Chemical and Mechanical Roughening Treatments of a Supra-Nano Composite Resin Surface: SEM and Topographic Analysis

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Abstract: Background: Repairing a restoration is a more advantageous and less invasive alternative to its total makeover. The aim of this study was to analyze the effects of chemical and mechanical surface treatments aimed at increasing the roughness of a supra-nano composite resin. Methods: 27 cylindrical blocks of microhybrid composite were made. The samples were randomly divided into nine groups (n = 3). The samples’ surface was treated differently per each group: acid etching (35% H3PO4, 30 s and 60 s), diamond bur milling, sandblasting and the combination of mechanical treatment and acid etching. The samples’ surface was observed by a scanning electron microscope (SEM) and a confocal microscope for observational study, and surface roughness (R_a) was recorded for quantitative analysis. Results: The images of the samples sandblasted with Al2O3 showed the greatest irregularity and the highest number of microcavities. The surfaces roughened by diamond bur showed evident parallel streaks and sporadic superficial microcavities. No significant roughness differences were recorded between other groups. The difference in roughness between the control group, diamond bur milled group and sandblasted group was statistically significant. (p < 0.01). Comparison between the diamond bur milled group and the sandblasted group was also significant (p < 0.01). Conclusion: According to our results, sandblasting is the best treatment to increase the surface roughness of a supra-nano composite.

Keywords: surface roughness; microhybrid composite; sandblasting; surface treatment; composite repair; minimal invasive dentistry

1. Introduction

Over the past few years, the quality of direct and indirect restorations has improved in terms of the adhesion strength, longevity and composition of the resin matrix and filler [1].

However, like all dental materials, the composites undergo deterioration processes as a consequence of mechanical (cyclic fatigue and wear) or thermal stresses and chemical degradation (enzymatic, hydrolytic and acidic) [2,3].

Fractures, marginal bacterial infiltration, dentin treatments, teeth or restoration color changes, indirect restorations or endodontic post detachment can also compromise the result and require a makeover [4–9].

In these cases, complete replacement can be a long and expensive procedure with the possibility of further healthy dental tissue loss and an increased risk of pulp exposure. Therefore, repairing the restoration by preserving parts or rebonding an indirect restoration can be an advantageous alternative as these techniques are less invasive and allow the prolongation of the efficiency of conservative therapies over time [5,10–16].

The prognosis of a repair and maintenance treatment depends on the adhesion strength achieved between the old restoration and the new composite material layer [10].

In clinical practice, the adhesion techniques and mechanisms at the dental tissues–composite or composite–composite interfaces are different [17].

The adhesion strength between two composite surfaces depends on their chemical composition and characteristics such as the roughness, conditioning procedures and ability to become wet of the polymerized surface [10].

Furthermore, contrary to what happens in the composite layering technique, during reparation, the material integration process is hindered by the relative lack of unpolymerized monomers [18–20].

After intraoral photopolymerization, the conversion rate from monomer to polymer is between 45% and 70% because not all monomers participate in the polymerization and the remaining monomers are available for new adhesion [21–23].

Considering that the number of unsaturated double bonds in monomers decrease over time, the adhesion strength reduces by between 25% and 80%, and consequently the effectiveness of the repair process is time-related [18–20].

The adhesion of new composite to an old restoration is achieved with a new bond established by residual monomers and micromechanical retention that exploits the surface irregularities of the old restoration.

In order to improve this type of adhesion, various surface treatments have been described in the literature such as surface roughening with diamond burs of different granulometry, sandblasting with aluminum oxide or silica sand, acid etching and the application of hydrogen peroxide or silane.

A systematic review of the data still appears insufficient to indicate the best method for repairing Bis-GMA-based resins [11,14,18,21,22]. The aim of this study is to analyze the effects of chemical and mechanical surface treatments and their combination on the roughness of a supra-nano composite resin surface.

2. Materials and Methods

27 cylindrical blocks (height 4 mm and diameter 6 mm) of supra-nano composite col. A1 (Estelite Sigma Quick, Tokuyama Dental, Japan) were made using a silicone mold matrix.

Considering a power level of 85% with a type-I error = 0.05, for parameter roughness (Ra), three samples for each independent group (surface-treated and independent control) were necessary.

The technical characteristics of the composite resin provided by the manufacturer were:

- A resin matrix (bisphenol A-glycidyl methacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA)) and particles of reinforcement (71% of the total volume) formed by spherical particles of silica and zirconia, with sizes ranging from 0.1 to 0.3 µm (average size of 0.2 µm).

The cylindrical blocks were obtained through two vertical composite increments of 2 mm inside the silicone matrix. Using a LED curing lamp (Valo, Ultradent, South Jordan, UT, USA) the composite layers were light-cured for 20" at a distance of 1 mm at 3.200 mW/cm². In order to prevent the inhibition of polymerization due to the presence of oxygen, and to create a homogeneous surface, the last layer of composite was covered by a glass plate before light-curing.

To make the samples’ surface uniform, all the composite blocks were polished under 4× magnification (EyeMag Pro S, Zeiss, Oberkochen, Germania), using Soft-Lex (3M ESPE, St Paul,
Minnesota) coarse-grained, medium, fine and superfine discs for 10 s each. After each step the samples were washed with distilled water and air dried.

The samples were randomly divided into 9 groups (n = 3). The surface of the samples from each group was treated with different roughening protocols:

A) Control group, no surface treatment.
B) Etching for 30 s.
C) Etching for 60 s.
D) Roughening with diamond bur.
E) Roughening with diamond bur and etching for 30 s.
F) Roughening with diamond bur and etching for 60 s.
G) Sandblasting.
H) Sandblasting and etching for 30 s.
I) Sandblasting and etching for 60 s.

Etching was performed with 35% orthophosphoric acid (Ultra-Etch, Ultradent) for 30 s in groups B, E and H or 60 s in groups C, F and I. After etching, each sample was washed with distilled water and air dried.

Milling was carried out with a diamond bur with granulometry 151 µm (6837 KR Komet) mounted on a handpiece for 3 s (groups D, E and F).

Sandblasting (20 s) was performed with an intraoral sandblaster using Al₂O₃ 50 µm (Dentoprep, Rønvig Dental).

The observational analysis of the treated surface of the samples was carried out with an SEM (Phenom G2, Phenom, Eindhoven, the Netherlands), at 2.100x magnification, with 5 kV voltage and secondary electrons (SE). This SEM does not require any treatment of the sample surfaces [24,25].

The samples were then observed and analyzed with a confocal microscope (Leica DCM 3D, Leica Microsystems) in order to measure roughness. In each sample an area of 2.5 mm² was selected using a systematic random sampling protocol for stereological and morphometrical analysis and the roughness (Ra) was calculated using the following formula:

\[ Ra = \frac{1}{L} \int_{0}^{L} |Z(x)| \, dx \]

For each experimental group the mean and quantitative parameter standard deviation (SD) were calculated.

Data were analyzed using the Student’s t-Test and differences of p < 0.05 were considered statistically significant.

The statistical analysis was performed using the SPSS 17.0 for Windows package and the Prism software package (GraphPad, La Jolla, CA, USA).

3. Results

3.1. SEM Analysis

The SEM observation showed that the different types of roughening protocol exhibited no substantial differences among samples of the same group.

Comparisons between groups showed different surface morphologies, displayed in Figure 1.
Comparisons between groups showed different surface morphologies, displayed in Figure 1. Figure 1. SEM images (2100×). Voltage: 5kV. Type of electron: secondary electron (SE). Groups A, B and C show a surface without irregularities; groups D, E and F show evident parallel streaks resulting from the bur action (arrows indicate the parallel streaks); groups G, H and I, the sandblasted samples, show the highest number of microcavities (arrows indicate microcavities).

The surface of the control group samples (group A) was homogeneous, without irregularities and without microcavities. Etching with 35% orthophosphoric acid for 30 s or 60 s (groups B and C) did not cause any observational modification of the composite surface. The surface roughened by diamond bur (group D) showed evident parallel streaks resulting from the bur action. The images of the sandblasted samples (group G) show greater irregularity and a high number of microcavities. Etching using 35% orthophosphoric acid for 30 s and 60 s (groups E, F, H and I) as an additional treatment to milling and sandblasting caused no change on the sample surface.

In all experimental groups, the optical confocal microscope analysis shows three-dimensional topographic images similar to the SEM observation (Figure 2).
Figure 2. Confocal microscope. Three-dimensional surface topography. Red expresses the peaks; blue shows the depressions. Groups A, B and C show a surface with low irregularities; groups D, E and F show a moderate presence of surface irregularities; groups G, H and I show the highest presence of surface irregularities.

3.2. Profilometric Analysis

The mean values of roughness (Ra) and the standard deviation (SD) for each experimental group are shown in Figure 3.

Figure 3. Roughness (Ra) mean values of experimental group.
There were no statistically significant differences in $R_a$ between groups A (94.2 ± 44.7 nm), B (78.6 ± 23.3 nm) and C (60.7 ± 35.3 nm).

The same result was obtained by comparing the values for groups D (1647.8 ± 471.9 nm), E (1812.3 ± 300.7 nm) and F (1603.8 ± 280.4 nm), and those for groups G (2955.6 ± 572.9 nm), H (2777.8 ± 447.6 nm) and I (2855.6 ± 494 nm).

The comparison of $R_a$ between group A (control) and groups D and G indicated statistically significant differences ($p < 0.01$). Additionally, the difference in $R_a$ between groups D and G was statistically significant ($p < 0.01$).

4. Discussion

The introduction into clinical practice of build-up and indirect restorations made of composite resins requires the knowledge of adhesion mechanisms, especially when the surfaces are made of already polymerized composite [26]. This analysis can also be linked to the necessity of following a conservative approach in order to increase the longevity of direct restorations and the possibility of further repairing processes [27]. The treatments used most often to enhance the adhesive’s action are chemical and mechanical surface roughening [14,18,19,21,26]. In this study, two mechanical and one chemical technique of surface conditioning and their combination were used. The mechanical techniques consisted of surface roughening by the action of a diamond bur or sandblasting with $\text{Al}_2\text{O}_3$.

The chemical conditioning technique instead consisted of an etching procedure on the samples’ surface with 35% orthophosphoric acid for 30 s or 60 s [28].

The images obtained with SEM and three-dimensional topography with an optical confocal microscope in all experimental groups agree, showing that among samples of the same groups the surface treatments cause substantially comparable morphological alteration. This highlights how the response of the same type of material to various techniques does not change and is therefore predictable.

The control group (treated exclusively by polishing with abrasive discs) presented a smooth, homogeneous surface without any irregularities or microcavities.

The surfaces roughened by a bur were irregular, with a limited number of microcavities distant from each other and with parallel streaks resulting from the action of the bur. The sandblasted samples showed greater irregularity and a high number of microcavities.

The comparison between the observations for these latter groups, according to previous studies, confirms the hypothesis that sandblasting with $\text{Al}_2\text{O}_3$ is the most suitable treatment for increasing the micromechanical retention of composite surfaces [29].

In our research, the treatment of etching alone, regardless of its duration, does not cause a significant improvement of composite roughness. Furthermore, there are not any substantial modifications on the surface even when the etching action is carried out on a previously mechanically conditioned surface. This outcome is independent of the duration of acid application. The observational data, therefore, seem to exclude any positive impact of the etching, according to Loomans et al., who stated that 35% orthophosphoric acid is not able to cause significant alterations to the resin filler. This component can be modified only by more aggressive acids, such as hydrofluoric acid [28].

The morphological findings (SEM and confocal) are confirmed by the profilometric analysis quantifying the average roughness ($R_a$) of the experimental groups.

The profilometric analysis (Figure 2) shows variable roughness among the experimental groups. The color scale from red to blue expresses the difference in height at different points of the same sample. To understand these data it is important to note that it is almost impossible to obtain a perfectly flat sample by hand, and this is particularly evident in the A, B and C groups, where the analysis shows the lowest $R_a$ values but red and blue areas are visible. This color expression afforded the researchers a visual way of understanding the surfaces’ pattern and without comparing their height. Groups A, B and C obtained the lowest $R_a$ values; moreover, there were no significant differences in the average roughness between surfaces treated with etching only and the control group. Even when the effect of the etching on milled or sandblasted surfaces was evaluated, the variation in roughness was not
statistically significant. The concordance between the observations shows how chemical treatment does not change the roughness of a smooth or previously mechanically roughened surface.

The average roughness increased significantly when comparing the control group to the samples (D, E and F) whose surface was roughened by the action of a diamond bur. This statistical evaluation was also valid for the sandblasted groups (G, H and I).

The comparison between the groups treated with mechanical conditioning shows that the sandblasted samples had significantly higher average roughness. These data agree with the observational findings and confirm that sandblasting is the best treatment possible for roughening a composite surface [28].

SEM observation allows us to directly observe the surface morphology to understand the modification related to different tools and the combination of materials and techniques [30]. The principal advantage of this technique is the possibility to directly observe samples without any major surface modification, especially samples with irregular and reflective surfaces, where producing bi-dimensional images does not give any information regarding surface roughness. In particular, the SEM used in this study gives a precise and reliable surface image not requiring any surface metallization prior to observation. The confocal laser microscope with profilometric analysis gives a 3D objective evaluation of the surfaces, providing a visual color scale that shows differences in height among different points of the sample and so provides quantitative data on the sample microsurface [31]. The combination between SEM analysis and the confocal laser provides the possibility to obtain and order numerical categories such as the smoothness, roughness and waviness of irregular and reflective surfaces along with a precise and detailed bi-dimensional image [32].

The main limitation of this study is related to the experimental samples that evaluated just one supra-nano composite. There is a need to compare the efficacy of this surface treatment on other composites, considering that different composite materials with different physical-chemical characteristics might respond to surface treatments in a specific manner.

In our experimental sample the specimens were not subjected to thermocycling procedures due to the in vitro experimental setting of the research. The thermocycling process gives the researchers the possibility to perform tests and evaluate the samples in a setting more similar to the clinical world. In the literature it has been described how aged composite shows lower adhesion values when compared to non-aged composite, probably due to hydrolytic degradation in the resin matrix that occurs in the oral environment, and due to the reduction of free radicals available that can react chemically with a fresh composite [20,33,34].

Moreover, further study is necessary to evaluate the bond strength of the composite surface and its effect.

5. Conclusions

Considering the results of our research, it is possible to conclude that among the treatments used to increase the roughness of a supra-nano composite surface, the most effective treatments are mechanical ones. Within this category of conditioning, sandblasting creates the best environment for the microretention of the adhesive system.

The use of orthophosphoric acid does not result in efficient surface roughness: the SEM images show a different appearance after 60 s acid etching; however, there is no statistically significant difference. According to our results, it can be concluded that sandblasting should be carried out to increase the surface roughness of an already polymerized supra-nano composite.

This treatment makes it possible to integrate the chemical forces generated between the monomers contained both in the adhesive and in the old composite surface with mechanical microretention.

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