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# Effect of whitening dentifrices on surface roughness of dental nanofiller-based composites. Stereomycroscopy and AFM analysis

Efeito de dentifrícios clareadores na rugosidade superficial de compósitos de nanoparticulados. Estereomicroscopia e AFM

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## CLINICAL RELEVANCE

The indiscriminate use of whitening toothpastes can shorten the longevity of composite resin restorations. Dentists should advise their patients about the abrasive effect of these products.

## ABSTRACT

**Objective:** the aim of this study was to assess the effects of whitening dentifrices on the surface roughness of three commercial nanofiller-based composite. **Material and Methods:** two optical analyses were performed: stereomicroscopy and atomic force microscopy (AFM). Disks (8.0 diam. x 2.0 mm thick.) of the nanocomposite Z350 (3M ESPE) and two nano-hybrid composites (EsthetXHD, Dentsply and Premisa, Kerr) were submitted to *in vitro* brushing (1000 cycles) with three whitening dentifrices (Colgate Luminous White, Oral-B 3D White e Close-Up Diamond Attraction) and a control (Colgate Total12). The results were analyzed by stereomicroscopy and rated according to the following criteria: 0 – no observed roughness, 1 – average roughness, 2 – great roughness. Data was submitted to Kruskall-Wallis non-parametric test followed by Tukey test ($p = 0.05$). Z350 specimens were assessed by AFM. **Results:** the surface roughness of composites brushed with whitening dentifrices was statistically higher than the roughness found in specimens brushed with conventional dentifrice. AFM analyses showed that, at a submicrometric scale, there was an increase in the number of peaks and irregularities in specimens brushed with whitening dentifrices. **Conclusion:** Whitening dentifrices may have an impact on the longevity of dental composites by an

## RESUMO

**Objetivo:** o objetivo deste estudo foi avaliar os efeitos da abrasão produzida por dentifrícios clareadores sobre as superfícies de três marcas comerciais de resinas compostas contendo partículas nanométricas. **Materiais e Métodos:** A resina nanoparticulada Z350 (3M ESPE) e as resinas nano-híbridas EsthetXHD (Dentsply) e Premisa (Kerr) foram submetidas à escovação *in vitro* com três diferentes dentifrícios clareadores (Colgate Luminous White, Oral-B 3D White e Close-Up Diamond Attraction). Os espécimes de resina foram confeccionados com 8mm de diâmetro e 2mm de espessura. Foram produzidos 45 corpos de prova para os grupos experimentais e mais 15 espécimes para o grupo controle (dentifrício Colgate Total12). Após a realização de 1.000 ciclos de escovação, os espécimes foram armazenados em saliva artificial. A análise das superfícies escovadas foi obtida por estereomicroscópio. As rugosidades foram mensuradas segundo os critérios: 0 – sem rugosidade, 1 – média rugosidade e 2 – com muita rugosidade. Os dados obtidos foram analisados estatisticamente por teste não-paramétrico de Kruskall-Wallis seguido pelo teste de Tukey ($p < 0,05$). Os espécimes confeccionados com Z350 foram avaliados por microscopia de força atômica (AFM). **Resultados:** A rugosidade superficial dos espécimes de resina composta foi estatisticamente maior quando escovados com os dentifrícios clareadores em relação ao dentifrício controle convencional. Análise por AFM demonstrou, em uma escala submicrométrica, um aumento no número de picos na superfície dos espécimes da resina em que foi realizada a escovação increase in its roughness. It is strongly recommended that clinicians guide their patients, warning them about the risks and consequences of their use.

## KEYWORDS

Abrasion; Dentifrices; Composites resin; Roughness; Whitening; Abrasive.

com dentifrícios clareadores em comparação com o grupo controle. **Conclusão:** Os dentifrícios clareadores aumentaram a rugosidade superficial das resinas compostas podendo, assim, diminuir a sua longevidade.

## PALAVRAS-CHAVE

Dentifrício; Resina composta; Clareamento dental; Abrasão.

## INTRODUCTION

Dental composites have continuously evolved since its inception, many decades ago. The main change experienced by this material during its development was related to the type and size of its fillers particles. The first composites that emerged during the 70s used quartz (average size of 20 $\mu$m) as the inorganic filler, and therefore, were difficult to wear or polish. [1]

In contrast, from the 2000s, the composites began to be produced with the aid of nanotechnology [2,3] and, thus, inorganic particles of silica and zirconia, with an average size of about 0.005-0.07$\mu$m, came into use. These composites have been presenting great optical characteristics mainly due to the ease of wear, which results in better polishing. [4-7] However, composites have also become more susceptible to excessive wear caused, for example, by tooth brushing with abrasive toothpastes.

Noteworthy it is also the fact that investments in oral health widely spread the use of dentifrices as a method to prevent tooth decay, periodontal diseases, halitosis and many problems related to the oral cavity. For this purpose, organic solvents and abrasives are added to toothpastes. The harshness of toothpastes should not be so high as to cause damage, even unintentionally, to hard or soft tissue of the oral cavity, or even to dental restorations. [8]

Nevertheless, the abrasiveness of current dentifrices can result in changes in tooth surface morphology, as well as, on the surface of restorative materials. These surface changes can favor the accumulation of plaque and also increase the restorative materials potential for degradation. [8] Wearing produced by an abrasive agent is influenced by a wide variety of properties of the inorganic filler such as its chemical composition, crystalline structure, hardness, solubility, concentration, particle shape, size and distribution. [8]

Both composites and dentifrices play important roles, nowadays, in aesthetic dentistry with the latter incorporating in its composition agents that promise, according to manufacturers, easy home bleaching. Dentifrices represent about 50% of over-the-counter (OTC) products available on the market for dental bleaching. [9,10] Most of these products have no oxidizing agents (hydrogen peroxide or carbamide) in its composition. Regarding bleaching toothpastes, superficial stains are removed from enamel surface by an increase in abrasive particles, notably hydrated silica, dicalcium phosphate dihydrate and alumina. [11,12] Less abrasive particles present in conventional toothpastes such as calcium carbonate or sodium bicarbonate are ineffective for this purpose. [13,14]

As a result of increased abrasiveness in bleaching dentifrices, a greater roughness was found on composites surfaces as reported in many *in vitro* studies. [15-17] The higher the wear resistance of a composite, the greater its' color stability and less likely to occur marginal leakage. [15]

Given the above, the objective of this work was to analyze the effect of various whitening dentifrices on the surface profile of nanofiller-based composites.

## MATERIAL AND METHODS

The composites chosen for this study were the following: Z350 (nanocomposite, 3M ESPE, St. Paul, MN, USA), Esthet X HD (nano-hybrid composite, Dentsply, Milford, DE, USA) and Premisa (nano-hybrid composite, Kerr; Orange, CA, USA). Three brands of whitening dentifrices containing different compositions were used (table 1): Colgate Luminous White (Colgate-Palmolive, São Bernardo do Campo, SP, Brazil), Close Up Diamond Attraction (Unilever, Ipojuca, PE, Brazil) and Oral-B 3D White (Procter & Gamble, Gross-Gerau, Hesse, Germany). Colgate Total 12 (Colgate-Palmolive, São Bernardo do Campo, SP, Brazil), a conventional dentifrice, was used as a control. Dentifrice compositions are described in table 1.

### *Confection of the resin disks*

Twenty specimens (disks) were made for each commercial composite, 2.0 mm thickness and 8.0 mm in diameter. The composites were lightactivated using a LED unit under 1500 mW/cm$^2$ (Radii Plus, SDI, Bayswater, Australia) and the exposure time was selected according to the manufacturers' instructions.

After polymerization the specimens had one side finished and polished with rubber abrasive points (Microdont, São Paulo, SP, Brazil) and diamond polishing paste (Poli I and Poli II, Kota, Cotia, SP, Brazil) to simulate a clinical procedure.

Specimens were then stored in artificial saliva (4.10-3M KH2PO4, 5.10-3M NaHPO4, 2,4.10-2M KHCO3, 1,7.10-2M NaCl, 5.10-4M MgCl2, 3.10-3M CaCl2, 2.10-4M citric acid and distilled water). Five specimens from each composite brand were brushed with different toothpaste from those listed in table 1.

### *Brushing*

Brushing conditions were simulated with a brushing machine developed in the Department of Biomaterials and Oral Biology from University of São Paulo, Brazil. Disks were fixed and brushed a thousand cycles at 70 cycles per minute in a slurry containing dentifrice and water (10 g/70 mL). Each specimen was brushed using 16 g of the mixture with a load of 200 g. The brushing machine was equipped with flat trim toothbrushes presenting soft bristles (Reach, Johnson & Johnson, São Paulo, Brazil).

### *Stereomicroscopy, AFM and Statistical Analysis*

Optical micrographs and sample examinations were conducted using an Olympus SZ61 stereomicroscope equipped with a CCD camera (Q-Color 3, Olympus) with a 40 x magnification.

Specimens' surface roughness was assessed by images obtained under the stereomicroscope. A single-blind study analysis was carried out by two calibrated evaluators. The inter-rater agreement was confirmed by the Kappa test (K= 0.875). The surface was classified according to the following rank:

0 – no apparent roughness

1 – average roughness

2 – great roughness

Ranked data were submitted to Kruskall-Wallis non-parametric ANOVA followed by Tukey [18] multiple pair wise comparison (significance to $p < 0.05$) in SigmaPlot software.

Nanoscale morphologies of four disks (Z350 composite) brushed with the different dentifrices were assessed by ScanAsyst mode atomic force microscopy (Bruker, Germany) with a silicon nitride cantilever probe (ScanAsyst-Air, Bruker, Germany). Images analyses (25 x 25 $\mu$m scan size) were carried out using WSxM 5.0 Develop 8.0. Software.

## RESULTS

### *Stereomicroscopy*

Some of the images achieved by stereomicroscopy are represented in Figure 1. Kruskall-Wallis analysis of variance and medians by dentifrice are shown in figure 2. Graph

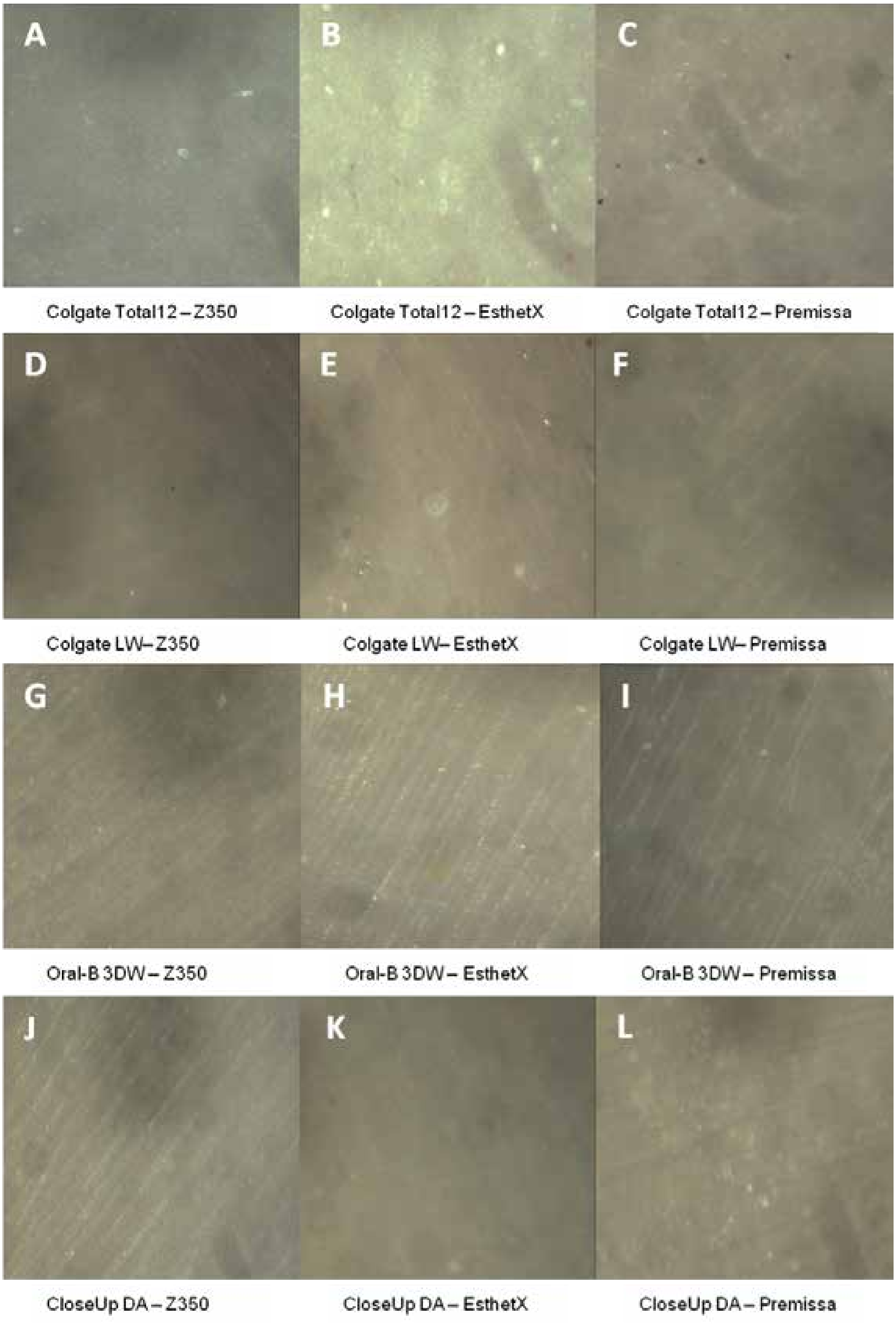


**Figure 1 -** Images (40 x) from the surface of the studied composites. For each image is indicated the dentifrice-composite brands.

in figure 2 points to the statistical difference between whitening dentifrices and the control (Colgate Total 12). Specimens brushed with whitening dentifrices showed higher roughness than control (rank 0), as shown in figure 1. Close Up Diamond Attraction and Colgate Luminous White presented similar roughness (rank 1). Specimens brushed with Oral-B 3D White presented the greatest roughness for this analysis (rank 2).

The specimens' distribution according to ranks is shown in Figure 3. The most prevalent score for the control dentifrice was 0 (zero – no apparent roughness). However, 20% of Premissa specimens had a score of 1 (one). When specimens were brushed with Colgate Luminous White, the most prevalent score was 1 (one), and some specimens made of Premissa composite (40%) presented a score of 2 (two). Only one specimen of Esthet X presented no apparent roughness. The surface analysis of the specimens brushed with Close Up Diamond Attraction revealed a wider distribution of scores among the studied composites. The specimens manufactured with Z350 composite presented, for 80 percent of the specimens, the highest score (2). The highest frequency of rough surfaces was observed in specimens brushed with Oral-B 3D White dentifrice (at least 60% of the specimens from any group obtained a score of 2).

### *Atomic Force Microscopy*

Images achieved by ScanAsyst AFM are shown in figure 4. Noteworthy it is the fact that AFM images corroborate with the results found in stereomicroscopy analysis. Specimens brushed with the control dentifrice (Colgate Total 12) presented a more regular surface, when compared to other assessed dentifrices.

Control specimens also presented fewer peaks, when compared to specimens brushed with Colgate Luminous White and Close Up Diamond Attraction, at a sub-micrometric scale. Oral-B 3D White produced an irregular surface without any pattern, and also with fewer peaks, when compared to other whitening dentifrices.

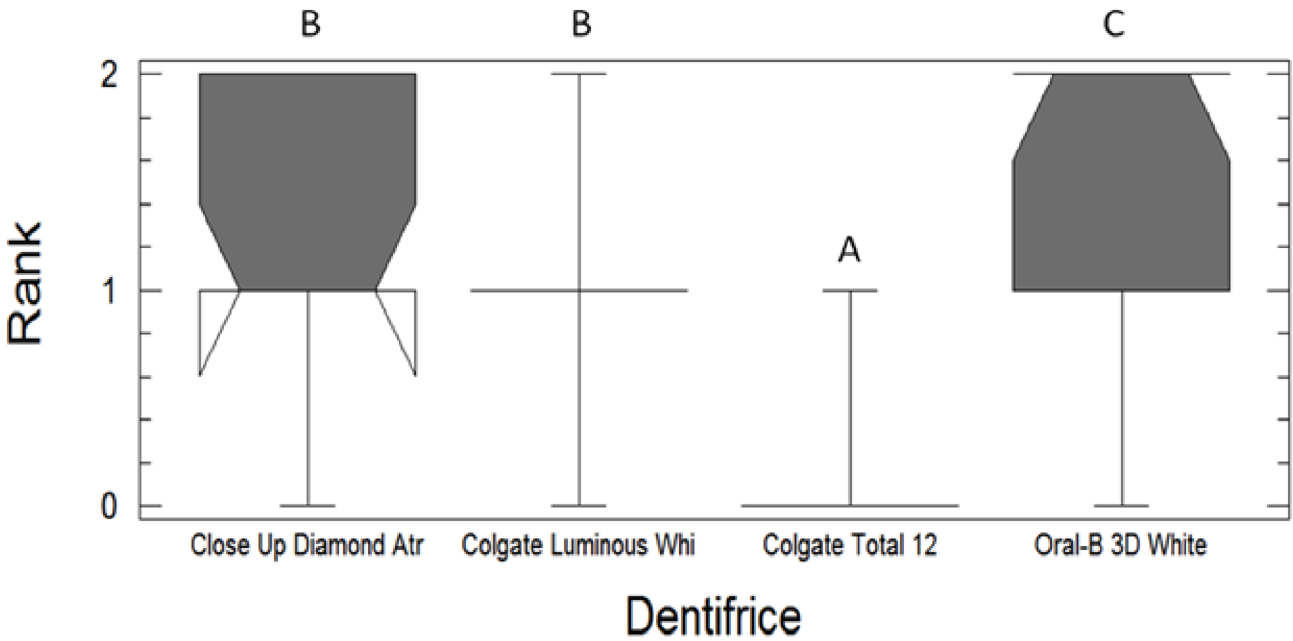


**Figure 2 -** Ranks of whitening dentifrices and control group (Kruskal-Wallis; p = 0.05). Different letters correspond to statistically significant difference.

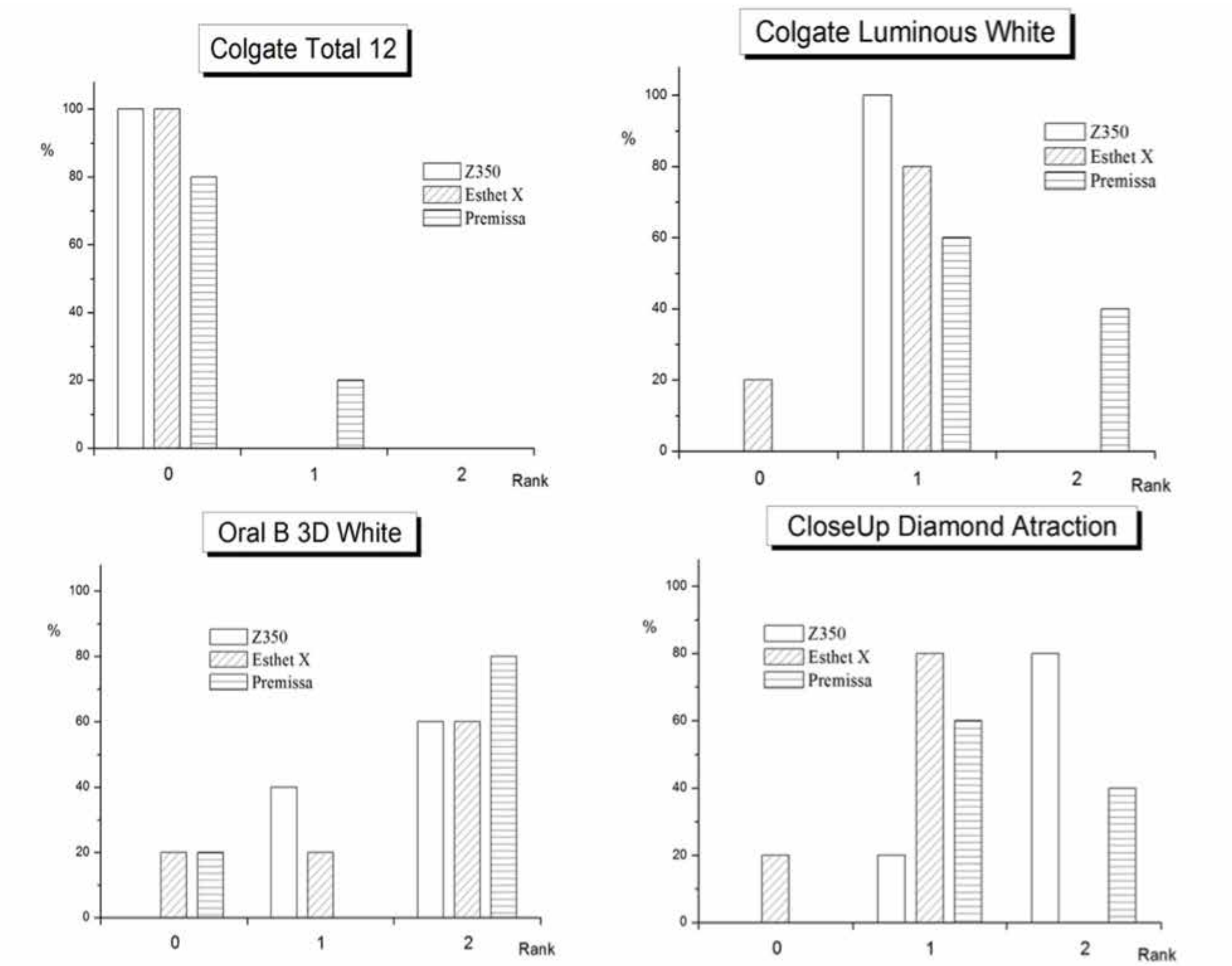


**Figure 3 -** Frequency and distribution of the specimens in ranks according to roughness analyses.

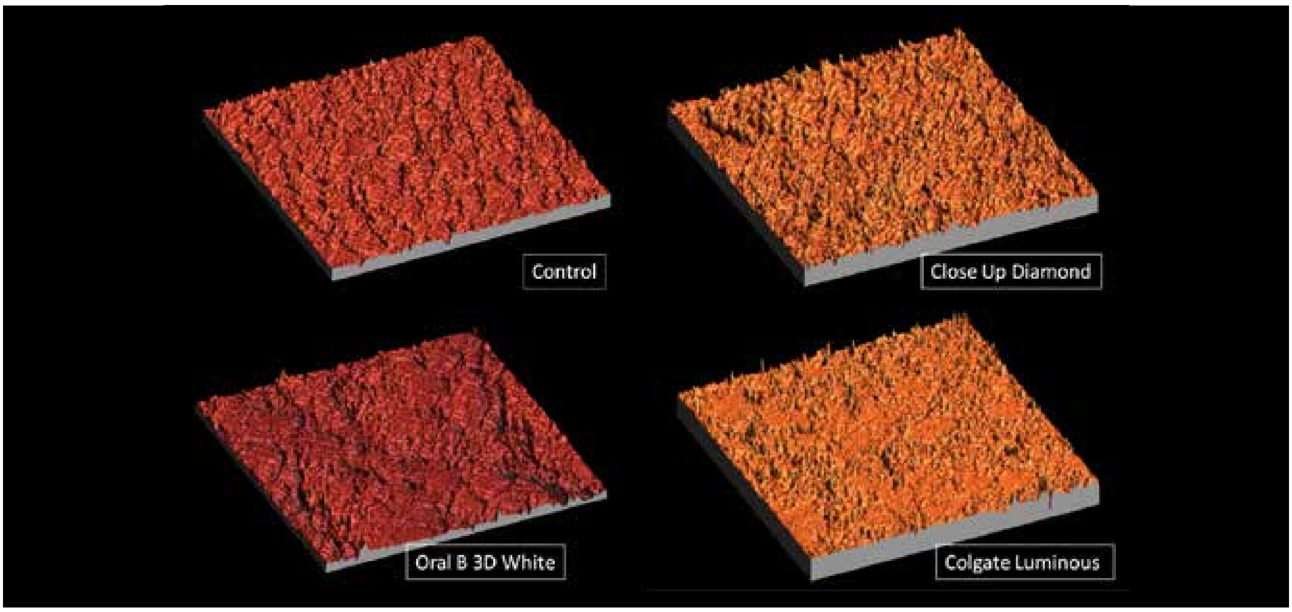


**Figure 4 -** AFM images of Z350 composite. The control group presented the most regular surface in comparison to the whitening dentifrices.

## DISCUSSION

Aesthetic appeal in modern society is extremely strong, which has resulted in a growing interest of pharmaceutical industries in developing new beauty products. In the dental field, specifically, the need for white teeth has led to the recent emergence of OTC products as whitening dentifrices, mouthwashes, whitening strips and bleaching chewing gum, which should be better investigated regarding possible side effects. Hasson et al, [19] in a systematic review, emphasized that few studies focusing on OTC products are conducted and that the majority of them present some conflict of interest, a fact that continues to happen today. Therefore, it is notable to say that there is the need for conducting independent studies.

Many chemical compounds have been used in the formulation of toothpastes, including surfactants, calcium chelators, enzymes, polymers, and oxidizing agents in order to remove or prevent teeth staining. [20] Few whitening dentifrices have hydrogen peroxide (1%) in its formulation which is the active principle of bleaching products used in the dental office. [10,21] The peroxide is in fact responsible for the disruption of unsaturated groups present in the pigment molecules that are deposited on dental tissue over time. Nevertheless, the indiscriminated use of hydrogen peroxide, without professional supervision, can cause irritation of the gingival tissues and lead to dentin hypersensitivity.[10,21] Moreover, when applied through a dentifrice, the peroxide has its effect limited due to the short time that it acts on the teeth's surface. [10,21]

Another substance used by cosmetic industries in whitening dentifrices is titanium dioxide. This agent acts as a pigment and can color the enamel's surface white, more than the effect in removing stains. Titanium dioxide is still easily removed from enamel surface. [10,21] Among the dentifrices evaluated in this study, Colgate Total 12 and Colgate Luminous White have titanium dioxide in its composition.

Blue covarine is another pigment used in some dentifrices with the ability of remaining on dental enamel in contact to the biofilm producing an optical effect that reduces the yellowing, and results in a bleaching perception. [22] Blue covarine is present in CloseUp Diamond Attraction dentifrice, used in this study. The other whitening dentifrices (Colgate Luminous White and Oral-B 3D White) do not present blue covarine in their composition, but they use the number 1 blue pigment with the same purpose.

Regardless of the addition of hydrogen peroxide or pigments to whitening dentifrice, abrasive substances are often present with the aim of increasing the friction during tooth brushing, which leads to the removal of great part of the extrinsically deposited pigments. [23-25] Nonetheless, the high abrasiveness of the whitening dentifrices can jeopardize resin composite surfaces as detected in this study, without contributing to whiten the teeth. Gerlach et al. [21] in an in vivo comparative study with different whitening OTC products (whitening strips, paint-on gels and whitening dentifrice without peroxide) concluded that the whitening effect of a dentifrice is not clinically significant.

Nanocomposites and nano-hybrid composites are more susceptible to wear [1], which favors polishing and, in this study, they proved to be susceptible to the abrasive effect caused by the whitening dentifrices. Specimens confectioned with any of the three studied composites (Z350, Esthet X, Premissa) presented a scratched surface after the brushing simulation as seen in Figure 1.

These results corroborate another work conducted in 2011, [26] which submitted two micro-hybrid composites (Z250, 3MESPE; Rok, SDI) to 20.000 brushing cycles using a whitening dentifrice (Colgate Max White or CloseUp Extra Whitening) or a control dentifrice (Colgate Total 12). After the *in vitro* brushing simulation, which corresponded to 24 months of in vivo brushing, the authors observed a greater surface roughness measured by rugosimeter on the composite specimens submitted to the whitening dentifrice than those submitted to the control. Monteiro et al. [27], detected the greater increase in surface roughness (with 5.000, 10.000 and 20.000 cycles) of a nano-filler composite surface when a whitening dentifrice (Oral-B Pro-Health Whitening) was used. In the present study, the greater roughness found in a nano-filler composite was also produced by a whitening dentifrice from the same manufacturer (Oral-B 3D White – Procter and Gamble).

It is also worth saying that one thousand brushing cycles, which specimens were submitted, corresponded to one month of in vivo brushing, enough to modify significantly the surface of the nanofiller-based composites with the use of the newer commercially available whitening toothpastes. The changes in specimens' surface were perceivable at a sub-micrometric scale (AFM), but also at a macroscopic scale (stereomicroscopy and even naked eye).

The increase of surface roughness in composite restorations may result in gingival irritation, biofilm buildup and accelerate the composite degradation.26,28 Besides, it can impair the aesthetics and produce the opposite effect to what is expected in a whitening treatment.

Since whitening dentifrices have the ability to remove extrinsic stains due to its abrasiveness, they could be indicated to maintain the whitening effect achieved in office or home bleaching techniques. [10,21] However, Hilgenberg et al [13] verified that the association between bleaching techniques and whitening dentifrices may increase some changes in dental enamel surface induced by the peroxide action. Therefore, the use of whitening dentifrices after a treatment with peroxide bleaching agents must be conducted carefully. Changes in enamel surface or restorative composites are higher when associated to abrasion caused by toothpastes. [29]

Regarding the composites assessed in this study, Z350 was chosen to AFM analysis because of the regular distribution of its nanoparticle fillers. Furthermore, Z350 exhibited great wear resistance in a previous study. [3] Despite this wear resistance of the nanofilled composites, the AFM images revealed an increase in the number and distribution of peaks at a submicrometric scale when using abrasive dentifrices (CloseUp Diamond and Colgate Luminous), as seen in figure 4. The apparent irregularity found in the AFM image for the control dentifrice (Figure 4, Control) was expected, as a previous study demonstrated that a nanofiller composite, even when polished, shows surface irregularities identified in AFM images [30]. These irregularities are represented by lower peaks, while those irregularities found in composites brushed with whitening dentifrices presented a more peaked profile. Another qualitative analysis conducted by Monteiro et al. [27], using MEV images, have shown an increase in surface roughness of Z350 composite specimens when a whitening dentifrice was tested. One of the specimens (brushed with Oral-B 3D White) did not show an increase in the number of peaks, but presented irregularity by means of the presence of valleys or grooves on the surface. These different profiles observed on surfaces under the action of the studied whitening dentifrices may be related to variations in the dentifrices composition, especially in the hardness and distribution of its abrasive particles.

The main purpose of this study was to show through images the abrasiveness of the whitening dentifrices and its effect on the surface roughness of nanofiller-based composites. The images achieved by stereomicroscopy, while did not bring accurate information about the peaks and valleys formed during abrasion, quite evidently showed the scratching ability of whitening dentifrices in comparison with the conventional dentifrice (control). In this study, as the differences observed in stereomicroscopy were clear enough, it can be inferred that the results would probably be confirmed by a quantitative analysis obtained by profilometry or rugosimeter.

Indeed, it was still possible to compare the images obtained with stereomicroscope with images generated by AFM, which allowed a better understanding of the changes that occurred on composites surface.

Despite the limitations of this study, it is possible to warn the clinicians about the possible effects of these toothpastes when used indiscriminately and without proper control.

## CONCLUSION

*In vitro* brushing of resin composites specimens with whitening dentifrices proved to significantly increase specimens' surface roughness when compared to *in vitro* brushing with a conventional dentifrice.

It is strongly recommended that clinicians guide their patients, warning them about the risks and consequences of their use.

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