent. 2016;41(6):e195-e208.
7. Yap AU, Tan SH, Wee SS, Lee CW, Lim EL, Zeng KY. Chemical degradation of composite restoratives. J Oral Rehabil. 2001;28(11):1015-1021.
8. Heintze SD, Zappini G, Rousson V. Wear of ten dental restorative materials in five wear simulators--results of a round robin test. Dent Mater. 2005;21(4):304-317.
9. Wongkhantee S, Patanapiradej V, Maneenut C, Tantbirojn D. Effect of acidic food and drinks on surface hardness of enamel, dentine, and tooth-coloured filling materials. J Dent. 2006;34(3):214-220.
10. Yap AU, Lim LY, Yang TY, Ali A, Chung SM. Influence of dietary solvents on strength of nanofill and ormocer composites. Oper Dent. 2005;30(1):129-133.
11. Glauser S, Astasov-Frauenhoffer M, Muller JA, Fischer J, Waltimo T, Rohr N. Bacterial colonization of resin composite cements: influence of material composition and surface roughness. European journal of oral sciences. 2017;125(4):294-302.
12. Food and Drug Administration. FDA guidelines for chemistry and technology requirements of indirect additive petitions. Washington, 1976.
13. Egilmez F, Ergun G, Cekic-Nagas I, Vallittu PK, Lassila LVJ. Does artificial aging affect mechanical properties of CAD/CAM composite materials. Journal of Prosthodontic Research. 2018;62(1):65-74.
14. Eweis AH, Yap AU, Yahya NA. Dynamic analysis of bulk-fill composites: Effect of food-simulating liquids. J Mech Behav Biomed Mater. 2017;74:183-188.
15. Yap A, Lee M, Chung S, Tsai K, Lim C. Effect of food-simulating liquids on the shear punch strength of composite and polyacid-modified composite restoratives. Operative dentistry. 2002;28(5):529-534.
16. Vouvoudi EC, Sideridou ID. Dynamic mechanical properties of dental nanofilled light-cured resin composites: Effect of food-simulating liquids. J Mech Behav Biomed Mater. 2012;10:87-96.
17. Vouvoudi EC, Sideridou ID. Effect of food/oral-simulating liquids on dynamic mechanical thermal properties of dental nanohybrid light-cured resin composites. Dent Mater. 2013;29(8):842-850.
18. Silva TMD, Sales A, Pucci CR, Borges AB, Torres CRG. The combined effect of food-simulating solutions, brushing and staining on color stability of composite resins. Acta Biomater Odontol Scand. 2017;3(1):1-7.
19. Pala K, Tekce N, Tuncer S, Serim ME, Demirci M. Evaluation of the surface hardness, roughness, gloss and color of composites after different finishing/polishing treatments and thermocycling using a multitechnique approach. Dent Mater J. 2016;35(2):278-289.
20. Kakaboura A, Fragouli M, Rahiotis C, Silikas N. Evaluation of surface characteristics of dental composites using profilometry, scanning electron, atomic force microscopy and gloss-meter. Journal of materials science Materials in medicine. 2007;18(1):155-163.
21. Lainovic T, Vilotic M, Blazic L, Kakas D, Markovic D, Ivanisevic A. Determination of surface roughness and topography of dental resin-based nanocomposites using AFM analysis. Bosnian journal of basic medical sciences. 2013;13(1):34-43.

22. Cabadag OG, Gonulol N. The Effects of Food-Simulating Liquids on Surface Roughness, Surface Hardness, and Solubility of Bulk-Fill Composites. Journal of Advanced Oral Research. 2021;12(2):245-253.

23. Tanthanuch S, Kukiattrakoon B, Eiam-O-Pas K, Pokawattana K, Pamanee N, Thongkamkaew W, et al. Surface changes of various bulk-fill resin-based composites after exposure to different food-simulating liquid and beverages. Journal of Esthetic and Restorative Dentistry. 2018;30(2):126-35.

24. Ertas E, Guler AU, Yucel AC, Koprulu H, Guler E. Color stability of resin composites after immersion in different drinks. Dent Mater J. 2006;25(2):371-376.

25. Kumari CM, Bhat KM, Bansal R, Singh N, Anupama A, Lavanya T. Evaluation of Surface Roughness and Hardness of Newer Nanoposterior Composite Resins after Immersion in Food-Simulating Liquids. Contemp Clin Dent. 2019;10(2):289-293.

26. Peutzfeldt A. Resin composites in dentistry: the monomer systems. Eur J Oral Sci. 1997;105(2):97-116.

27. Yap AU, Wattanapayungkul P, Chung SM. Influence of the polymerization process on composite resistance to chemical degradation by food-simulating liquids. Oper Dent. 2003;28(6):723-727.

28. Larsen IB, Munksgaard EC. Effect of human saliva on surface degradation of composite resins. Scand J Dent Res. 1991;99(3):254-261.

29. Blackham JT, Vandewalle KS, Lien W. Properties of hybrid resin composite systems containing prepolymerized filler particles. Operative dentistry. 2009;34(6):697-702.

30. de Fatima Alves da Costa G, Melo A, de Assuncao IV, Borges BCD. Impact of additional polishing method on physical, micromorphological, and microtopographical properties of conventional composites and bulk fill. Microsc Res Tech. 2020;83(3):211-222.

31. Can Say E, Yurdaguven H, Yaman BC, Ozer F. Surface roughness and morphology of resin composites polished with two-step polishing systems. Dental materials journal. 2014;33(3):332-342.

32. Kooi TJ, Tan QZ, Yap AU, Guo W, Tay KJ, Soh MS. Effects of food-simulating liquids on surface properties of giomer restoratives. Oper Dent. 2012;37(6):665-71.

33. Cabadag˘ ÖG, Gönülol N. The Effects of Food-Simulating Liquids on Surface Roughness, Surface Hardness, and Solubility of Bulk-Fill Composites. 2021;12(2):245-53.

34. Güler AU, Güler E, Yücel AC, Ertaş E. Effects of polishing procedures on color stability of composite resins. J Appl Oral Sci. 2009;17(2):108-112.

35. Llena C, Fernández S, Forner L. Color stability of nanohybrid resin-based composites, ormocers and compomers. Clin Oral Investig. 2017;21(4):1071-1077.

36. Sideridou I, Tserki V, Papanastasiou G. Study of water sorption, solubility and modulus of elasticity of light-cured dimethacrylate-based dental resins. Biomaterials. 2003;24(4):655-65.

37. Thoma DS, Ioannidis A, Fehmer V, Michelotti G, Jung RE, Sailer I. Threshold Values for the Perception of Color Changes in Human Teeth. Int J Periodontics Restorative Dent. 2016;36(6):777-783.