Mechanical properties and polished surface characteristics of a structural colored resin composite

Kiyoto Mizutani

Nihon University Graduate School of Dentistry,

Major in Operative Dentistry

(Directors: Prof. Masashi Miyazaki and Assoc. Prof. Toshiki Takamizawa)

# Contents

Summary	<b>P.</b> 1
Introduction	<b>P.</b> 4
Materials and methods	P. 5
Results	P. 9
Discussion	P. 13
Conclusions	P. 16
References	<b>P. 17</b>
Tables and Figures	P. 20

This thesis is based on the published article listed below with additional data.

Mizutani K, Takamizawa T, Ishii R, Shibasaki S, Kurokawa H, Suzuki M, Tsujimoto A, Miyazaki M (2021) Flexural properties and polished surface characteristics of a structural colored resin composite. Oper Dent 46, e117-e131.

#### Summary

A new type of resin composite has been developed based on the structural color concept and bottom-up nanotechnology used to make specialized filler particles. 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. The use of the structural colored resin composite has met with simplify the shade matching process and render it more costeffective as it only requires a single universal shade. However, little information is available about the mechanical and surface properties of this structural colored resin composite. This study aimed to determine the mechanical properties and surface characteristics under different finishing and polishing procedures of the structural colored resin composite.

The structural colored resin composite, Omnichroma (OM), and two conventional resin composites, Filtek Supreme Ultra (FS) as a typical nanofilled resin composite and Tetric EvoCeram (TE) as a typical nanohybrid resin composite, were used in this study. The flexural properties of the tested resin composites were measured according to the ISO 4049 specifications. The flexural strength ( $\sigma_F$ ), elastic modulus (*E*), and the modulus of resilience (*R*) were determined. The surface Knoop hardness number (*KHN*) of the cured resin composites was measured. Specimens polymerized with a curing unit were stored in the dark at 25°C for 24 h. The prepared specimens were randomly divided into seven groups (n = 10 each) for different finishing and polishing methods as follows: ground with #320-grit silicon carbide paper (BAS); finished with a superfine diamond point (SFD); finished with a tungsten carbide bur (TCB); SFD polished with a one-step point-type polishing system (SFD+CMP); TCB polished with CMP (TCB+CMP); SFD polished with a multi-step polishing system (SFD+SSD); and TCB polished with SSD (TCB+SSD).

The surface roughness area (Sa) before and after finishing and polishing procedures was obtained using a three-dimensional laser scanning microscope. The surface gloss (GU) of

each group was measured using a gloss meter after the *Sa* measurements were obtained. The contact angles and surface free energies ( $\gamma_s$ ) of the prepared specimens were measured in order to evaluate the surface characteristics in different finishing and polishing procedures. Scanning electron microscopy (*SEM*) observations of representative specimens from the tested resin composites were performed after argon-ion etching to visualize the morphological features of the filler particles. Representative specimens from the three resin composites were also observed after the different finishing and polishing procedures.

The average  $\sigma_F$  were ranked as follows, with significant differences between each value: FS > TE > OM. The average *E* were ranked as follows, with significant differences between each value: FS > TE > OM. The average *R* were ranked in the following order: OM > TE > FS. Although no significant difference was found between FS and TE, OM showed a significantly higher *R* than the other resin composites, in contrast to  $\sigma_F$  and *E*. FS had significantly higher *KHN*, whereas OM had significantly lower *KHN* than the other resin composites.

Two-way ANOVA revealed that the finishing and polishing methods and the type of resin composite significantly affected the *Sa*. The groups finished with SFD showed significantly higher *Sa* when compared with the other groups, regardless of the type of resin composite used. Most groups polished with SSD showed significantly lower *Sa* than the groups polished with CMP, regardless of the type of finishing used (SFD or TCB). The finishing and polishing methods and the type of resin composite used significantly affected the *GU* (p < 0.001). The *GU* of the BAS, SFD, and TCB were significantly lower than those of the groups polished with CMP and SSD. The finishing and polishing method, as well as the type of resin composite used, significantly influenced the  $\gamma_S$  (p < 0.001). The specimens in most of the groups polished with SSD showed significantly higher  $\gamma_S$  values than those in 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  $\gamma_S$  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. In *SEM* observations, the shapes, sizes, and distributions of the inorganic fillers were found to be material dependent. All the resin composites exhibited smoother surfaces after finishing with TCB when compared to those finished with SFD. When comparing the different polishing methods (CMP and SSD), smoother surfaces were observed in the SSD in the case of the OM and FS specimens, regardless of the finishing method.

In the results of this laboratory study, although the structural colored resin composite OM showed significantly lower  $\sigma_F$ , *E*, and *KHN*, it had a significantly higher *R* compared to the other resin composites. These results suggested 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 significantly affected the surface properties of the resin composite, as measured by the *Sa*, *GU*, and *SFE*. *SEM* observations demonstrated that the shapes, sizes, and distributions of the fillers varied among the resin composites, and different surface appearances were observed depending on the 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 secure better surface properties than the other finishing and polishing methods.

# Introduction

It is important to take into account both the esthetic and mechanical properties of a resin composite restoration and its durability in the oral environment (1-3). The mechanical properties of a resin composite are closely related to its fracture resistance and wear behavior (4,5). Changes in the surface properties of a restoration lead to staining, plaque accumulation, restoration degradation, and gingival inflammation (6,7). Therefore, to obtain the desired esthetics and at the same time ensure the longevity of the restoration, the polishing ability of the resin composite should be determined. Ishii *et al.* (8) 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.

A new type of resin composite has been developed based on the structural color concept and bottom-up nanotechnology used to make specialized filler particles (9). These fillers are in the form of spherical particles, having an average diameter of 260 nm, which produce the yellow to red colors required to match the natural tooth color (9,10). 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 (11). The use of the structural colored resin composite has met with simplify the shade matching process and render it more cost-effective as it only requires a single universal shade (9). However, little information is available about the mechanical and surface properties of this structural 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.

This study aimed to determine the mechanical properties of the structural colored resin composite based on a flexural strength test and microhardness test. The surface properties were then examined in terms of SFE, surface roughness (Sa), and gloss (GU) 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.

#### Materials and methods

#### Study materials

The structural colored resin composite, Omnichroma (OM, Tokuyama Dental), and two other conventional resin composites, Filtek Supreme Ultra (FS, 3M Oral Care) as a typical nanofilled resin composite and Tetric EvoCeram (TE, Ivoclar Vivadent) as a typical nanohybrid resin composite, were used in this study (Table 1). A halogen-quartz-tungsten curing unit (Optilux 501; SDS Kerr) was used to avoid any effects of the reported non-uniformity of light-emitting diode curing units (12). The light irradiance (above 600 mW/cm<sup>2</sup>) of the curing unit was confirmed using a dental radiometer (Model 100; SDS Kerr).

## Flexural strength test

The flexural properties of the resin composites were measured according to the ISO 4049 specifications (13). A transparent matrix tape was placed on a glass slide, and a stainless-split mold ( $25 \times 2 \times 2$  mm) was positioned on it. The resin composites were then inserted into the mold and covered with the matrix tape. The middle third of the specimen was irradiated for 30 s after which the remaining thirds were irradiated for 30 s each. The polymerized specimen was removed from the mold, and all the sides were polished using a #1,200-grit silicon carbide (SiC) paper (Fuji Star type DCCS; Sankyo Rikagaku). The prepared specimens were stored in distilled water at 37°C in the dark for 24 h. Twelve specimens per test resin composite were subjected to the three-point bending test (span length = 20 mm) using a universal testing

machine (5500R; Instron) at a crosshead speed of 1.0 mm/min until breaking point. The flexural strength ( $\sigma_F$ ) and elastic modulus (*E*) were determined using built-in computer software connected to the testing machine. In addition, the modulus of resilience (*R*) was obtained (14).

# Measurement of the Knoop hardness number (KHN)

The surface microhardness of the cured resin composites was measured at 24 h after making the specimens. The resin composite paste was placed into a polytetrafluoroethylene cylindrical mold (6 mm in diameter and 2 mm in height) and covered with transparent matrix tape. The specimens were light-irradiated for 20 s. One flat surface from each specimen was polished using a sequence of SiC papers up to #2,000-grit. Ten flat specimens were prepared from each group and stored under dark conditions for 24 h in a 100% humidity environment at 37°C. *KHN* was measured from the indentation after application of a 1.96 N load for 15 s dwell time using a microhardness tester (Via-S; Matsuzawa). Three measurements per specimen were performed at different locations near the center of the specimen, and the mean values were calculated for each specimen.

## 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 matrix tape, and the resin paste was condensed into the mold from the open end. The open end was then covered with matrix tape, and pressure was applied, followed by light irradiation for 30 s. The polymerized specimen was removed from the mold and was later stored in the dark at  $25^{\circ}$ C for 24 h 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 #320-grit SiC paper under running water. The specimens in three groups were finished using a superfine diamond point (SFD, SF102R; ISO #017, Shofu), and those from another three groups were finished using a tungsten carbide bur (TCB, FG7714; ISO #014, Kerr). Those in the remaining group were only ground with SiC #320-grit grinding paper and were set as the baseline (BAS). The finishing procedures were performed using a high-speed handpiece (TwinPower Turbine; J. Morita Mfg.) with water spray. Light hand pressure was applied in multiple directions, and the burs were changed after five times using. From the three groups finished by each method, one was set aside for measurement. One of the two remaining groups was polished using the one-step point-type polishing system (CMP, CompoMaster; Shofu), and the other using the multi-step polishing system (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) 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 Sa

The surfaces of all the specimens were observed using a three-dimensional laser scanning microscope (LSM, VK-8700; Keyence). The spectral maximum of the excitation light source was observed to be at 658 nm; the intensity of the excitation power and the amplification of the photomultiplier were kept constant during the observation period. Profilometric measurements were conducted in a region (1.0 mm  $\times$  1.0 mm) at the center of the specimen. The *Sa* was obtained using the built-in computer software (VK Analyzer; Keyence). Three measurements were taken, and the means were then determined for each group.

#### Measurement of GU

The GU of each group was measured using a gloss meter (GM-26D; Murakami Color Research Laboratory) 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 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) with a built-in charge-coupled device camera (12). 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 (15). 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

The polymerized specimens were polished using abrasive disks and a series of diamond pastes down to a particle size of 0.25  $\mu$ m. The mirror-polished surfaces were subjected to argon-ion beam etching (IIS-200ER; Elionix) for 40 s, with the ion beam directed perpendicular to the polished surface (accelerating voltage = 1 kV, ion current density = 0.4 mA/cm<sup>2</sup>). The surfaces were then coated with a thin film of Au in a vacuum evaporator (Quick Coater SC-701; Sanyu Electron). Images were obtained using a scanning electron microscope (FE-8000; Elionix) at an operating voltage of 10 kV. In order to understand the surface texture

of the finishing and polishing instruments before use, Au coated surfaces were observed by *SEM*.

### **Statistical Analysis**

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). 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 honestly significant difference test at a significance level of 0.05. Significant differences in flexural properties and *KHN* were observed using the one-way ANOVA test, whereas differences in *Sa*, *GU*, 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 Pearson product-moment correlation coefficient was also used to perform pairwise comparisons, to understand the relationships between the tested parameters of the surface properties.

#### 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  $\sigma_F$  than the other resin composites. The average *R* ranged from 0.78 to 1.01 MJ/mm<sup>3</sup> in the following order: OM > TE > FS. Although no significant difference was found between FS and TE, OM showed a significantly higher *R* than the other resin composites, in contrast to  $\sigma_F$  and *E*.

### KHN

*KHN* of the resin composites are presented in Table 3. *KHN* ranged from 47.7 to 80.0 in the following order: FS > TE > OM. FS had a significantly higher *KHN*, whereas OM had a significantly lower *KHN* than the other resin composites.

# Sa

The influence of different finishing and polishing methods on the *Sa* is shown in Table 4. Two-way ANOVA revealed that the finishing and polishing methods and the type of resin composite significantly affected the *Sa* (p < 0.001). 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* than the other groups, regardless of the type of resin composite used. On the other hand, the TCB + SSD showed the lowest *Sa* compared to the other groups, and significant differences in FS and TE were observed between the TCB + SSD and the other groups. In addition, most groups polished with SSD showed significantly lower *Sa* than 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. However, the *Sa* of the OM polished with CMP and SSD were found to be significantly lower than those of the other resin composites, regardless of the finishing method.

# GU

The effects of the different finishing and polishing methods on the GU are presented in Table 5. Two-way ANOVA test revealed that both factors, finishing and polishing methods and the type of resin composite used, significantly affected the GU (p < 0.001). In addition, a significant two-way interaction between these two factors was observed (p < 0.001). The GUof the BAS, SFD, and TCB 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 than those finished with SFD. In addition, the specimens finished with TCB demonstrated significantly higher GU than those finished with SFD. The OM polished with SSD showed significantly higher GU than those polished with CMP. However, the FS specimens that had been polished with SSD showed significantly lower GU compared to those polished with CMP, regardless of the finishing method. On the other hand, the TE polished with SSD showed a different trend in their GU when comparing the specimens finished with SFD and TCB.

### SFE

The effects of the different finishing and polishing methods on the *SFE* are presented in Table 6 and Fig. 1. Two-way ANOVA revealed that the finishing and polishing method, as well as the type of resin composite used, significantly influenced the  $\gamma_S$  (p < 0.001). 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  $\gamma_S$  compared to those in the other groups. The polished OM specimens showed significantly higher  $\gamma_S$  compared to those in the BAS and other finished groups. In the case of the FS specimens, although the specimens in the TCB + SSD presented significantly higher  $\gamma_S$  than those in the other groups, no significant differences were observed among the specimens in the other groups. The  $\gamma_S$  of the TE polished with CMP were found to be lower than those in the other groups. FS tended to have higher  $\gamma_S$ when compared with the other resin composites, regardless of the finishing and polishing methods.

With regard to each component of the  $\gamma_S$  (Fig. 1), all three resin composites tended to have higher  $\gamma_S^p$  when polished with SSD than with the other finishing and polishing methods. The FS specimens had higher  $\gamma_S^h$  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 surface properties are presented in Table 7. Extremely strong negative correlations between *Sa* and *GU* in the combined data from the three resin composites and for each resin composite, and between *Sa* and  $\gamma_s$  in the OM 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 presented in Figs. 2-4. The shapes, sizes, and distributions of the inorganic fillers were found to be resin composite dependent. OM consisted of nanosized spherical fillers (approximately 0.25  $\mu$ m) and round prepolymerized fillers that employ the same nanosized spherical fillers (Fig. 2). FS consisted of spheroidal aggregates (0.5-5.0  $\mu$ m) of nanosized filler particles (Fig. 3), whereas TE consisted of irregular fillers (0.5-2.0  $\mu$ m) made up of nanosized filler particles that were packed into prepolymerized fillers at a high density (Fig. 4).

Representative *SEM* images of the surfaces of the instruments are shown in Fig. 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 Figs. 6-8. All the resin composites exhibited smoother surfaces after finishing with TCB than with SFD. For FS and TE, scratches and plucked-out fillers were observed following finishing 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 SSD in the case of the OM and FS, regardless of the finishing method. On the contrary, in the case of the TE specimens, there was no difference in surface smoothness between the SSD and CMP.

# Discussion

In this study, the mechanical and surface properties of the structural colored resin composite OM were investigated and compared with those of the conventional resin composites FS and TE. The findings of this study were consistent with the results of previous studies, which investigated the flexural properties of nanohybrid and nanofilled resin composites, including the resin composites used in this study (16,17). FS showed significantly higher  $\sigma_F$  and *E* than TE in the current study. In general, materials with high flexural strength have high elastic moduli (18,19). OM presented significantly lower *E* than the other materials despite having a higher filler content. Barszczewska-Rybarek (20) reported that UDMA had a lower *E* than bis-GMA and TEGDMA. UDMA is the resin monomer used in OM. 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  $\sigma_F$  and *E* due to the various types present in resin composites. OM has been known to consist of uniformly sized spherical fillers with constant interparticle spacing. Therefore, crack propagation in OM tended to be simple, leading

to lower fracture resistance. On the other hand, R is thought to represent the ability of a material to absorb energy when it is elastically deformed under external stress without failing (18). OM showed a significantly higher R than 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, that the flexural properties of OM would not differ from those of other resin composites, was rejected.

In the results of this study, the two factors, that is finishing and polishing procedure and type of resin composite, significantly influenced the *Sa*. In particular, the groups finished with SFD showed significantly higher *Sa* than 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 (8). This finding may be explained by the different surface textures of the finishing instruments (Fig. 5) resulting in different finishing mechanisms. The TCB cut away the surface, whereas diamond points grind the surface with many abrasive diamond particles (Figs. 6 and 7) (21,22). Furthermore, the finishing methods significantly affected the polished groups, because most of the specimens in the TCB showed lower *Sa* than those in the SFD, regardless of whether CMP or SSD was used.

Gloss is defined based on the degree of specular reflection of light (23). The finishing and polishing methods and type of resin composite used significantly affected GU in the current study. Although the OM polished with SSD showed significantly higher GU than those polished with CMP, the opposite findings were observed in the FS, regardless of the finishing method used. Nonetheless, a smoother surface is not necessary to obtain a high surface gloss, and the relationship depends on the polishing procedures and materials used (24). Although 40 to 60 GU was identified as the typically desired gloss in an American Dental Association professional product review (25), Cook and Thomas (26) 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 final polishing was performed (27). Therefore, if the clinically acceptable GU is assumed to be 40 to 60 GU, the SSD for OM and CMP for FS may be acceptable.

Although the  $\gamma s^p$  and  $\gamma s^h$  in each group showed wide variation, some trends were observed between the materials or the finishing and polishing methods. FS had a higher  $\gamma s^h$  component than the other resin composites in all the groups. This trend is likely brought about by the different surface chemistries of 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 (28). This 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.

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

# Conclusions

- 1. Although the structural colored resin composite OM showed significantly lower  $\sigma_F$  and E, it had a significantly higher R than the other resin composites.
- 2. The finishing and polishing methods along with the type of resin composite used significantly affected the surface properties of the resin composite, as measured by the *Sa*, *GU*, and *SFE*.
- 3. *SEM* observations demonstrated that the shape, size, and distribution of the fillers varied among the resin composites, and different surface appearances were observed with different finishing and polishing methods.
- In the case of OM, the use of the multiple polishing system (SSD) after finishing with TCB may secure superior surface properties when compared with the other finishing and polishing methods.

#### References

- Jefferies SR (2007) Abrasive finishing and polishing in restorative dentistry; A state-ofthe-art review. Dent Clin North Am 52, 379-397.
- 2. Endo T, Finger WJ, Kanehira M, Utterodt A, Komatsu M (2010) Surface texture and roughness of polished nanofill and nanohybrid resin composites. Dent Mater J 29, 213-223.
- 3. Ferracane JL (2011) Resin composite-state of the art. Dent Mater 27, 29-38.
- Heintze SD, Ilie N, Hickel R, Reis A, Loguercio A, Rousson V (2017) Laboratory mechanical parameters of composite resins and their relation to fractures and wear in clinical trials–A systematic review. Dent Mater 33, e101-e114.
- Imai A, Takamizawa T, Sugimura R, Tsujimoto A, Ishii R, Kawazu M, Saito T, Miyazaki M (2019) Interrelation among the handling, mechanical, and wear properties of the newly developed flowable resin composites. J Mech Behav Biomed Mater 89, 72-80.
- 6. Bollen CM, Lambrechts P, Quirynen M (1997) Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacteria plaque retention; A review of the literature. Dent Mater 13, 258-269.
- Reis AF, Giannini M, Lovadino JR, Ambrosano GM (2003) Effects of various finishing systems on the surface roughness and staining susceptibility of packable composite resins. Dent Mater 19, 12-18.
- Ishii R, Takamizawa T, Tsujimoto A, Suzuki S, Imai A, Barkmeier WW, Latta MA, Miyazaki M (2020) Effects of finishing and polishing methods on the surface roughness and surface free energy of bulk-fill resin composites. Oper Dent 45, e91-e104.
- Saegusa M, Kurokawa H, Takahashi N, Takamizawa T, Ishii R, Shiratsuchi K, Miyazaki M (2021) Evaluation of color matching ability of a structural colored resin composite. Oper Dent 46, 306-315.

- Pereira Sanchez N, Powers JM, Paravina RD (2019) Instrumental and visual evaluation of the color adjustment potential of resin composites. J Esthet Restor Dent 31, 465-470.
- Luo J, Qu D, Tikhonov A, Bohn J, Asher SA (2010) Monodisperse, high refractive index, highly charged ZnS colloids self assemble into crystalline colloidal arrays. J Colloid Interface Sci 345,131-137.
- Price RB, Ferracane JL, Shortall AC (2015) Light-curing units: A review of what we need to know. J Dent Res 94, 1179-1186.
- International Organization for Standardization (2009) ISO 4049:2009. Dentistry Polymerbased restorative materials. International Organization for Standardization, Geneva, Switzerland.
- Peutzfeldt A, Asmussen E (1992) Modulus of resilience as predictor for clinical wear of restorative resins. Dent Mater 8, 146-148.
- Hata T, Kitazato Y, Saito T (1987) Estimation of the surface energy of polymer solids. J Adhes 21, 177-194.
- 16. Sideridou ID, Karabela MM, Vouvoud EC (2011) Physical properties of current dental nanohybrid and nanofill light-cured resin composites. Dent Mater 27, 598-607.
- 17. Ilie N, Rencz A, Hickel R (2013) Investigations towards nanohybrid resin-based composites. Clin Oral Investig 17, 185-193.
- Powers JM, Wataha JC (2013) Dental Materials: Properties and Manipulation, 10th ed., Elsevier Mosby, St. Louis, 15-21.
- 19. Shibasaki S, Takamizawa T, Nojiri K, Imai A, Tsujimoto A, Endo H, Suzuki S, Suda S, Barkmeier WW, Latta MA, Miyazaki M (2017) Polymerization behavior and mechanical properties of high-viscosity bulk fill and low shrinkage resin composites. Oper Dent 42, e177-e187.

- 20. Barszczewska-Rybarek IM (2009) Structure-property relationships in dimethacrylate networks based on Bis-GMA, UDMA and TEGDMA. Dent Mater 25, 1082-1089.
- Jung M (1997) Surface roughness and cutting efficacy of composite finishing instruments.
  Oper Dent 22, 98-104.
- 22. Roeder LB, Tate WH, Powers JM (2000) Effect of finishing and polishing procedures on the surface roughness of packable composites. Oper Dent 25, 534-543.
- Rodrigues-Junior SA, Chemin P, Piaia PP, Ferracane JL (2015) Surface roughness and gloss of actual composites as polished with different polishing systems. Oper Dent 40, 418-429.
- 24. Cazzaniga G, Ottobelli M, Ionescu AC, Paolone G, Gherlone E, Ferracane JL, Brambilla E (2017) In vitro biofilm formation on resin-based composites after different finishing and polishing procedures. J Dent 67, 43-52.
- 25. da Costa JB, Goncalves F, Ferracane JL (2011) Comparison of two-step versus four-step composite finishing/polishing disc systems: Evaluation of a new two-step composite polishing disc system. Oper Dent 36, 205-212.
- 26. Cook MP, Thomas K (1990) Evaluation of gloss meters for measurement of moulded plastics. Polymer Test 9, 233-244.
- 27. Kaizer MR, Oliveria-Ogliari A, Cenci MS, Opdam NJM, Moraes RR (2014) Do nanofill or submicron composites show improved smoothness and gloss? A systematic review of in vitro studies. Dent Mater 30, e41-e78.
- 28. Venz S, Dickens B (1991) NIR-spectroscopic investigation of water sorption characteristics of dental resins and composites. J Biomed Mater Res 25, 1231-1248.

Tables and Figures

Code	e 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 Supreme Ultra (Shade; A2: N870378)	bis-GMA, UDMA, TEGDMA, bis-EMA, PEGDMA, zirconia/silica clusters (0.6-10 μm), silica (20 nm silica filler), ziroconia (4-11nm)	Nano filled (78.5 wt%, 63.3 vol%)	3M Oral Care, St. Paul, MN, USA
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 nn	Nano hybrid (75-76 wt%, 53-55 vol%) n)	Ivoclar Vivadent, Schaan, Lichtenstein
Cod	e Finishing system	em Model		Manufacturer
SFD	Superfine grit diamond point	SF102 R (ISO #017, particle size less than 25 $\mu m)$		Shofu, Kyoto, Japan
тсв	Tungsten carbide bur	FG7714 (ISO #014, 12 blades)	FG7714 (ISO #014, 12 blades)	
Cod	e Polishing system	Model		Manufacturer
СМР	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: green (fine): $\varphi$ 12-mm disk; 20 µm aluminum oxide SSD R: red (superfine): $\varphi$ 12-mm disk; 7 µm aluminum oxide		Shofu

Table 1: Materials used in this study

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

Table 2: Flexural properties of the tested resin composites							
Code	Flexural strength; $\sigma_F$ (MPa)	Elastic modulus; <i>E</i> (GPa)	Resilience; R (MJ/mm <sup>3</sup> )				
ОМ	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				

Values in parentheses indicate standard deviation.

Same small case letter in vertical columns indicates no difference at 5% significance level.

Table 3: <i>KHN</i> of the tested resin composites						
Code	KHN					
ОМ	47.7 (1.3)c					
FS	80.0 (1.0)a					
TE	55.6 (1.5)b					

Values in parentheses indicate standard deviation.

Same small case letter in vertical columns indicates no difference at 5% significance level.

Table 4: Influence of finishing and polishing methods on surface area roughness (Sa, $\mu$ m)							
	BAS	SFD	ТСВ	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

Values in parentheses indicate standard deviation.

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.

Table 5: Influence of finishing and polishing methods on surface gloss $(GU)$							
	BAS	SFD	TCB	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

Values in parentheses indicate standard deviation.

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.

Table 6: Influence of finishing and polishing methods on total SFE ( $\gamma_s$ )							
	BAS	SFD	TCB	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

Values in parentheses indicate standard deviation.

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.

Table 7: Relationship between the parameters of surface properties						
	-	GU	γs			
Combined	data of three resi	in composites				
Sa	r	-0.847	-0.274			
	<i>p</i> -value	< 0.001	0.230			
CU	r		0.318			
60	<i>p</i> -value		0.160			
OM						
Sa	r	-0.972	-0.897			
Sa	<i>p</i> -value	< 0.001	0.006			
GU	r		0.930			
	<i>p</i> -value		0.002			
FS						
S.a.	r	-0.713	-0.265			
Sa	<i>p</i> -value	0.072	0.566			
CU	r		0.222			
GU	<i>p</i> -value		0.632			
ТЕ						
Sa	r	-0.921	0.026			
	<i>p</i> -value	0.003	0.956			
CU	r		-0.145			
60	<i>p</i> -value		0.756			



Fig. 1. The total SFE ( $\gamma_S$ ) values and the three corresponding parameters of the resin composites.



Fig. 2. Representative *SEM* images of OM after argon-ion etching  $(5,000 \times \text{ and } 30,000 \times)$ .



Fig. 3. Representative *SEM* images of FS after argon-ion etching  $(5,000 \times \text{ and } 30,000 \times)$ .



Fig. 4. Representative *SEM* images of TE after argon-ion etching  $(5,000 \times \text{ and } 30,000 \times)$ .



Fig. 5. Representative *SEM* images of the instrument surfaces. SFD, Superfine diamond point (50x and 1,000x); TCB, Tungsten carbide bur (50x and 1,000x);

CMP, One-step point-type polishing system, Compomaster (1,000x); SSD G, Multi-step polishing system, Super-Snap fine green (1,000x); SSD R, Multi-step polishing system, Super-Snap super fine red (1,000x);



Fig. 6. Representative SEM images of OM after different finishing and polishing methods (2,500x).



Fig. 7. Representative SEM images of FS after different finishing and polishing methods (2,500x).



Fig. 8. Representative SEM images of TE after different finishing and polishing methods (2,500x).