

# Effect of Different Surface Coating Applications on The Surface Roughness and Color Stability of Resin-Based Composites: An SEM Study

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## Farklı Yüzey Örtücü Uygulamalarının Rezin Bazlı Kompozitlerin Yüzey Pürüzlülüğü ve Renk Stabilitesine Etkisi: SEM Çalışması

### ABSTRACT

**Objective:** To investigate color change and surface roughness of resin-based composites treated with a surface coating agent.

**Methods:** Forty specimens were prepared from each resin [G-ænial Posterior (microhybrid composite/GP) and SDR flow\*(bulk-fill composite/SDR)]. Following baseline color and surface roughness (Ra) measurements, the specimens were randomly divided into 4 groups (n=10) according to surface coating agents [(Permaseal, Biscover LV, Prebond SE, and control group)]. Following the application procedures, color and roughness measurements were repeated. The specimens were discolored for 144 hours in a coffee solution, renewing daily. Final measurements were performed. Color change values ( $\Delta E_{00}$ ) and Ra were calculated. Surface topography was determined using scanning electron microscopy. Two-way analyses of variance, Tukey's post-hoc test and Student t-test were performed, with a  $p<0.05$  regarded as indicative of significance.

**Results:** SDR showed more color change and surface roughness than GP and both materials presented unacceptable (AT>1.8) and perceptible (PT>0.8) discoloration. The highest discoloration was observed for Permaseal and Prebond SE in terms of  $\Delta E_2$  and  $\Delta E_3$ . GP-Biscover LV, SDR-Prebond SE combinations showed the lowest and clinically acceptable (AT<1.8)  $\Delta E$  values. There were no significant differences between surface coating agents in terms of surface roughness ( $p>0.05$ ). GP-Permaseal and SDR-Prebond SE combinations were exhibited less surface roughness.

**Conclusion:** A bulk-fill composite is more prone to discoloration than a microhybrid composite. At each period, the bulk-fill composite exhibited greater surface roughness than the microhybrid composite. Biscover LV showed more acceptable results in terms of color stability and roughness than other surface coating agents (Permaseal and Prebond SE).

**Keywords:** Sealant, Permaseal, Biscover LV, Prebond, Discoloration, Roughness

### ÖZ

**Amaç:** Yüzey örtücü ajanı ile muamele edilmiş rezin esaslı kompozitlerin renk değişimi ve yüzey pürüzlülüğünü araştırmak.

**Yöntemler:** Her rezin materyalinden [G-ænial Posterior (mikrohibrit kompozit/GP) and SDR flow\*(bulk-fill kompozit/SDR)] kırk numune hazırlandı. Başlangıç renk ve yüzey pürüzlülüğü (Ra) ölçümlerinin ardından, numuneler yüzey örtücü ajanlarına [(Permaseal, Biscover LV ve Prebond SE) ve kontrol grubu distile su olacak şekilde rastgele 4 gruba (n=10) ayrıldı. Uygulama işlemlerinin ardından renk ve pürüzlülük ölçümleri tekrarlandı. Numuneler, günlük olarak yenilenen kahve solüsyonunda 144 saat boyunca renklendirildi. Son ölçümler yapıldı. Renk değişim değerleri ( $\Delta E_{00}$ ) ve Ra hesaplandı. Yüzey topografisi, taramalı elektron mikroskobu kullanılarak belirlendi. İki yönlü varyans analizleri, Tukey post hoc testi ve Student-t testi yapıldı ve  $P<0,05$  anlamlılık göstergesi olarak kabul edildi.

**Bulgular:** SDR, GP'den daha fazla renk değişimi ve yüzey pürüzlülüğü gösterdi ve her iki materyal de kabul edilemez (AT>1,8) ve algılanabilir (PT>0,8) renk değişikliği gösterdi.  $\Delta E_2$  ve  $\Delta E_3$  açısından en yüksek renk değişimi Permaseal ve Prebond SE için gözlemlendi. GP-Biscover LV ve SDR-Prebond SE kombinasyonları en düşük ve klinik olarak kabul edilebilir (AT<1,8)  $\Delta E$  değerlerini gösterdi. Yüzey pürüzlülüğü açısından yüzey örtücü ajanları arasında anlamlı fark yoktu ( $p>0,05$ ). GP-Permaseal ve SDR-Prebond SE kombinasyonları daha az yüzey pürüzlülüğü sergiledi.



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**Sonuç:** Bulk-fil rezin kompoziti, mikro-hibrit kompozite göre renk bozulmasına daha yatkındır. Her periyotta, Bulk-fil kompozit, mikro-hibrit kompozitten daha fazla yüzey pürüzlülüğü sergiledi. Biscover LV, hem renk stabilitesi hem de pürüzlülük açısından diğer yüzey örtücü ajanlarına (Permaseal ve Prebond SE) göre daha kabul edilebilir sonuçlar gösterdi.

**Anahtar Kelimeler:** Örtücü, Permaseal, Biscover LV, Prebond, Renk değişikliği, Pürüzlülük

## INTRODUCTION

Color and surface properties of resin-based composites (RBCs) are important factors affecting the long-term prognosis of restorations.<sup>1</sup> Additionally, resin composites' organic structure and filler particle ratio directly affect the surface's roughness and propensity for external discoloration. Rough restoration surfaces can cause plaque accumulation, gingival irritation, and secondary caries formation. These surfaces are easily discolored by the absorption and adsorption of oral fluids.<sup>2</sup>

Another significant issue with RBCs materials is the formation of micro-gaps.<sup>3</sup> Factors such as polymerization shrinkage, characteristics of the restorative material, finishing and polishing processes, morphological and histological structure of enamel and dentin, application method of composite resin, number of bonded surfaces, the position of the cavity and occlusion can cause micro-gap formation.<sup>4</sup> Contrary to conventional incremental 2mm material thickness, bulk-fill composites enable insertion in single-layer thicknesses of 4-6mm, reducing the number of clinical steps and internal/external marginal gap development.<sup>5</sup>

Surface coating agents (surface sealants, composite glaze materials, bonding adhesive agents) have been developed to cover micro-porosities on the restoration surfaces, increase marginal integrity and abrasion resistance, and ensure color stability by preventing the absorption of pigments.<sup>6</sup> These materials, with their low viscosity and high wettability, can penetrate through micro-cracks and form a shiny, slippery surface on the restoration.<sup>7</sup> Manufacturers aim to eliminate the oxygen inhibition layer on the composite surface and to reduce plaque formation and staining with the clinical applications of these agents.<sup>7</sup> Although not specified in the manufacturer's recommendations, in clinical practice, dentists commonly use bonding agents in the finishing process to provide smoother restoration surfaces.<sup>8</sup> However, these materials contain hydrophilic monomers and solvents that can damage some properties of RBCs (such as discoloration).<sup>9</sup>

The effects of surface sealant applications on RBCs have been evaluated in many studies.<sup>4,10</sup> Nevertheless, there is a lack of information about the effect of using bonding adhesive and surface sealant materials on the color stability and surface structure of RBCs restorations. The aim of the present study is to evaluate the color change and surface roughness of RBCs surface covered with different surface coating materials. The definitions of the study's null hypotheses were as follows: (1) application of surface coating agents does not cause color change on RBCs, (2) surface sealants or bonding adhesive agents do not have an effect on the surface roughness of RBCs.

## METHODS

A microfilled hybrid (mFR) resin composite [G-ænial Posterior (GP)], and a bulk-fill resin composite [SDR flow<sup>+</sup> (SDR)] were evaluated in this study. And the properties of these materials are given in Table 1.

### Specimen preparation

Figure 1 is a simplified representation of study design. The sample size was calculated with the G\*Power program (version 3.1.9.4, Heinrich Heine, University of Düsseldorf, Düsseldorf, Germany). A supposed

significance level of 0.05 and an effect size of 0.25 was applied and a total of 80 specimens were prepared to obtain 10 specimens at the final subgroups of each material.

Teflon molds in size of 6\*2 mm were used to prepare specimens for each resin composite (GP and SDR). A mylar strip band and a glass plate were used to obtain smooth surfaces on the specimens. All the specimens were polymerized using a LED (D-Light Pro, GC, Japan) with irradiation of 1200 mW/cm<sup>2</sup> for 20 sec. A polishing system (Super-Snap Rainbow Technique Kit, Shofu Inc., Kyoto, Japan) was applied to a single surface of the samples in each group. Then the specimens were post-polymerized in distilled water at 37°C for 24 hours.

### Color measurement

A digital spectrophotometer (Vita Easyshade V, Vita Zahnfabrik, Bad Säckingen, Germany) was used to measure baseline colors (T<sub>0</sub>) of the specimens. The device's probe was positioned in the center of the specimens, which are on a white surface without reflection. "L, C, and H" values were averaged after being measured three times for each specimen. The spectrophotometer was re-calibrated in accordance with the manufacturer's instructions after every nine measurements.

### Surface roughness measurement

Initial surface roughness (Ra<sub>0</sub>) measurements (T<sub>0</sub>) were determined with a mechanical contact profilometer (Mitutoyo, SurfTest SJ-410, Japan) with a measuring distance of 4 mm and a cut-off value of 0.8 mm. Before each measurement, calibration of the profilometer was performed using a reference block with a Ra value of 3.05 µm. Three measurements were obtained at the center of each specimen surface were averaged and Ra<sub>0</sub> values were recorded in µm.

### Surface coating agents' applications

Following the baseline color and surface roughness measurements, the specimens were randomly divided into four groups (n=10): Permaseal, Biscover LV, Prebond SE, and control group. The three surface coating agents (Table 1) were applied to the polished surfaces in accordance with the manufacturer's recommendations.

Following the application of the surface coating materials, color, and surface roughness (Ra<sub>1</sub>) measurements (T<sub>1</sub>) were repeated.

### Staining procedure

The specimens were immersed in a 20 ml diffuse coffee solution prepared using 5 g of coffee (Nescafe Gold, Nestle, Istanbul, Turkey) and 300 ml of boiling water for a total of 144 hours. The staining beverage was re-prepared daily. Following this process, the specimens were rinsed with water for 10 s and gently dried. Distilled water was preferred as a control group to appreciate the intrinsic changes.

When the immersion period was completed, the final color and surface roughness (Ra<sub>2</sub>) measurements (T<sub>2</sub>) were performed. To evaluate the color differences between the baseline and final measurements after staining,  $\Delta E_{00}(T_1, T_0)$  values were calculated via the following formulation:

$$\Delta E_{00} = \left[ \left( \frac{\Delta L}{k_L S_L} \right)^2 + \left( \frac{\Delta C}{k_C S_C} \right)^2 + \left( \frac{\Delta H}{k_H S_H} \right)^2 + R_T \left( \frac{\Delta C}{k_C S_C} \right) \left( \frac{\Delta H}{k_H S_H} \right) \right]^{1/2}$$

In this study, CIEDE2000 (1:1:1) formula was used. The color changes were analyzed based on an 'Acceptability Threshold' (AT) of 50:50% (AT: $\Delta E_{00}=1.8$ ) and a 'Perceptibility Threshold' (PT) of 50:50% (PT: $\Delta E_{00}=0.8$ ) for all the resin-based materials.<sup>11</sup>

**Table 1.** Chemical composition of restorative materials and surface coating agents used in the study

Resin-based composites							
Product	Lot Number	Manufacturer	Shade	Classification	Composition		
					Monomer Composition	Filler Type	Filler Amount (wt%/vol %)
G-ænial Posterior (GP)	1709223	Kuraray Noritake Dental Inc., Okayama, Japan	A3	Microfilled hybrid (mFR)	Bis-GMA, TEGDMA, and hydrophobic aromatic dimethacrylate	Glass ceramics, surface-treated alumina micro filler, silica, particle size 6 µm	92/82
SDR™ Flow* (SDR)	1903000872	Dentsply, Konstanz, Germany	U	Bulk-Fill flowable composite	Modified UDMA, ethoxylated bisphenol-A-dimethacrylate (EBPADMA), TEGDMA, butylated hydroxytoluene (BHT), UV stabiliser, titanium dioxide and iron oxides, camphoroquinone.	Ba-Al-F-B-Si glass and St-Al-F-Si-glass, particle size 10 µm	70.5/47.4
Surface coating agents							
Product	Lot Number	Manufacturer	Composition		Application procedure		
Permaseal	BM6TJ	Ultradent Products, UT, USA	Bis-GMA 60%, TEGDMA 40%, 1-dimethylaminoethyl metacrylate <3%		37% phosphoric acid (Panora 200 Phosphoric Acid, Imicryl, Konya, Turkey) was applied to the resin composite specimens for 20 seconds. The specimens were washed for 15 seconds and dried for 10 seconds. Then, a thin layer of PermaSeal surface sealant was applied to the specimen surfaces for 5 seconds with the help of the brush included in the package. After 5 seconds of gentle airflow, polymerization was achieved for 30 seconds.		
Biscover LV	2200001852	Bisco, IL, USA	Dipentaerythritol penta-acrylateesters and ethanol		37% phosphoric acid (Panora 200 Phosphoric Acid, Imicryl, Konya, Turkey) was applied on the resin composite samples for 15 seconds. The specimens were washed for 15 seconds using an air-water syringe and dried for 10 seconds. A thin layer of Biscover LV was then applied to the specimen surfaces using disposable adhesive application brushes. 15 seconds was waited without applying air to remove the solvents and then polymerization was achieved for 30 seconds.		
Prebond SE	216644	President Dental, München, Germany	Funtional MDP monomer, methacrylate, photoinitiators, ethanol, water		The bottle was shaken well before use. A thin layer of Prebond SE was then applied to the specimen surfaces using disposable adhesive application brushes for 20 seconds. The specimens were dried with gentle airflow and then polymerized for 20 seconds.		

**Abbreviations:** Bis-GMA = bisphenol-glycidyl methacrylate; TEGDMA = triethyleneglycol dimethacrylate; UDMA = urethane dimethacrylate; MDP = methacryloyloxydecyl dihydrogen phosphate

**Table 2.** The mean  $\Delta E_{00}$  values  $\pm$  standard deviations after treatment with surface coating agents and immersion in coffee

Materials	Surface coating agents				Results	Total
	Control (Distilled water)	Permaseal	Biscover LV	Prebond SE		
<b><math>\Delta E_1</math> (T1-T0)</b>						
GP	1.00 $\pm$ 0.55 <sup>B,b</sup>	1.12 $\pm$ 0.57 <sup>b</sup>	0.65 $\pm$ 0.12 <sup>B,b</sup>	1.96 $\pm$ 0.72 <sup>A,a</sup>	$p^a=0.000$	1.18 $\pm$ 0.71
SDR	0.51 $\pm$ 0.39 <sup>A,b</sup>	1.50 $\pm$ 0.55 <sup>a</sup>	1.12 $\pm$ 0.43 <sup>A,a,c</sup>	0.88 $\pm$ 0.57 <sup>B,b,c</sup>	$p^a=0.001$	1.0 $\pm$ 0.59
	$p^b=0.038$	$p^b=0.152$	$p^b=0.004$	$p^b=0.002$		$p^b=0.230$
Total	0.75 $\pm$ 0.53 <sup>z</sup>	1.31 $\pm$ 0.58 <sup>x,y</sup>	0.88 $\pm$ 0.39 <sup>y,z</sup>	1.42 $\pm$ 0.84 <sup>x</sup>	$p^a=0.001$	
<b><math>\Delta E_2</math> (T2-T0)</b>						
GP	5.14 $\pm$ 1.06 <sup>A,a,b</sup>	6.12 $\pm$ 1.22 <sup>B,a</sup>	4.24 $\pm$ 0.97 <sup>b</sup>	5.83 $\pm$ 0.6 <sup>B,a</sup>	$p^a=0.001$	5.33 $\pm$ 1.20
SDR	3.83 $\pm$ 0.86 <sup>B,b</sup>	9.38 $\pm$ 3.38 <sup>A,a</sup>	6.46 $\pm$ 3.27 <sup>a,b</sup>	8.94 $\pm$ 2.25 <sup>A,a</sup>	$p^a=0.000$	7.15 $\pm$ 3.39
	$p^b=0.007$	$p^b=0.01$	$p^b=0.055$	$p^b<0.001$		$p^b=0.000$
Total	4.48 $\pm$ 1.16 <sup>y</sup>	7.75 $\pm$ 2.99 <sup>z</sup>	5.35 $\pm$ 2.61 <sup>y</sup>	7.38 $\pm$ 2.26 <sup>z</sup>	$p^a=0.000$	
<b><math>\Delta E_3</math> (T2-T1)</b>						
GP	5.08 $\pm$ 0.99 <sup>A,a</sup>	5.99 $\pm$ 1.40 <sup>B,a</sup>	3.69 $\pm$ 0.96 <sup>B,b</sup>	6.11 $\pm$ 0.84 <sup>B,a</sup>	$p^a=0.000$	5.22 $\pm$ 1.42
SDR	3.72 $\pm$ 0.74 <sup>B,b</sup>	9.72 $\pm$ 3.39 <sup>A,a</sup>	6.82 $\pm$ 3.62 <sup>A,a,b</sup>	8.49 $\pm$ 2.66 <sup>A,a</sup>	$p^a=0.000$	7.19 $\pm$ 3.55
	$p^b=0.003$	$p^b=0.005$	$p^b=0.016$	$p^b=0.015$		$p^b=0.000$
Total	4.40 $\pm$ 0.1.10 <sup>z</sup>	7.85 $\pm$ 3.17 <sup>x</sup>	5.26 $\pm$ 3.04 <sup>y,z</sup>	7.30 $\pm$ 2.27 <sup>x,y</sup>	$p^a=0.000$	

$\Delta E_1$ : Color change between sealant application and baseline;  $\Delta E_2$ : Color change between discoloration and baseline;  
 $\Delta E_3$ : Color change between discoloration and sealant application. Lower letters indicate the difference between lines, capital letters indicate the differences between rows.  
AT: $\Delta E_{00}$ =1.8 and PT: $\Delta E_{00}$ =0.8  
GP: G-ænial posterior, SDR: SDR™ Flow\*.  
 $p^a$  values are based on One-way ANOVA test,  $p^b$  values are based on The Student t-test, \* $p<0,05$  is significant.

**Table 3.** Comparison of mean surface roughness (Ra) values for material groups and tested surface coating agents

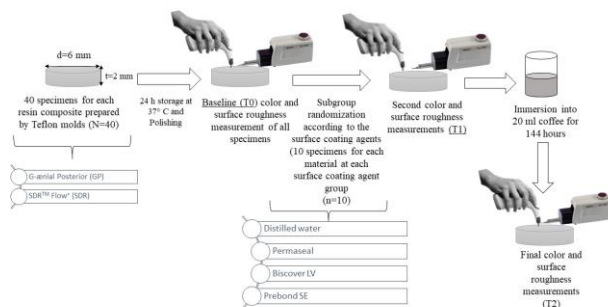
Materials	Surface coating agents				Results	Total
	Distilled water	Permaseal	Biscover LV	Prebond SE		
<b>G- aenial posterior</b>						
Ra0	1.99 ±1.21	2.23 ±1.09	1.17 ±0.60	1.79 ±0.1	p <sup>a</sup> =0.120	1.79±1.04
Ra1	1.91 ±1.22	1.19 ±1.03	2.31 ±1.55	1.72 ±1.10	p <sup>a</sup> =0.258	1.78±1.26
Ra2	2.51 ±1.79	1.28 ±0.82	1.90 ±1.22	1.71 ±0.86	p <sup>a</sup> =0.472	1.85±1.74
	p <sup>b</sup> =0.384	p <sup>b</sup> =0.097	p <sup>b</sup> =0.262	p <sup>b</sup> =0.988		p <sup>b</sup> =0.996
Total	2.14 ± 1.41	1.57 ± 1.07	1.79 ± 1.62	1.74 ± 1.33	p <sup>a</sup> = 0.436	1.81±1.37
<b>SDR</b>						
Ra0	3.09 ±2.5	2.27 ±1.28	2.76 ±2.23	1.53 ±1.27	p <sup>a</sup> =0.296	2.42±1.92
Ra1	2.54 ±1.59	2.85 ±2.0	1.79 ±0.81	2.69 ±1.81	p <sup>a</sup> =0.684	2.47±2.04
Ra2	1.87 ±1.16	2.30 ±1.53	2.51 ±1.71	2.80 ±1.41	p <sup>a</sup> =0.556	2.37±1.45
	p <sup>b</sup> =0.407	p <sup>b</sup> =0.584	p <sup>b</sup> =0.179	p <sup>b</sup> =0.06		p <sup>b</sup> =0.926
Total	2.50 ± 2.17	2.47 ± 1.60	2.35 ± 1.91	2.34 ± 1.57	p <sup>a</sup> = 0.979	2.42±1.81

Lower letters indicate the difference between lines and capital letters indicate the difference between columns.  
 Ra0: Baseline surface roughness, Ra1: Surface roughness after surface coating agent application, Ra2: Surface roughness after staining procedure.  
 p<sup>a</sup> values are based on One-way ANOVA test, p<sup>b</sup> values are based on Repeated Measures for One Way ANOVA, \*p<0,05 is significant. The acceptability threshold of surface roughness was considered as 0.2 µm.

**Table 4.** Factors affecting color change ( $\Delta E_{00}$ ) and surface roughness at different times

	Source	Type III sum of squares	df	Mean square	F	Sig.
$\Delta E_1$	Material	0.632	1	0.632	2.373	0.128
	Surface coating agent	6,327	3	2.109	7.919	0,000
	Material*Surface coating	8.097	3	2.699	10.134	0.000
	<i>R Squared = ,440 (Adjusted R Squared = ,385)</i>					
$\Delta E_2$	Material	66.394	1	66.394	16.689	0.000
	Surface coating agent	149.171	3	49.724	12.499	0.000
	Material*Surface coating	68.416	3	22.805	5.732	0.001
	<i>R Squared = ,498 (Adjusted R Squared = ,449)</i>					
$\Delta E_3$	Material	77.598	1	77.598	16.877	0.000
	Surface coating agent	161.618	3	53.873	11.717	0.000
	Material*Surface coating	78.488	3	26.163	5.690	0.001
	<i>R Squared = ,490 (Adjusted R Squared = ,440)</i>					
Ra	Material	0.288	2	0.144	0.072	0.930
	Surface coating agent	0.288	2	0.144	0.073	0.930
	Material*Surface coating material	8.498	6	1.416	0.713	0.640

$\Delta E$  values are based on the One-way ANOVA test, and Ra values are based on Repeated Measures for One-way ANOVA.

**Figure 1.** Schematic illustration of study design.

### Surface topography evaluation by Scanning Electron Microscope (SEM)

Scanning electron microscope images were taken from randomly chosen specimens from all experimental groups following the staining procedure. Specimens were sputter-coated with palladium in the ion plating unit (Polaron SC500 sputter coater, FISONs Instrument, UK) and were observed by SEM device (Zeiss GEMINI 500, Zeiss, Oberkochen, Germany) at the Erciyes University Technology Research and Application Center. The entire surface of the specimen was scanned, and the most representative areas were photographed at a magnification of 1,500x.

### Statistical analyses

Statistical analyses were performed using the Statistical Package for the Social Sciences (version 25.0, IBM SPSS Corp., Armonk, NY, USA). The normality of the  $\Delta E_{00}$  and Ra ( $\mu\text{m}$ ) data was determined using the Shapiro-Wilk test. Two-way analysis of variance (ANOVA) was used to determine the significance of color change caused by surface coating materials in restorative materials. Intra-group color change differences of materials were performed by Student t-test. Two-way analysis of variance (ANOVA) and repeated measures for the two-way ANOVA test were employed to indicate the evaluation of surface roughness due to the parametric test assumptions being fulfilled. Student t-test was used to determine the surface roughness differences of the materials in the same time period. P=0.05 was set the level of statistical significance.

## RESULTS

### Assessments of resin-based materials' color changes

The mean  $\Delta E_{00}$  values and standard deviations of the restorative materials after treatment with surface coating agents (T1- T0) and immersion in coffee (T2- T0) are shown in Table 2. Regarding  $\Delta E_1$  values, no significant difference was detected between resin composite materials used in the present study (p<sup>b</sup>>0.05). The discoloration of the GP and SDR specimens were below AT threshold (<1.8), while the values were above the PT threshold (>0.8). Regarding the  $\Delta E_2$  and  $\Delta E_3$  values, there were significant differences between restorative materials (p<sup>b</sup>=0.000) due to more color change observed in SDR than in GP. Also, both materials presented unacceptable and perceptible discoloration.

### Assessments of surface coating agents' color changes

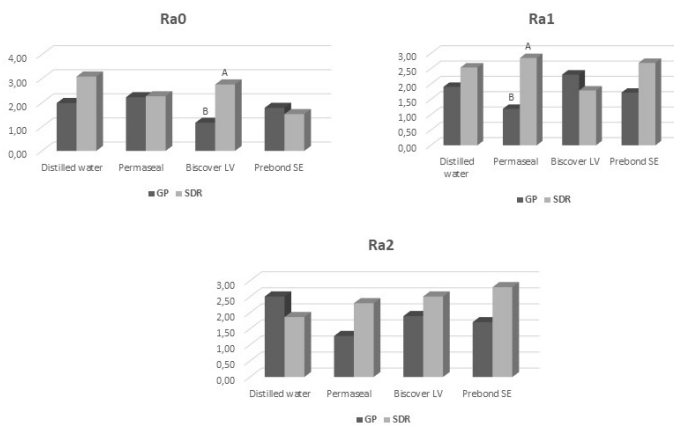
There were significant differences among surface coating agents, regarding  $\Delta E_1$  values ( $p^a=0.001$ ). All values were below AT threshold (<1.8). Only distilled water showed imperceptible color change. Also, there were significant differences among the surface coating agents, regarding the  $\Delta E_2$  and  $\Delta E_3$  values ( $p^a=0.000$ ). All values were above AT (>1.8) and PT (>0.8) thresholds. The highest discoloration was observed for Permaseal and Prebond SE in terms of  $\Delta E_2$  and  $\Delta E_3$  (Table 2).

### Assessments of resin-based materials' surface roughness

The difference in surface roughness between restorative materials in the same period is schematized in Figure 2. Regarding Ra0 and Ra1, there were significant differences between resin composites due to the SDR exhibiting more surface roughness in Biscover LV and Permaseal groups ( $p=0.001$  and  $p=0.002$ , respectively). In addition, the Ra values of both resin composites were above the threshold value ( $Ra=0.2 \mu m$ ). Regarding Ra2 values of materials, no significant difference was found.

### Assessments of surface coating agents' surface roughness

The mean surface roughness (Ra) values and standard deviations of the restorative materials after treatment with surface coating agents and immersion in coffee are summarized in Table 3. No significant differences were detected between surface coating agents for both GP and SDR. Permaseal showed the lowest roughness in GP specimens, whereas Prebond SE was in SDR specimens. However, all Ra values of surface coating agents were above the threshold value ( $Ra=0.2 \mu m$ ).



**Figure 2.** Average Ra values of resin composite materials after surface coating agent applications and staining procedure. GP: G-aenial Posterior, SDR: SDR™ Flow+ Ra0: Baseline surface roughness, Ra1: Surface roughness after surface coating agent application, Ra2: Surface roughness after staining procedure.

### Assessment of resin-based materials/ surface coating agents' interactions

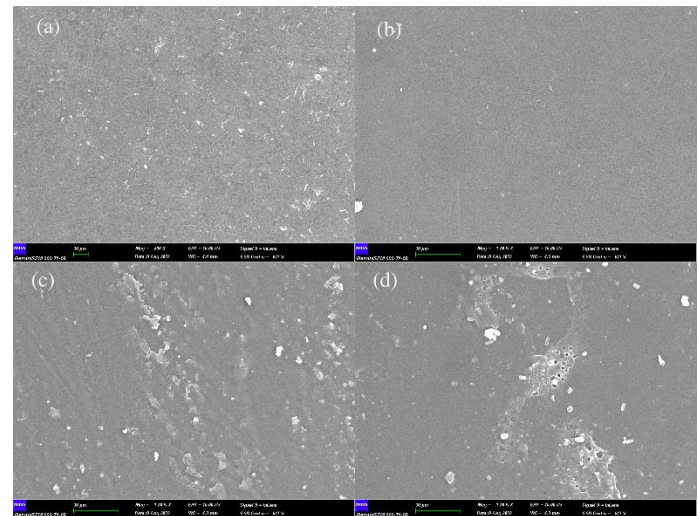
The factors affecting color change ( $\Delta E_{00}$ ) and surface roughness (Ra) at different times were summarized in Table 4. Regarding the interactions of composite resin materials/surface coating agents, there were significant differences in all evolution periods of  $\Delta E$  values ( $p=0.000$ ,  $p=0.001$ , and  $p=0.001$ ; respectively). When the origin of the differences was evaluated (Table 2), it was seen that GP- Biscover LV and SDR-Prebond SE combinations showed the lowest and clinically acceptable  $\Delta E$  values. Also, the combinations with the control group exhibited the lowest color change values.

Considering the interaction between surface coating agents and restorative material, there was no significant difference regarding the surface roughness at each time (Table 4). Although no significance, GP-Permaseal and SDR-Prebond SE combinations were exhibited less surface roughness (Table 3).

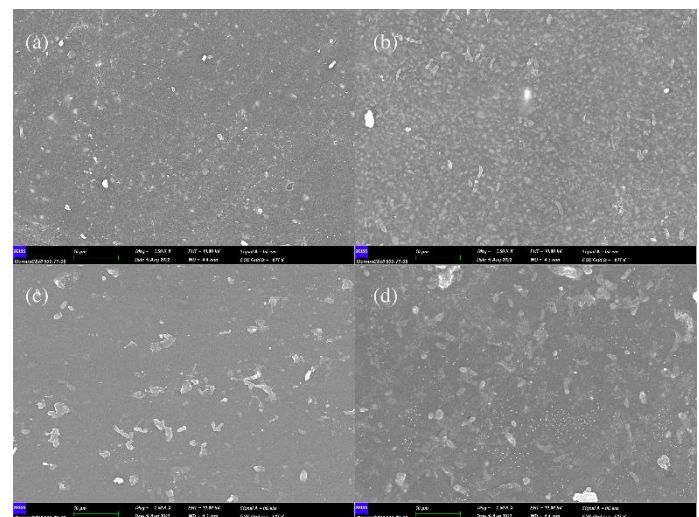
### Assessment of SEM images

Scanning electron microscope images of a specimen from each group of the GP and SDR are shown in Figure 3 and Figure 4, respectively.

SEM micrographs of the SDR specimens treated with surface coating agents revealed a more surface porosity compared to specimens immersed in distilled water. However, this condition was the opposite for GP resin composite. The SDR resin composite specimen displayed slight alterations compared to the GP resin composite specimen. The GP resin composite treated with Permaseal specimen showed fewer surface alterations compared to other surface coating agents and distilled water (Figure 3). Among the surface coating agents, similar surface alterations were observed for the SDR resin composite specimen (Figure 4).



**Figure 3** SEM images of a G-aenial Posterior specimen. (a) control group, (b) Permaseal group, (c) Biscover LV group, (d) Prebond SE group.



**Figure 4** SEM images of a SDR™ Flow+ specimen. (a) control group, (b) Permaseal group, (c) Biscover LV group, (d) Prebond SE group.

## DISCUSSION

Surface topography and color stability are the major factors determining the clinical performance of resin-based materials. The surface of dental restoration should have as smooth as possible to reduce plaque accumulation and discoloration.<sup>12</sup> In this context, this study sought to answer the effects of surface sealant applications on the color stability and surface roughness of different resin-based composites.

Visual and instrumental methods are available to investigate the color differences of dental materials, however instrumental techniques have been widely recommended due to visual color assessment can create inconsistencies inter-observers in color perception.<sup>13</sup> The clinical spectrophotometer including the CIEDE2000 system was performed to measure the color changes of specimens in this study.<sup>14</sup> The CIEDE2000 formula is more preferred in recent studies evaluating color stability than the CIE L\*a\*b\* formula, as it offers a better fit and provides a comprehensible indication of perceptibility and acceptability.<sup>13,14</sup>

The acceptability and visual perceptibility threshold values are significant in detecting color differences of dental tissues and materials in clinical dentistry.<sup>15</sup> In the current study, the PT and AT in analyzing color changes were specified as  $\Delta E_{00}=0.8$  and  $\Delta E_{00}=1.8$  respectively, as reported by Paravina et al.<sup>16</sup> No color change should be determined after being subject to the test environment for the material to achieve complete color stability ( $\Delta E=0$ ).<sup>17</sup> Additionally, in the current study, the CIEDE2000 (1:1:1) formula was used instead of the CIEDE2000 (2:1:1) formula because of insufficient data on acceptability and perceptibility threshold values for the CIEDE2000 (2:1:1).<sup>18</sup>

In most studies evaluating the color stability of resin composites exposure to staining solutions, samples were immersed in tea, red wine, coffee, and other beverages for extended periods (hours or days) without interruption.<sup>19</sup> Among these staining solutions, coffee is reported to be one of the most effective agents that can mimic the daily routine *in-vitro*.<sup>20</sup> The mechanism of coffee-induced color change in resin-based materials is the adsorption and absorption of yellow pigments through the organic phase of the materials.<sup>21</sup> It has been reported in the literature that 72 hours of simulated coffee consumption corresponds to 3 months of daily consumption<sup>20</sup>, while immersion for 15 days simulates one-year daily coffee consumption<sup>22</sup>. The immersion time acknowledged in the current study was 144 hours uninterruptedly, which is corresponding to approximately 6 months of clinical aging, in accordance with Korkut et al.<sup>19</sup> It has been also reported that hot coffee solution is a more active agent for discoloration.<sup>20</sup> Therefore, in the present study, to stimulate oral conditions, the specimens were continuously exposed to the staining solution at 37°C and the solution was refreshed daily.

In the present study, a micro-filled hybrid (mFR) resin composite (G-ænial Posterior) was chosen as the control material to compare with the bulk-fill resin composite (SDR). The findings of the current study revealed that the color stability of RBCs was affected following the application of surface coating agents. Therefore, the first null hypothesis of this study is rejected. G-ænial Posterior resin composite, presented a significantly low amount of color change than SDR (Table 2). However, the discoloration was at a clinically unacceptable level in both materials ( $AT>1.8$ ). Some researchers have stated that increased composite thickness is responsible for the higher color change observed in bulk-fill resin composites compared to conventional resin composites.<sup>17,23</sup> Based on this information, flowable bulk-fill specimens were prepared with a thickness of 2 mm instead of a 4 mm single layer. Packable composites are generally reported to have less color change than the flowable composite used in thinner layers.<sup>17</sup> Not in consistent with the present study, Bilgili Can D. & Özarslan M.<sup>24</sup> stated that the most color change was observed in G-ænial Posterior specimens. Staining of resin composites has been stated to be closely related to the resin phase. Bisphenol-glycidyl methacrylate (Bis-GMA) is less stain-resistant than Urethane dimethacrylate (UDMA) due to its high water absorption characteristics.<sup>25</sup> Bis-GMA monomer has a viscous and bulky bifunctional matrix.<sup>26</sup> Therefore, it is diluted by the addition of a more reactive monomer, triethylene glycol dimethacrylate (TEGDMA). The dilution process allows a higher amount of nanofillers to be added to the

resin matrix.<sup>27</sup> The increased filler and monomer (hydrophobic aromatic dimethacrylate) content is thought to be the reason for less discoloration in GP specimens due to reduced water absorption rates in the material.

The surface structure of restorative materials affects plaque accumulations, wear resistance and physical properties.<sup>12</sup> Surface roughness depends on the type of resin matrix, amount, type, shape, size and distribution of inorganic filler particles, filler and resin matrix combination, finishing and polishing procedure, abrasive hardness and application methods.<sup>28</sup> Surface roughness measurements can be performed qualitatively (qualitative) such as scanning electron microscopy (SEM), surface profile analysis (Profilometer), and quantitatively (quantitative) methods.<sup>29</sup> However, it has been reported that supporting profilometry findings with qualitative methods increases the reliability of the findings due to these methods provide more detailed surface information than profilometry.<sup>30</sup> Considering these findings, SEM imaging was performed to support and detail the profilometer findings in the current study (Figure 3 and 4). According to the profilometer findings, there was no significant difference regarding the surface roughness between the restorative materials at each treatment stage (Table 3). However, surface roughness values of the materials were above the cut in each period (T0, T1, and T2) ( $Ra>0.2 \mu\text{m}$ ). According to these findings, the second hypothesis is also rejected. The highest mean Ra values in all periods of the current study were seen in SDR ( $Ra=2.47 \mu\text{m}$ ). Previous studies stated that a perfectly smooth surface cannot be obtained after finishing-polishing methods in tooth-colored restorative materials.<sup>12</sup> Although the threshold surface roughness was mentioned for bacterial plaque retention as  $0.2 \mu\text{m}$ , no significant difference was found in plaque on surfaces with Ra values between  $0.7$  and  $1.4 \mu\text{m}$ .<sup>29</sup> Due to the presence of specimens showing roughness above these values in the current study, surface smoothness could be checked with a method such as light microscopy after finishing-polishing methods, and if the polishing was found to be insufficient, this process could be repeated with different finishing-polishing kits. This condition is the first limitation of the present study.

All treatment groups presented discoloration above AT including the control group. The degree of water absorption and the hydrophilic/hydrophobic structure of the resin matrix may contribute to the specimens' staining sensitivity following immersion in distilled water.<sup>23</sup> There are studies in the literature using distilled water and/or artificial saliva as a control group<sup>31</sup>, and in both, it was observed that the specimen's color change was within clinically acceptable limits due to material aging. Artificial saliva does not contain any chemical enzymes that will affect the resin matrix and cause the softening of the dimethacrylate polymers in its structure and the hydrolysis of methacrylate ester linkages.<sup>32</sup> For this reason, distilled water was used as a control group to compare the effect of surface sealants on color and roughness changes in restorative materials in the current study, as in most studies investigating color change.

The level of color changes in the surface coating agent groups was higher than the control group. In contrast to this finding, Korkut et al.<sup>19</sup> and Pedroso et al.<sup>4</sup> observed that the discoloration of surface sealant applied to resin composite specimens was lower. However, this finding was variable according to the resin composite type and the surface coating agent type in the current study. The highest color change was observed in Permaseal and Prebond SE, and the lowest in Biscover LV (Table 2). According to the baseline color measurements, the color change observed after the Biscover LV application was below the clinical AT (1.8) and very close to the PT (0.8). This supports the argument stated in previous studies<sup>10,19</sup> that the reason for the less color change of Biscover is that it has a shorter polymerization time and contains dipentaerythritolpentaacrylate. On the other hand, the adhesive system (Prebond SE) presented worse color stability than the surface sealants

(Permaseal and Biscover LV) in the current study. Self-etching adhesives present a high content of hydrophilic monomers.<sup>8</sup> This may lead to higher water and coffee pigment absorption. In addition, solvents present in the structure of adhesive systems, which can lead to insufficient polymerization if not evaporated well, may cause worse performance.<sup>8</sup>

Surface coating agents have been recommended for composite resins to obtain smoother surfaces. However, it is difficult to obtain a smooth surface on restorations with the application of liquid agents.<sup>3</sup> In the present study, it was observed that the surface coating agents showed better results in terms of roughness than the control group. The main reason for this finding may be that surface coating agents reduce surface irregularities and defects and increase smoothness. The lowest roughness was observed in Permaseal, but the difference between groups was not significant and similar results were obtained when compared with other coating agents. Opposing to our results, Ruschel et al.<sup>33</sup> reported that Permaseal had significantly higher roughness than other surface coating agents (Fortify and Biscover). Also, Rizzante et al.<sup>10</sup> stated that Biscover showed lower roughness than the other groups (Fortify, Lasting touch, Fill glaze). On the other hand, the performances of the self-etching adhesive system and surface sealants were similar in terms of roughness in the current study. In accordance with the results of this study, Cortopassi et al.<sup>8</sup> reported that the surface roughness decreased in adhesive systems.

Considering the interaction between the type of surface coating agents and the type of restorative material, the findings of the present study revealed that there was a significant difference at each stage of the color measurements. The combinations of GP (microhybrid)-Biscover LV, SDR (bulk-fill)-Biscover LV, and SDR (bulk-fill)-Biscover LV were successful in terms of color stability in the present study. Miotti et al.<sup>34</sup> reported similar results of Biscover LV and microhybrid resin combination. Another study contrastly reported that the surface sealant application negatively affected the color stability of microhybrid composite resins.<sup>1</sup> On the other hand, SDR-Permaseal combination showed most discoloration in our study. Variations in the formulation and roughness of the resin composites tested in the studies may have caused these contradictory results. In addition, material combinations with distilled water exhibited less color change generally. In light of these results, it can be said that applying surface sealants may not always provide favorable outcomes.

Considering the interaction between the type of surface coating agents and the type of restorative material, the present study revealed no significant difference regarding the surface roughness at each period. GP-Permaseal and SDR-Biscover LV/Prebond combinations were effective regarding roughness. A previous study reported that surface sealant application reduced roughness in all tested composites.<sup>35</sup> These results may depend on the type of composite resin and surface coating agent used, or differences in finishing and polishing processes.

This in vitro study also has other limitations: Use of a single staining solution to simulate intraoral conditions, only 144 hours of storage time and no thermocycle application for aging. In addition, it should be considered that different finishing-polishing systems, prolonged exposure time of the specimens in the staining beverages, and many other factors (such as dietary and oral hygiene habits, the effect of saliva, toothbrushing, or occlusion) might influence the surface alterations of restorative materials. Therefore, further clinical studies should be designed to verify the discoloration degree and surface roughness of resin composites in the oral environment.

Within the limitations of the present study, the findings can be summarized as follows: In this in vitro experimental model designed to determine the effect of daily coffee consumption on the surface and color change of resin composites treated with surface coating agents, the time simulating a period of 6 months was deemed sufficient. A bulk-fill resin composite is more prone to discoloration than micro-filled

hybrid (mFR) resin composite. At each period, the bulk-fill composite exhibited greater surface roughness than the microhybrid composite. Biscover LV showed more acceptable results in terms of both color stability and roughness than other surface coating agents (Permaseal and Prebond SE). The combination of resin composites used in the current study with Biscover LV might be considered the most effective combination for inhibiting discoloration. However, the microhybrid composite-Permaseal combination was found to be successful in reducing the surface roughness. Clinicians should be aware that the consumption of coffee for long periods may lead to more discoloration in resin composites and surface coating agents can be applied over the surface of resin composites to decrease the color stability and surface roughness.

**Etik Komite Onayı:** Bu in-vitro çalışma, yazarların herhangi biri tarafından insan katılımcılarla veya hayvanlarla gerçekleştirilen herhangi bir çalışma içermemektedir. Tüm yöntemler ilgili kurallara ve düzenlemelere uygun olarak gerçekleştirildi.

**Hasta Onamı:** Bu tür in-vitro çalışma için resmi onam gerekli değildir.

**Hakem Değerlendirmesi:** Dış bağımsız.

**Yazar Katkıları:** Fikir –A.T.E.A.; Tasarım – E.D., A.T.E.A.; Denetleme – E.D., A.T.E.A.; Kaynaklar – E.D., A.T.E.A.; Veri Toplanması ve/veya İşlemesi – E.D., A.T.E.A.; Analiz ve/veya Yorum – E.D., A.T.E.A.; Literatür Taraması – E.D., A.T.E.A.; Makaleyi Yazan – E.D., A.T.E.A.; Eleştirel İnceleme – E.D., A.T.E.A.

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**Ethics Committee Approval:** This in-vitro study does not contain any studies with human participants or animals performed by any of the authors. All methods were performed in accordance with the relevant guidelines and regulations.

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**Peer-review:** Externally peer-reviewed.

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