Surface Roughness of Composite Resins after Simulated Toothbrushing with Different Dentifrices
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Abstract:
Background: The aim of the study was to evaluate, in vitro, the surface roughness of two composite resins submitted to simulated toothbrushing with three different dentifrices.

Materials and Methods: Totally, 36 samples of Z350XT and 36 samples of Empress Direct were built and randomly divided into three groups (n = 12) according to the dentifrice used (Oral-B Pro-Health Whitening [OBW], Colgate Sensitive Pro-Relief [CS], Colgate Total Clean Mint 12 [CT12]). The samples were submitted to 5,000, 10,000 or 20,000 cycles of simulated toothbrushing. After each simulated period, the surface roughness of the samples was measured using a roughness tester.

Results: According to three-way analysis of variance, dentifrice (P = 0.044) and brushing time (P = 0.000) were significant. The composite resin was not significant (P = 0.381) and the interaction among the factors was not significant (P > 0.05). The mean values of the surface roughness (µm) followed by the same letter represent no statistical difference by Tukey’s post-hoc test (P <0.05): Dentifrice: CT12 = 0.269; CS Pro- Relief = 0.300⁴; OBW = 0.390⁵. Brushing time: Baseline = 0.046⁴; 5,000 cycles = 0.297⁷; 10,000 cycles = 0.354⁷; 20,000 cycles = 0.584⁷.

Conclusion: Z350 XT and Empress Direct presented similar surface roughness after all cycles of simulated toothbrushing. The higher the brushing time, the higher the surface roughness of composite resins. The dentifrice OBW caused a higher surface roughness in both composite resins.

Key Words: Composite resins, dentifrices, tooth abrasion, toothbrushing

Introduction
Dentifrices have been a source of concern and subject of study for many professionals, since it is one of the main resources used in daily oral care by the population. Dentifrices have different components, such as detergents, fluoride, therapeutic ingredients, flavors, and abrasives. Among the abrasives, the most common are calcium carbonate and silica.¹ These abrasives have an important role in cleaning teeth, removing bacteria and stains from the tooth surface. However, the dentifrice should promote optimal tooth surface cleaning with minimal abrasion,² since dentifrices with high amounts of abrasives can damage hard tissue, soft tissue and restorations, causing gingival recession, cervical abrasion, and dentin hypersensitivity.³,⁴

Currently, patients increasingly value esthetic and seek simplified and safe results for their oral problems.³ Thus, the composite resins have been widely used in restorative treatments. However, it should be considered that the success and longevity of these restorations are related to the material, the dentist, and the patient.⁶ The patients are responsible to care for their dietary habits, preventive measures, availability of fluoride, and oral hygiene since carious lesions have been the most common cause of direct composite resin restorations replacements.⁷

Studies have been conducted to evaluate the surface roughness of composite resins caused by the abrasivity of some dentifrices. Amaral et al.⁸ evaluated the action of abrasive dentifrices on esthetic restorative materials after simulated toothbrushing cycles. The researchers found a significant difference between the abrasivity of dentifrices, but not among the composite resins. The dentifrices that used silica and carbonate were less abrasive compared to the ones containing bicarbonate. Liljeborg et al.⁹ assessed the acrylic surface roughness caused by different values of relative dentin abrasivity (RDA), which is the abrasivity characteristic of dentifrices provided by the manufacturer. In this study, an association was analyzed between the value of RDA, provided by the manufacturer, with the surface roughness (Ra) found for each dentifrice after simulated toothbrushing. Surprisingly, the correlation between RDA and Ra value was too low. Dentifrice with low RDA (40) showed higher Ra (5.73 µm), while the other with high RDA (130) obtained Ra of 1.84 µm; a third dentifrice with the lowest value of RDA (30) showed a Ra value of 1.13 µm.

A large supply of new dentifrices has been available on the market nowadays, which makes the choice more difficult for patients. Furthermore, the speed of production has surpassed independent studies without ties to manufacturers.¹⁰
Concerned about the integrity of composite resin restorations from the abrasivity of dentifrices available, this study aims to answer the following questions: (a) does toothbrushing with dentifrice change the surface roughness of composite resins? (b) is the surface roughness the same, regardless of the type of dentifrice or composite resin evaluated? Therefore, the aim of the study was to evaluate, in vitro, the surface roughness of two composite resins submitted to simulated toothbrushing with three different dentifrices in periods of 6 months, 1 year, and 2 years. The null hypothesis is that toothbrushing with dentifrices does not influence the surface roughness of composite resins.

Materials and Methods

Selection of composite resin specimens
The composite resins Empress Direct (Ivoclar Vivadent, Schaan, Liechtenstein) and Z350 XT (3M/ESPE, St. Paul, MN, USA), enamel shade A1, were selected. The specifications of each composite resin brand are described in Table 1.

The composite resin specimens were made using a silicone matrix with orifices of 5 mm in diameter and height. The matrix was positioned on a glass plate and filled with composite resin. A polyester strip was then placed on the composite resin followed by a glass plate in order to obtain a flat surface. The composite resin was then light-cured with the light-emitting diode light unit Radii-cal (SDI, Australia) for 20 s at a distance of 1 mm from the surface of the specimen. The composite resin surface in contact with the polyester strip received the toothbrushing with dentifrices. For each composite resin, 36 specimens were fabricated in distilled water at 37°C for 24 h to complete the polymerization and simulate conditions of the oral cavity environment.

### Table 1: Specifications and manufacturer of composite resins.

| Composite resin   | Organic phase                                                                 | Inorganic phase                                                                 | Manufacturer               |
|------------------|-------------------------------------------------------------------------------|--------------------------------------------------------------------------------|---------------------------|
| Empress direct   | Dimethacrylates (20 to 21.5% weight, opalescent 17% weight), copolymer (77.5-79% weight, opalescent 83% weight). Additives, catalysts, stabilizers and pigments (<1.0% weight) | Barium glass, ytterbium trifluoride, mixed oxides, silicon dioxide 75-79% weight, 52-59% vol. (opal 60.5% or 45% volume). Average particle size of 550 nm | Ivoclar Vivadent, Liechtenstein |
| Z350 XT          | Bis-GMA, UDMA, TEGDMA, and Bis-EMA                                           | Silica size of 20 nm and zirconia, size 4-11 nm. 72.5% weight (55.5% volume) in translucent shades. 78.5% weight (63.3% volume) for other shades | 3M/ESPE, St. Paul, MN, EUA |

Experimental groups

The 36 specimens of each composite resin were randomly divided into three groups ($n = 12$). Each group was subject to simulated toothbrushing with three different dentifrices: Oral-B Pro-Health Whitening (OBW), Colgate Sensitive (CS) Pro-Relief, Colgate Total Clean Mint 12 (CT12). The dentifrice CT12 was considered the control group. The composition of each dentifrice is shown in Table 2.

Analysis of surface roughness

The initial surface roughness of each specimen was measured with a roughness tester SL-201 (Mitutoyo Surf test Analyzer, Tokyo, Japan). Three consecutive measurements of the specimen were taken in different regions (one central, one right, and one left), with cut-off of 0.25, obtaining the mean average from the three measurements.

Simulated toothbrushing

The simulated toothbrushing machine developed by Idea Institute of PUCRS was used for this study. Each specimen was fixed in the center (orifice) of an acrylic plate (55 mm × 25 mm × 4 mm), respectively for the diameter and height, enabling the test surface to remain 1 mm beyond the edge of the orifice, which housed the specimen. Utility wax was applied to fix the specimens. Each plate was placed in an acrylic tank, which was attached to the brushing machine by metal pins. The acrylic tank was filled with a mixture composed of 1 g of dentifrice paste per 1 ml of distilled water. Soft bristle Classic Colgate (Colgate-Palmolive, São Bernardo do Campo, SP, Brazil) toothbrushes were used, with a load of 200 g. The speed of brushing was 250 cycles per minute, carried out in 5,000 cycles, 10,000 cycles, and 20,000 cycles of simulated toothbrushing, cumulatively, that corresponds to approximately 6 months, 12 months, and

### Table 2: Composition and manufacturer of dentifrices tested.

| Commercial brand | Composition                                                                 | Manufacturer                                                                 |
|------------------|-----------------------------------------------------------------------------|------------------------------------------------------------------------------|
| CT12 Clean Mint  | 1450 ppm of fluoride, triclosan 0.3%, water, sorbitol, silicon dioxide (abrasive), sodium lauryl sulfate, PVM/MA copolymer (Gantrez), flavor, carrageenan, sodium saccharin, sodium hydroxide, white colorant | Colgate-Palmolive, São Bernardo do Campo, SP, Brazil |
| CS Pro-Relief    | Arginine, sodium monofluorophosphate, 1450 ppm fluoride, calcium carbonate, water, sodium bicarbonate, sorbitol, sodium lauryl sulfate, sodium monofluorophosphate, flavor, cellulose gum, potassium ascorbate, sodium silicate, starch xanthan gum, sucralose, titanium dioxide | Colgate-Palmolive, São Bernardo do Campo, SP, Brazil |
| OBW              | Stannous fluoride (1100 ppm fluoride), sodium fluoride (350 ppm fluoride), glycerin, hydrated silica, sodium hexametaphosphate, propylene glycol, PEG-6, zinc lactate, flavor, sodium lauryl sulfate, sodium gluconate, silica, phosphate triisodium, mica, saccharin sodium, carnauba wax, xanthan gum, titanium dioxide | Procter and Gamble Manufacturing GMBH, Gross-Gerau, Germany |

CT12: Colgate Total 12, CS: Colgate sensitive, OBW: Oral B Pro-Health Whitening, PVM-MA: Poly styrol methyl ether maleic acid
24 months of brushing, respectively. The dentifrice pastes and toothbrushes were changed every cycle.

After every brushing cycle, the specimens were washed in running water and ultrasonically cleaned in distilled water for 10 min, followed by drying with compressed air. The roughness of the surface was measured again.

The surface roughness reading was perpendicular to the brushing direction of the toothbrush bristles. For the correct positioning of the specimen in the brushing machine and to always ensure readability in the same direction (perpendicular to the brushing), a mark with a diamond bur and high-speed handpiece was made on the border of each specimen.

Analysis by scanning electron microscopy (SEM)
From each of the composite resins, 12 additional specimens were obtained, as previously described. The 12 specimens were randomly divided into three groups (n = 4), each group being subjected to the simulated toothbrushing with dentifrice as described above. Of the four samples, one was analyzed by SEM before simulated toothbrushing, the second after 5,000 cycles, the third after 10,000 cycles, and the fourth after 20,000 cycles of simulated toothbrushing. The specimens were dried in dehumidifier with silica gel for 72 h, metallized with gold (Balzers, Liechtenstein) and observed under a scanning electron microscope (JSM 6060, Eindhoven, Netherlands) at ×4,000 magnification for qualitative analysis of the surface.

Statistical analysis
The values of surface roughness were submitted to the Kolmogorov-Smirnov normality test. Three-way analysis of variance (ANOVA) was used to test the effect of composite resin, dentifrice, and brushing time on the surface roughness, followed by Tukey’s test at a significance level of 5%.

Results
Three-way ANOVA analysis showed that the dentifrice (P = 0.044) and the brushing time (P = 0.000) had a significant effect on the surface roughness. The composite resin was not significant (P = 0.381) and the interaction among the factors was not significant (P > 0.05).

CT12 had the lowest surface roughness mean (0.269 µm), which was not significantly different from CS (0.300 µm). The highest surface roughness mean was obtained with OBW (0.390 µm), which was not significantly different from the CS (Table 3).

The highest surface roughness mean was obtained after 20,000 brushing cycles (0.584 µm), differing statistically from the other cycles. The 5,000 cycles (0.297 µm) and 10,000 cycles (0.354 µm) had no statistical difference. The lowest surface roughness mean was obtained for the measurement performed at baseline (0.046 µm), differing statistically from the other cycles (Table 4).

Analysis by SEM
The SEM images of Figure 1a-d correspond to the Z350 XT composite resin. There was substantial surface smoothness of the composite resin (Figure 1a) before toothbrushing. After 5,000 cycles of brushing, dentifrice OBW (Figure 1d) caused significant surface roughness of the composite resin compared to dentifrice CT12 (Figure 1b) and CS (Figure 1c).

Figure 2 corresponds to Empress Direct composite resin. The increasing number of brushing cycles caused a higher removal of the resin matrix and thus greater exposure of the filler particles.

Discussion
The clinical wear of a restoration may result from numerous factors, such as functional and centric contacts, attrition of the

| Dentifrices     | Mean value (µm) | Standard deviation |
|-----------------|-----------------|--------------------|
| CT12 Clean Mint | 0.269*          | 0.244              |
| CS Pro-Relief   | 0.300+          | 0.252              |
| OBW             | 0.390*          | 0.317              |

Means followed by the same letter do not differ statistically according to Tukey’s test at a significance level of 5%. OBW: Oral B Pro-Health Whitening, CS: Colgate Sensitive, CT12: Colgate Total 12, (a) abrasion with Colgate Total 12 Clean Mint; (b) abrasion with Colgate Sensitive Pro-Relief; (c) abrasion with Oral B Pro-Health Whitening.

| Time            | Mean value (µm) | Standard deviation |
|-----------------|-----------------|--------------------|
| Baseline        | 0.046*          | 0.022              |
| 5,000 cycles    | 0.297*          | 0.270              |
| 10,000 cycles   | 0.354*          | 0.267              |
| 20,000 cycles   | 0.584*          | 0.526              |

Means followed by the same letter do not differ statistically according to Tukey’s test at a significance level of 5%.

Table 3: Surface roughness mean value (µm) of different dentifrices.

Table 4: Surface roughness mean (µm) according to the brushing time.
According to Cury et al., there are dentifrices with low, medium, and high abrasivity. Among the lower abrasivity are the dentifrices in the form of gel, containing silica as the abrasive agent. However, when this mineral is combined with other abrasives such as calcium carbonate, sodium pyrophosphate, titanium oxide, and sodium phosphate, it is considered a high abrasive dentifrice. This finding is consistent with the results of this study, in which the dentifrice OBW, responsible for a statistically significant abrasivity, has silica as an abrasive agent in combination with titanium dioxide, classifying it as a highly abrasive dentifrice.

According to Gusmão et al., the main cause factor of abrasivity is not limited to the type nor quantity of abrasive present in the dentifrice, but the physical characteristics of the mineral as the size and the shape of the particles. Silica, for example, when used in fine particles with regular shapes, preserves its mild abrasivity mineral characteristic, but when coarse and irregular particles are incorporated, it is highly abrasive. Thus, only the gel or cream formula, or the type of the abrasive agent present in the dentifrice has not been sufficient to characterize its abrasivity to the tooth structures and composite resins.

The simulated toothbrushing abrasion is considered an established model in the literature, because it is an important in-vitro wear factor, able to simulate a clinical condition. According Sexson and Phillips, for each session of brushing a patient performs approximately 15 cycles. Thus, maintaining an oral hygiene based on two daily sessions of brushing, 10,000 cycles are performed by the end of 1 year.

In the present study, the brushing time was statistically significant. The highest surface roughness mean was obtained after 20,000 cycles, the equivalent of 24 months of brushing (0.584 µm), differing statistically from 5,000 cycles, equivalent to 6 months of brushing (0.297 µm) and 10,000 cycles, equivalent to 12 months of brushing (0.354 µm). This finding concurs with other studies. In the present study, a nanohybrid composite resin (Empress Supreme XT, which was considered a clinically successful composite resin. This finding is not equivalent with the present

The lowest surface roughness mean was obtained with the dentifrice CT12 (0.269 µm), confirming Barbieri et al. study that the same dentifrice was responsible for a low abrasion of composite resins analyzed. The CT12 contains silica as an abrasive agent that exhibits low abrasivity. Amaral et al. study concluded that dentifrices based on silica or calcium carbonate are less abrasive than those containing sodium bicarbonate. This possibly justifies the fact that the dentifrice CS, which has bicarbonate in the composition, caused a higher surface roughness compared to CT12, although no statistical difference was found.

The dentifrice OBW showed the highest Ra (0.390 µm) mean, different from CT12. This result corroborates the findings of Amaral et al. and Barbieri et al., in which the whitening dentifrices also results in higher surface roughness both containing silica abrasives in combination with other agents. The study of Menezes et al. found a significant difference in Ra caused by whitening dentifrices on bovine root dentin, stating that the change of Ra depends on the dentifrice used.

Figure 2: Surface morphology of composite resin Empress Total 12 Clean Mint (×4,000) (a) 0 cycle; (b) 5,000 cycles; (c) 10,000 cycles.
study, in which both composites (Empress Direct and Z350 XT) performed similarly to a performance evaluation of 24 months (20,000 cycles). This difference in results among studies is probably related to the amount of toothbrushing cycles performed on each study.

According to the manufacturer, the composite resin Z350 XT consists of a combination of zirconia and silica nanoparticles in nanoclusters reducing the interstitial space between the particles and increasing the amount of load. The resin matrix with nanoparticles results in better physical properties and greater wear resistance, which means that during the abrasion these nanoclusters wear at a speed similar to the surrounding resin matrix. The result is a smooth, glossy, and durable polished surface.

Through the analysis of the SEM images, it was found that both composite resins had higher surface irregularities, higher resin matrix wear, and exposure of the filler particles when brushed with the dentifrice OBW. This wear was higher with the increase in the number of brushing cycles. These wear patterns are commonly found in evaluations by SEM after abrasive test.13-16,18

Regarding the clinical significance of this study, it is evident that the surface roughness of composite resins changes according to the type of dentifrice used and the time of brushing, and the whitening dentifrice OBW had greater capacity to change the surface of composite resins.

Conclusions
Within the limitations of the methodology used, it was possible to conclude that:
- The composite resins Z350 XT and Empress Direct showed similar surface roughness after all cycles of simulated toothbrushing
- The higher the brushing time, the higher the surface roughness of composite resins
- The dentifrice OBW caused a higher surface roughness in both composite resins.

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