The Effect of a Modeling Resin and Thermocycling on the Surface Hardness, Roughness, and Color of Different Resin Composites

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ABSTRACT

Statement of Problem: The application of modeling resin could affect the surface quality and color of resin composites. Purpose: To evaluate the effects of modeling resin on the microhardness, roughness, and color of composite restorations, with and without thermocycling.

Methods: Sixty disc-shaped specimens for each resin composite were prepared in three groups: Group 1: A resin composite disc was cured against a polyester matrix and finished/polished; Group 2: A composite instrument was wetted with Bisco Modeling Resin (Bisco, Schaumburg, IL, USA) to smooth the composite surface, which was cured against a polyester matrix and finished/polished; Group 3: A composite instrument was wetted with modeling resin to smooth the composite surface, which was cured against a polyester matrix. Microhardness, roughness, and color were measured 24 hours after curing and after 10,000 thermocycles.

Results: Modeling resin significantly influenced the microhardness of GrandioSO (Voco, Cuxhaven, Germany) and Gradia Direct Posterior (GC America, Alsip, IL, USA), and the surface roughness of GrandioSO, Filtek Silorane (3M ESPE, St Paul, MN, USA), and Aelite All Purpose Body (Bisco) (p < 0.05). The microhardness of the Group I resin composites was affected by thermocycling (p < 0.05); however, thermocycling had no significant effect on surface roughness (p > 0.05). Tested composites showed clinically perceptible color changes after thermocycling. In Group I, Filtek Ultimate (3M ESPE) showed the lowest color change (p < 0.05), and in Group 2, Filtek Silorane showed the highest significant color changes (p < 0.05).

Conclusions: Modeling resin did not affect the microhardness, surface roughness, and color of Aelite LS Posterior (Bisco), Filtek Ultimate (3M ESPE), and Clearfil Majesty Esthetic (Kuraray Medical Inc, Tokyo, Japan) specimens. Also, thermocycling process only affected microhardness of tested resin composites.

CLINICAL SIGNIFICANCE

The effect of modeling resin on surface microhardness, roughness, and color stability of composite materials depends on the type of resin composite. In clinical practice, the adverse effects of modeling resin might be alleviated by a proper finishing and polishing procedure.

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INTRODUCTION

Resin composites have been widely used as dental restorative materials since the mid-1960s. A dental

composite basically consists of four main components: an organic polymer matrix, inorganic filler particles, a silane coupling agent for binding the filler to the matrix, and chemicals that promote or modulate the

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polymerization reaction.¹ The physical and mechanical properties of a dental composite are highly dependent upon the material's formulation as defined by the manufacturer but are also heavily influenced by the extent of the curing reaction and the care taken in placement, both of which are controlled by the clinician.²

In parallel with the development of adhesive dentistry, the physical and mechanical properties of resin composite restorations have been improved as a result of the changes in inorganic fillers and polymeric matrices. Attempts have been made to modify the monomer matrix from the conventional dimethacrylate monomer systems to the recently introduced epoxy-based resin systems.³ Over the last few decades, the most significant changes in resin composites have been made through improvements in the filler systems. Efforts to change the types of fillers or filler sizes and their surface sizing by silanization have been made.⁴ These changes provide for several physical and mechanical property improvements that facilitate the realization of the ideal resin composite restorative material.

Clinicians have tried to minimize the stickiness by wiping the composite instrument with one of several lubricants indicated for that purpose: isopropyl alcohol, acetone, dentin/enamel adhesive, and proprietary commercial products.^{5–7} Recently, manufacturers have introduced composite instrument wetting resins to facilitate composite handling, adaptation, and contouring. Practitioners coat dental hand instruments with these resins or dentin bonding agents to aid material handling.⁸ However, the effect of wetting resins on the physical and mechanical properties of composites is unknown.

Acceptable levels of the surface roughness, color stability, and microhardness of dental composite materials are essential for clinical success. In a restorative procedure, an important objective is to obtain restorations with smooth surfaces. Surface roughness has a major influence on plaque accumulation, secondary caries, and gingival irritation.^{9,10} Further, it may directly influence wear behavior and the marginal integrity of composite restorations.^{9,11} A roughened surface of a resin composite restoration is also likely be stained by exogenous sources, such as coffee, tea, or red wine, leading to the discoloration of the material.¹⁰ Additionally, the color stability of resin composites may be affected by the chemical differences of the resin components, such as their polymeric structure and photoinitiation system.^{12–14} On the other hand, surface hardness is related to material wear resistance and the ability to maintain form stability.¹⁵ Also, the surface hardness measurement has been found to be a good predictor for resin conversion, and was also reported to be especially sensitive to small changes in the polymer cross-linking in areas of high conversion.¹⁶

In the oral cavity, restorative materials are subjected to thermal stress. The cyclic thermal stresses, together with the presence of water and other fluids, may degrade the filler matrix interfaces and also lead to stress corrosion of the fillers.¹⁷ Although there have been several studies concerning the effects of thermocycling on the microhardness, roughness, and color of composite restorations,^{18–20} the effect of modeling resins on these features of resin composites has not been investigated. The objectives of this study were to evaluate the effect of a modeling resin on the microhardness, roughness, and color of composite restorations with and without thermocycling. The null hypotheses tested were: (1) the use of modeling resin does not affect the microhardness, roughness, and color of composite restorations; and (2) thermocycling does not influence the physical properties of composite restorations, such as microhardness, roughness, and color.

MATERIALS AND METHODS

The six dimethacrylate-based and one silorane-based dental resin composites, all of A2 shade, and the modeling resin used in this study are described in Table 1.

Sixty disc-shaped specimens (8.0 mm diameter \times 2.0 mm height) for each resin composite

TABLE 1. The resin composite materials and modeling resin used in the present study

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Dental resin composite/modeling resin	Composite type	Manufacturer	Organic matrix	Filler particle type	Filler particle size	% filler Wt/vol
GrandioSO	Nanohybrid	Voco, Cuxhaven, Germany	Bis-GMA, Bis-EMA, TEGDMA	Glass ceramic filler, Functionalized silicon dioxide nanoparticles, pigments (iron oxide, titanium dioxide)	1 µm 20–40 nm	89/73
Gradia Direct Posterior	Microhybrid	GC America, Alsip, IL, USA	UEDMA	Barium silicate glass, silica dioxide, prepolymerized filler	0.85 µm	77/65
Aelite LS Posterior	Hybrid	Bisco, Schaumburg, IL, USA	Bis-EMA, TEGDMA	Glass filler, amorphous silica	0.04–3.5 µm	89/75
Filtek Silorane	Microhybrid	3 M ESPE, St. Paul, MN USA	Silorane	Quartz filler, yttrium fluoride	0.1–2 µm	76/55
Aelite All Purpose Body	Microhybrid	Bisco, Schaumburg, IL, USA	Bis-EMA, TEGDMA	Glass filler, amorphous silica	0.04–0.7 µm	76/55
Filtek Ultimate	Nanofilled	3 M ESPE, St. Paul, MN, USA	Bis-GMA, UEDMA, TEGDMA, Bis-EMA	Silica, zirconia filler, zirconia/silica cluster filler	20 nm silica and 4 to 11 nm zirconia particles, average cluster particle size 0.6 μm to 10 μm	78.5/63.3
Clearfil Majesty Esthetic	Nanohybrid	Kuraray Medical Inc., Tokyo, Japan	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate	Silanated barium glass filler, prepolymerized organic filler	20 nm 1.5 µm	78/66
Modeling Resin		Bisco, Schaumburg, IL USA	UEDMA, ethoxylated Bis-GMA	Amorphous silica	N/A	30/
Bis-GMA = Bisnhenol A-GN	voidvl Methacrylate: Bis-F	=MA = ethoxylated hishhenol-A c	limethacrvlate TEGDMA = Triethvle	ne alvrol dimethacrulate 1 IEDM2	1 - 1 Inathana Dimathacrulata	

were prepared. For 20 disc-shaped composite specimens, the stainless steel mould was filled with uncured resin composite and covered on both sides with a polyester matrix strip (Mylar Strip, SS White Co., Philadelphia, PA, USA). For the other 40 disc-shaped composite specimens, one drop of modeling resin was dispensed into a clean mixing well. Following the placement of the resin composite into the stainless steel mould, a composite instrument (round-ended plugger with a diameter of 2 mm) was dipped into the modeling resin. With modeling resin on the instrument, the resin composite was sculpted for 5 seconds and covered with a polyester matrix strip.

The mould was compressed with finger pressure between two glass microscope slides (1 mm thick) to remove excess material and to obtain a flat surface. The top surfaces of all samples were photopolymerized for 40 seconds through the glass slide and polyester matrix with a halogen light unit (VIP, Bisco Inc., Schaumburg, IL, USA) calibrated at 600 mW/cm². The light intensity of the curing light was checked regularly during specimen preparation, by using a radiometer (Hilux Curing Light Meter, Benlioglu Dental Inc., Ankara, Turkey). The composite specimens were removed from the moulds after they were light cured. The top surfaces of the specimens were marked with a permanent marker. Then, the specimens were stored in distilled water at 37°C for 24 hours. The polymerized composite specimens were divided into three groups.

Group 1 (finished and polished resin composite discs): The top surfaces of 20 composite specimens without modeling resin were ground with 600 grit silicon carbide (SiC) paper for 20 seconds. Then, the surfaces were polished under dry conditions using a complete sequence of 12.5 mm Sof-Lex Polishing Discs (3 M ESPE, St. Paul, USA), from medium to superfine. Each disc was used for 30 seconds with a hand piece rotating at 10,000 rpm. After each polishing step, specimens were thoroughly rinsed with water for 10 seconds to remove debris, air-dried for 5 seconds, and polished with another disc of lower grit for the same period, until the final polishing. *Group 2 (finished and polished resin composite discs with modeling resin)*: The top surfaces of 20 composite specimens with modeling resin were finished and polished as for Group 1.

Group 3 (resin composite discs with modeling resin under a polyester matrix strip): The top surfaces of 20 composite specimens with modeling resin were not submitted to any finishing or polishing procedures after curing against the polyester matrix strip.

Microhardness Measurements

For the microhardness tests, 30 disc-shaped specimens for each resin composite according to the three groups (N=10) were used. The Vickers hardness number (VHN) (kg/mm²) was determined using a microhardness tester (Shimadzu HMV-2, Japan) before the thermocycling process. Three indentations were made on the surface under a 200 g load with a 15-second dwell time. The average hardness value for each specimen was then calculated. Next, the measured specimens were immersed in a water bath and repeatedly thermocycled between 5 and 55°C with a dwell time of 30 seconds in each bath. The same measurements were performed after 10,000 cycles. Each measurement was performed near the previously measured position to maintain consistency.

Surface Roughness and Color Measurement

For the surface roughness and color measurements, a total of 30 disc-shaped specimens for each resin composite according to the three groups (n = 10) was used. The surface roughness and color measurements were made before (baseline) and after thermocycling.

The surface properties assessed included the roughness average, (R_a , μm), by a two-dimensional profilometer (Surtronic 3⁺, Taylor Hobson, Leicester, UK). A diamond stylus of 5 μm and a stylus angle of 90° traversed a length of 1.25 mm with a cut-off length of 0.25 mm. Five measurements in the center of each sample at crossing directions were performed.

After profilometric examination, spectrophotometric analysis was carried out. Before each measurement, the specimens were cleaned in distilled water for 1 minute and dried under a flow of air. Values were recorded in the Commission Internationale de l'Eclairage (CIE) CIELAB color system relative to CIE standard illuminant A (incandescent light) using a VITA Easyshade Compact (VITA Zahnfabrik, Bad Sackingen, Germany, Model DEASYCHP). Before measuring the color of the specimens, the Vita Easyshade was calibrated using its calibration block according to the manufacturer's instructions. The probe tip was placed perpendicular and flushed to the surfaces of the specimens to obtain accurate measurements. Measurements were performed at the centers of the resin composite discs and repeated three times. The CIELAB system is an approximately uniform color space with coordinates for lightness: namely, white/black (L*), red/green (a*), and yellow/blue (b*). The mean of the values obtained was calculated, and the L*, a*, and b* parameters were determined. All measurements were made on a white Plexiglass background in order to eliminate background light.21

Color changes (ΔE^*) after thermocycling were calculated as $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. Changes in CIE L*, a*, and b* values ($\Delta L^*, \Delta a^*, \Delta b^*$) during thermocycling were calculated as "the value after thermocycling – the value before thermocycling."

A three-way analysis of variance (ANOVA) was performed for each resin composite type to observe the effect of the independent variables on surface microhardness and surface roughness: the factors were the resin composite, the resin composite preparation group, and the thermocycling process (before or after).

Two-way ANOVA was used to evaluate the effect of the resin composite and the resin composite preparation group on color stability, including the possibility of interactions between the two factors.

When the difference was statistically significant (p < 0.05), post-hoc pair-wise multiple comparisons with Bonferroni's correction were performed with the

probability level set to $\alpha = 0.05$ for statistical significance. All analyses were performed using a commercially available software package (SPSSWIN 15.0; SPSS, Chicago, IL, USA).

RESULTS

Surface Microhardness Results

The mean VHN values and standard deviations for the resin composites tested under the experimental conditions used in this study are shown in Table 2. According to the three-way ANOVA test, all analyzed factors (resin composite, resin composite preparation group, and thermocycling process) had a statistically significant influence on the resin composite hardness (p < 0.001). The analysis of factor interactions showed that all two-way interactions had a statistically significant effect on the results obtained (p < 0.001).

Differences in microhardness were observed among the composites in all application groups. In Groups 1 and 2 at baseline, the GrandioSO and Aelite LS Posterior specimens showed the highest (p < 0.05), and the Gradia Direct Posterior specimens showed the lowest VHN values when compared with the other tested resin composites. At the baseline, the VHN values of the Filtek Ultimate in Groups 1 and 2 were 101 ± 10 and 101 ± 7 , and after thermocycling, 89 ± 4 and 88 ± 7 , respectively. Pair-wise multiple comparisons with the Bonferroni test (p < 0.05) revealed that in Groups 1 and 2, Filtek Ultimate exhibited a significantly higher hardness value than all other materials, except GrandioSO and Aelite LS Posterior. The Aelite LS Posterior resin composite discs with the modeling resin under the polyester matrix strip showed significantly higher VHN values than all others materials, except for the Filtek Ultimate specimens in the Group 3 baseline (p < 0.05).

In Group 1, the GrandioSO (146 ± 6) and the Gradia Direct Posterior (43 ± 3) baseline VHN values were significantly higher than those in Group 2 and Group 3. For the other tested materials, there was no statistical

Resin Composite	Group I		Group 2		Group 3				
	Baseline	тс	P *	Baseline	тс	P*	Baseline	тс	P *
GrandioSO	146±6	129±8	p<0.05	137±3	122±10	p < 0.05	28±5	27±3	p>0.05
	A a	AI		Ab	A ¹		ACD c	ACDEF ²	
Gradia Direct Posterior	43±3	4 ±	p<0.05	39±2	37±2	p<0.05	18±3	16±0.4	p<0.05
	Ва	В		ВЬ	B ²		Ac	B ³	
Aelite LS Posterior	143±6	122±10	p<0.05	4 ± 8	129±13	p>0.05	42±14	34±11	p>0.05
	A a	A ¹		A a	A		Вb	CE ²	
Filtek Silorane	73±2	61±3	p<0.05	73±5	63±5	p<0.05	22±1	21±1	p<0.05
	C a	C'		Ca	C'	1	AD b	ABDF ²	
Aelite All Purpose Body	74±4	67±3	p<0.05	77±8	74±2	p>0.05	29±6	27±5	p>0.05
	C a	C'		C a	C ²		ACF b	DCEF ³	
Filtek Ultimate	101±10	89±4	p<0.05	101±7	88±7	p<0.05	38±8	33±7	p>0.05
	Da	D		Da	D'		BCG b	EG ²	
Clearfil Majesty Esthetic	52±5	45±5	p<0.05	52±4	44±2	p<0.05	31±7	25±5	p>0.05
	Ba	BI		Ea	BI		DFG b	FG ²	

TABLE 2. Microhardness values (VHN, kg/mm²)(mean \pm SD) of the tested materials

 p^* represent statistical significant differences in each group between the baseline and thermocycled specimens (TC) within the same composite. Means followed by distinct capital letters represent statistical significant differences in each column (p < 0.05).

Means followed by distinct lower case letters (comparison of the baseline values between the groups) represent statistical significant differences in each row (p < 0.05).

Means followed by distinct superscript numbers (comparison of the thermocycled specimens' values between the groups) represent statistical significant differences in each row (p < 0.05).

difference between the Group 1 and Group 2 VHN values. In Group 3, the lowest hardness values were recorded for all resin composites tested at baseline and after thermocycling (p < 0.05).

Within the same resin composite and with respect to thermocycling, there were significant differences in microhardness values for all tested composites in Group 1 (p < 0.05). In Group 2, thermocycling affected the VHN values for the composite specimens GrandioSO, Gradia Direct Posterior, Filtek Silorane, Filtek Ultimate, and Clearfil Majesty Esthetic (p < 0.05). However, in Group 3, only the Gradia Direct Posterior and Filtek Silorane specimens were affected by the thermocycling process (p < 0.05).

Surface Roughness Results

Table 3 shows the mean surface roughness (R_{a} , μm) values together with their standard deviations for the resin composites tested under different conditions. Three-way univariate ANOVA revealed that the three main factors (resin composite, resin composite preparation group, and thermocycling) and the interaction between the resin composite and the resin composite preparation group were significant determinants of surface roughness. No statistically significant differences in surface roughness were observed among the two-way interactions between resin composite and thermocycling, or resin composite preparation group and thermocycling.

Resin composite	Group I		Group 2		Group 3	
	Baseline	тс	Baseline	тс	Baseline	тс
GrandioSO	0.168 ± 0.02	0.183±0.033	0.144±0.016	0.150±0.01	0.066 ± 0.006	0.071±0.008
	A a	A	Аb	A ²	Аc	A ³
Gradia Direct Posterior	0.126±0.014	0.144±0.024	0.129±0.02	0.137±0.013	0.071±0.012	0.078±0.019
	BCF a	BI	A a	A	AB b	AB ²
Aelite LS Posterior	0.218±0.024	0.226±0.021	0.196±0.024	0.206 ± 0.025	0.082±0.011	0.089±0.015
	D a	C'	B a	Bi	Вb	AB ²
Filtek Silorane	0.1±0.013	0.109±0.015	0.123±0.012	0.136±0.015	0.071±0.008	0.074 ± 0.005
	CE a	D'	Ab	A ²	AB c	AB ³
Aelite All Purpose Body	0.112±0.016	0.126±0.016	0.140±0.01	0.147±0.013	0.060 ± 0.006	0.075±0.01
	BE a	BD1	Ab	A ²	Ac	AB ³
Filtek Ultimate	0.119±0.018	0.130±0.017	0.133±0.037	0.143±0.038	0.071±0.007	0.091±0.022
	BEF a	BD1	A a	A	AB b	B ²
Clearfil Majesty Esthetic	0.139±0.02	0.155±0.02	0.125±0.02	0.134±0.021	0.069±0.008	0.080±0.009
	Fa	ABI	A a	A ²	Аb	AB ³

TABLE 3. Surface roughness values (R_a , μm) (mean \pm SD) of the tested materials

TC=thermocycled specimens.

Means followed by distinct capital letters represent statistical significant differences in each column (p < 0.05).

Means followed by distinct lower case letters (comparison of the baseline values between the groups) represent statistical significant differences in each row (p < 0.05).

Means followed by distinct superscript numbers (comparison of the thermocycled specimens' values between the groups) represent statistical significant differences in each row (p < 0.05).

Significant differences in surface roughness were found between the tested composites and the tested composite application groups. In Group 1, the silorane-based resin composite $(0.1 \pm 0.013 \,\mu\text{m})$ showed the smoothest surface, which was significantly lower than GrandioSO, Aelite LS Posterior, and Clearfil Majesty Esthetic (p < 0.05). Furthermore, the roughest surface was found for Aelite LS Posterior $(0.218 \pm 0.024 \,\mu\text{m}) \ (p < 0.05)$. The same effect was shown after 10,000 thermocycles for Filtek Silorane and Aelite LS Posterior; however, Filtek Silorane had a significantly lower roughness value than GrandioSO, Gradia Direct Posterior, Aelite LS Posterior, and Clearfil Majesty Esthetic. The mean R_a of GrandioSO at baseline $(0.168 \pm 0.02 \,\mu\text{m})$ was significantly higher than the other tested composites, except Aelite LS Posterior. In Group 2, for both baseline and after the

thermocycling process, Aelite LS Posterior was significantly rougher than the other tested composites (p < 0.05). Aside from this value, there were no significant differences among the mean R_a of the resin composites used in this study at baseline and after thermocycling. For Group 3, Aelite All Purpose Body ($0.060 \pm 0.006 \ \mu m$) produced the lowest surface roughness at baseline and the surface roughness was only significantly lower than Aelite LS Posterior ($0.082 \pm 0.011 \ \mu m$). After thermocycling, a significant difference was found only between the GrandioSO and Filtek Ultimate samples.

With respect to the composite application groups, the significantly lowest roughness value was obtained for each resin composite when the specimen was cured under a polyester matrix strip (Group 3) (p < 0.05).

Resin composite	Group I	Group 2	Group 3
GrandioSO	1.49±0.31	1.62 ± 0.34	1.58±0.23
	AD a	A a	A a
Gradia Direct Posterior	1.68±0.38	1.64±0.36	1.59 ± 0.35
	ABD a	A a	A a
Aelite LS Posterior	1.96±0.16	1.52±0.28	1.65 ± 0.2
	ACE a	AC b	Ab
Filtek Silorane	1.79±0.18		3.43±0.12
	ABC a	2.53±0.45Bb	Вс
Aelite All Purpose Body	2.17±0.4	1.79±0.19	1.45 ± 0.24
	CE a	AD b	Ab
Filtek Ultimate	1.28±0.19	1.10±0.26	2.39±0.51
	D a	Ca	Сb
Clearfil Majesty Esthetic	2.34±0.35	2.2±0.23	1.88±0.29
	Ea	BD a	Аb

TABLE 4. Color change values (ΔE) (mean \pm SD) of the tested materials after thermocycling

Means followed by distinct capital letters represent statistical significant differences in each column (p < 0.05), and means followed by distinct lower case letters represent statistical significant differences in each row (p < 0.05).

GrandioSO, Filtek Silorane, and Aelite All Purpose Body mean surface roughness values were significantly different according to the application Group 1 or Group 2 for baseline and after thermocycling. The specimens of GrandioSO resin composites prepared with modeling resin showed smoother surfaces than specimens prepared without modeling resin. For the Filtek Silorane and Aelite All Purpose Body resin composites, Group 2 specimens exhibited higher surface roughness than Group 1 specimens. There were no statistical differences among the specimens for the baseline in Groups 1 and 2 for the other composites (p > 0.05).

Color Results

The mean values for the color changes in the different groups after thermocycling (10,000 cycles) are summarized in Table 4. The ANOVA showed the interaction between the variables "resin composite" and "resin composite preparation groups" (p < 0.001).

Color changes of the resin composites were in the range 1.1–3.4 ΔE units. Color change was influenced by the resin composite type (p < 0.05). In Group 1, the highest mean ΔE value was observed in the Clearfil Majesty Esthetic, and this was significantly different from the GrandioSO, Gradia Direct Posterior, Filtek Silorane, and Filtek Ultimate (p < 0.05) specimens. In Group 2, the Filtek Silorane ($\Delta E = 2.53$) demonstrated a statistically significant difference from the rest of the materials except for the Clearfil Majesty Esthetic $(\Delta E = 2.2)$ (*p* < 0.05). Color changes in the resin composites in Group 3 showed the highest ΔE value for the Filtek Silorane ($\Delta E = 3.43$) specimens, whereas the second highest mean ΔE value was obtained from the Filtek Ultimate ($\Delta E = 2.39$) specimens, and both were significantly higher than the rest of the materials.

When the application groups were compared for the Filtek Silorane and Filtek Ultimate Group 3 specimens, color changes were significantly higher than those in Groups 1 and 2. The color change of Clearfil Majesty Esthetic in Group 3 was significantly lower than those in the other application groups. For the Aelite All Purpose Body and Aelite LS Posterior specimens, the color changes in Group 1 were statistically different from the Group 2 and Group 3 specimens. GrandioSO and Gradia Direct Posterior exhibited no significant difference in color change between the application groups.

DISCUSSION

Numerous dental resin composites based on various formulas and containing a variety of different components are available commercially. Due to the different viscosities of composite materials, clinicians need to use special hand instruments or wetting resins to apply the composite materials. The purposes of this study were to evaluate the effect of a modeling resin and thermocycling on the microhardness, surface roughness, and color of different composites.

Hardness determines the degree of deformation of a material, and it is generally accepted as an important property and a valuable parameter for comparison with the tooth structure.²² The hardness of composite materials has been related to the degree of conversion when the same material is evaluated under different polymerization conditions.²³ However, it is important to state that differences in hardness among different resin composites cannot be attributed to differences in the degree of conversion. On the basis of the present results and previous studies,^{24,25} the composite type had a significant effect on hardness values because of the variation in composition, type, size, and loading of fillers. Increased filler loading has been shown to result in increased hardness values.²⁶ In Group 1, as expected, GrandioSO and Aelite LS Posterior showed significantly higher microhardness values than the other tested composites. Both of these composites had the highest filler content. These results were compatible with the results of a previous study.²⁷ Yeh et al. found that the

Grandio microhardness value was higher than the tested composites Filtek Z350, Estelite Sigma, and Premisa. Estelite Sigma and Premisa had higher filler weight than Filtek Z350 but had lower microhardness values.²⁷ These results might be due to the prepolymerized filler particles of these two composites. Filler loading can be deceiving, however, because it may not be indicative of the presence of prepolymerized filler particles.²⁸ In our study, Clearfil Majesty Esthetic and Gradia Direct Posterior, which had prepolymerized particles, showed the lowest values for microhardness. Larger filler particles may strengthen the resin composite.²⁹ Filler size is only one of several factors affecting the properties of resin composites. The type, shape, amount, and coupling to the resin matrix of filler particles may also affect performance.²⁸

For Group 2, the microhardness values were in the same order as in Group 1. Comparison of the two groups revealed that the Group 2 preparation did not affect the microhardness of the tested composites, except for the GrandioSO and Gradia Direct Posterior. The silorane-based composite matrix consists of siloxane and oxirane components, and there is a debate about the interaction of dimethacrylate-based resins and silorane-based composites because of the differing chemical compositions of the matrices. In our study, the surface hardness of the silorane-based composites with or without modeling resin were similar; the Bis-GMA-based modeling resin did not influence the surface hardness of the silorane-based composites. Finishing and polishing processes remove the resin-rich layer of the composite specimens cured under a polyester matrix strip and might remove the modeling resin-silorane interface. The first hypothesis of this study, suggesting that the use of modeling resin does not affect the microhardness of resin composites, had, then, to be partially rejected. One explanation for the differences in hardness of the resin composites relies on the filler concentration.^{25,30} In the present study, the Filtek Silorane showed significantly lower microhardness than the Filtek Ultimate. The results of the present investigation are consistent with those of Poggio et al.³¹ The concentration of filler particles in Filtek Silorane is 76% by weight, and the filler consists of a combination of fine quartz particles and

radiopaque yttrium fluoride. The filler in Filtek Ultimate is silica, zirconia, and zirconia/silica clusters in a concentration of 78.5% by weight, and is classified as a nanofilled resin composite. This difference might be due to the filler concentration, type of filler and resin matrix. The present study showed that significantly lower microhardness values were achieved with resin composite discs which were prepared with modeling resin under a polyester matrix strip. This finding was in agreement with other studies. Korkmaz et al. and Erdemir et al. found that the microhardness differences were in the ranges of 15 and 27% between the composite specimens prepared under polyester matrix strip and composite specimens which were finished and polished.^{32,33} In our study, the differences between Group 1 and Group 3, and Group 2 and Group 3 were in the range of 40 and 80%. This was probably due to the filler volume of the modeling resin. The modeling resin caused a high resin-rich layer on the surfaces of the tested composites.

Thermocycling is a combination of hydrolytic and thermal degradation, and a method that simulates temperature-related breakdown by repeated sudden temperature changes.³⁴ A material's durability can be affected by thermocycling.³⁵ Water absorption affects the mechanical properties of composites toward hydrolytic degradation.³⁶ It can also cause microfractures in the interface between the fillers and the resin matrix, as well as induce superficial stress because of a high temperature gradient variation close to the surface.^{17,37} To mimic the oral environment, 10,000 thermocycles were employed in this study, and the effect of thermocycling on the microhardness of the resin composites was tested. The effect of 10,000 thermocycles on microhardness has been reported for several dental composites. Similar to our results, thermocycling significantly affects the microhardness of resin composites.¹⁸ In the current study, after thermocycling, the mean microhardness of tested composites in Group 1 was significantly decreased. The same effect was shown in Group 2, except for the Aelite All Purpose Body and Aelite LS Posterior specimens. A decrease in microhardness could be expected after thermocycling due to water absorption. Water acts like a plasticizer and, thereby, weakens the polymer

structure. It also degrades the matrix/filler interface directly by hydrolytic breakdown of the silane/filler interface and the surface of the filler particles.^{34,38}

The term "surface quality" reflects a set of widely different properties such as roughness, color, gloss, polarity, and morphology.³⁹ The determinants of the micromorphologies of the resin composites include the size, type, amount, and hardness of the composite filler particles, as well as the factors related to the flexibility of the backing material in which the abrasives are embedded, the hardness of the abrasives, and the instruments and their geometries.⁴⁰ In the present study, resin composite discs were either cured under a polyester matrix strip, or finished with wet, 600-grit SiC papers (30 µm average particle size) and polished with flexible aluminum oxide discs. Dental finishing instruments are often loaded with 30 µm abrasive particles, but using SiC papers for finishing allowed more facile standardization than rotating instruments.⁴¹ Also, it is commonly held that flexible aluminum oxide discs are the best instruments for attaining low surface roughness on composite surfaces.^{40,42} This is due to the better hardness of the aluminum oxide particles than most of the filler particles in resin composites.40 According to the surface roughness assessment, the average roughness $(R_a, \mu m)$ values of the finished and polished resin composite discs ranged from 0.10 µm for Filtek Silorane to 0.218 µm for Aelite LS Posterior. Bollen et al. stated that the threshold surface roughness for bacterial plaque retention was 0.2 µm, and a clinical study showed that the majority of patients could detect differences of about 0.3 µm in mean surface roughness.43,44 The surface roughness of the polished resin composite discs was under 0.2 µm, except for Aelite LS Posterior. This material's particle size was in the range of 0.04–3.5 µm with a filler loading of 89 wt%. Larger particles present in the composites contribute more to the surface roughness than do the average-sized particles.45,46 Previous studies reported that the surface of the Filtek Silorane was smoother than the other composites.^{46,47} Similar to our results, the posterior hybrid composite Filtek P60 showed the highest and the Filtek Silorane showed the lowest roughness values.⁴⁶ This value might be due to the Filtek Silorane's particular monomer chemistry, low

filler content (76%), and relatively smaller particle size (0.47 µm). In our study, the GrandioSO resin composite discs without modeling resin were significantly rougher than the other tested materials. Janus et al. reported that Grandio specimens polished with aluminum oxide discs were significantly rougher than the other composites tested in their study. They revealed that the numerous large voids that resulted from the plucking out of the voluminous fillers at the surface of Grandio, as observed by scanning electron microscopy, contributed to these results.⁴⁸ The present study indicated that there was no significant difference between the Filtek Ultimate and Aelite All Purpose Body specimens. In accord with our findings, Jung et al. reported that Filtek Supreme showed no better surface roughness than that of a traditional hybrid composite after polishing.49

In the present study, the effects of modeling resin on the surface roughness of seven different resin composites were compared. Except for the composites GrandioSO, Filtek Silorane, and Aelite All Purpose Body, there were no significant differences between Groups 1 and 2. Thus, the first hypothesis, suggesting that modeling resin does not influence the surface roughness of composites, can only be partially accepted. The surface roughness results of the current study demonstrated that the surface finish obtained with a polyester matrix strip was significantly smoother than the surfaces polished with aluminum oxide discs. However, this resin-rich surface layer had poor physical, mechanical, and biological properties. Therefore, this layer should be eliminated during the finishing and polishing procedures.⁴⁵ Surface roughness and microhardness values have been reported to be directly proportional to each other.⁵⁰ These findings agree with the results of this study, in that Aelite LS Posterior and GrandioSO showed higher hardness and surface roughness values among all the tested composites. In contrast to our results, Topcu et al. found that Clearfil Majesty Posterior showed the highest significant microhardness and lowest significant surface roughness values.²⁴

The thermocycling process involves repeated cooling and heating. These repeated temperature changes may induce degradation of the matrix-filler bond due to the different thermal expansion coefficients of the fillers and the resin matrices.^{18,47} One of the null hypotheses of this study was that the thermocycling process does not influence the surface roughness of resin composite discs. The results of the present study showed no significant difference between thermocycled and non-thermocycled composite specimens. Therefore, we must accept the null hypothesis. Similar to our results, Hahnel et al. reported that, after thermocycling, no significant difference in surface roughness was observed compared to baseline.⁴⁷ On the other hand, our results were inconsistent with those obtained by Minami et al., who performed 50,000 thermocycles in their study.¹⁹ These differences between the studies could be explained by the thermocycling cycle and dwell time. Ten thousand cycles of 30 seconds cooling and heating take approximately 7 days, whereas 50,000 cycles with 60 seconds dwell times require 64 days in an aging medium. The fillers and resin matrix have different thermal expansion properties; therefore, they react differently to thermal changes. This could affect the mechanical properties such as microhardness; however, there was no difference in the surface roughness, which was determined using a profilometry method in our study. Because not all surface irregularities can be detected by means of profilometry, further studies might be necessary using different methods, such as atomic force microscopy, to evaluate surface irregularities.

Unacceptable color match is one of the reasons for composite replacement. The evaluation of color stability and discoloration are commonly used outcome measurements that rate the success and failure of resin composite restorations in clinical practice.⁵¹ Discoloration of resin composites can be classified as either internal or external.⁵² Internal discoloration is due to the alteration of the resin matrix, filler, loading, and particle size distributions, and the type of photoinitiator.⁵³ The intrinsic color of esthetic materials may change when materials are aged under various physical-chemical conditions such as thermal changes and humidity.⁵⁴ External discoloration, which can be caused by oral habits such as tobacco use and certain dietary patterns, along with bad oral hygiene and the adsorption or absorption of water-soluble stains throughout the resin matrix,^{55,56} can be easily removed by polishing. However, internal discoloration is irreversible.⁵⁷

The color changes of composite materials can be determined both visually and by specific instruments.^{55,56} The methodology used in the present study was in accordance with previous studies that used spectrophotometry and the CIE L*a*b* coordinate system, which is a recommended method for dental purposes.^{21,58} The CIE L*a*b* coordinate system was chosen to evaluate the color variation (ΔE) because it is well suited for the determination of small color changes and has advantages such as repeatability, sensitivity, and objectivity.^{58,59} In the present study, changes in color after 10,000 thermocycles in seven different composites, applied in three groups, were evaluated. The mean ΔE ranged between 1.28-2.34 for Group 1, 1.1-2.53 for Group 2, and 1.45-3.43 for Group 3. Studies have shown that a value of $\Delta E > 1$ is visually perceptible when observing the color differences in esthetic restorations, whereas $\Delta E > 3.3$ was found to be the critical value for the visual perception of the restoration.⁶⁰ The data in the present study indicated that all composites in the three application groups had lower ΔE values than the acceptable threshold level of 3.3, except for the Filtek Silorane ($\Delta E = 3.43$) in Group 3. This might be due to the different monomer composition of the Filtek Silorane and the modeling resin. Distinct from the predominant radical polymerization initiation in conventional methacrylate-based composites, the silorane matrix is formed by the cationic ring-opening polymerization of silorane monomers. The "silorane" molecule represents a hybrid that is made of both siloxane and oxirane structural moieties.³

The color changes in Groups 1 and 2 after thermocycling were small, and these values were statistically significant and different among the composites tested. However, none would reveal a clinically discernible color shift. Thermocycling in distilled water is a method to determine the internal discoloration of resin composites because thermal change can also induce physicochemical reactions in the composites. Lee and Lee evaluated the changes in the optical properties of eight resin composites in a total of 41 shades after 5,000 thermocycles. They found that the color changes in 39 shades of the composites were below the acceptable threshold level of 3.3; the exceptions were shades A2 and A3 of Filtek Z350.²⁰ In general, thermocycling results in color changes of resin composites within clinically acceptable limits.⁶¹ In addition to the number of thermocycles, other factors such as the dwell time, the interval between dwells, the condition of the solution for dwelling, and the temperature range could change the effects of thermocycling.²⁰

In the present study, Filtek Ultimate showed the lowest color change, and Clearfil Majesty Esthetic showed the highest color change in Group 1. There was no significant difference between Filtek Ultimate, GrandioSO, and Gradia Direct Posterior in Group 1. In Group 2, the lowest color change was observed significantly in Filtek Ultimate specimens, except for the Aelite LS Posterior specimens, and the highest color change was observed in the Filtek Silorane and Clearfil Majesty Esthetic specimens. The degree of color changes after thermocycling varied among the resin composites tested. The differences in the resin matrix, such as its monomers, the concentration/type of the activators, the initiators, the inhibitors, and the oxidation of unreacted carbon-carbon double bonds and fillers, might have affected the color stability of the tested composites. The color and translucency changes of eight different composites after 2,000 thermocycles were evaluated: ΔE ranged between 0.4–1.3, and the lowest color changes were observed in Filtek Supreme and Tetric Ceram specimens.⁶¹ Lee and Lee evaluated the color changes of A2 shade Grandio, Gradia Direct Posterior, and Filtek Supreme after 5,000 thermocycles and found ΔE values of 1.6, 1.7, and 2.3, respectively.²⁰

Previous studies used different aging protocols and storage mediums to evaluate the color changes of dental composites.^{53,60} After 1 week storage in distilled water, Clearfil Majesty Esthetic and Filtek Supreme XT, a similar material to Filtek Ultimate, showed small color changes. The contradictory result for Clearfil Majesty Esthetic may be explained by differences in the working method. The 1 mm thick samples in the study were light cured for 40 seconds at 3,000 mW/cm² to eliminate any possible influence of unreacted camphorquinone.⁶² Çelik et al. calculated the color changes of 10 resin composites after 30 days storage in distilled water. They reported that the color change of Filtek Supreme was below the acceptable threshold level of 3.3, whereas Clearfil Majesty Esthetic exhibited a greater color change.⁵⁸ It has been reported that Filtek Supreme, Clearfil Majesty Posterior, Clearfil APX, and Filtek Z250 showed clinically acceptable color changes after 1 month storage in distilled water.⁵⁶ However, after 6 months, these composite materials (except Clearfil Majesty Posterior) demonstrated clinically perceptible color changes ($\Delta E > 3.3$). Erdemir et al. associated these findings to water sorption by the organic matrix over time.⁵⁶ When we compared the color changes of the composites in Groups 1 and 2, there were significant differences between the Aelite LS Posterior, Aelite All Purpose Body, and Filtek Silorane specimens. The null hypothesis that the use of modeling resin does not affect the color of composites was partially accepted. In Group 2, the color changes of Aelite LS Posterior and Aelite All Purpose Body were lower than in Group 1. This might be due to the composition of the modeling resin, which consisted of UDMA (urethane dimethacrylate), Bis-EMA (ethoxylated bisphenol-A dimethacrylate), and amorphous silica (30% by weight). These components might have been incorporated into the chemical structure of the composites, which could have enhanced the color stability. Additionally, this discrepancy in the chemical structure between the Silorane and the modeling resin might explain the higher discoloration in Group 2. On the other hand, the Filtek Silorane and Filtek Ultimate specimens in Group 3 showed significantly higher discoloration than in Group 1 and Group 2. When a dental resin-based composite material is cured against a polyester matrix strip or a glass cover, a resin-rich layer forms on the upper surface of the resin-based composite. The resin-rich layer forms during placement of the matrix strip which flattens the surface of the resin-based composite and, therefore, forces the filler particles further from the surface. This has been suggested to result in decreased filler loading in the uppermost layer

of the resin-based composite,⁶³ which is more susceptible to discoloration.⁶⁴ This increased discoloration susceptibility in the resin-rich layer can be tentatively explained by a decreased level of polymerization, resulting from trapped oxygen between the surface of the resin-based composite and the matrix strip, thus increasing discoloration.^{64,65}

CONCLUSION

In summary, this study showed some of the changes in the surface hardness, roughness, and color of resin composites caused by modeling resin and thermocycling. Within the limitations of the present study, we conclude that:

- 1 Microhardness, surface roughness, and color stability of the tested composites showed differences due to the variations in their polymer matrices and filler types. The smoothest surface and lowest hardness were produced with a polyester matrix strip in all the composite materials.
- 2 Modeling resin did not affect: (1) the microhardness of the composites, except for GrandioSO and Gradia Direct Posterior; (2) the surface roughness, except for GrandioSO, Filtek Silorane, and Aelite All Purpose Body; and (3) the color stability, except for Filtek Silorane. The adverse effects expected from the use of modeling resin could be removed by finishing and polishing of the restorative materials.
- 3 Modeling resin affected the GrandioSO more than the other materials by the three parameters evaluated in the study.
- 4 The in vitro aging with thermocycling affected the microhardness; however, it did not affect the surface roughness. It was also noted that thermocycling with distilled water did not influence the color stability of the tested composites.

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