# Flexural Properties and Polished Surface Characteristics of a Structural Colored Resin Composite

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#### Clinical Relevance

Polishing the structural colored resin composite Omnichroma with an aluminum oxide flexible disk after finishing with a tungsten carbide bur significantly improved its surface properties when compared with the other methods.

#### **SUMMARY**

Objective: The aim of this study was to determine the flexural properties and surface characteristics of a structural colored resin composite after different finishing and polishing methods, in comparison to those of conventional resin composites.

Methods and Materials: A structural color resin composite, Omnichroma (OM, Tokuyama Corp, Chiyoda City, Tokyo, Japan), and two comparison resin composites, Filtek Supreme Ultra (FS, 3M, St Paul, MN, USA) and Tetric EvoCeram

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Hiroyasu Kurokawa, DDS, PhD, Department of Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan (TE, Ivoclar Vivadent, Schaan, Liechtenstein), were used. The flexural properties of the resin composites were determined in accordance with the ISO 4049 specifications. For surface properties, 70 polymerized specimens of each resin composite were prepared and divided into seven groups of 10. Surface roughness (Sa), gloss (GU), and surface free energy (SFE) were investigated after the following finishing and polishing methods. Three groups of specimens were finished with a superfine-grit diamond bur (SFD), and three with a tungsten carbide bur (TCB). After finishing, one

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of the two remaining groups was polished with a one-step silicone point (CMP), and the other with an aluminum oxide flexible disk (SSD). A group ground with SiC 320-grit was set as a baseline.

Results: The average flexural strength ranged from 116.6 to 142.3 MPa in the following order with significant differences between each value: FS > TE > OM. The average E ranged from 6.8 to 13.2 GPa in the following order with significant differences between each value: FS > TE > OM. The average R ranged from 0.77 to 1.01 MJ/mm<sup>3</sup> in the following order: OM > FS > TE. The Sa values of the OM groups polished with CMP and SSD were found to be significantly lower than those of the other resin composites, regardless of the finishing method. The GU values appeared to be dependent on the material and the finishing method used. The OM specimens polished with SSD showed significantly higher GU values than those polished with CMP. Most of the resin composites polished with SSD demonstrated significantly higher yS values compared to the other groups. Extremely strong negative correlations between Sa and GU in the combined data from the three resin composites and each resin composite and between Sa and yS in the OM specimens were observed; GU showed a strong positive correlation with  $\gamma$ S in the same material.

Conclusion: These findings indicate that both flexural and surface properties are material dependent. Furthermore, the different finishing and polishing methods used in this study were observed to affect the Sa, GU, and SFE of the resin composites.

## INTRODUCTION

In order to achieve esthetic dental restorations, the color matching ability of resin composites used to restore teeth is considered to be of utmost importance. However, in some instances, it is difficult to color match the restoration to the tooth as the latter's color depends on the location and the optical properties of the tooth substrate. In order to mimic the color of the restored tooth, different shades of resin composites are mixed using the layering technique. This technique consists of several filling steps, which require a certain amount of clinical skill to obtain the desired color.

It is important to take into account both the esthetic and mechanical properties of the resin composite restoration and its durability in the oral environment.<sup>5-7</sup> The mechanical properties of a resin composite are

closely related to its fracture resistance and wear behavior. Sp. Changes in the surface properties of a restoration lead to staining, plaque accumulation, restoration degradation, and gingival inflammation. Therefore, to obtain the desired esthetics and at the same time ensure its longevity, the polishing ability of the resin composite should be determined. Ishii et al. have examined the surface properties of resin composites and reported that the surface free energy (SFE) might reflect some of the important characteristics of the material, as with surface topography evaluation.

Structural color is thought to be a chromogenic phenomenon in which the visible color is derived from the structural features of the substance at a scale comparable to or smaller than the wavelength of visible light.13 In contrast to colorants such as pigments or dyes, substances that produce structural color do not have a color of their own, but they can display visible colors owing to light interferences generated by their ultrastructure.14 These materials can be classified into two categories, iridescent and non-iridescent, in accordance with their optical properties. The iridescent color arises from photonic crystals, which have periodic structures with regular lengths of the order of the wavelength of visible light.<sup>15</sup> On the other hand, non-iridescent materials have angle-independent characteristics wherein the color impression is the same for different illuminations and observation angles. 16

A new type of resin composite has been developed based on the structural color concept and the bottomup nanotechnology used in making unique filler particles.<sup>17</sup> These fillers are in the form of spherical particles, having an average diameter of 260 nm, which produce the yellow to red color shades required to match the natural tooth color. 17,18 The refractive index of the uniformly sized spherical filler exceeds that of the resin matrix, leading to the expression of a stronger structural color due to the scattering of the incident light. 19 Using the structural colored resin composite has been seen to simplify the shade matching process and render it more cost-effective as it only requires a single universal shade to be used. Saegusa and others, 17 who evaluated the color-matching ability of the structural colored resin composite and compared it with conventional ones using artificial teeth, have confirmed the excellent color-matching ability of the structural colored material. In addition, they suggested that these might have the potential to fix any color mismatch caused by using conventional materials. However, little information is available about the mechanical and surface properties of this structurally colored resin composite. As the color of the composite is achieved through modification of the filler structure, there is a

possibility that these properties will differ from those of conventional resin composites, and the properties of these composites must be taken into account in clinical usage. Therefore, it is important to measure them.

This study aims to determine the mechanical properties of the structural colored resin composite based on a flexural strength test, which should help to understand the effects of different finishing and polishing procedures on the surface properties of this material. The surface properties were then examined in terms of surface free energy (SFE), surface roughness, and gloss measurements; morphological assessments were made via scanning electron microscopy (SEM). The null hypotheses were as follows: 1) the flexural properties of the structural colored resin composite would not differ from those of conventional resin composites, and 2) the surface properties of the structural colored resin composite would not be affected by the finishing and polishing methods and would not differ from those of the conventional resin composites.

#### **METHODS AND MATERIALS**

#### Study Materials

The structural colored resin composite, Omnichroma (OM, Tokuyama Dental, Tokyo, Japan), and two other conventional resin composites, Filtek Supreme Ultra (FS, 3M Oral Care, St. Paul, MN, USA) as a nanofilled resin composite and Tetric EvoCeram (TE, Ivoclar Vivadent, Schaan, Lichtenstein) as a nanohybrid resin composite, were used in this study (Table 1). A halogen-quartz-tungsten curing unit (Optilux 501; SDS Kerr, Danbury, CT, USA) was used to avoid any effects of the reported non-uniformity of light-emitting diode curing units. The light irradiance (above 600 mW/cm²) of the curing unit was confirmed using a dental radiometer (Model 100; SDS Kerr).

#### Flexural Strength Test

The flexural properties of the tested resin composites were examined as per the ISO 4049 specifications.<sup>21</sup> A transparent polymer matrix tape (Matrix tape and Dispenser, 3M Oral Care) was placed on a glass slide, and a stainless-split mold (25 × 2 × 2 mm) was positioned on it. The resin composites were then inserted into the mold and covered with the matrix tape. Pressure was manually applied to the composites using another glass plate. The mold was then positioned on a glass slide. The middle third of the specimen was irradiated for 30 seconds after which the remaining thirds were irradiated for 30 seconds each. The polymerized specimen was removed from the mold, and all the sides were polished using a 1200-grit SiC (Fuji Star type

DCCS; Sankyo Rikagaku, Saitama, Japan) paper. The prepared specimens were stored in distilled water at 37 °C in the dark for 24 hours. Twelve specimens per test resin composite were subjected to the three-point bending test (span length = 20 mm) using a universal testing machine (model 5500R; Instron, Canton, MA, USA) at a crosshead speed of 1.0 mm/min until breaking point. The flexural strength ( $\sigma_F$ ) and elastic modulus (E) were determined from the stress-strain curve created using built-in computer software (Bluehill ver. 2.5; Instron) connected to the testing machine. In addition, the modulus of resilience (R) was calculated using the following equation<sup>22</sup>:

$$R = \sigma_E^2/2E$$

# Specimen Preparation for Surface Property Evaluation

The specimens were prepared in cylindrical Teflon molds (height = 2.0 mm, diameter = 10.0 mm). One end of the mold was sealed using the matrix tape, and the resin paste was condensed into the mold from the open end. The open end was then covered with the matrix tape, and pressure was applied, followed by light irradiation for 30 seconds with a curing unit. The polymerized specimen was removed from the mold and was later stored in the dark at 25 °C for 24 hours before finishing and polishing. Seventy specimens were prepared for each resin composite.

### **Finishing and Polishing Procedures**

The specimens were randomly divided into seven groups (n=10 each). All specimens in the seven groups were ground flat with a 320-grit SiC paper under tap water. The specimens in three groups were finished using a superfine-grit diamond bur (SFD; SF102R, ISO #017; Shofu, Kyoto, Japan), and those from another three groups were finished using a tungsten carbide bur (TCB; FG7714, ISO #014; Kerr, Orange, CA, USA). Those in the remaining group were ground with SiC 320-grit grinding paper and set as the baseline (BAS).

The finishing procedures were performed using a high-speed handpiece (TwinPower Turbine; J. Morita Mfg, Kyoto, Japan) with water spray. Light hand pressure was applied in multiple directions, and the burs were changed after five uses. Three specimens, each from the three groups (SFD, TCB, and BAS), were set aside for measurements. Two specimens, one each from the SFD and TCB groups, were polished using the one-step point-type polishing system comprising a silicone polisher impregnated with a diamond particle (CMP; CompoMaster, Shofu). Likewise, two specimens (one each from the SFD and TCB groups)

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Code	Resin Composite (Lot No.)	Main Components	Type of Resin Composite (Filler Content)	Manufacturer
ОМ	Omnichroma (18B28)	UDMA, TEGDMA, silica-zirconia filler (260 nm)	Supra-nano filled (79 wt%, 68 vol%)	Tokuyama Dental, Tokyo, Japan
FS	Filtek Suprem Ultra (Shade; A2: N870378)	bis-GMA, UDMA, TEGDMA, bis-EMA, PEGDMA, zirconia/silica clusters (0.6-10 µm) silica (20 nm silica filler), zirconia (4-11 nm)	Nano filled (78.5 wt%, 63.3 vol%)  3M Oral Care, St. Paul, MN, U	
TE	Tetric EvoCeram (Shade; A2: T21387)	bis-GMA, UDMA, bis-EMA barium glass, ytterbium trifluoride, mixed oxide and pre-polymer (range: 40 nm-3 µm, average: 550 nm)	Nano hybrid (75-76 wt%, 53-55 vol%)	Ivoclar Vivaden, Schaan, Lichtenshtein
Code	Finishing Bur	Model	Manufacturer	
SFD	Super fine grit diamond bur	SF102R (ISO#017, particle size less than 25 µm)	Shofu, Kyoto, Japan	
TCB	Tungsten carbide bur	FG7714 (ISO#014, 12 blades)	Kerr, Orange, CA, US	SA
Code	Polishing System	Model	Manufacturer	
CMP	Compomaster (one step system)	One step diamond polisher (6 µm) silicone base (25%), diamond  particles (75%)	Shofu	
SSD	Super-Snap (multi step system)	SSD G: Fine, green: $\sigma$ 12-mm disk; 20- µm aluminium oxide SSD R: Superfine, red: $\sigma$ 12-mm disk; 7-µm aluminium oxide	Shofu	

Abbreviations: UDMA, urethane dimethacrylate, TEGDMA: triethyleneglycol dimethacrylate; bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy) phenyl) propane; bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; PEGDMA: poly ethylene glycol diether dimethacrylate.

were polished using the multistep polishing system comprising aluminum oxide-impregnated disks (SSD; Super-Snap Rainbow Technique Kit, Shofu). All the polishing procedures were performed using a slow-speed handpiece (TorqTech CA-DC; J. Morita Mfg.) at 5,000 rpm and contact pressure of 1.0 N, which was monitored with a digital balance (AT200; Mettler-Toledo, Greifensee, Switzerland) underneath the specimen. All specimens were finished and polished by a single operator to reduce variability between samples.

The final groups of specimens from each type of composite were as follows: ground with 320-grit SiC paper (BAS); finished with SFD (SFD); finished with TCB (TCB); SFD polished with CMP (SFD+CMP); TCB polished with CMP (TCB+CMP); SFD polished

with SSD (SFD+SSD); and TCB polished with SSD (TCB+SSD).

## **Measurement of Surface Area Roughness**

Before the Sa measurements, the finished and polished specimens were cleaned with distilled water in an ultrasonic bath (Quantrex 301; L&R Ultrasonics, Kearny, NJ, USA) for 3 minutes and dried in oil-free air. The surfaces of all the specimens were observed using a three-dimensional laser scanning microscope (LSM, VK-8700; Keyence, Osaka, Japan). The spectral maximum of the excitation light was observed to be at 658 nm; the intensity of the excitation light and the amplification of the photomultiplier were kept constant during the observation period. Profilometric measurements were conducted in a region (1.0 mm ×

1.0 mm) at the center of the specimen. The *Sa* value was obtained using the built-in computer software (VK Analyzer; Keyence) connected to the LSM. Three measurements were taken, and the means were then determined for each group.

#### **Measurement of Surface Gloss**

The GU of each group was measured using a gloss meter (GM-26D; Murakami Color Research Laboratory, Tokyo, Japan) after the *Sa* measurements were obtained. The gloss meter was calibrated with a blackboard of known gloss value. Subsequently, the GU measurements were taken with an incident angle of 60 degrees, and the values for each group were determined.

# Measurement of Surface Free Energy (SFE)

The contact angles of the specimens were measured in order to evaluate the surface characteristics. The contact angles of the three test liquids with known SFE parameters, 1-bromonaphthalene, di-iodomethane, and distilled water were measured using a contact angle meter (Drop Master DM500; Kyowa Interface Science, Saitama, Japan) with a built-in charge-coupled device camera. <sup>12</sup> The equilibrium contact angle  $(\theta)$  of each test liquid on the specimens was then measured using the sessile-drop method at room temperature (23±1°C). Sessile liquid drops (1.0 μL) were dispensed with a micropipette, and the surface characteristics were calculated based on the fundamental concepts of wetting. The Young-Dupré equation has associated contact angle to the work of adhesion for a solid (S) and liquid (L) that are in contact  $(W_{SI})$ , the interfacial free energy between the S and the L  $(\gamma_{SL})$ , and the free energies of the L and S ( $\gamma_L$  and  $\gamma_S$ , respectively) and is expressed as follows:

$$W_{\rm SL} = \gamma_{\rm L} + \gamma_{\rm S} - \gamma_{\rm SL} = \gamma_{\rm L} (1 + \cos\theta).$$

An extension of the Fowkes equation following the Kitazaki-Hata approach<sup>23</sup> yields the following equations:

$$\gamma_{\rm SL} = \gamma_{\rm L} + \gamma_{\rm S} - 2 \; \big(\gamma_{\rm L}^{\rm d} \; \gamma_{\rm S}^{\rm d}\big)^{1/2} - 2 \; \big(\gamma_{\rm L}^{\rm p} \; \gamma_{\rm S}^{\rm p}\big)^{1/2} - 2 \; \big(\gamma_{\rm L}^{\rm h} \; \gamma_{\rm S}^{\rm h}\big)^{1/2},$$

$$\gamma_{L}=\gamma_{L}{}^{d}+\gamma_{L}{}^{p}+\gamma_{L}{}^{h},\,\gamma_{S}=\gamma_{S}{}^{d}+\gamma_{S}{}^{p}+\gamma_{S}{}^{h},$$

where,  $\gamma^d$ ,  $\gamma^p$ , and  $\gamma^h$  are the components of the SFE ( $\gamma$ ) arising from the dispersion force, the polar (permanent and induced) force, and the hydrogen-bonding force, respectively. The  $\theta$  values were determined for the three test liquids, and the surface-energy parameters of the treated resin composites were obtained using

the above equations using add-on software (FAMAS, Kyowa Interface Science).

#### **SEM Observations**

To visualize the morphological features of the fillers in the resin composites, the polymerized specimens were polished using abrasive disks and a series of diamond pastes (DP-Paste; Struers, Ballerup, Denmark) down to a particle size of 0.25 μm. The mirror-polished surfaces were subjected to argon-ion beam etching (IIS-200ER; Elionix, Tokyo, Japan) for 40 seconds, with the ion beam directed perpendicular to the polished surface (accelerating voltage = 1 kV, ion current density = 0.4 mA/cm²). The surfaces were then coated with a thin film of Au in a vacuum evaporator (Quick Coater Type SC-701; Sanyu Denchi, Tokyo, Japan). Images were obtained using an SEM (FE-8000; Elionix, Vienna, Austria) at an operating voltage of 10 kV at magnifications of 5,000× and 30,000×.

In order to understand the surface texture of the finishing and polishing instruments before use, Aucoated surfaces were observed by SEM at magnifications of 50× and 1,000×. Furthermore, SEM observations of representative specimens from the three resin composites were performed after the different finishing and polishing procedures. The coated surfaces were visualized by SEM at a magnification of 2,500×.

#### **Statistical Analysis**

A power analysis has indicated that at least 10 samples for flexural properties and eight samples for surface properties and SFE measurements were needed. Thus, this study was initially performed with 12 specimens for flexural property testing and 10 for other measurements. After gathering the data, *post hoc* power tests were performed using two statistical software systems (G Power calculator and SigmaPlot ver. 13.0; Systat Software, Inc, Chicago, IL, USA).

After confirming that the distribution was normal using the Kolmogorov-Smirnov test, data from each experiment were subjected to the analysis of variance (ANOVA) test; this was followed by the Tukey honest significant difference test at a significance level of 0.05. Significant differences in flexural properties were observed using the one-way ANOVA test, whereas differences in surface roughness, surface gloss, and total SFE were assessed using the two-way ANOVA test; the type of resin composite and the polishing method were used as factors for the two-way ANOVA. The statistical analyses were carried out using a commercially available statistical software program (SigmaPlot ver. 13.0).

The Pearson product-moment correlation coefficient was also used to perform pairwise comparisons, in

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Table 2: Flexural Properties of the Tested Resin Composites <sup>a,b</sup>							
	Flexural Strength; $\sigma_F$ Elastic modulus; $E$ Resilience; $R$						
(MPa) (GPa) (MJ/mm³)							
OM	116.6 (4.8) c	6.8 (0.6) c	1.01 (0.11) a				
FS	142.3 (5.8) a	13.2 (0.6) a	0.78 (0.06) b				
TE	125.3 (5.2) b	9.3 (0.8) b	0.85 (0.11) b				

Abbreviations: FS, Filtek Supreme Ultra; OM, Omnichroma; TE, Tetric EvoCeram.

order to understand the relationships between the tested parameters of the surface properties (SigmaPlot version 13.0, Systat).

#### **RESULTS**

#### **Flexural Properties**

The flexural properties of the resin composites are presented in Table 2. The average  $\sigma_F$  ranged from 116.6 to 142.3 MPa in the following order with significant differences between each value: FS > TE > OM. The average E ranged from 6.8 to 13.2 GPa in the following order with significant differences between each value: FS > TE > OM. FS showed significantly higher E and  $\sigma_F$  values compared to the other resin composites. The average E ranged from 0.78 to 1.01 MJ/mm³ in the following order: OM > TE > FS. Although no significant difference was determined between FS and TE, OM showed a significantly higher E than the other resin composites, in contrast to  $\sigma_F$  and E.

#### **Surface Area Roughness**

The influence of different finishing and polishing methods on the *Sa* values is shown in Table 3. A two-way ANOVA revealed that the finishing and

polishing methods and the type of resin composite can significantly affect the Sa values (p<0.001). The two-way interaction between these two factors was also found to be significant (p<0.001). The groups finished with SFD showed significantly higher Sa values when compared with the other groups, regardless of the type of resin composite used. On the other hand, the TCB+SSD group showed the lowest Sa values compared to the other groups, and significant differences in FS and TE were observed between the TCB+SSD group and the other groups. In addition, most groups polished with SSD showed significantly lower Sa values compared to the groups polished with CMP, regardless of the type of finishing used (SFD or TCB).

Among the tested resin composites, no significant differences were observed between the BAS and TCB groups. However, the *Sa* values of the OM groups polished with CMP and SSD were found to be significantly lower than those of the other resin composites, regardless of the finishing method.

#### Gloss

The effects of the different finishing and polishing methods on the GU values are shown in Table 4. The two-way ANOVA test revealed that both factors, ie, finishing and polishing methods and the type of resin

Table 3: Influence of Finishing and Polishing Methods on Surface Area Roughness (Sa, µm) <sup>a,b</sup>							
	BAS	SFD	TCB	SFD+CMP	TCB+CMP	SFD+SSD	TCB+SSD
ОМ	1.32 (0.02) aB	1.51 (0.02) bA	1.05 (0.02) aC	0.92 (0.03) cD	0.85 (0.01) cE	0.78 (0.02) cF	0.78 (0.02) cF
FS	1.31 (0.02) aB	1.60 (0.05) aA	1.05 (0.02) aC	1.00 (0.02) bD	0.99 (0.04) bD	0.93 (0.04) bE	0.88 (0.02) bF
TE	1.30 (0.01) aB	1.46 (0.03) cA	1.03 (0.02) aCD	1.07 (0.05) aC	1.01 (0.03) aD	1.00 (0.02) aD	0.85 (0.04) aE

Abbreviations: BAS, baseline; CMP, diamond particle; FS, Filtek Supreme Ultra; OM, Omnichroma; SFD, superfine grit diamond bur; SSD, aluminum oxide-impregnated disks; TCB, tungsten carbide bur; TE, Tetric EvoCeram.

<sup>a</sup>Values in parentheses indicate standard deviation.

<sup>b</sup>Same small case letter in vertical columns indicates no difference at 5% significance level. Same capital letter in horizontal rows indicates no difference at 5% significance level.

<sup>&</sup>lt;sup>a</sup>Values in parentheses indicate standard deviation.

<sup>&</sup>lt;sup>b</sup>Same small case letter in vertical columns indicates no difference at 5% significance level.

Table 4: Influence of Finishing and Polishing Methods on Surface Gloss (GU) <sup>a,b</sup>							
	BAS	SFD	ТСВ	SFD+CMP	TCB+CMP	SFD+SSD	TCB+SSD
ОМ	3.7 (0.2) bF	1.8 (0.1) cG	22.9 (0.6) aE	34.1 (1.0) bD	44.3 (0.4) bC	50.6 (1.0) aB	53.3 (1.2) aA
FS	7.4 (0.2) aF	2.7 (0.1) aG	12.4 (0.2) bE	53.7 (0.6) aB	65.2 (0.2) aA	31.8 (0.3) bD	40.7 (0.5) cC
TE	3.4 (0.1) cF	2.0 (0.1) bG	22.6 (0.2) aE	35.5 (0.4) bC	44.1 (0.8) bB	32.8 (0.8) bD	48.5 (0.3) bA

Abbreviations: BAS, baseline; CMP, diamond particle; FS, Filtek Supreme Ultra; OM, Omnichroma; SFD, superfine grit diamond bur; SSD, aluminum oxide-impregnated disks; TCB, tungsten carbide bur; TE, Tetric EvoCeram.

composite used, have significantly affected the GU (\$\rho<0.001). In addition, a significant two-way interaction between these two factors was observed (p<0.001). The GU values of the BAS, SFD, and TCB groups were significantly lower than those of the groups polished with CMP and SSD. Furthermore, the specimens in the groups finished with TCB had significantly higher GU values compared to those finished with SFD. In addition, the specimens finished with TCB demonstrated significantly higher GU values when compared with those finished with SFD. The GU values also appeared to be dependent on the material and the finishing method used. The OM specimens polished with SSD showed significantly higher GU values than those polished with CMP. However, the FS specimens that had been polished with SSD showed significantly lower GU values compared to those polished with CMP, regardless of the finishing method. On the other hand, the TE specimens polished with SSD showed a different trend in their GU values when compared with the specimens finished with SFD and TCB.

#### Surface Free Energy

The effects of the different finishing and polishing methods on the SFE values are shown in Table 5

and Figure 1. A two-way ANOVA revealed that the finishing and polishing method, as well as the type of resin composite used, significantly influenced the  $\gamma_s$  value (p<0.001). The two-way interaction between the factors was also found to be significant (p<0.001).

The specimens in most of the groups polished with SSD showed significantly higher  $\gamma_s$  values compared to those in the other groups. The polished OM specimens showed significantly higher  $\gamma_s$  values compared to those in the BAS and other finished groups. In the case of the FS specimens, although the specimens in the TCB + SSD group presented with significantly higher  $\gamma_s$  value than those in the other groups, no significant differences were observed among the specimens in the other groups. The  $\gamma_s$  values of the TE specimens polished with CMP were found to be lower than those in the other groups. FS tended to have higher  $\gamma_s$  values when compared with the other resin composites, regardless of the finishing and polishing methods.

With regard to each component of the  $\gamma_s$  (Figure 1), although the dispersion force  $(\gamma_s^d)$  was constant at approximately 40 mN·m<sup>-1</sup> in all the groups, variations in the polar force  $(\gamma_s^p)$  and hydrogen-bonding force  $(\gamma_s^h)$  were observed among the groups. However, all three resin composites tended to have higher  $\gamma_s^p$  values when

Table 5: Influence of Finishing and Polishing Methods on Total SFE $(\gamma_g)^{a,b}$							
	BAS	SFD	ТСВ	SFD+CMP	TCB+CMP	SFD+SSD	TCB+SSD
ОМ	45.2 (2.0) bC	43.5 (1.9) bD	44.6 (2.2) cCD	48.4 (1.8) bB	48.2 (0.9) bB	50.8 (1.2) aA	50.6 (1.2) bA
FS	51.2 (1.8) aB	51.8 (1.5) aB	51.0 (0.9) aB	51.2 (1.1) aB	51.7 (1.5) aB	51.4 (1.2) aB	53.9 (2.2) aA
TE	49.4 (1.4) aBC	50.5 (1.6) aAB	48.2 (1.4) bCD	47.0 (1.5) bD	46.6 (1.2) cD	51.8 (1.0) aA	51.4 (1.6) bA

Abbreviations: BAS, baseline; CMP, diamond particle; FS, Filtek Supreme Ultra; OM, Omnichroma; SFD, superfine grit diamond bur; SFE, surface-free energy; SSD, aluminum oxide-impregnated disks; TCB, tungsten carbide bur; TE, Tetric EvoCeram.

<sup>&</sup>lt;sup>a</sup>Values in parentheses indicate standard deviation.

<sup>&</sup>lt;sup>b</sup>Same small case letter in vertical columns indicates no difference at 5% significance level. Same capital letter in horizontal rows indicates no difference at 5% significance level.

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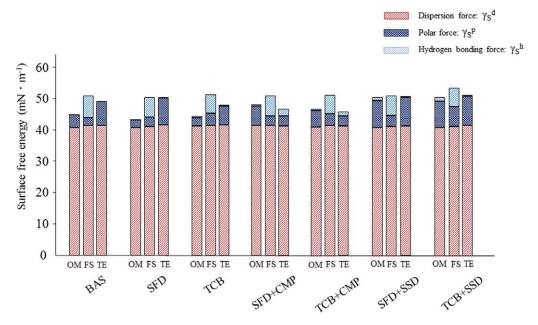


Figure 1. The total SFE  $(\gamma_s)$  values and the three corresponding parameters of the resin composites. Abbreviations:  $\gamma_s$  total surface free energy; BAS, baseline; CMP, diamond particle; FS, Filtek Supreme Ultra; GU, surface gloss; OM, Omnichroma; SFD, superfine grit diamond bur; SSD, aluminum oxide-impregnated disks; TCB, tungsten carbide bur; TE, Tetric EvoCeram.

polished with SSD when compared with the other finishing and polishing methods. The FS specimens had higher  $\gamma_S^h$  components than the other resin composites, regardless of the finishing and polishing methods.

# Interrelations Between the Tested Parameters of the Surface Properties

The Pearson product-moment correlation coefficient (r) and p values for the relations between the tested parameters of the surface properties are presented in Table 6. Extremely strong negative correlations between Sa and GU in the combined data from the three resin composites and each resin composite and between Sa and  $\gamma_s$  in the OM specimens were observed; GU showed a strong positive correlation with  $\gamma_s$  in the same material, and those correlations were revealed to be individually statistically significant. However, the other comparisons showed weak correlations or no correlations.

#### **SEM Observations**

SEM images of the mirror-polished surfaces after argon-ion etching are illustrated in Figures 2 through 4. The shapes, sizes, and distributions of the inorganic fillers are found to be resin composite dependent. OM consisted of nanosized spherical fillers (approximately 0.25  $\mu$ m) and round pre-polymerized fillers that employ the same nanosized spherical fillers (Figure 2). FS consisted of spheroidal aggregates (0.5-5.0  $\mu$ m)

of nanosized filler particles (Figure 3), whereas TE consisted of irregular fillers (0.5-2.0 µm) made up of nanosized filler particles that were packed into prepolymerized fillers at a high density (Figure 4).

Representative SEM images of the surfaces of the instruments are shown in Figure 5. SFD consisted of embedded irregular diamond particles that were less than 20 µm in size. On the other hand, TCB consisted of sharp uniform blades. The one-step polishing instrument CMP consisted of impregnated irregular diamond particles, and the interfaces between the particles and matrix appeared to be loose. In the case of the multiple polishing system, SSD, the sizes of the aluminum oxide particles were found to be varied in the different disks (SSD G and SSD R). The particles in SSD R were tightly packed when compared to those in CMP and SSD G.

Representative SEM images of the resin composite surfaces after the different finishing and polishing methods are shown in Figures 6-8. All the resin composites exhibited smoother surfaces after finishing with TCB when compared to those finished with SFD. For FS and TE, scratches and plucked-out fillers were observed following polishing with SFD. In particular, plucked-out aggregated fillers were seen in the FS specimens, whereas large glass fillers were observed in the TE specimens.

When comparing the different polishing methods (CMP and SSD), smoother surfaces were observed in the

Table 6: Relationships Between the Parameters of Surface Properties						
		GU	γ <sub>s</sub>			
C	ombined data of tl	nree resin compos	sites			
Sa	r	-0.847	-0.274			
Sa	p-value	<0.001	0.230			
GU	r		0.318			
GO	p-value		0.160			
ОМ						
Sa	r	-0.972	-0.897			
	p-value	<0.001	0.006			
GU	r		0.930			
	p-value		0.002			
FS						
Sa	r	-0.713	-0.265			
	p-value	0.072	0.566			
GU	r		0.222			
	p-value		0.632			
TE						
Sa	r	-0.921	0.026			
	p-value	0.003	0.956			
GU	r		-0.145			
	p-value		0.756			

Abbreviations:  $\gamma_{S'}$  total surface free energy; BAS, baseline; CMP, diamond particle; FS, Filtek Supreme Ultra; GU, surface gloss; OM, Omnichroma; r, correlation coefficient; Sa, surface roughness; SFD, superfine grit diamond bur; SSD, aluminum oxide-impregnated disks; TCB, tungsten carbide bur; TE, Tetric EvoCeram

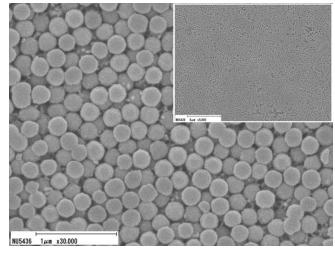


Figure 2. SEM images of resin composite surfaces after argon-ion etching Omnichroma (OM) – 5000× and 30,000×. Abbreviation: SEM, scanning electron microscopy.

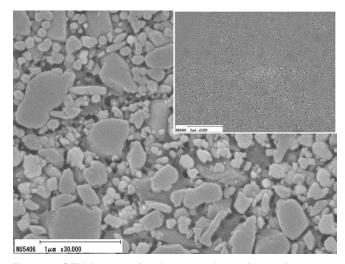


Figure 3. SEM images of resin composite surfaces after argonion etching. Filtek Supreme Ultra (FS) – 5000× and 30,000×. Abbreviation: SEM, scanning electron microscopy.

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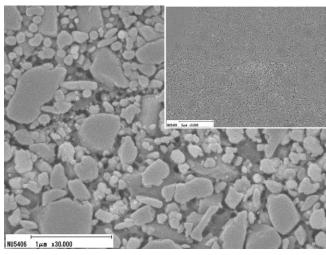


Figure 4. SEM images of resin composite surfaces after argon-ion etching Tetric EvoCeram (TE) – 5000× and 30,000×. Abbreviation: SEM, scanning electron microscopy.

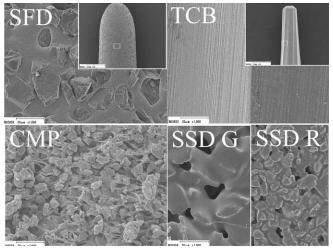


Figure 5. SEM images of resin composite surfaces after argon-ion etching. Representative SEM images of the instruments' surfaces. CMP, One-step diamond polisher, Compomaster (1,000x); SEM, scanning electron microscopy; SFD, Super fine grit diamond bur (50x and 1,000x); SSDG, Multistep polishing system, Super-Snap fine green (1,000x); SSDR, Multistep polishing system, Super-Snap super fine red (1,000x); TCB, Tungsten carbide bur (50x and 1,000x). Abbreviation: SEM, scanning electron microscopy.

SSD groups in the case of the OM and FS specimens, regardless of the finishing method. On the contrary, in the case of the TE specimens, there exists no difference in surface smoothness between the SSD and CMP groups.

#### DISCUSSION

Understanding the various properties of resin composites before clinical use will aid in selecting the optimum resin composite, which will in turn lead to ideal outcomes in the intraoral environment.<sup>7,8</sup> In this study, the flexural

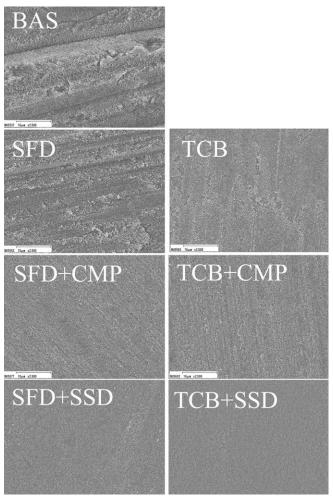


Figure 6. Representative SEM images of OM after different finishing and polishing methods. BAS, Ground with SiC #320 (2,500×); SEM, scanning electron microscopy; SFD, Finished with Super-fine grit diamond (2,500×); SFD+CMP, Polished with Compomaster after finishing with Super-fine grit diamond bur (2,500×); SFD+SSD, Polished with Super-Snap after finishing with Super-fine grit diamond bur (2,500×); TCB, Finished with Tungsten carbide bur (2,500×); TCB+CMP, Polished with Compomaster after finishing with Tungsten carbide bur (2,500×); TCB+SSD, Polished with Super-Snap after finishing with Tungsten carbide bur (2,500×). Abbreviation: SEM, scanning electron microscopy.

and surface properties of the structural colored resin composite OM were investigated and compared with those of the conventional resin composites FS and TE.

When considering the longevity of resin composite restorations in intraoral conditions, fracture-related material properties are important. Flexural strength testing is a well established standardized method, and it is preferred as a way to screen the mechanical properties of materials due to ease of testing. These values are helpful in comparing materials under controlled conditions and also provide some help

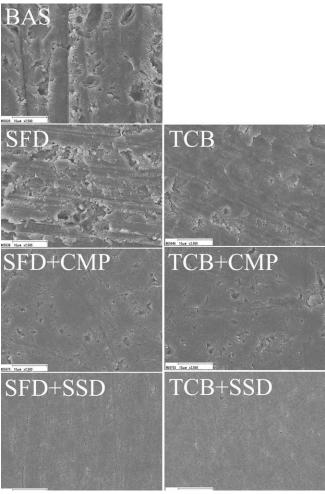


Figure 7. Representative SEM images of FS after different finishing and polishing methods. BAS, Ground with SiC #320 (2,500×); SEM, scanning electron microscopy; SFD, Finished with Super-fine grit diamond bur (2,500×); SFD+CMP, Polished with Compomaster after finishing with Super-fine grit diamond bur (2,500×); SFD+SSD, Polished with Super-Snap after finishing with Super-fine grit diamond bur (2,500×); TCB, Finished with Tungsten carbide bur (2,500×); TCB+CMP, Polished with Compomaster after finishing with Tungsten carbide bur (2,500×); TCB+SSD, Polished with Super-Snap after finishing with Tungsten carbide bur (2,500×). Abbreviation: SEM, scanning electron microscopy.

in predicting restoration longevity. The mechanical properties of resin composites are influenced by the resin monomers, filler characteristics, and surface treatment of the fillers. The findings of this study were found to be consistent with the results of the previous studies, which investigated the flexural properties of nanohybrid and nanofilled resin composites, including the resin composites used in this study. FS showed significantly higher  $\sigma_F$  and E values compared to TE in the current study. Thus, it can be speculated that the fine nanosized spherical fillers and the aggregated

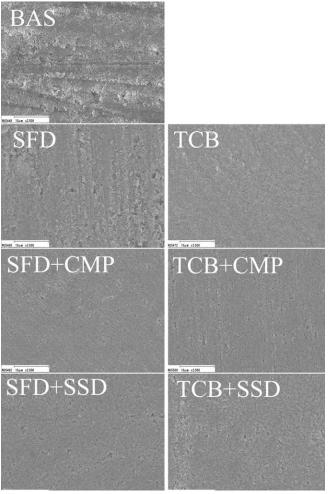


Figure 8. Representative SEM images of TE after different finishing and polishing methods. BAS, Ground with SiC #320 (2,500×); SEM, scanning electron microscopy; SFD, Finished with Super-fine grit diamond bur (2,500×); SFD+CMP, Polished with Compomaster after finishing with Super-fine grit diamond bur (2,500×); SFD+SSD, Polished with Super-Snap after finishing with Super-fine grit diamond bur (2,500×); TCB, Finished with Tungsten carbide bur (2,500×); TCB+CMP, Polished with Compomaster after finishing with Tungsten carbide bur (2,500×); TCB+SSD, Polished with Super-Snap after finishing with Tungsten carbide bur (2,500×). Abbreviation: SEM, scanning electron microscopy.

fillers with a wide range of sizes in FS might contribute to higher fracture resistance.

In general, materials with high flexural strength have high elastic moduli. <sup>26,27</sup> OM presented with significantly lower *E* values compared to the other materials despite having a higher filler content. Of the monomers used in the tested composites, 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl) propane (bis-GMA) has a relatively higher molecular weight and lower mobility compared to urethane dimethacrylate (UDMA) and triethyleneglycol dimethacrylate (TEGDMA).

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Izabela and others,28 who examined the mechanical properties of three common homopolymers, have reported that UDMA had a lower E than bis-GMA and TEGDMA. UDMA is the resin monomer used in OM. However, although the type of resin monomer used can influence the flexural properties of a resin composite, it is difficult to identify the exact resin monomer and its effects on  $\sigma_{E}$  and E due to the various types present in resin composites; moreover, the interactions between resin monomers and inorganic fillers may differ between composites. OM has been known to consist of uniformly sized spherical fillers with constant interparticle spacing. FS consists of non-aggregated silica (20 nm) and zirconia fillers (4 to 11 nm), whereas OM consists of silicazirconia fillers (260 nm). The fillers in OM were found to have greater interparticle spaces (Figures 2 and 3) than those in FS. Therefore, crack propagation in OM tended to be simple, leading to lower fracture resistance. On the other hand, R is thought to be the ability of a material to absorb energy when it is elastically deformed under external stress without failing.26 OM showed a significantly higher R value compared to the other resin composites. This may be attributed to the presence of UDMA, which acts as a very flexible backbone with weak hydrogen-bonding due to the urethane groups. Therefore, the first null hypothesis, which indicates that the flexural properties of OM would not differ from those of other resin composites, was rejected.

The surface characteristics of resin composites, in addition to the mechanical properties, must be understood to predict their longevity in the intraoral environment, because the surfaces of restorations are directly subjected to various degradation factors. 10,11 From the results of this study, the two factors, ie, finishing and polishing procedure and type of resin composite used, have significantly influenced the Sa values. In particular, the groups finished with SFD showed significantly higher Sa values compared to those finished with TCB, regardless of the type of resin composite. This result was in line with a previous study that investigated the surface roughness of bulk-fill resin composites after different finishing and polishing procedures.<sup>12</sup> This finding may be explained by the different surface textures of the finishing instruments (Figure 5) resulting in different finishing mechanisms. The tungsten carbide burs cut away the surface, whereas diamond burs grind the surface with many abrasive diamond particles.<sup>29,30</sup> SEM observations clearly showed that some filler particles were plucked from the surfaces in the case of SFD, whereas in the case of TCB polishing, the filler particles remained embedded in the surface, resulting in a smoother surface (Figures 6 and 7). Furthermore, the finishing methods have significantly affected the polished groups, because most of the specimens in the TCB group showed lower *Sa* values compared to those in the SFD group, regardless of whether CMP or SSD was used.

The multiple-step polishing system (SSD) created smoother surfaces compared with the one-step polishing system (CMP; Figures 6 through 8). This might be attributed to the distribution (Figure 5) and the hardness of the particles in the different polishing instruments. Watanabe and others<sup>31</sup> indicated that, although the one-step polishing system has the capacity to polish resin composites as effectively as multistep polishing systems, the smoothest surface was obtained with multistep polishing systems. Among the resin composites, OM has showed significantly lower Sa values compared to the other resin composites for all the polishing methods. It can be inferred that the superior polishability of OM is probably due to the uniform nanoscale spherical fillers and the constant interspaces between them.

The surface gloss of restorations has been considered an important parameter for esthetics.<sup>32</sup> Gloss is defined based on the degree of specular reflection of light.<sup>33</sup> The finishing and polishing methods and type of resin composite used have significantly affected the GU in the current study. The GU obtained after polishing is known to be dependent on the type of material and the finishing method used. Although the OM specimens polished with SSD showed significantly higher GU values compared to those polished with CMP, the opposite findings were observed in the FS specimens, regardless of the finishing method used. Cazzaniga and others<sup>34</sup> obtained similar results wherein FS specimens polished with aluminum oxide disks showed lower surface roughness compared to those polished with one-step rubber points; however, a significantly higher surface gloss was obtained when polished with the one-step rubber points. In general, surface roughness and gloss are thought to have a negative correlation. 35,36 Nonetheless, a smoother surface is not necessary to obtain a high surface gloss, and the relationship depends on the polishing procedures and materials used.34 Although 40 to 60 GU was identified as the typically desired gloss in an American Dental Association professional product review,37 Cook and Thomas38 reported that a finish and polish below 60 GU was generally considered as poor; the acceptable values lie between 60 and 70 GU. However, a systematic review on surface gloss reported that few investigations showed a gloss of over 60 GU, even when the final polishing was performed.<sup>32</sup> Therefore, if the clinically acceptable GU is assumed to be 40 to 60 GU, SSD groups for OM and CMP groups for FS may be acceptable.

The SFEs were determined to understand the surface changes wrought by the different finishing and polishing methods from the perspective of surface chemistry. The finishing and polishing methods and the type of resin composite used significantly influenced the  $\gamma_s$  in this study. Therefore, the second null hypothesis, which implies that the surface properties of OM would not be affected by the finishing and polishing methods and would not differ from the other resin composites, was rejected. For all the resin composites, the groups polished with SSD showed significantly higher γ<sub>s</sub> values when compared with the other groups. Fundamentally, the wettability of a substance increases with the increase in the γ<sub>s</sub> value.<sup>39,40</sup> In composite resins, inorganic fillers are hydrophilic, whereas the resin matrix is hydrophobic.<sup>12</sup> The increase in the  $\gamma_s$  values in the SSD groups may be attributed to the high level of inorganic filler exposure on the surface due to the polishing method. A flexible disk coated with aluminum oxide might be able to cut the filler particles effectively, leaving parts of the filler particles exposed at the surface. This process produces uniform surface conditions without the plucking of the fillers, improving its resistance to various degradation factors.

γ<sub>S</sub> represents the water and hydroxyl components of the substrate, whereas  $\gamma_s^p$  is related to the electronic and metallic interactions and the dipolar interactions. 39,40 Although the  $\gamma_S^{\ p}$  and  $\gamma_S^{\ h}$  values in each group showed wide variations, some trends were observed between the materials or the finishing and polishing methods. FS had a higher number of  $\gamma_s^h$  components than the other resin composites in all the groups. This trend is likely brought about by the different surface chemistries between the exposed fillers and the resin matrix. The reasons why FS showed higher  $\gamma_s^h$  might be related to the relatively higher vol% of inorganic filler components and the somewhat larger nanofiller clusters, in addition to the presence of relatively hydrophilic resin monomers. In a previous study, which investigated the water sorption of common homopolymers, the descending order of water sorption was reported as TEGDMA > bis-GMA > UDMA. 41 The study suggested that these differences could be attributed to the presence of hydrophilic ether linkages in TEGDMA, hydroxyl groups in bis-GMA, and urethane linkages in UDMA. Although the parts of resin monomers related to hydrophilicity might increase the  $\gamma_s^h$ , it is difficult to determine the details of the interactions between  $\gamma_S^p$  or  $\gamma_S^h$  and the inorganic fillers or resin matrix due to the complexity of the resin composites.

Regarding the interrelations between the tested parameters of the surface properties, extremely strong correlations were observed between *Sa* and GU in all the

resin composites, as reported previously.  $^{35,36}$  Therefore, in order to obtain an esthetic and smooth surface on the restoration, it is important to conduct appropriate finishing and polishing procedures. On the other hand, extremely strong correlations were observed between Sa and  $\gamma_S$  and GU and  $\gamma_S$  in the OM specimens only, indicating that finishing and polishing methods may have a larger impact on the surface properties of OM when compared to the other resin composites.

Structural colors depend on the refractive index distribution and the differences in the refractive indices of the resin matrix and the inorganic filler. <sup>17,19</sup> Although appropriate finishing and polishing procedures may be necessary to generate a structural color in OM, further studies are needed to determine the influence of the different finishing and polishing procedures on color matching.

#### CONCLUSION

Although the structural colored resin composite OM showed significantly lower  $\sigma_{F}$  and E values, it had a significantly higher R compared to the other resin composites. These results suggest that cavity status should be taken into consideration when using OM in clinical situations. The finishing and polishing methods along with the type of resin composite used have significantly affected the surface properties of the resin composite, as measured by the Sa, GU, and SFE values. SEM observations demonstrated that the shapes, sizes, and distributions of the fillers varied among the resin composite, and different surface appearances were observed with the different finishing and polishing methods used. In the case of the OM specimens, the use of the multiple polishing system (SSD) after finishing with a TCB may improve its surface properties when compared with the other finishing and polishing methods.

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#### **Conflict of Interest**

The authors have no financial interest in any of the companies or products mentioned in this article.

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