

EVALUATION OF THE ROUGHNESS OF COMPOSITE RESINS SUBMITTED TO DIFFERENT SURFACE TREATMENTS

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ABSTRACT

The aim of this study was to evaluate the surface roughness of restorative composite resins after polishing with aluminum oxide discs and applying an adhesive layer. The following composite resins were used: Filtek Z250 (hybrid, 3M ESPE, A2) and Filtek Supreme XT (nanofilled, 3M ESPE, A2E). Thirty specimens of each composite were made using a condensation silicone mold (5.0 x 2.0 mm) into which the composites were inserted and submitted to light pressure. After polymerization using the halogen light source Curing Light 2500 (3M) for 40 seconds, the specimens were assigned to the following groups: G1-Z250/CO – control, did not receive any treatment; G2-Z250/SL – the specimens underwent finishing and polishing with Sof-Lex discs; G3-Z250/ADE, application of an adhesive layer on the top of the specimen and light curing for 20 seconds. Groups G4, G5 and G6 followed the same treatment sequence, but using Filtek

Supreme XT. The specimens were stored in deionized water at 37°C for 24h. Three readings of surface roughness were made for each specimen. The results were submitted to variance analysis by Two-Way ANOVA Test and Tukey HSD Test. The mean values obtained were: G3 (0.2325 ± 0.1484 μm) and G6 (0.2266 ± 0.0463 μm), which were higher than the other groups and did not differ statistically from each other. Groups G1 (0.1023 ± 0.0464 μm), G4 (0.1083 ± 0.0241 μm), G5 (0.1160 ± 0.0252 μm) and G2 (0.1360 ± 0.0131 μm) had the lowest average roughness and did not differ statistically among each other. It was concluded that the Sof-Lex discs performed better for the surface treatment of the composites resins tested, producing similar values of surface roughness for both composites. Covering with dentin adhesive increased the surface roughness in both composites.

Keywords: Polymers, methacrylates, dental polishing.

EVALUACIÓN DE LA RUGOSIDAD DE RESINAS COMPUESTAS DESPUÉS DE DIFERENTES TRATAMIENTOS DE SUPERFICIE

RESUMEN

El propósito del presente estudio fue evaluar la rugosidad superficial de resinas compuestas después de ser pulidas con discos de óxido de aluminio y de aplicar una capa de adhesivo. Se utilizó resina Filtek Z250 y Filtek Supreme XT. Se fabricaron treinta especímenes de cada resina utilizando una matriz de silicona (5,0 x 2,0 mm). Después de su polimerización por 40 segundos, se formaron los siguientes grupos: G1-Z250/CO – control, que no recibió ningún tratamiento; G2-Z250/SL – los especímenes fueron acabados y pulidos con discos Sof-Lex; G3-Z250/ADE – se aplicó una capa de adhesivo en la parte superficial de los especímenes polimerizada por 20 segundos. Los grupos G4, G5 y G6 siguieron el mismo patrón, utilizando resina Filtek Supreme XT. Tres lecturas de rugosidad superficial fueron hechas en cada espécimen. Se

evaluaron mediante las pruebas de ANOVA Two-Way y Tukey HDS ($p = 0,05$), obteniendo los siguientes valores: G3 (0.2325 ± 0.1484 μm) y G6 (0.2266 ± 0.0463 μm) obtuvieron valores superiores a los otros grupos sin diferencia estadística. G1 (0.1023 ± 0.0464 μm), G4 (0.1083 ± 0.0241 μm), G5 (0.1160 ± 0.0252 μm) y G2 (0.1360 ± 0.0131 μm) obtuvieron los menores valores de rugosidad superficial sin diferencia estadística. Se concluyó que los discos Sof-Lex presentaron un mejor desempeño para el tratamiento superficial de las resinas compuestas, siendo capaces de producir valores similares de rugosidad de la superficie de ambos compuestos. La aplicación de una capa hidrofóbica de monómeros en las resinas produjo una elevada rugosidad superficial.

Palabras clave: Polímeros, metacrilatos, pulido dental

INTRODUCTION

Due to the increasing aesthetic requirements of patients and the evolution of the physical, optical and mechanical properties of composite resins, this material has been consecrated as one of the most widely used in dental practice. Other advantages include corrosion resistance, non-conduction of electric current and mercury-free structure¹. Since the advent of composite resins, in attempt to reduce surface roughness in order to obtain better optical properties, manufacturers have reduced the mean size of the inorganic

particles^{2,3}. In composite resins with particles ranging from 0.02 to 0.04 μm, classified as microfilled, about 50% of the volume of the material is resin. They have excellent surface smoothness⁴. However, their physical and mechanical properties are inferior to those of conventional resins. Hybrid composite resins contain particles ranging from 0.6 to 1.0 μm and were developed in order to achieve better surface smoothness and excellent physical and mechanical properties^{5,6}. A new category of composite resins introduced to the market was developed using a nanoparticle

technology. The nanomers are inconspicuous particles with sizes ranging from 20 to 75 nm and the nanoclusters consists of silica and zirconia nanoparticles interlinked, reaching a mean size of 0.6 μm^1 . This formulation, combining nanoparticles and nanoclusters, reduces the interstitial spaces among the inorganic particles, providing better physical properties and polish maintenance¹, which can be seen in the surface texture, and reduces the material degradation over the years⁷. This technology also achieved enough mechanical properties to allow its use in both anterior and posterior teeth^{7,8}.

The aesthetic and clinical properties of composite resins depend not only on their structure, but also on the finishing and polishing, which are of great importance in achieving greater longevity of the restoration. The marginal finishing as well as the surface roughness and integrity may affect bacterial plaque retention⁹ and evolve into periodontal disease^{1,10,11}. Furthermore, the surface roughness may directly influence the deterioration¹² and the marginal integrity of the restorations¹³.

The finishing process removes excess material, obtaining particles larger than 25 μ , while polishing obtains particles smaller than 25 μm^{14} . Some authors report that since the introduction of composite resin, many studies have been conducted in order to develop a finishing and polishing procedure to achieve a smooth surface on the restoration^{15,16}.

Various finishing and polishing systems are available on the market, including abrasive diamond tips, silicon discs, aluminum oxide discs, abrasive rubbers and several polishing pastes containing thin abrasive particles^{12,17}.

Attar¹, Perez et al.¹⁰ e Cilli et al.¹⁸ mention a liquid polishing system introduced on the market with the purpose of reducing the need for manual polishing. It is a light-curable composite resin formulation which does not form an oxygen-inhibited surface layer (BisCover, Bisco Inc., Schaumburg, IL, USA). Several studies have been conducted using pit and fissure sealants, dentin adhesives and surface protective agents¹⁹⁻²¹ in order to assess the capacity of these materials to reduce microleakage in restorations. However, information about the real action of these materials on the surface roughness of odontological composites is still uncertain in literature.

Other surface sealants are commercially available in several combined monomers such as Bis-GMA (bisphenol glycidyl dimethacrylate), TEGDMA (tri-

ethylene glycol dimethacrylate) and UDMA (urethane dimethacrylate)¹⁸. When the restoration is finished, these low-viscosity resins are applied to the surface of the material and the adjacent dental enamel, permeating the marginal microgaps, providing marginal sealing and a better quality surface, which leads to less accumulation of bacterial plaque and prevents the restoration from becoming stained²².

Dentists also often apply a dentin adhesive layer as a post-finishing step of the composite restorations to obtain a smooth, glossy surface, which motivated this study to investigate the surface roughness of a nanofilled composite resin and a hybrid composite resin after the application of a resinous adhesive layer on the surface, and compare it to the surface roughness obtained using a polishing system available in the market, as well as to check the surface characteristics of these composites after the surface treatment, by means of scanning electron microscopy (SEM) analysis.

MATERIAL AND METHODS

The dental composites used in this study were Filtek Supreme XT (3M ESPE, St Paul, MN, USA) and Filtek Z250 (3M ESPE, St Paul, MN, USA). The materials used for the surface treatment were Adper Scotchbond Multi-Purpose (3M ESPE, St Paul, MN, USA) and Sof-Lex Finishing and Polishing System (3M ESPE, St Paul, MN, USA). Table 1 shows the components of these materials.

Using a condensation silicone mold placed on a glass plate and a polyester matrix strip, 30 specimens, measuring 5.0 mm in diameter and 2.0 mm in depth, were made for each composite resin, resulting in 60 specimens. The composites were inserted into the mold in a single portion, using an auxiliary insertion spatula for composite resin.

After filling the mold with the composite, with the aid of an amalgam condenser, slight axial force was exerted on the surface layer of composite resin, allowing the extrusion of excess material. Thereafter, on ten specimens of each composite resin, a new polyester matrix strip was placed at the upper portion of the specimen before the light curing (control groups). A new matrix strip was not used for all the rest. The specimens were then light cured for 40 seconds with the halogen curing unit Curing Light 2500 (3M ESPE, St Paul, MN, USA).

For each photopolymerization, the intensity of the light source was tested and exceeded 800 mW/cm^2 . After curing, the ten specimens of each composite

resin light cured under the polyester matrix strip received no surface treatment and were assigned to the control groups G1 (Z250/CO) and G4 (Supreme/CO). The other ten specimens of each composite resin formed the groups G2 (Z250/SL) and G5 (Supreme/SL), which were polished by the Sof-Lex Finishing and Polishing System (3M ESPE, St Paul, MN, USA). The discs permeated with aluminum oxide were used in the medium (40 μm), fine (24 μm) and ultra fine (8 μm) grit sequence, according to the manufacturer's instructions. The remaining specimens of each composite resin formed the groups G3 (Z250/ADE) and G6 (Supreme/ADE). These received as surface treatment the application of the Adper Scotchbond Multi-Purpose adhesive layer (3M ESPE, St Paul, MN, USA) and then they were light cured for 20 seconds using the same curing unit.

After receiving the surface treatment, the specimens were stored in deionized water at 37 °C for 24 hours. Then they were washed and air dried before the surface roughness test.

The mean surface roughness (Ra) of each specimen was measured by the profilometer Kosaka Lab Surf-Corder SE 1700, with three readings of the specimen surface being made at intervals of 45°, resulting in 180 readings for all the experiment.

A representative specimen of each group was prepared by covering with gold for analysis by scanning electron microscopy (JEOL 5600LV). The scanning electron photomicrographs were taken at a magnification of X1000.

The data obtained in this surface roughness experiment (μm) after submitting the composites to different surface treatments were evaluated by the Two-Way ANOVA analysis of variance test at the significance level of 0.05, in order to determine whether there were statistically significant differences among the mean values of surface roughness and the surface treatment type. However, to determine which of these means differed statistically from each other, the Tukey HSD test was used at the significance level of 0.05.

Table 1: Materials used in the study, composition and lot number.

Materials	Composition	Lot
Filtek Supreme XT (Nanofilled)	Organic Portion: Bis-GMA (Bisphenol glycidylmethacrylate), Bis-EMA (Ethoxylated bisphenol dimethacrylate), UDMA (urethane dimethacrylate) TEGDMA (Triethylene glycol dimethacrylate)	SBU
	Inorganic Portion: Non-agglomerated silica 20 nm nanoparticles and nanoclusters with mean size range from 0.6 to 1.4 μm	
Filtek Z250 (Hybrid)	Organic Portion: Bis-GMA, UDMA, Bis-EMA and camphorquinone	7YU
	Inorganic Portion: Zirconium/silica particles with mean size of 0.6 μm	
Adper™ Scotchbond Multi-Purpose	Adhesive: Solution of Bis-GMA, 2-hydroxyethylmethacrylate (HEMA) and camphorquinone	7543
Sof-Lex Finishing and Polishing System	Aluminium oxide abrasive coated in poliurethane	1958D

RESULTS

As shown in Table 2/ Fig. 1, there are statistically significant differences among the mean values for surface roughness of the tested composite resins according to the surface treatment type.

The smoothest surfaces were obtained in μm with resins as Filtek Z250 ($0.1023 \pm 0.0464 \mu\text{m}$) and Filtek Supreme XT ($0.1083 \pm 0.0241 \mu\text{m}$) light cured

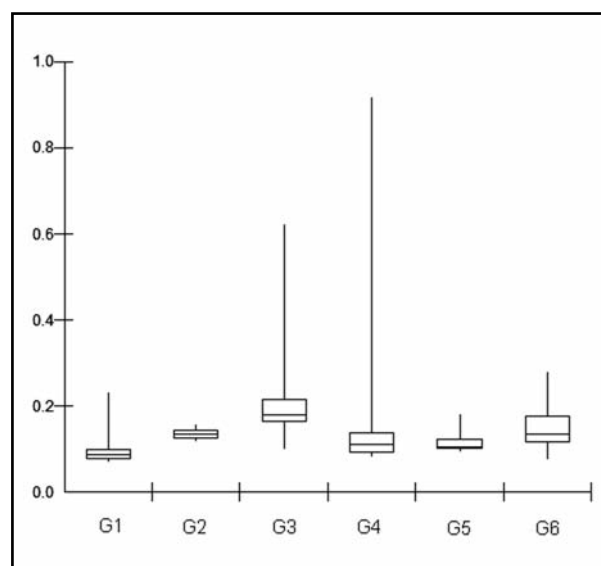


Fig. 1: Box-Plot - Median and quartiles of surface roughness of composites tested according to the surface treatment. ANOVA and Tukey, significance level of 0.05.

against a polyester matrix strip with no surface treatment, followed by the resin Filtek Supreme XT ($0.1160 \pm 0.0252 \mu\text{m}$) and Filtek Z250 ($0.1360 \pm 0.0131 \mu\text{m}$) after finishing and polishing by Sof-Lex discs, with no statistically significant difference among these means ($p > 0, 05$).

The roughest surfaces were found for the resin Filtek Z250 treated with an adhesive layer on its surface ($0.2325 \pm 0.1484 \mu\text{m}$), which differed significantly from the control group and the surface treatment with Sof-Lex ($p < 0.05$) and Filtek Supreme XT after the same surface treatment ($0.2266 \pm 0.0463 \mu\text{m}$). It also differed significantly from the control groups and the treatment with Sof-Lex discs ($p < 0.05$).

Therefore, the surface roughness values for Filtek Z250 and Filtek Supreme XT, after the surface treatment by applying an Adper Scotchbond Multi-Purpose adhesive layer, were statistically higher than those for the control groups and the groups treated with Sof-Lex discs (Table 2).

Table 2: Mean values and standard deviations of surface roughness of composites tested after different surface treatments.

Materials/Treatment	Mean (Standard deviation)
G1 - Z250/Control	0.1023 (0.0464) A
G4 - Supreme/Control	0.1083 (0.0241) A
G5 - Supreme/Sof-Lex	0.1160 (0.0252) A
G2 - Z250/Sof-Lex	0.1360 (0.0131) A
G6 - Supreme/Adhesive	0.2266 (0.0622) B
G3 - Z250/Adhesive	0.2325 (0.1484) B

Means followed by different capital letters differ at a 5% level of significance by the Tukey test. () Standard deviation.

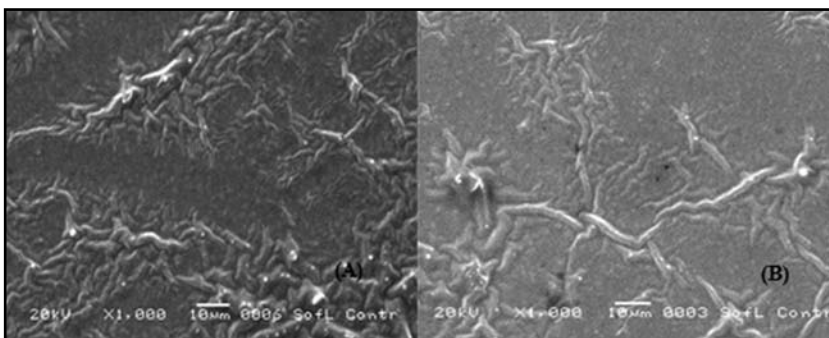


Fig. 2: Photomicrograph of representative area of the control groups. (A) Filtek Supreme, (B) Filtek Z250.

DISCUSSION

According to the results of this study, the smoothest resin surfaces were obtained after the photopolymerization of the composites through the polyester matrix strips (Table 2/ Fig. 2) and after the treatment with Soft-Lex discs. The application of a dentine adhesive layer as a type of surface treatment had a distinct effect, causing higher surface roughness levels in both composites.

Other studies presented better surface roughness values using a polyester matrix strip on the composite resin layer, for example Joniot et al.¹⁵, Türkun and Türkün²³, Barbosa et al.¹⁷ and Gedik et al.¹⁶, Watanabe et al.²⁴, Celik and Ozgünaltay²⁵ and Jefferies and Boston²⁶. Nevertheless, according to Eide and Tveit²⁷, this smooth surface can rarely be maintained in a clinical situation because of the need for contouring, occlusal adjustment and removal of excess material at marginal areas^{4,6,28,29}, resulting in higher roughness of the restoration surface. Moreover, Luts, Setcos and Phillips³⁰ emphasize that the surface created against the polyester matrix, rich in resinous monomers, is less resistant to abrasion and may have empty gaps. Therefore, the removal of this surface layer increases surface resistance³¹.

According to Gedik et al.¹⁶ and Jung, Sehr and Klimek⁶ in fact there is some difficulty during the finishing and polishing of composite resins, due to the difference in hardness between the resinous matrix and the inorganic particles, so that, in many cases, these components do not respond equally to abrasion. Therefore, the surface micromorphology of dental composites after finishing and polishing procedures depends on the inorganic particle properties, such as size and hardness, and also on its content^{29,32}. These characteristics, associated to the shape and distribution of the inorganic particle sizes in the

composites, play a major role in the biological resistance of the restorations, because smooth surfaces produce little frictional deterioration in occlusal contact areas, avoiding the wear between the composite and the antagonist enamel³³.

According to Ergücü and Türkün³⁴, when hybrid composites are polished, hard particles are exposed on the surface while the soft resin matrix is

removed. That is why the inorganic particles should be as close as possible to each other, to protect the resinous matrix from abrasive action.

Finishing and polishing polyurethane discs covered with aluminum oxide, which are highly flexible, were widely used to polish the composite resin restorations. According to Watanabe et al.²⁴, Venturini et al.³⁵ and Cenci et al.³⁶, the ability of producing a smooth surface using aluminum oxide discs lies in its capacity to cut the inorganic particles and the resinous matrix equally. Nevertheless, its efficacy depends on the anatomical shape and the accessibility of the restoration surface to be polished^{35,36}. In this work, the mean values of surface roughness found for the composite resins Filtek Z250 and Filtek Supreme XT, after the surface treatment using Sof-Lex discs, were statistically similar. Due to the nature and size of the Filtek Supreme XT and Filtek Z250 inorganic particles, it was noted that the Sof-Lex discs were capable of maintaining a level of surface smoothness as high as the one obtained using the polyester matrix strip, with no statistically significant difference between them (Table 2/ Fig. 3 A and B).

Again according to Jung, Sehr and Klimek⁶, the surface quality of a nanofilled composite can be attributed to its high content of inorganic particles. Mitra, Wu and Holmes³⁷ reported that although the average size of nanoclusters is similar to that of hybrid universal composites particles, nanofill particles are fundamentally different from hybrid composite particles, which are typically large and thick. Due to the intense chemical interaction amongst the nanoparticles in the resinous matrix, during the nanocomposite's wear there is a rupture of primary individual nanoparticles, not a rupture of larger particles as in the hybrid composites. As a result, the surface shows extremely small irregularities, which are smaller than the wavelength of light.

Liquid polishers are low-viscosity resins without inorganic

content that provide high gloss to composite resin restorations, improving the final aesthetics. They have been widely used to optimize restoration surfaces, acting on their structural microleakages and microclefs³⁸. Therefore, they refine the smoothness of the surface, contribute to marginal sealing, fill the microgaps and thus reduce the marginal permeation of the restorations³⁹, as well as improve the material's resistance to wear.

It has also been argued in favor of applying a dentin adhesive as a post-polishing step for composite restorations, to obtain a smooth, glossy surface, and mainly to reduce additional costs to both dentist and patient¹⁸.

In this study, the application of an Adper Scotch-bond Multi-Purpose adhesive layer on both the composite resin surfaces tested significantly increased the surface roughness, once the polymerization of the adhesive was inhibited by oxygen, leading to the formation of a non-polymerized resin layer, rich in resinous monomers with air bubbles (Table 2/ Fig. 4), making the surface of the composite resins extremely irregular.

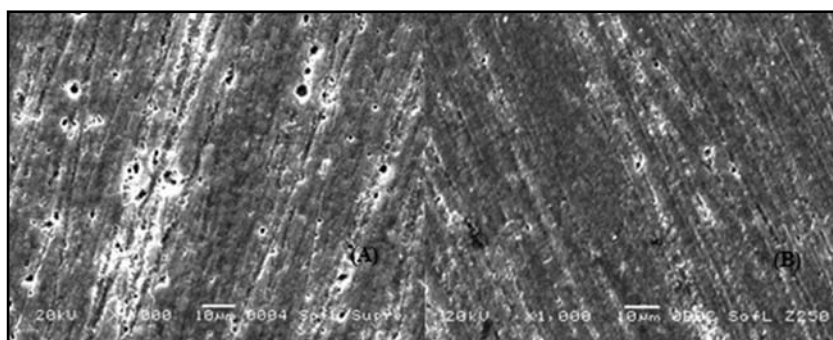


Fig. 3: Photomicrograph of representative area of the specimen after finishing and polishing with Sof-Lex discs. (A) Filtek Supreme, (B) Filtek Z250.

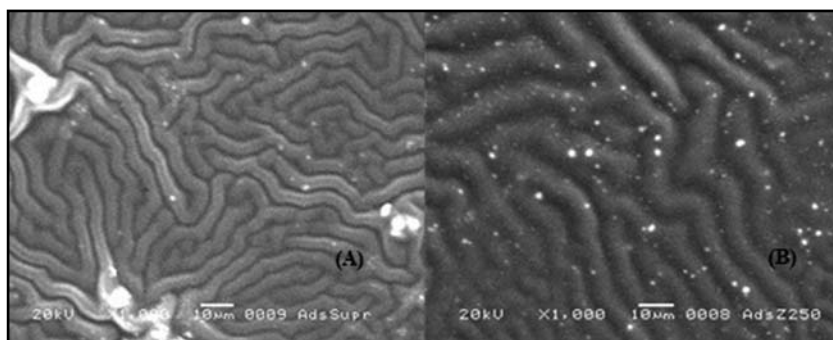


Fig. 4: Photomicrograph of representative area of the specimen after applying a layer of adhesive resin. (A) Filtek Supreme, (B) Filtek Z250.

According to Guler et al.⁴⁰, most of the liquid polishers have low resistance to wear and discoloration. Some products become unstuck from the substrates immediately after the application, due to the weak interaction between material and the substrate and many of them cannot be light cured by LED.

In a similar work, Takeuchi et al.⁴¹ investigated the influence of the surface sealant Protect-it! (JenericPenetron) and the dentin adhesive Single Bond (3M ESPE) on the surface roughness of the composite resin Filtek-P60 (3M ESPE) before and after the simulated tooth brushing abrasive test. The authors highlighted a significant difference in the surface sealant agent's performance before and after the tooth brushing and concluded that the use of a sealant did not ensure the integrity of the surface. Perez et al.¹⁰ evaluated the effect of the sealant BisCover on the surface roughness of different restoring materials after different polishing and finishing

procedures. The results showed that the BisCover (Bisco) coverage did not reduce the surface roughness values of Filtek Supreme after using Soft-Lex discs. On the other hand, Attar¹ found a significant reduction in surface roughness for all the composites tested when the surface sealant BisCover was used after the polishing steps.

According to the methodology used and the limitations of this study, it was concluded that the Soft-Lex discs performed better for the surface treatment of the composite resins tested, and was capable of producing similar surface roughness values on both composite resins.

The application of a cover of Adper Scotchbond MultiPurpose dentin adhesive increased the surface roughness of both composites.

Scanning electron microscopy (SEM) confirmed the findings of the quantitative analysis of the data obtained in this study.

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