

Evaluation of Surface Roughness of a Nanofill Resin Composite After Simulated Brushing and Immersion in Mouthrinses, Alcohol and Water

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This study investigated the alteration of surface roughness of the nanofill composite Filtek Z350 3M/ESPE®, caused by simulated brushing associated with the use of mouthrinses with or without alcohol. Sixty specimens were prepared and distributed into six groups: distilled water, ethylic alcohol, Listerine® Vanilla Mint, Plax® without alcohol, Oral B® without alcohol and a control group. Each group was submitted to two intercalary 5,000 simulated brushing cycles. At the end of each cycle, the specimens were washed in tap water and immersed for two cycles of six hours equivalent to one year of daily use of the solution for 2 minutes. It was possible to verify significant alteration in surface roughness of the composite influenced by ethylic alcohol. It was not significant for distilled water and the mouthrinses.

Keywords: *surface roughness, mouthrinses, simulated brushing*

1. Introduction

Nanofill composites were introduced on the dental market with the aim of providing less polymerization shrinkage and higher resistance to traction, compression and fracture as well as an improvement in optical properties, lower attrition, and greater retention of gloss¹. The technology of such resins may also improve the continuity between the dental structure and the nanoparticles, providing more balance between the mineralized hard tissue of teeth and those of the advanced restorative biomaterials².

One of the factors that determine the clinical longevity of a restoration is its surface characteristics³. Ideally a restoration must provide a smooth and regular surface, but it is not always possible, as the composite resins are frequently subject to certain deleterious actions in the oral cavity through the processes of abrasion (brushing), attrition and erosion (citrus drinks, fruit, soft drinks)⁴. Furthermore, the materials are exposed to exogenous substances containing a variety of chemicals, including acids, bases, salts, alcohol, oxygen, etc. entering the environment during oral food and fluid intake and oral hygiene. The chemical and duration of exposure are important determinants that may have some influence on the polymer chain molecules of materials. Several factors related to chemical structure and molecular chains of polymers are important in determining how these materials will be affected by the aqueous environment. Important chemical characteristics include the hydrophilicity of the polymer, and the differences in the solubility parameters between polymer and solvent. Important structural parameters include the density of cross links and the porosity of the chain. Moreover, the presence of reinforcing structures may significantly influence the solubility and sorption of the structure⁵.

These adverse effects could affect both the internal and external composition⁶, such as surface texture and color⁷⁻⁹. The phenomena of sorption and solubility may serve as precursors to a variety of chemical and physical processes producing deleterious effects on the structure and function of the polymeric chain⁵. These effects may include changes such as volume expansion; physical changes,

such as softening and plasticization; and chemical changes, such as oxidation and hydrolysis. The properties of the polymer chains can be permanently altered by these events, and compromise the performance of these materials¹⁰. There is concern that the effects of the action of solvent and hydrolytic degradation may lead to decreased longevity of restorations. But equally disturbing is the possibility of biological effects spreading from the polymers of dental restorations⁵.

The brushing associated with toothpaste is the main method of oral hygiene, bringing many benefits, in addition to a reduction in the incidence of caries. However, studies have shown that the movement of agents associated with the toothbrushing abrasive in a dentifrice and the toothbrush bristles, can cause damage to the brushed substrate, capable of altering the restorative material roughness¹¹⁻¹⁶. The abrasives in dentifrices have been related to dental wear (abrasion) and over time, can also cause an increase in the surface roughness of restorative materials, leading to greater plaque retention and composite pigmentation^{12,13,15}.

Mouthrinses have also been used for centuries for the purpose of providing oral health and cosmetic benefits¹⁷ and in the last few years their use has attracted the curiosity of the researchers because of their ability to modify the surface of composite resins. Studies have shown that these products with and without alcohol can affect the hardness of composite and glass ionomer cement and have become a possible threat to oral health¹⁸. However, studies have shown that alcohol in mouthrinses is not the only factor that can lead to modification of polymers. The effect of commercial mouthrinses on wear and hardness is dependent on the material¹⁹.

Taking into consideration the importance of roughness with respect to the esthetics and function of restorations, the aim of this study was to evaluate the relationship between the mechanical action of brushing with toothpaste linked to three types of oral mouthrinses, water and alcohol and identify possible changes in the resin composite surface.

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2. Experimental Procedure

2.1. Materials

The materials used in this study were: nanocomposite resin Filtek Z350 (3M/ESPE[®]) color A3, tooth brushes (TEK[®]), toothpaste Colgate[®] Total 12 Clean Mint, and oral mouthrinses (Listerine[®] Vanilla Mint, Plax[®] without alcohol and Oral B[®] without alcohol). The materials, composition and batch number are listed in Tables 1 and 2.

2.2. Methods

The samples were prepared with the resin composite Filtek Z350 3M/ESPE[®], which was inserted directly into in a cylindrical polished metal matrix, 5 mm in diameter and 2 mm thick. A glass slab interposed with a polyester strip was placed on the composite resin to obtain a smooth surface with the polish of natural resin. A weight of 10 grams was used for 10 minutes, to accommodate the composite and obtain a flat surface. After this time, the weight was removed and the resin polymerized in accordance with the manufacturer's recommendations, i.e. 40 seconds, 36 J.cm⁻², covering the total area with a high intensity visible light source (Optilux-Demetron 450[®]) at all times keeping the tip as close as possible to the resin surface. Sixty-eight samples were prepared and 8 of these were discarded due to presenting surface imperfections and bubbles.

The samples were stored in distilled water at 37 °C, and then randomly divided into 06 groups of 10 samples each. The samples were fixed onto an acrylic base two by two.

The bases were identified and subjected to the mechanical brushing test in a MSET machine (Elquip, São Carlos, SP, Brazil). Brushes with soft bristles and rounded edges (TEK[®], Johnson & Johnson[®], São Paulo, Brazil) were fixed to the apparatus and kept alongside the samples, using slurry of Colgate[®] Total 12 Clean Mint dentifrice with

distilled water in a ratio of 1:3 by weight²⁰. Simulated brushing was performed with a linear movement, speed of 4.5 cycles per second, a cycle understood as being the complete back-and-forth movement of the toothbrush. Each experimental group (1) Listerine[®] Vanilla Mint, (2) Plax[®] without alcohol, (3) Oral B[®] alcohol-free, (4) alcohol 96, and (5) distilled water, was subjected to 02 intercalated time periods of 5,000 brushing cycles equal to a total of 10,000 cycles (representing the total time of 1 year of brushing) each time period performed in 40 minutes²⁰. At the end of each cycle, the samples were washed under running water to remove the toothpaste. Between each brushing cycle, the samples were immersed for 6 hours, in 2 immersion cycles (12 hours), equivalent to one year of daily use of the solution for two minutes^{18,21}. The groups in the mouthrinses were shaken every hour in order to prevent chemical balance on the composite surface, and kept at a temperature of 37 °C¹⁸. After this time the samples were stored in distilled water at 37 °C until the readout was taken on the surface roughness gauge. The control group (6) was not subjected to any chemical or mechanical action.

The pH of each solution was measured (pH-meter Calcheck HI221). For each solution three readouts were taken and the values recorded. The mean pH values were: Listerine[®] Vanilla Mint - pH: 4.08; Plax without alcohol - pH: 5.05; Oral B[®] without alcohol - pH: 6.32.

To verify the change in roughness of the samples, a surface roughness gauge (Mitutoyo Sj-400-Japan[®]) was used and data were recorded by the computer program Surfpack version SJ-1300 Speed-0.5; Range-800.00. The mean roughness was recorded in Ra, defined as: $Ra = (1/L) \int_0^L |h(x)| dx$ # A.1. Each sample was subjected to three readouts, one in each direction in order to scan the entire sample. All data were transferred and stored in Microsoft Office Excel 2007[®] files. Data analysis was performed using statistical tests: F (ANOVA) with Tukey's paired comparisons, and Levene's F test. The calculations were performed using the statistical program SPSS version 13. The margin of error used in the decision of the statistical tests was 5.0%.

Table 1. Resin composite and composition according to the manufacturer's information.

Material	Organic matrix (% w)	Inorganic filler (% w.v ⁻¹)	Manufacturer/ Batch number
Filtek Z350	Bis-GMA (10-15), UDMA, TEGDMA(10-15) e Bis-EMA (1-5)	Nanoagglomerate of zirconia/silica (0.6 µm a 1.4 µm); Silica not agglomerated/ not aggregated (20 nm) (78.5/59.5)	3M/ESPE, St. Paul, Minnesota, USA 8NU

Table 2. Mouthrinses and their composition according to the manufacturers' information.

Material	Composition	Manufacturer/ Batch number
Listerine Vanilla Mint	Thymol 0.064%, eucalyptol 0.092%, methyl salicylate 0.060%, menthol 0.042%, water, sorbitol, alcohol 21.6%, poloxamer 407, benzoic acid, mentha piperita oil, essential oil of mint viridis, propyleneglycol alginate, sucralose, sodium benzoate, aroma, Cl 42090, Cl 15985.	Jonhson & Jonhson [®] 01018L
Plax without alcohol	Water, glycerin, propyleneglycol, sorbitol, PEG-40, hydrogenated castor oil, sodium benzoate, aroma, phosphoric acid, sodium fluoride (225 ppm of fluoride), cetylpyridineum chloride, sodium saccharin.	Colgate [®] Br122
Oral B without alcohol	Water, glycerin, PEG-40, hydrogenated castor oil, methylparaben, flavoring, cetylpyridineum chloride monohydrate 0.053%, sodium fluoride 0.050% (226 ppm of fluoride), sodium saccharin, sodium benzoate, propylparaben, Cl 42090.	Procter e Gamble [®] 7297852521

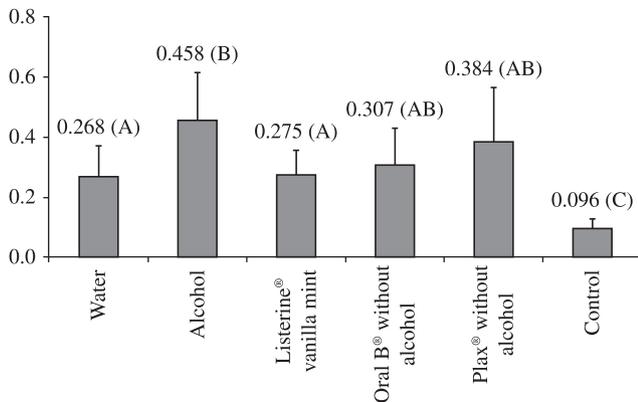


Figure 1. Results for roughness (mean values in μm) according to the products employed. *Different letters in parenthesis indicate statistical difference among the groups according to Tukey's test.

3. Results and Discussion

An increase in surface roughness of materials used in the oral environment has many consequences¹⁶. In this research the samples were not subjected to any surface treatment, in order to avoid the influence of finishing techniques on the results. Only a polyester strip was used on the resin composite before polymerization with the intention of obtaining a smooth surface. Any form of additional polishing could lead to an increase in surface roughness²².

A significant increase in surface roughness of the Filtek Z350 resin (3M/ESPE®) control group was observed when compared with the other groups (Figure 1). There were also significant changes between the alcohol group and groups with water and Listerine Vanilla Mint product (Figure 1). These changes may be related to a probable elution of the non-reacted monomers and a degrading effect on the polymer chain, which is expected after composite resins are exposed to chemicals, water, artificial saliva, alcohol, solvents, acids or bases^{5,21,23} because there is a change in the interactions of secondary links that increase the volume of the polymer chain, and a potential weakness due to fewer chain-chain interactions that increase plasticization. The reduction in hardness and other properties is the result of separation of polymer chains from a molecule that does not form a chemical connection with the primary chain, but simply serves to occupy space. This initial effect is greater on the surface⁵.

The action of mouthrinses on hardness and roughness depends on the composition of the restorative material, which can be attributed to different chemical compositions and the composition of the organic matrix¹⁹. Several factors related to polymer chemical structure and molecular chains are important in determining how these materials will be affected by an aqueous environment⁵. It is known that there is a difference in hydrophilicity between the matrix monomers and the degree of difference is presented in the following order: TEGDMA>Bis-GMA>UDMA>HMDMA²⁴, and TEGDMA is more susceptible to enzymatic hydrolysis than Bis-GMA or Bis-EMA⁵. Thus it is expected that the considerable sorption of water by resin Filtek Z350 3M/ESPE®, leads to an increase in roughness, as it is composed predominantly of monomers that are more susceptible to hydrolysis, i.e. 10-15% of Bis-GMA and 10-15% of TEGDMA (Table 1).

This study analyzed one year use of mouthrinse solution for two minutes daily. This is considered a short term methodology; nevertheless it was possible to observe changes. It is important to highlight that the manufacturers' instructions do not always inform

the consumer that this practice should be restricted to only once a day. When the mouthrinses were used in studies with longer exposure time, a statistically significant difference in the sorption of liquids was observed²⁵. It could be affirmed that the chemical composition and the duration of exposure are important determinants with influence on the polymer chain molecules and thus the longer the period of exposure to the products, the more intense are the adverse effects^{5,27}. The increased roughness in the results of the present study was shown in the following order: higher when alcohol was used (0.458), followed by samples of Plax® without alcohol (0.384) and was lowest in the control (0.096). The control was taken as reference for the roughness assessment, because it was not subjected to the process of immersion in liquid or simulated brushing. In the samples exposed to alcohol, the increase in the roughness of the composite can be attributed to the high alcohol concentration, which can lead to softening of restorative materials. The acid pH of the mouthrinses may have contributed to the degradation of the surface, as observed by other authors^{27,28,31}.

The increased roughness in the results of the present study may also be related to the mechanical factor. There are studies that have shown the deleterious effect on restorative materials when brushing is associated with the use of fluorides or mouthrinses^{26,33}. In a study of Filtek Supreme resin (3M ESPE®) subjected to various cycles of simulated brushing using Close-Up® toothpaste for a short and long term, without immersion in liquid, it was demonstrated that the short term lead to a significant change in the resin surface, but in the long term this change was less evident¹⁶. Nanocomposites were introduced with the so-called advantage of increased polish and gloss retention, as only small particles would be dislodged during wear, leaving the surfaces with defects smaller than the wavelength of light³². The higher short-term wear can be explained by the microstructure of the resin (type of loading and particle distribution); in nanoparticle composites there is uniformity in the size of fillers¹⁶.

Although this research was conducted *in vitro*, this has the advantage of providing data of a single variable of interest to be studied without the interference of other factors, because clinically, the effects of mechanical brushing associated with mouthrinses on restorative materials may be modified by variables that are reproduced *in vivo*. Saliva, for example, can dilute or reduce the effect of the mouthrinse^{18,34}. Moreover, studies have shown that an aqueous medium, such as the oral environment, may interfere with the characteristics of composite resins and even lead to hydrolytic degradation over time^{35,36}. Thus, it is important for other *in vitro* and *in vivo* studies to be developed to assess different variables that could show the full extent of their influence on the physical and chemical behavior of these composites.

4. Conclusion

According to the results it can be concluded that among the mouthrinses tested none significantly influenced the surface roughness of Filtek Z350 resin, although all presented acidic pH. After evaluating the roughness of Filtek Z350 resin, alcohol was shown to have a direct influence on surface roughness, but no significant influence was observed for water or the three tested mouthrinses.

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