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Evaluation of the Effect of Two Polishing Techniques and Thermocycling Process on Surface Roughness, Hardness, and Color Stability of Composites

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ORIGINAL ARTICLE

Evaluation of the Effect of Two Polishing Techniques and Thermocycling Process on Surface Roughness, Hardness, and Color Stability of Composites

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ABSTRACT

Objective: The aim of this study was to evaluate the effect of two polishing techniques and 10.000 thermocycles on the color stability, surface roughness, and hardness of two nanohybrid (Tetric N-Ceram, Escom100) and one bulkfill (Filtek) resin composites. **Methods:** A total of 60 specimens were prepared using three resin composites and 20 discs from each composite. Specimens for each composite were randomly divided into two different polishing groups (Optrapol rubber and Sof-Lex discs) $(n=10)$. Surface roughness (Ra, μ m), microhardness (VHN), and color change (ΔE00) values were measured pre- and post-thermocycling. Two-way analysis of variance (ANOVA) was used to evaluate the effect of independent variables. Bonferroni test was used for multiple comparisons ($p<0.05$). **Results:** Escom100 with Sof-Lex found the highest mean ΔE00 and Filtek bulk-fill composite with Optrapol found the lowest mean ΔE00. Escom100 with Sof-Lex exhibited the lowest Ra values in all groups. Sof-Lex discs exhibited smoother surfaces than Optrapol in all groups.. Among the polishing groups, Optrapols' VHN values were higher than Sof-Lex's (p<0.05). **Conclusions:** Filtek bulk-fill with Optrapol in terms of color change and microhardness; Escom100 (nanohybrid) with Sof-Lex in terms of smoothness, can be recommended for clinical use. After thermocycling, surface roughness values increased and surface hardness values decreased in all composite resins.

Key words: color stability, composite resin, surface hardness, surface roughness, thermocycling

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INTRODUCTION

Resin composites are frequently used in dentistry worldwide due to their aesthetic quality and superior physical properties. With the development of resin composites, many efforts have been made to improve the clinical behavior of this type of restorative material.¹ Recently, the development of bulk-fill resin composite materials decreased the application time during restoration. The utilization of this material with the bulk technique in dentistry resulted in more easy and quick clinical practices.² Resin composite materials are usually categorized according to their various properties, such as the filler type, content, the size of an average particle, and the extent of distribution besides physical and mechanical characteristics.3 It is known that the types and size of filler particle influence the physical and mechanical properties of the material and protect the organic matrix against the force applied to the direct restoration, and therefore have a direct influence on the surface properties of the composites.⁴ Different distinct filler shapes have been employed to reduce polymerization shrinkage especially to achieve better color stability adequate wear resistance, and clinically acceptable surface smoothness, resulting in better aesthetic results. The resin matrix and filler particles do not wear at the same rate due to different physical properties.5 However, composite resin materials have some limitations; for instance, particle degradation might occur after polishing procedures,⁵ accumulation of plaque, discoloration, marginal fractures, surface roughness, water sorption, and polymerization shrinkage.6

Due to the importance of treatments, dentists can choose a wide variety of finishing/polishing systems. However, there are different systems available in the market.7 A previous study reported that multi-step

aluminum oxide discs produced the best surface smoothness.⁸ However, due to the fact that the shape of the discs had a flat surface, especially in posterior restorations, it did not allow for the creation of the proper anatomy. Additionally, other finishing and polishing systems exist to treat posterior and anterior areas in one, two, or multiple steps. In the last years, many attempts focused on optimizing the finishing and polishing instruments.⁷ The finishing/polishing application procedures are affected by influencing the surface quality, aesthetics, and the long-term processing treatment of the composite materials.⁹ Additionally, it is crucial to generate the smoothest surface possible, as the tongue can sense variations in surface roughness as little as 0.3µm .¹⁰ The mechanical and physical properties of resin composites depend on the concentration, particles size, and distribution of fillers. Recently, on the resin matrix, while the particle size of the materials has decreased, the amount of filler has increased. Newly developed nanocomposites are called nanofil/nanohybrid composites, which contain nanoparticles. Nanocomposites can be used in both anterior and posterior regions. However, in addition to the organic content of restorative materials, the shape, type, and size of the inorganic particles in their content can also affect the rate of change of surface roughness and hardness values in the composite.11 The ideal composite materials would have surfaces with a higher hardness and a lower roughness, resulting in acceptable and sustainable longevity.12,13

In the oral environment, as a result of the simulation of temperature fluctuation defects that may occur due to deterioration, color change and wear of the materials can be observed. Transfer to clinical conditions by in vitro studies mimicking the oral environment enables thermocycling testers to simulate temperature changes inside the mouth. In these devices, the desired temperature values are made in certain cycle numbers. 14 Restorative materials are based on the best clinical studies that can be evaluated. Material variability, patient complaints, return problems, high cost, and long follow-up of patients limit clinical studies. Therefore, clinical studies that mimic the natural oral environment are static or in vitro, and the inclusion of dynamic, artificial aging methods should be supported by studies.

There is a lack of knowledge regarding the performance of bulk fill materials compared to nanohybrid composites with finishing and polishing materials in literature. The aim of the study was to evaluate the effect of surface hardness, roughness, and color stability of composites (two nanohybrid composites and one bulk-fill composite) after two polishing techniques and a thermocycling process. The null hypothesis polishing techniques and thermocycling process do not affect the color, surface roughness, and hardness of the resin composites.

METHODS

Resin composite materials were used in the Filtek bulk-fill (3M ESPE, St. Paul, MN, USA), the nanohybrid Tetric N-Ceram (Ivoclar Vivadent, Schaan, Liechtenstein), and the nanohybrid Escom100 (Spident, Gojan-dong, Namdong-gu, Incheon, Korea) composites with shade A1. The two polishing systems, Optrapol (Ivoclar Vivadent, Schaan, Liechtenstein) and Sof-Lex discs (3M ESPE, St. Paul, MN, USA), were used in the present study. The composition and manufacturer of the resin composite materials are described in Table 1, and the composition and manufacturer of the polishing systems are described in Table 2. Disc-shaped specimens of 10mm in diameter and 2mm in thickness were prepared using a teflon mold. The composites were pressured by finger and compressed by mylar strip bands, and excess material was removed to obtain a flat surface. All of the specimens were polymerized via LED (Elipar S10, 3M ESPE, Germany) for 20 seconds (s) according to the manufacturer's instructions. Then, 1200-grit silicon carbide abrasive paper (SiC) was used for 10 s under stream water before application using the polishing systems. A total of 60 discs were prepared, using 20 discs from each composite resin group. Specimens for each composite were divided into two different polishing groups $(n = 10)$. The polishing procedures were applied for the one surface of the specimens; Sof-Lex discs (four different colors) were applied for 10 s to one group, and Optrapol rubber was applied for 40 s to the other group. New discs and polishing cups were used. Then, all of the specimens were rinsed for 10 s and then stored at 37ºC in distilled water for 24 hours. Initial measurements (color, roughness, and microhardness values) were determined in all specimens, which were then immersed in hot and cold water baths while repeatedly thermocycling between 5 and 55°C, with a dwell time of 30 s in each bath (10,000 cycles). The final measurements (color, roughness, and microhardness values) of all the specimens were determined after thermocycling.

Color measurements

Color measurements were conducted on the specimens before and after thermocycling with a spectrophotometer (VitaEasyshade, VITA Zahnfabrik, BadSäckingen, Germany), which was calibrated according to the manufacturer's instructions. Color differences can be quantified using the following CIEDE2000 formula:15

$$
\Delta E_{00} = \sqrt{\left(\frac{\Delta L^{'}}{K_L S_L}\right)^2 + \left(\frac{\Delta C^{'}}{K_C S_C}\right)^2 + \left(\frac{\Delta H^{'}}{K_H S_H}\right)^2 + R_T \left(\frac{\Delta C^{'}}{K_C S_C}\right) \left(\frac{\Delta H^{'}}{K_H S_H}\right)^2}
$$

where *ΔL*′, *ΔC*′, and *ΔH*′ are the differences in lightness, chroma, and hue, respectively. *SL*, *SC*, and *SH* are considered weighting functions that adjust the total color difference for variations in the location of the color difference pair in the *L*′, *a*′,

Table 1. Compositions and manufacturers of composite resins

DDDMA: Didode cyldimethylammonium bromide, UDMA: Urethane dimethacrylate, Bis-EMA: Bisphenol-A ethoxylated dimethacrylate, Bis-GMA: Bisphenol-A glycidyl dimethacrylate.

Table 2. Compositions and manufacturers of finishing-polishing systems

and *b'* coordinates. K_L , K_C , and K_H , the parametric factors, are correction terms for experimental conditions. Finally, *RT* is a rotation function that accounts for the interaction between chroma and hue differences in the blue region.¹⁵

Surface roughness measurements

The initial and final surface roughness measurements were used with a profilometer (MarSurf PS1, Mahr, Göttingen, Germany). The average roughness (Ra, µm) was calculated at the center of the specimen in two directions that were perpendicular to each other three times in the pre- and post-thermocycling.

Surface microhardness measurements

The initial and final surface hardness measurements of Vickers hardness values (VHN) (kg/mm2) were calculated using a microhardness test device (LHV-1D, Bursam NDT, Bursa, Turkey). A 300g load with a 10 s dwell period was used on the surface to make three various indentations, and the average value from each specimen was assessed.

Statistical analysis

In this study, statistical analyses were performed with the SPSS 23.0 package program. First, the normality of the distribution was checked with the Kolmogorov-Smirnov test. For the data, the p value obtained as a result of the Kolmogorov-Smirnov test was p>0.05; it was concluded that the data showed a normal distribution and the parametric test was found appropriate. A two-way analysis of variance (ANOVA) test was used to evaluate the effect of independent variables' color, surface roughness, and hardness; the evaluated factors were the composite and polishing system. When a difference was statistically significant, a Bonferroni test was used as a post-hoc for multiple comparisons. A paired sample t-test analysis was used to compare the pre- and post-thermocycling surface roughness and hardness values. The data are expressed as mean \pm standard deviation. Correlations between color change and roughness were calculated using a Pearson correlation analysis. The statistical significance level was accepted as $p<0.05$.

RESULTS

Color differences

The color changes of the resin composites ranged between 1.3 and 6.6 ΔE00. In the Sof-Lex group, the highest mean ΔE00 value of the Escom100 composite was found to be significantly different from the values of the Tetric N-Ceram and Escom 100 ($p<0.05$) group. In the Optrapol group, the lowest mean ΔE00 value of the Filtek bulk-fill composite was found to be significantly different (p <0.05). In the polishing groups that were compared, similar color changes for the Filtek bulk-fill and Tetric N-Ceram groups were shown. For Escom100, Optrapol presented a lower mean color changes than the Sof-Lex group $(p<0.05)$ (Table 3). According to the results of the color change, the significant difference indicated the common effect between the polishing groups and resin composite variables that were interactive (Table 4).

Table 3. Mean color changes (ΔE00) and standard deviation of the tested materials after thermocycling

Means followed by distinct capital letters represent statistically significant differences in each column ($p < 0.05$). There is no difference between receive the same letters.

Table 4. Two-way ANOVA results of finishing/polishing type and composite type (interactive) in terms of color change (∆E00) after thermocycling

Source	Type III Sum of Squares	df	Mean Square		
Finishing/Polishing	21.789		217.89	5.750	.020
Composite	101.830		50.915	13.437	.000
Finishing/ Polishing*Composite	45.430		22.715	5.994	.004

Table 5. Surface roughness values (Ra, µm) (mean±std.deviation) of the resin composites

TC=Thermocycling; p* represent statistically significant differences in each group of the same resin composites (between before and after thermocycling of specimens values)

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

Means followed by different lower letters (comparisons of before thermocycling, specimen values between the groups) represent statistically significant differences in each row (p <0.05).

Means followed by different superscript numbers (comparisons of after thermocycling, specimen values between the groups) represent statistically significant differences in each row (p <0.05).

Table 6. Two-way ANOVA results of finishing/polishing type and composite type (interactive) in terms of surface roughness pre- and post- thermocycling

Surface roughness measurements

Among the polishing groups, the pre- and postthermocycling surface roughness values where shown to be significantly different. When compared to the resin composites, Escom100 with Sof-Lex, during

pre- and post-thermocycling, presented the lowest mean Ra values ($p<0.05$). The Sof-Lex finishing/polishing system exhibited the smoothest surfaces compared to the Optrapol finishing/polishing system for all of the resin composites ($p<0.05$). When compared to

Sof-Lex disc				Optrapol rubber		
Composites	Before TC	After TC	p	Before TC	After TC	p
Filtek Bulk-Fill	62.60 ± 4.38	55.72 ± 3.59	< 0.05	62.91 ± 3.72	56.43 ± 4.85	< 0.05
	Aa	A ¹		Aa	A ¹	
Tetric N-Ceram	48.42 ± 3.08	44.21 ± 5.61	>0.05	52.97 ± 4.02	47.03 ± 3.95	< 0.05
	Ba	B		Bb	B ¹	
Escom100	55.43 ± 5.12	46.95 ± 3.51	< 0.05	62.26 ± 4.16	53.22 ± 7.02	< 0.05
	Ca	B ¹		Ab	A^2	

Table 7. Surface hardness values (VHN) (mean±std.deviation) of the resin composites

TC=Thermocycling

p* represent statistically significant differences in each group of the same resin composites (between before and after thermocycling of specimens values)

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

Means followed by different lower letters (comparisons of before thermocycling, specimen values between the groups) represent statistically significant differences in each row (p<0.05).

Table 8. Two-way ANOVA results of finishing/polishing type and composite type (interactive) in terms of surface hardness pre- and post- thermocycling

the Ra values during pre- and post-thermocycling, the Ra values increased with aging for all of the resin composites. In addition, except for three groups, the Ra values were significantly different for the pre- and post-thermocycling values (p <0.05). Thermocycling had significantly influenced the pre- and postthermocycling process in finishing the polishing groups' composites ($p<0.05$). According to the results of the analysis, the significant difference indicated a common effect between the polishing groups and resin composite variables that were interactive during preand post-thermocycling (Table 5, Table 6).

Surface microhardness measurements

Pre-thermocycling, the Tetric N-Ceram showed the lowest values when compared to the other composites (p<0.05), except for Optrapol with Escom 100 and Filtek bulk-fill; Sof-Lex with Filtek bulk-fill showed significantly higher values than the other composites at pre- and post-thermocycling $(p<0.05)$. When compared the among polishing groups' pre-thermocycling, Optrapol group had higher values than the Sof-Lex group (p<0.05). Moreover, except for Tetric N-Ceram with Sof-Lex, the other groups were significantly affected by the thermocycling $(p<0.05)$. Thermocycling had significantly influenced the pre- and post- processes

when completing the polishing groups' composites (p<0.05). The significant difference indicated a common effect between the polishing groups and resin composite variables that were interactive during the pre-thermocycling process (Table 7, Table 8).

Means followed by different superscript numbers (comparisons of after thermocycling, specimen values between the groups) represent statistically significant differences in each row $(p<0.05)$.

Pearson correlation analysis results

A Pearson correlation test showed that there was no relationship effect on the change differences of color and surface roughness (during pre- and post-thermocycling) of the polishing groups of the composites (p>0.05).

DISCUSSION

This study evaluated the effect of two polishing techniques and 10.000 thermocycles on the color stability, surface roughness, and microhardness of 2 nanohybrid (Tetric N-Ceram, Escom100) and 1 bulkfill (Filtek) resin composites. Polishing techniques

and thermocycling process affected the color, surface roughness, and hardness of the resin composites, and from the results, our null hypothesis was rejected.

The thermal cycling test is used in in vitro studies to mimic the temperature changes to which composite restorations are exposed in the oral environment.14 Thermal cycling tests have been reported in studies where the average number of cycles in the mouth between 5°C–55°C is 20–50 cycles in 1 day; 10,000 cycles can correspond to 1 year.^{14,16}

The color stability of resin composites is a critical factor for the longevity of the restoration. The color change depends on many factors, such as the matrix contents of resin, filler particle rate, color adsorption and absorption, solution type, and physical-chemical reactions.17 In dentistry, ΔEab is often used to evaluate color differences. However, the CIELAB color space takes on equal weight for all color coordinates.15 Studies have shown a discrepancy in sensitivity for different color coordinates within the CIELAB color space.^{18,19} Therefore, ΔE00, which considers parametric factors, was proposed to evaluate color differences.^{15,20} In addition, it was demonstrated that ΔE00 presents a better correlation with visual perception than ΔEab.21,22 Recently, the published literature was assessed by CIEDE2000 color differences with 50%:50% perceptibility ($PT = 0.81 \triangle E00$ units) and $50\% : 50\%$ acceptability ($AT = 1.77 \triangle E00$) thresholds.²³ In the present study, all tested composite groups exhibited color changes within the range of 1.3 and 6.6 $\triangle E00$ values during the pre- and post-thermocycling processes. The color change of composite resins is affected by extrinsic and intrinsic factors. Extrinsic factors include the duration/time and intensity of the light emission curing process and exposure to environmental factors, such as ultraviolet radiation, water, and heat. Intrinsic factors include the content of the resin matrix, filler loading and particle size distribution, type of photoinitiator, and remaining C=C bonds.24 The bulk-fill composite's exhibition of better color stability may be due to its organic filler sizes and submicron-nanometer and filler load. In addition, this may be related to the fact that UDMA shows lower viscosity and water absorption than Bis-GMA.25,26

The finishing/polishing procedure can influence the surface properties (hardness and roughness) and longevity restorations. In the present study, each specimen was either polymerized under a mylar strip or finished underwater with 1200 grit silicon carbide paper (average abrasive particle size: 30 μm). To finish the resin composite surface and to simulate the clinical scenario, similar particle-sized abrasives were incorporated into most dental finishing instruments.^{27,28} The ideal surface quality of the materials can differ depending on multiple factors, including filler size, filler loading, polishing systems' procedures, and the structure construction of the resin-matrix for low

surface roughness and high surface hardness in resin composites.29 For finishing systems to be effective in restorative composite materials, the abrasive particles need to be harder than the filler contents of the material. In other circumstances, the polishing systems will only remove the soft matrix of the resin and cause the filler particles to overhang from the surface.²⁷

Several studies reported that the Ra values of the surface ranged between 0.7–1.4μm, the accumulation of plaque did not differ significantly.9,28 In the current study, we found the roughness of the surface throughout all polishing groups was in the range of 0.1–0.9μm. Although the studies used different polishing systems, the Ra values obtained for the roughness parameter are comparable with the values we obtained in our study. Therefore, in the current study, all the evaluated restorative composite materials had acceptable Ra values for each polishing system tested. There are several studies that researched the associations among the size of the load particle, shape to the polishing capacity, and roughness of the composites.³⁰ Though the diamond abrasives in Optrapol provided a good surface finish, they were found to be rougher than the surface finish produced by aluminum oxide incorporated in Sof-Lex discs. This may occur due to the nondisplacement of composite filler particles by Sof-Lex discs, as compared to Optrapol rubber, which is less flexible. Aluminum oxide in Sof-Lex discs promotes the homogenous abrasion of fillers and the resin matrix. Additionally, the Sof-Lex discs may be efficiently adapted to the surface of the composite resin. Studies have reported the achievement of a surface roughness on composites through the use of flexible aluminum oxide discs, which is commonly accepted as the best option. $9,31$ Multi-step polishing instruments use smaller particles with each step to remove scratches from the previous step until a gloss surface is achieved. 32 Lu et al., 33 reported that the smoothest surfaces were created using aluminum oxide-coated discs that can perform an equal amount of abrasion from both an organic resin matrix and inorganic filler in composites. Several studies have reported that multistep systems perform better than one-step systems.^{34,35} One-step finishing/polishing systems can be achieved with a single polishing material, and smooth surfaces are provided in a shorter time.³⁶ One study stated that the texture of the final surface depends on the technique and material used;³⁷ however, there is no consensus on the materials or techniques necessary to provide the smoothest surfaces for the composite resins used.³⁸

A systematic review stated that the surface roughness of nanofiller or submicron composite resins is not superior to that of conventional microhybrids. The literature, in general, indicates that nanofiller (and probably submicron) particles outperform microhybrids due to their smaller size. This can be explained by the fact that different finishing and polishing methods were reported, or different approaches were used to evaluate surface properties, preventing comparisons between studies.⁴ All of the materials with Optrapol had demonstrated similar surface roughness values. However, in the Sof-Lex groups, Escom100 presented lower roughness values than the Filtek bulk-fill and Tetric N-Ceram roughness values. Escom100 and Tetric N-Ceram (nanohybrid) have approximately the same filler particle volume but showed differences to their roughness value after thermocycling. This result may be related to the chemical composition of the resin matrix and filler loading. Moreover, the hydrolytic degradation of silane may lead, over time, to surface degradation on composite materials and affect the material's abrasion resistance. Although there is no difference between the materials applied in the rubber group, the disc application may have contributed to the surface smoothness of the nanohybrid composites.

Berger et al.,³⁹ observed that the roughness of the surface and staining were closely associated with the technique and the polishing materials in restorations, and their influence on the size and distribution of the load particles was lesser than the former ones. The roughness of the resin surface was altered via a mastication process, while the factors causing discoloration and deposits could stay longer on rough resin surfaces.40 Furthermore, the changes in the color of the resin composites, surface roughness, and hardness properties in the oral cavity might be relatively more significant than the results obtained in this study. An investigation of color stability and mechanical properties of the composite resins used in different polishing systems in dentistry is very important in determining the areas where these materials will be applied.41,42 In the current study, composite resins demonstrated an increase in surface roughness values after thermocycling, and no correlation was found between roughness and color change. These findings are similar to previous studies.43

Microhardness tests have been used to estimate information regarding the resistance and mechanical properties of materials by opposing dental structures or materials.⁹ Different methods have been used to evaluate the hardness of the surface of restorative materials used in dentistry.44 Vickers are the most commonly used test methods for surface microhardness measurements of resin composites.45 There is no consensus for VHN hardness for it to be considered ideal. According to some authors, resin composites of VHN hardness values surpassing 50 VHN can be considered ideal. Other than Tetric N-Ceram with Sof-Lex, the other composites of VHN hardness values reached above 50 VHN at pre- thermocycling. In this study, VHN values were decreased in all of the specimens after thermocycling. This finding was consistent with previous studies.^{9,44} Tuncer et al.,⁴¹ reported that mean values of specimens decreased after the thermocycling

process due to water absorption. Water acts as a plasticizing molecule within the composite matrix, causing a softening of the polymer resin component by swelling the network and reducing the frictional forces between polymeric chains.⁶ Water absorption by the resin matrix and oral temperature can alter the cohesion between the matrix and inorganic particles, resulting in the degradation of these materials by reducing their mechanical properties.46,47 In the current study, the highest mean value of surface microhardness was recorded for Filtek bulk-fill. Zirconia particles may have affected the increase in the VHN values of the Filtek bulk-fill resin material. Tornavoi et al.,⁴⁶ reported that, among different composite resins, the hardness values of composite resins with zirconia content were better than other composites. The microhardness of composite resins depends on a number of factors, such as the content of the organic matrix and the type and shape of the filler particles; the surface microhardness in resin composites is directly related to filler particle ratios.48 In this context, the difference in material contents in our study reveals differences in VHN values.

As a limitation of the current study, we found that specimens significantly differed in changes of color, surface roughness, and microhardness regarding the various chemical composition and filler content particles of the composite resins. Nevertheless, it is known that the overall clinical success rate of composites is affected by multiple interfering components, and it does not seem likely that in vitro testing would help obtain accurate predictions, although some correlations have been reported to affect the success of composites. Due to these facts and the results of this study, we suggest that further studies are needed to reach accurate answers to these questions.

CONCLUSION

Under the conditions of the 10.000 thermocycling, the color change ΔE00 values of the nanohybrid and bulk fill composites ranged between 1.3 and 6.6. Escom100; the Sof-Lex group was found to have the highest mean $\Delta E00$ (6.64 \pm 2.40), and the Filtek bulk-fill group with Optrapol was found to have the lowest mean ΔE00 (1.31 \pm 0.75). There was a significant interaction between the resin composites and finishing/polishing systems for color, surface roughness, and hardness. Escom100 with the Sof-Lex exhibited the lowest Ra values in all groups. Among the polishing groups, the Sof-Lex group exhibited the smoothest surfaces when compared to the Optrapol group. After thermocycling, aging surface roughness values increased and surface hardness values decreased in all of the resin composites. Dentists must take into consideration the content of the resin composites and finishing/polishing systems.

CONFLICT OF INTERESTS

No conflict of interest was declared by the authors. The authors declared that this study received no financial support.

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