

## Surface roughness and morphology of resin composites polished with two-step polishing systems

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The purpose of this study was to investigate surface roughness (Ra) and morphology of supra-nanofilled [Estelite Omega (EO), Estelite  $\Sigma$  Quick (EQ)], micro-hybrid [Esthet.X HD (EHD), G-aenial (GAE)] and nano-hybrid [Clearfil Majesty Posterior (CMP), Charisma Diamond (CD), Beautifil II (BII)] composites polished with two-step polishing systems [Enhance/PoGo (EP); Venus Supra (VS)]. Composite discs, 30 of each type, were prepared. Each composite group was divided into three subgroups: Mylar (control), EP and VS. Ra was evaluated with profilometer. In each composite group, the control had the lowest Ra. With both polishing systems, EO and EQ had significantly the lowest Ra, whereas CMP and BII had the highest. Except for GAE, CD and BII, the differences in Ra between EP and VS in each composite group were significant, showing smoother surfaces for EP. Supra-nanofilled composites created smoother surfaces than nano-hybrids, and their performance was similar or slightly better than that of micro-hybrids.

**Keywords:** Surface roughness, Polishing system, Atomic force microscopy

### INTRODUCTION

The surface quality of resin composite restorations is one of the most important factors determining their clinical success in the oral cavity. The natural gloss and final esthetic of the restoration, abrasivity and wear kinetics<sup>1</sup>; improved mechanical properties<sup>2</sup>; and tactile perception and comfort of the patient<sup>3</sup> are highly associated with surface properties. Moreover, smooth surfaces and margins reduce the risk of biofilm adhesion and maturation, recurrent caries, gingival irritation<sup>4-8</sup> and staining<sup>9</sup>.

The smoothest possible surface is obtained when the resin composite polymerizes against a Mylar matrix without subsequent finishing or polishing<sup>10,11</sup>; however, such a surface has a resin-rich layer, poor mechanical properties, is susceptible to increased wear and discoloration and should be eliminated<sup>2,9</sup>. In addition, in clinical situations, most restorations need to be adjusted to their final shape. Thus, finishing and polishing of restorations are crucial.

Commercially available finishing and polishing systems have a wide variety of abrasives, such as silicon carbide, aluminum oxide, diamond and silicon dioxide, which are impregnated in rubber and aluminum oxide or diamond silica-coated abrasive discs that use one, two or multiple application steps<sup>12</sup>. In several *in vitro* studies, multi-step aluminum-oxide discs exhibited the smoothest surfaces<sup>13-16</sup>; however, due to their geometry, the anatomically contoured surfaces of composite restorations are difficult to polish<sup>12</sup>. In contrast, one- and two-step polishing systems that use elastomeric or rubberized polishers in various shapes, sizes and dimensions come into direct contact with the restoration

surface and complement the access limitations of the aluminum-oxide discs. Two-step polishing systems that use diamond abrasive-impregnated polishers appear to be particularly effective in achieving high surface smoothness similar to<sup>12,17</sup> or better than that achieved by the multi-step aluminum-oxide-coated abrasive disc systems<sup>18</sup>. Thus, the success of the one-step polishing systems was found to be closely related to the initial finishing regimen<sup>18</sup>.

The composition of resin composites has evolved significantly since the materials were first introduced to dentistry more than 50 years ago<sup>19</sup>. Modification of filler concepts, reduction of the filler particle size and increase in filler loading seem to be the most significant changes<sup>20</sup>. Apart from traditional hybrid and micro-hybrid composites, nano-fill and nano-hybrid composites represent the state of the art in terms of filler formulation<sup>19</sup>. Regarding surface roughness after polishing, nano-hybrids may not perform like nano-filled composites<sup>17,21</sup>, but their performance is similar to or slightly better than that of micro-hybrids<sup>21</sup>. To our knowledge, there is no comparison in the literature of surface roughness among the micro-hybrid, nano-hybrid and supra-nanofilled composites polished with two-step polishing systems. Therefore, the purpose of this *in vitro* study was to investigate the surface roughness and the morphology of two supra-nanofilled (Estelite Omega, Estelite  $\Sigma$  Quick), two micro-hybrid (Esthet.X HD, G-aenial) and three nano-hybrid (Clearfil Majesty Posterior, Charisma Diamond, Beautifil II) resin composites polished with aluminum-oxide/diamond-abrasive-impregnated (Enhance/PoGo) and diamond-abrasive-impregnated (Venus Supra) two-step polishing systems. The null hypotheses of this study

are 1) there would be no significant differences in the surface roughness among the two polishing systems for each composite; and 2) there would be no significant difference in the surface roughness among the different types of composites for each polishing system.

## MATERIALS AND METHODS

Seven commercially available resin composites, chosen for their different types of filler particles: two supra-nanofilled [Estelite Omega (EO; Tokuyama Dental Co, Tokyo, Japan), Estelite  $\Sigma$  Quick (EQ; Tokuyama Dental Co, Tokyo, Japan)], two micro-hybrid [Esthet.X HD (EHD; Dentsply Caulk, Milford, DE, USA), G-aenial (GAE; GC Dental Products Corp., Aichi, Japan)] and three nano-hybrid [Clearfil Majesty Posterior (CMP; Kuraray Medical Co, Tokyo, Japan), Charisma Diamond (CD; Heraeus Kulzer, Hanau, Germany),

Beautifil II (BII; Shofu Co, Kyoto, Japan)] and aluminum oxide/diamond-abrasive-impregnated (Enhance/PoGo; Dentsply Caulk, Milford, DE, USA) and diamond-abrasive-impregnated (Venus Supra; Heraeus Kulzer, Hanau, Germany) two-step polishing systems were used in this study. The properties of the resin composites and the composition of the two-step polishing systems and their application modes are listed in Tables 1 and 2. A total of 210 composite discs (shade A2), 30 from each resin composite, 5-mm in diameter and 2-mm-thick were prepared. Each material was inserted into a cylindrical metal mold and pressed between two opposing Mylar matrices, which were then covered with a glass slide 1 mm thick to extrude excess material and to produce a smooth, flat surface. The specimens were then polymerized through the glass slide using a halogen curing unit (Optilux 501, Kerr, CA, USA) according to the manufacturer's instructions with a light intensity

Table 1 The properties of the resin composites tested

Materials	Filler Type	Resin Matrix	Filler Loading Vol%-Wt%	Filler Size	Manufacturer	Lot
Estelite Omega (EO)	supra-nano filled	Silica, zirconia	Bis-GMA, TEGDMA 78% 82%	Mean 0.2 $\mu\text{m}$	Tokuyama Dental Co, Tokyo, Japan	9016
Estelite $\Sigma$ Quick (EQ)	supra-nano filled	Silica, zirconia	Bis-GMA, TEGDMA 71% 82%	0.1–0.3 $\mu\text{m}$ Mean 0.2 $\mu\text{m}$	Tokuyama Dental Co, Tokyo, Japan	E688
Esthet.X HD (EHD)	micro-hybrid	Barium boron fluoroaluminosilicate glass	Bis-GMA, TEGDMA, Bis-EMA 60% 77%	0.4–0.7 $\mu\text{m}$ Mean 0.6 $\mu\text{m}$	Dentsply Caulk, Milford, DE, USA	1201312
G-aenial (GAE)	micro-hybrid	Strontium-lanthanoid fluoride pre-polymerized fillers, silica, fumed silica	UDMA, Dimethacrylate co-monomer 62% 76%	16–17 $\mu\text{m}$ , 850 nm, 16 nm	GC Dental Products Corp., Aichi, Japan	1202012
Clearfil Majesty Posterior (CMP)	nano-hybrid	Glass ceramics, surface treated alumina microfiller, silica	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate 82% 92%	1.5 $\mu\text{m}$ 20 nm	Kuraray Medical Co, Tokyo, Japan	00152
Charisma Diamond (CD)	nano-hybrid	Ba-A-F borosilicate glass, SiO <sub>2</sub> nanofiller	TCD-DI-HEA, UDMA 64% 81%	5 nm–20 $\mu\text{m}$ Mean 0.6 $\mu\text{m}$	Heraeus Kulzer, Hanau, Germany	010037
Beautifil II (BII)	nano-hybrid	S-PRG, multifunctional Aluminofluoroborosilicate glass	Bis-GMA, TEGDMA, UDMA 68% 83%	0,1–4 $\mu\text{m}$ Mean 0.8 $\mu\text{m}$	Shofu Co, Kyoto, Japan	031224

Bis-GMA: bisphenol-A-diglycidyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate; S-PRG: surface reaction type pre-reacted glass-ionomer.

Table 2 Composition of the two-step polishing systems and their application modes

Polishing systems	Matrix	Abrasives	Particle Size	rpm	Manufacturer	Lot
Enhance	Polymerized urethane dimethacrylate	Al <sub>2</sub> O <sub>3</sub>	40 µm	10,000	Dentsply Caulk,	111024
PoGo	Polymerized urethane dimethacrylate	Diamond	7 µm	10,000	Milford, DE, USA	
Venus Supra	Urethane polymer	Diamond (70%)	40 µm	7,500	Heraeus Kulzer,	230024
Pre-polisher	Urethane polymer	Diamond (65%)	4–8 µm	7,500	Hanau, Germany	
High Gloss Polisher						

of 500 mW/cm<sup>2</sup>. Following storage in distilled water for 24 h at 37°C, the specimens in each composite group were randomly divided into three subgroups ( $n=10$ ): 1) Mylar matrix group (control), 2) Enhance/PoGo group, 3) Venus Supra group.

The Mylar matrix group received no polishing treatment. In the Enhance/PoGo group, the specimens were first wet-polished for 20 s with Enhance at a low speed (10,000 rpm), thoroughly rinsed with water for 10 s to remove debris and then air-dried for 5 s. Then, the specimens were wet-polished with PoGo at 10,000 rpm for 40 s, rinsed for 10 s and air-dried for 5 s. In the Venus Supra group, the specimens were wet-polished with a pre-polisher for 20 s at low speed (7,500 rpm), thoroughly rinsed with water for 10 s to remove debris, air-dried for 5 s and then high-gloss polished for 40 s at low speed (7,500 rpm). The same protocol was repeated for rinsing and drying.

Disc-shaped polishers were preferred with both finishing treatments because they come into direct contact with the surfaces of the specimens. Each polisher was used only once, with the same low-speed hand piece (Kavo 80E, Kavo Dental, Charlotte NC, USA) for all specimens. Prior to polishing the specimens, composite surfaces were pre-roughened with 320-grit silicon carbide (SiC) paper for 30 s<sup>10,22</sup>. Pre-roughening was standardized using a polishing machine (Buehler, IL, USA) at a rotation speed of 400 rpm, and constant moving action was applied under water coolant to prevent heat build-up. A new SiC paper was used for each specimen and discarded after each application. All specimen preparation, finishing and polishing procedures were performed by the same operator. The surface roughness of the specimens was evaluated with a profilometer (Perthometer M1 Mahr, Göttingen, Germany). For each specimen, five measurements at different locations and in different directions, with a cut-off length of 0.25 mm, a tracing length of 0.8 mm and a stylus speed of 0.1 mm/s, were recorded, and the roughness value (Ra; µm) was calculated as the average of these five readings. The second operator, who was blind to the polishing systems, as well as to the type of composite, performed all of the roughness evaluations. During the experimental period, the surface-roughness tester was periodically calibrated (Mahr GmbH, Göttingen, Germany).

#### Statistical analysis

The effect of polishing systems on the surface roughness in each composite group was statistically analyzed using one-way ANOVA, followed by Tukey's test. The comparison of the composites in terms of different polishing systems was performed by two-way ANOVA and a *post-hoc* Bonferroni test at a significance level of  $p<0.05$  (SPSS, 20.0; Chicago, IL, USA).

#### Scanning electron (SEM) and atomic force (AFM) microscopy

For surface characterization, two representative specimens from each group with Ra values close to the mean values were selected. One specimen was coated with gold and examined under a scanning electron microscope (JSM-5600, JEOL, Tokyo, Japan), whereas the other was observed with a commercial atomic force microscope (Veeco metrology Group Inc., Santa Barbara, CA, USA), using the contact mode. Cantilevers with a constant spring of 0.1 N/m and Nanoprobe SPM Tips, OTR 8-35 type were used. Deflection and height-mode images were obtained simultaneously with a resolution of 512×512 pixels. Images were acquired in 10×10-µm sizes and analyzed with specific software (Nanoscope v616r1, Veeco Metrology Inc., Santa Barbara, CA, USA and WSxM 4.0 Develop 11.1, Nanotec Electronica S.L. Trea Cantas, Spain).

## RESULTS

Mean surface-roughness values (Ra, µm), standard deviations ( $\pm$ SD), and statistical analysis of the control and polished resin composites are shown in Table 3.

In each composite group, the smoothest surfaces were obtained in the Mylar matrix group (control), whereas both polishing systems created significantly rougher surfaces than their corresponding control groups ( $p<0.05$ ). However, when the Mylar matrix groups were compared, no significant differences were found among the composites ( $p>0.05$ ).

Results of the two-way ANOVA indicated that the composite ( $p<0.0001$ ), the polishing system ( $p<0.0001$ ), and the interaction between them were statistically significant ( $p<0.0001$ ). Regarding the Enhance/PoGo polishing system, significantly smoother surfaces ( $p<0.05$ ) were obtained with the supra-nanofilled composites EO, EQ and the micro-hybrid composite EHD, which were not significantly different from each

Table 3 Mean surface roughness values (Ra,  $\mu\text{m}$ ), standard deviations ( $\pm\text{SD}$ ) and statistical analysis of the control and polished supra-nanofilled, micro-hybrid and nano-hybrid composites

Materials	Control	Two-step Polishing Systems	
	Mylar Matrix	Enhance/PoGo	Venus Supra
Estelite Omega (EO)	0.027 $\pm$ (0.003) <sup>a,A</sup>	0.063 $\pm$ (0.010) <sup>a,B</sup>	0.078 $\pm$ (0.009) <sup>a,C</sup>
Estelite $\Sigma$ Quick (EQ)	0.025 $\pm$ (0.003) <sup>a,A</sup>	0.060 $\pm$ (0.004) <sup>a,B</sup>	0.074 $\pm$ (0.004) <sup>a,C</sup>
Esthetix HD (EHD)	0.023 $\pm$ (0.002) <sup>a,A</sup>	0.060 $\pm$ (0.004) <sup>a,B</sup>	0.102 $\pm$ (0.022) <sup>b,C</sup>
G-aenial (GAE)	0.027 $\pm$ (0.03) <sup>a,A</sup>	0.085 $\pm$ (0.01) <sup>b,B</sup>	0.078 $\pm$ (0.013) <sup>a,B</sup>
Clearfil Majesty Posterior (CMP)	0.041 $\pm$ (0.007) <sup>a,A</sup>	0.120 $\pm$ (0.010) <sup>a,B</sup>	0.147 $\pm$ (0.042) <sup>c,C</sup>
Charisma Diamond (CD)	0.035 $\pm$ (0.003) <sup>a,A</sup>	0.109 $\pm$ (0.009) <sup>a,B</sup>	0.112 $\pm$ (0.01) <sup>b,B</sup>
Beautifil II (BII)	0.039 $\pm$ (0.004) <sup>a,A</sup>	0.129 $\pm$ (0.028) <sup>c,B</sup>	0.140 $\pm$ (0.027) <sup>c,B</sup>

Different superscript letters in each column and different capital letters in each row indicate significant differences at  $p < 0.05$ .

other ( $p > 0.05$ ). With this polishing system, the nano-hybrids CMP and BII exhibited significantly the highest Ra values ( $p < 0.05$ ), which did not significantly differ from each other ( $p > 0.05$ ). In addition, the nano-hybrid CD and the micro-hybrid GAE exhibited intermediate Ra values that were significantly different from both the smoothest and roughest composites ( $p < 0.05$ ).

With the Venus Supra polishing system, the supra-nanofilled composites EO, EQ and the micro-hybrid composite GAE showed significantly lower Ra values than any other composites, ( $p < 0.05$ ), and the difference between them was not significant ( $p > 0.05$ ). There were no significant differences between the Ra values of the nano-hybrids CMP and BII ( $p > 0.05$ ), which exhibited the roughest surfaces of all of the composites tested ( $p < 0.001$ ). The surface roughnesses of the micro-hybrid EHD and the nano-hybrid CD were significantly different from those of the roughest and smoothest composites ( $p < 0.05$ ), but no significant difference was observed between them ( $p > 0.05$ ).

Except for the micro-hybrid composite GAE ( $p = 0.332$ ) and the nano-hybrid composites CD ( $p = 0.616$ ) and B II ( $p = 0.411$ ), the differences in surface roughness between the Enhance/PoGo and Venus Supra polishing systems in each composite group were significant, showing smoother surfaces for the Enhance/PoGo polishing system ( $p < 0.05$ ).

#### Scanning electron-microscopy observations

Scanning electron micrographs of the Mylar matrix (control) groups showed homogeneous surface texture with some matrix imperfections and a resin rich layer (Figs. 1a, 3a, 5a, 7a, 9a, 11a, 13a).

Enhance/PoGo polishing system created smooth surfaces on the supra-nanofilled composites EO and EQ (Figs. 1b and 3b, respectively), whereas the presence of several narrow scratch lines and white spots were characteristic of Venus Supra polishing system (Figs.

1c and 3c, respectively). Enhance/PoGo exhibited a relatively uniform surface with some slight scratch lines on the micro-hybrid EHD (Fig. 5b), whereas narrow, deep scratch lines that caused irregularity were evident after polishing with Venus Supra (Fig. 5c). Both polishing systems created similar morphologies on the microhybrid GAE, a homogeneous surface with white spots (Figs. 7b, 7c). A high density of inorganic fillers with some filler debonding was evident on the nano-hybrid CMP after polishing with Enhance/PoGo (Fig. 9b); however, debonding of filler particles was more prominent after polishing with Venus Supra (Fig. 9c). Resin removal and filler protrusions were observed on the nano-hybrid composites CD and BII after polishing with Enhance/PoGo (Figs. 11b and 13b, respectively), whereas Venus Supra exhibited resin removal along with some filler debonding (Figs. 11c and 13c, respectively).

#### Atomic force microscopy (AFM) observations

The Mylar matrix (control) groups showed uniform surfaces, with some matrix imperfections (Figs. 2a, 4a, 6a, 8a, 10a, 12a, 14a). A small number of air voids were evident on supra-nanofilled composites EO and EQ (Figs. 2a and 4a respectively) and nano-hybrid composites CMP, CD and BII control groups (Figs. 10a, 12a and 14a, respectively) which were not evident on SEM. For all composites, polished specimens generally presented a more irregular topography than their control groups.

The Enhance/PoGo polishing system created slight uniform irregularities on the supra-nanofilled composites EO and EQ (Figs. 2b and 4b, respectively) and the micro-hybrid composite EHD (Fig. 6b). On the other hand, the Venus Supra polishing system created several narrow scratch lines on EO (Fig. 2c), undulating surface topography on EQ (Fig. 4c) and deep scratch lines on EHD (Fig. 6c). The micro-hybrid composite GAE exhibited deep and superficial scratch lines on AFM with the Enhance/PoGo (Fig. 8b) and

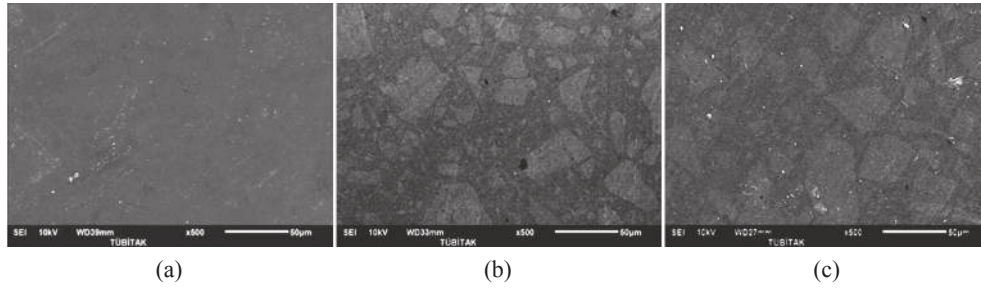


Fig. 1 Scanning electron micrographs of EO (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

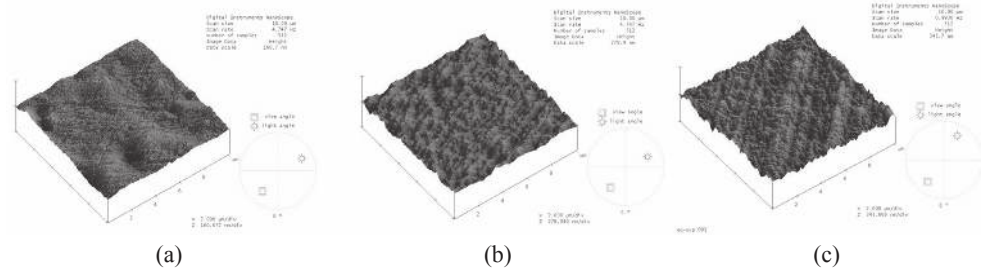


Fig. 2 Atomic force microscope micrographs of EO (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

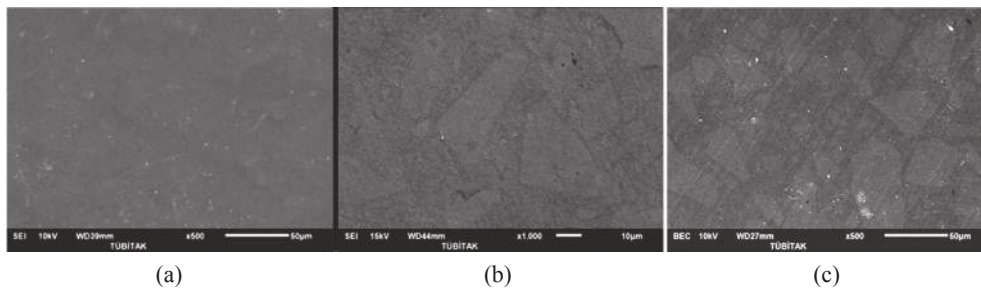


Fig. 3 Scanning electron micrographs of EQ (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

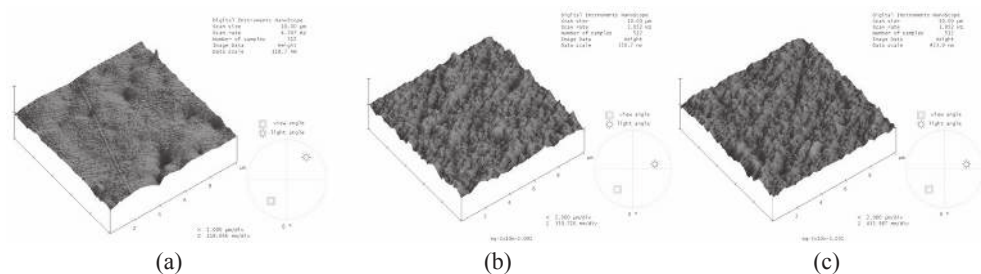


Fig. 4 Atomic force microscope micrographs of EQ (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

Venus Supra (Fig. 8c) polishing systems, which were not evident on SEM. Regarding the nano-hybrid composite CMP, both polishing systems revealed resin abrasion between the fillers, along with deep voids that represent debonded fillers (Figs. 10b and 10c). Undulating surface

topography on CD was observed with both of the polishing systems (Figs. 12b and 12c); however, debonded fillers were evident only after polishing with Venus Supra (Fig. 12c). Irregular surface topography due to the protrusion of fillers was observed on BII with Enhance/

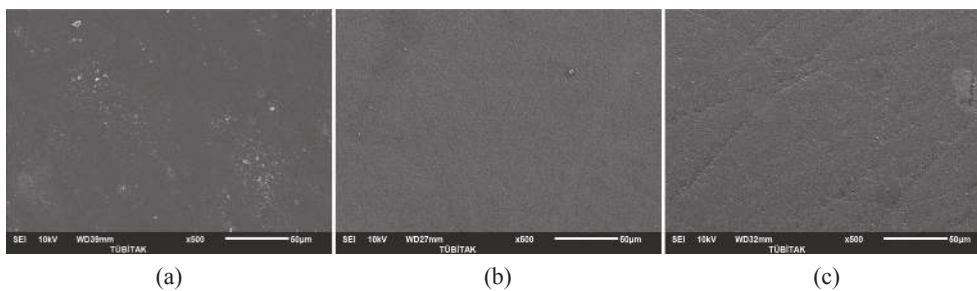


Fig. 5 Scanning electron micrographs of EHD (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

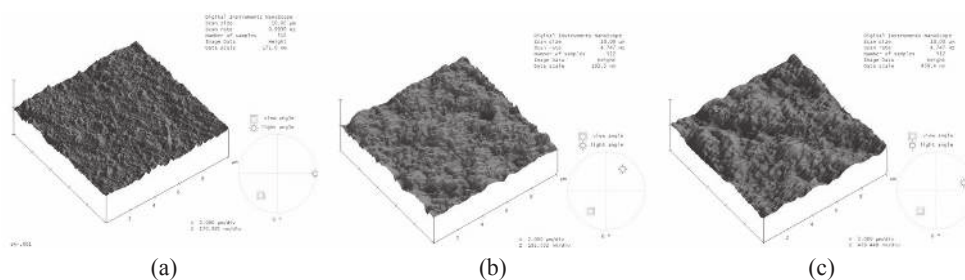


Fig. 6 Atomic force microscope micrographs of EHD (a) Mylar, (b) Enhance/ PoGo, (c) Venus Supra groups.

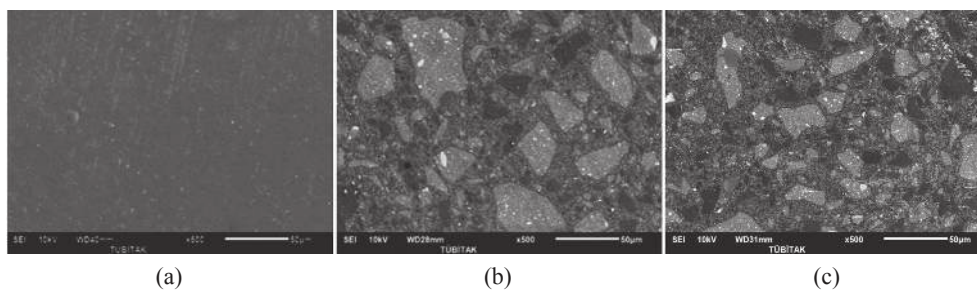


Fig. 7 Scanning electron micrographs of GAE (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

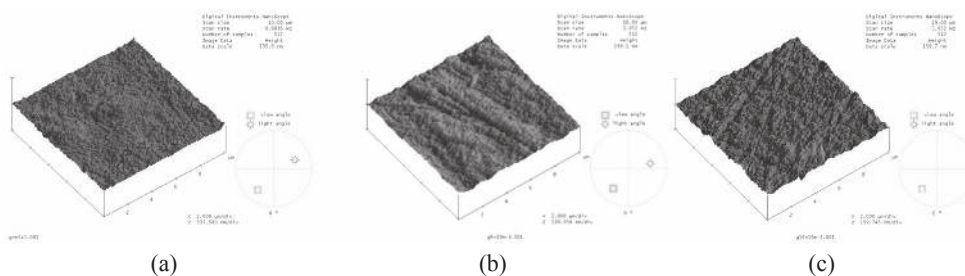


Fig. 8 Atomic force microscope micrographs of GAE (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

PoGo (Fig. 14b), whereas deep voids representing debonded fillers were evident after polishing with Venus Supra (Fig. 14c).

## DISCUSSION

Surface roughness is the most frequently used parameter in assessing the surface quality of different restorative materials. Due to the limitations of the

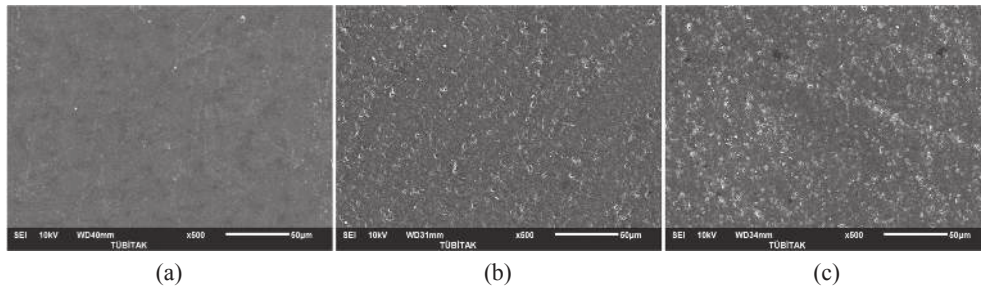


Fig. 9 Scanning electron micrographs of CMP (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

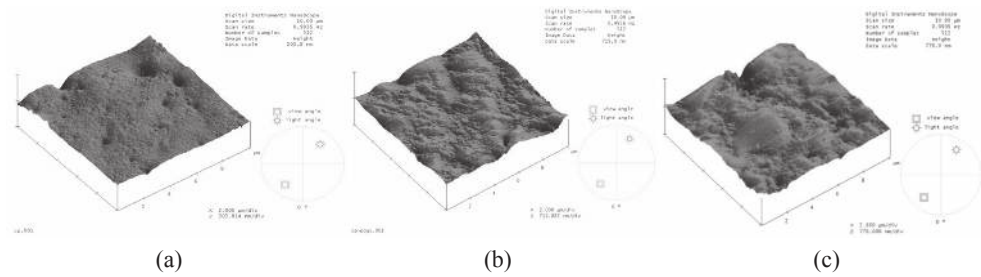


Fig. 10 Atomic force microscope micrographs of CMP (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

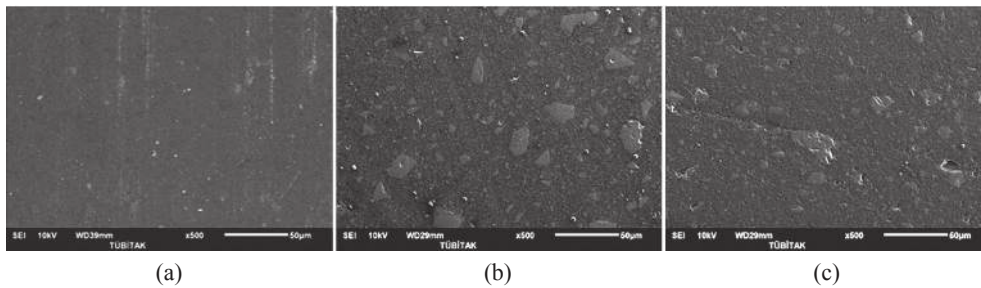


Fig. 11 Scanning electron micrographs of CD (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

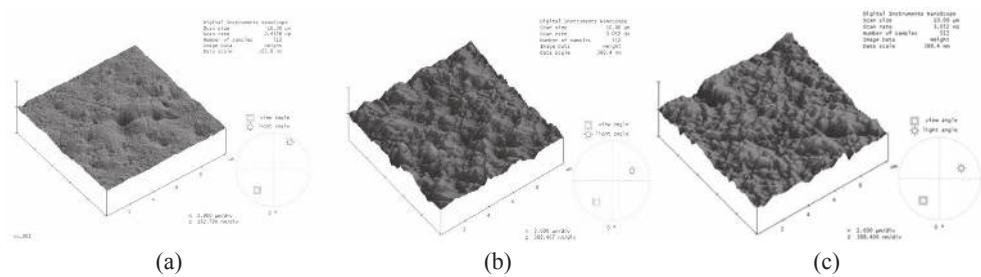


Fig. 12 Atomic force microscope micrographs of CD (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

quantitative measurement methods, the results are often verified qualitatively with scanning electron microscopy (SEM) to demonstrate shape and contour changes that may not be shown by the profilometer<sup>23)</sup>; however, SEM also has limitations in defining the

surface topography because it does not allow for visualization of the three-dimensional surface texture<sup>24)</sup>. Therefore, atomic force microscopy (AFM) has recently been employed in dental-materials research to provide three-dimensional detailed topographical images of

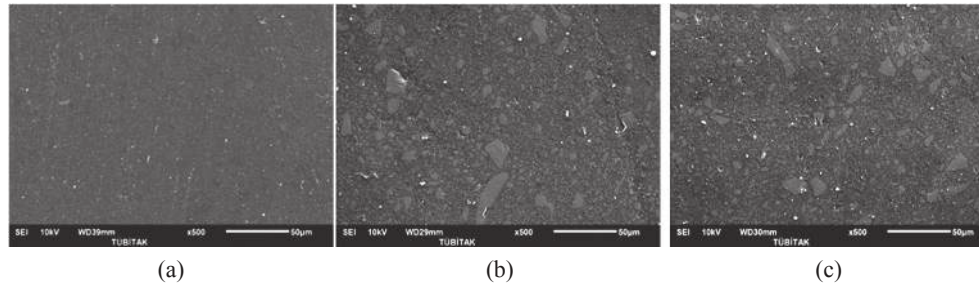


Fig. 13 Scanning electron micrographs of BII (a) Mylar, (b) Enhance/PoGo, (c) Venus Supra groups.

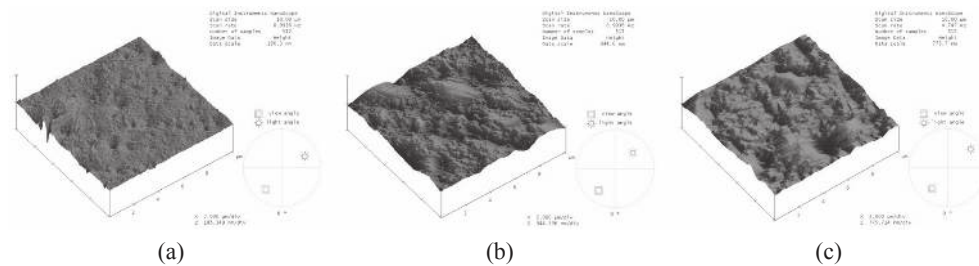


Fig. 14 Atomic force microscope micrographs of BII (a) Mylar, (b) Enhance/PoGo and (c) Venus Supra groups.

surface roughness at a nanometer resolution<sup>25</sup>). This study aimed to investigate the surface roughness and morphology of supra-nanofilled, nano-hybrid and micro-hybrid resin composites polished with two different types of two-step polishing systems (aluminum-oxide/diamond-abrasive impregnated and diamond-abrasive impregnated) using both quantitative (profilometer) and qualitative (SEM, AFM) methods.

The fabrication, shape and dimension of the specimens, inter-individual differences between various operators<sup>26</sup>, polishing time, applied force, rotation speed of the handpiece and water spray can significantly affect the results<sup>22</sup>. Pre-roughening with diamond burs results in a non-homogeneous surface texture<sup>22</sup> and creates different surface roughnesses on different materials<sup>22,27</sup>. Therefore, in this study, pre-roughening was standardized using a polishing machine with 320-grit SIC paper that generated roughness similar to that created with a 30/40- $\mu\text{m}$  diamond bur, which represents clinical contouring and finishing<sup>22</sup>. With both polishing systems, according to the manufacturer's instructions, the finishing step was accomplished in 20 s and the polishing step was completed in 40 s. To eliminate inter-individual differences in manual polishing that could substantially affect the results, all of the finishing and polishing procedures were performed by the same operator. All of the roughness evaluations were performed by a second operator who was blind both to the materials and to the polishing systems.

Several studies have demonstrated that the smoothest composite surfaces were achieved with the Mylar matrix; however, clinicians seldom leave composite restorations unfinished and unpolished, which

would significantly increase the surface roughness<sup>10,11</sup>. These findings are in agreement with the results of this study. Both of the two-step polishing systems created higher Ra values than for those same composites with the Mylar matrix-finished surfaces; however, the Mylar matrix-created surfaces are less characteristic of the bulk material used, and the surface roughness is mostly related to the Mylar itself<sup>28</sup>. Comparison of the Mylar matrix groups among the tested composites supports the hypothesis, showing no significant differences between the composites ( $p>0.05$ ). Consistent with the qualitative results, SEM observations also revealed homogeneous surface textures, with some matrix imperfections and a resin-rich layer (Figs. 1a, 3a, 5a, 7a, 9a, 11a, 13a), whereas AFM detected a low surface profile (Figs. 2a, 4a, 6a, 8a, 10a, 12a, 14a) for all tested composites.

Based on the results, the first null hypothesis that there would be no significant differences in surface roughness between two two-step polishing systems for each composite was accepted only for the micro-hybrid GAE ( $p=0.332$ ) and the nano-hybrids CD ( $p=0.616$ ) and B II ( $p=0.411$ ). Regarding supra-nanofilled composites EO and EQ, micro-hybrid EHD and the nano-hybrid CMP, the differences between the Enhance/PoGo and Venus Supra polishing systems in each composite group were significant, showing smoother surfaces for Enhance/PoGo ( $p<0.05$ ). PoGo is a one-step polishing system and can be used without any finishing treatment; however, according to the manufacturer, finishing can be accomplished with  $\text{Al}_2\text{O}_3$ -abrasive-impregnated Enhance and polishing can be performed with diamond-impregnated PoGo. For that reason, in this study, Enhance/PoGo was classified as a two-step



polishing system. On the other hand, Venus Supra is a two-step polishing system that consists of a diamond-impregnated pre-polisher and a diamond-impregnated high-gloss polisher. The efficiency of finishing/polishing systems is related to the type of abrasive material, particle size, hardness, shape of the abrasive and the speed and pressure used during application<sup>11</sup>. Therefore, for both of the two-step polishing systems, disc-shaped polishers were preferred because they come into direct contact with the specimens. During application, the time was fixed at 20 s for the first step and 40 s for the second step, whereas the rotation speed was set according to the manufacturer's instructions. As the second step of the two-step polishing systems (PoGo and Venus Supra high gloss polisher) involves diamond-impregnated polishers with nearly the same grit size (7  $\mu\text{m}$  and 4–8  $\mu\text{m}$ , respectively), the differences in Ra values could be explained either by the quantity of abrasives used in the instrument or by the type of abrasive material used for the finishing. Enhance contains an  $\text{Al}_2\text{O}_3$  abrasive (40  $\mu\text{m}$ ), and the Venus Supra pre-polisher is diamond-impregnated (40  $\mu\text{m}$ ). The hardness of the  $\text{Al}_2\text{O}_3$  abrasive is significantly higher than that of most of the filler particles used in resin composites<sup>29</sup>. This difference may lead to equal abrasion of the filler particles with the resin matrix, leaving a smooth surface<sup>30</sup>. On the other hand, diamond is harder than  $\text{Al}_2\text{O}_3$ ; therefore, it may cause deeper scratches on the composite's surfaces, resulting in higher roughness<sup>14</sup>. This result is consistent with the SEM and AFM observations of the supra-nanofilled composites EO (Figs. 1b and 2b) and EQ (Figs. 3b and 4b) and the micro-hybrid composite EHD (Figs. 5b and 6b), on which Enhance/PoGo created a smoother surface topography than Venus Supra. Similarly, Endo *et al.*<sup>17</sup> and Jung *et al.*<sup>18</sup> described detrimental surface alteration effect of relatively large diamond particles in finishing instruments on resin composites.

In addition to the finishing and polishing treatments, the surface roughness of the composites is also influenced by several material factors, such as the type, shape, size and distribution of the inorganic fillers<sup>10,31</sup>. The surface roughness has been decreased by decreasing the filler size<sup>18</sup> and increasing the filler content<sup>11</sup>. Use of a finer filler size results in less interparticle spacing, more protection of the softer resin matrix and less filler plucking<sup>21</sup>; however, during polishing, it is still difficult to avoid the occurrence of irregularities at the interface between the filler particles and the resin because they have different levels of hardness<sup>32</sup>. According to the results of this study, the second null hypothesis, that there would be no significant differences in surface roughness among the different types of composite for each polishing system, was rejected. With both polishing systems, the supra-nanofilled composites EO and EQ presented the lowest Ra values, whereas the nano-hybrids CMP, CD and BII showed significantly higher values ( $p < 0.05$ ). The Ra data after polishing correlated well with the mean filler size of these materials. The mean filler size of EO and EQ (0.2  $\mu\text{m}$ ) was the lowest among the tested materials,

which may explain why it yielded the lowest Ra values; nano-hybrid composites with larger filler sizes, *e.g.*, CMP (0.02–1.5  $\mu\text{m}$ ), CD (0.6  $\mu\text{m}$ ) and BII (0.8  $\mu\text{m}$ ) yielded higher Ra values. Another possible explanation for the smoothness of the surfaces achieved with supra-nanofilled composites can be the spherical shape of their fillers<sup>33</sup>. Composites filled with this type of filler have resulted in lower roughness and higher gloss values than nano-hybrid composites filled with irregularly shaped fillers<sup>34,35</sup> that are similar to the tested nano-hybrid composites (CMP, CD, BII) that contain irregular glass fillers. The differences in surface morphology after polishing between the supra-nanofilled composites and nano-hybrid composites were clearly observed on SEM and AFM. EO and EQ showed smooth surfaces on SEM and lower surface profile on AFM after using Enhance/PoGo (Figs. 1b–2b and 3b–4b, respectively) and presented narrow scratch lines after using Venus Supra (Figs. 1c–2c and 3c–4c, respectively). On the other hand, resin matrix abrasion, filler protrusions and some filler debonding were the characteristic features of the nano-hybrid composites CMP (Figs. 9b, c–10b, c), CD (Figs. 11b, c–12b, c) and BII (Figs. 13b, c–14b, c) with both of the polishing systems. Consistent with the present data, Ergücü *et al.*<sup>10</sup> and Endo *et al.*<sup>17</sup> showed higher Ra values and rougher surfaces, that are characterized with protrusion and debonding of fillers, for the nano-hybrids compared to a nano-filled composite.

When the tested nano-hybrids were compared, CMP, CD and BII exhibited similar Ra values with Enhance/PoGo. Similarly, Jung *et al.*<sup>18</sup> indicated no significant differences between nano-hybrids after polishing with Enhance/PoGo. In contrast, CMP and BII yielded significantly higher Ra values than CD with Venus Supra. Although CMP, CD and BII all contain irregular glass fillers, they differ from each other in terms of other types of fillers, filler loading and type of resin matrix (Table 1). CMP includes glass ceramics and alumina nanofiller and has the highest filler loading (82% vol; 92% wt) among the tested composites. Higher filler content is expected to protect the resin matrix from excessive abrasion, resulting in smoother surfaces<sup>11</sup>; however, fillers that are much harder than the resin matrix may cause prominent matrix abrasion during polishing<sup>36</sup>, which was also observed with the other nano-hybrids. The abrasion of the softer resin matrix may result in a lack of support of the fillers, leading to further filler debonding and roughening of the surface<sup>36</sup>. As shown in SEM and AFM, debonding of the inorganic filler particles was more prominent with CMP (Figs. 9c and 10c) than with CD (Figs. 11c and 12c), which corresponded well to its high surface roughness with Venus Supra. On the other hand, BII comprises surface-reaction-type pre-reacted glass-ionomer filler, with a relatively large mean filler size (0.8  $\mu\text{m}$ ) compared to the smaller mean filler size of CD (0.6  $\mu\text{m}$ )<sup>35</sup>. The greater Ra values of BII corresponded to the larger fillers that were exposed after polishing with Venus Supra and, consequently, yielded a rougher surface profile (Fig. 14c) than CD (Fig. 12c).

In general, it is difficult to distinguish nano-hybrids from micro-hybrids because nano-hybrids also contain a range of filler sizes<sup>37</sup>. In this study, the micro-hybrid GAE yielded significantly lower Ra values than the nano-hybrids CMP, CD and BII with both of the polishing systems, whereas the micro-hybrid EHD exhibited significantly smoother surfaces than the nano-hybrids only with Enhance/PoGo. In contrast to the nano-hybrids, the micro-hybrids EHD and GAE did not present any filler protrusion or filler debonding on SEM and AFM (Figs. 5b, c–6b, c and Figs. 7b, c–8b, c). The non-uniform abrasion of the resin matrix and the fillers of the nano-hybrids may explain the difference in roughness between the micro-hybrid and nano-hybrid composites. These results are in accordance with those of Gönülol and Yılmaz<sup>16</sup>; nano-hybrids exhibited similar or rougher surfaces compared to a micro-hybrid composite using seven different polishing systems.

GAE revealed similar Ra values for both of the polishing systems. Neither resin removal nor filler debonding was observed in SEM and AFM (Figs. 7b, 7c and 8b, 8c, respectively). On the other hand, EHD exhibited significantly rougher surfaces with Venus Supra than with the Enhance/PoGo polishing system, consistent with the observations from the SEM (Figs. 5b and 5c, respectively) and AFM (Figs. 6b and 6c, respectively). The differences in Ra values between these two micro-hybrids were also significant for each polishing system (Table 3). GAE and EHD have almost the same filler loading (62% Vol; 76% Wt and 60% Vol; 77% Wt, respectively). Their differences in roughness can be attributed to the type and size of the inorganic fillers and the type and ultimate degree of cure of the resin matrix<sup>9,10,31</sup>. The lower hardness of UDMA-based resins compared to Bis-GMA-based resins has been attributed to differences in their degree of polymerization, molecular rigidity and final strength<sup>38</sup>. Therefore, the incorporation of 2 types of pre-polymerized fillers with relatively lower hardness than the glass fillers<sup>11,39</sup> and UDMA as a major component of the resin matrix may account for the similar abrasion of the fillers with the resin matrix in GAE.

With both polishing systems, the supra-nanofilled composites EO and EQ behaved similarly to or slightly better than the micro-hybrids EHD and GAE. Micro-hybrids might have been expected to show higher Ra values because of their larger filler sizes (EHD 0.6  $\mu\text{m}$ ; GAE 16–17  $\mu\text{m}$ , 16 nm, 850 nm) than the supra-nanofilled composites (0.2  $\mu\text{m}$ ). In addition, the smaller, the specific surface areas of spherical fillers require less resin matrix to wet them and thus allow for higher filler loading<sup>14</sup> in EO and EQ than EHD and GAE; however, comparison between these two groups showed no material and polishing system dependent effect.

AFM can provide three-dimensional data on surface topography which cannot be visualized by SEM<sup>25</sup>. Thus, in this study, air voids in control groups of EO, EQ, CMP, CD, BII and polishing scratches on GAE were detected on AFM. These features were not visible in the SEM images. The differences between SEM and AFM

techniques suggest that AFM can offer more detailed definition of surface topography.

Based on studies using mechanical profilometry devices, the critical threshold Ra value for the simultaneous increase in plaque accumulation is 0.2  $\mu\text{m}$ <sup>4</sup>, whereas a surface roughness of 0.25–0.5  $\mu\text{m}$  can be detected by the patient's tongue<sup>3</sup>. According to the results, the mean surface roughness achieved with the Enhance/PoGo and Venus Supra polishing systems on the supra-nanofilled, micro-hybrid and nano-hybrid composites were below the clinically acceptable threshold value and were highly satisfactory; however, under the dynamic conditions of the oral environment, an increase in surface roughness is expected. Therefore, further evaluation of the impact of aging on surface roughness is necessary.

## CONCLUSION

Under the limitations of this *in vitro* study, the following may be concluded:

1. Supra-nano spherical filled composites polished with two-step polishing systems created smoother surfaces than nano-hybrid composites and performed similarly to or slightly better than the micro-hybrids.
2. The surface roughness of micro-hybrid and nano-hybrid composites seems to be dependent on materials and polishing systems.
3. An aluminum oxide/diamond-abrasive-impregnated two-step polishing system created smoother surfaces than the diamond-abrasive-impregnated two-step polishing system on supra-nano spherical filled composites.

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