

# Evaluation of Surface Characteristics and Weight Variation of Different Composite Resins after Simulated Toothbrushing

Jéssika Raissa Medeiros De Almeida, Aion Mangino Messias<sup>1</sup>, Diana Ferreira Gadelha, Sergei Godeiro Fernandes Rabelo Caldas, Marília Regalado Galvão Rabelo Caldas

Department of Dentistry, Federal University of Rio Grande do Norte, Natal, <sup>1</sup>Department of Dentistry, São Paulo State University, São Paulo, Brazil

## Abstract

**Objective:** To evaluate the surface characteristics of restorations performed after simulated toothbrushing and to compare the results between the composites. **Materials and Methods:** In total, 80 samples were made (7 mm × 4 mm) from the composites: Bulk Fill One, Bulk Fill Flow, Z350 XT, and Z350 XT flow (3M/ESPE, St. Paul, MN, USA). Half of the specimens was submitted to 60,000 cycles of simulated toothbrushing, while the other half was the control group ( $n = 10$ ). The surface was evaluated through Scanning electronic microscopy (SEM), surface roughness (Ra), and weight loss, before and after the simulated toothbrushing. To evaluate the statistical analysis, a two-factors variance test and the Tukey's posttest were performed. **Results:** In the analysis performed by SEM, it was observed the presence of both small and medium protruding particles in all groups after simulated toothbrushing, with the exception of the group which used Z350 flow composite. All materials presented increasing on Ra after simulated toothbrushing. Regarding weight variation, there was not any significant statistical difference in all materials. **Conclusions:** The abrasive process occasioned by toothbrushing leads to changes on the particles' disposition, which causes changes on the surface and increase on the Ra. On the other hand, there was not any difference regarding to weight variation.

**Keywords:** Scanning electronic microscopy, surface properties, wear

## INTRODUCTION

Nowadays, in Restorative Dentistry, there is an increasing of the aesthetic exigence by the patients, the development of new materials, and the consequent progress focused on mechanical proprieties<sup>[1]</sup> of the materials. In this way, changes in these proprieties are observed, related to size, morphology, and components of the fillers,<sup>[2]</sup> allowing resin composites to be widely used both for anterior and posterior restorations.<sup>[3,4]</sup>

In some clinical situations, there is the need of using a composite less viscous to promote better adaptation on the walls of the cavity. Due to this reason, low-viscosity resins (flow) were developed and introduced on the market on the late 90s. They present a filler volume reduced (approximately 37%–53%) – when compared to 50%–70% of the conventional composites, and that is what confers fluidity to this material.<sup>[5]</sup> Low-viscosity resins have many indications, such as liners, repairs on amalgam restorations, sealants, or yet class V restorations. It is important to emphasize that, due to its low mechanical properties, this material should not be used in areas submitted to masticatory forces.<sup>[6]</sup>

To supply another need on the dental office, low and high viscosity Bulk fill composites emerged on the market, and initially were developed to fill cavities with an unique increment up to 4 or 5 mm.<sup>[1,4]</sup> This fact allowed the optimization of the clinical time and the replacement of the traditional incremental technique, which is based on multiple increments of 2 mm<sup>[7]</sup> and also decreased the risk of contamination by blood, saliva, or oral fluids, as well as the postoperative sensitivity.<sup>[8,9]</sup> There are reports that Bulk fill composites present superior physical and mechanical proprieties to resist to the masticatory forces<sup>[1,10]</sup> because of the lower stress of polymerization contraction and this fact is due to the incorporation of other metacrylate monomers, such as AUDMA and AFM, which

**Address for correspondence:** Prof. Marília Regalado Galvão Rabelo Caldas, Av. Sen. Salgado Filho, 1787 - Lagoa Nova, Natal - RN, 59056-000, Brazil. E-mail: mariliaregalado@hotmail.com

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limits the contraction zones.<sup>[10]</sup> Besides that, the increase of the polymerization depth was reached through the improvement on the translucency of the material,<sup>[9]</sup> which might be achieved by decreasing the amount of particles and increasing the size of the particles, or yet by the addition of different photoinitiators to the same composite.<sup>[9]</sup>

The clinical success and the longevity of the restorations depend on many factors, such as the type of restorative material, the ability of the operator, the particularities of each patient,<sup>[3,4]</sup> as well as the surface properties, for example: surface roughness (Ra), characteristic which might be enhanced through finishing and polishing procedures.

Superficial texture is an important factor related to longevity<sup>[4]</sup> since studies have been demonstrating that a rough surface may affect color and gloss of the restoration, leading to a greater accumulation of biofilm, favoring to the development of secondary caries and inflammation on the periodontal tissues.<sup>[4,11-13]</sup> In this way, is extremely necessary to perform polishing procedures, aiming to enhance the surface properties of the restorative materials.<sup>[12]</sup>

Furthermore, another factor that may result in damage to the surface of resin restorations is toothbrushing. Despite this process has an important role on the oral health of the individual, it is known that the toothbrushing causes wear on the surface of the restoration, turning it rougher, and leading to the undesirable effects mentioned above. The amount of wear depends mainly on the toothbrushing habits, toothbrush type (soft, medium, or hard bristles), and the type of dentifrice utilized.<sup>[1,14]</sup>

Many studies have been focusing on the mechanical proprieties of bulk-fill composites. Even though there are still a few reports on the literature concerning to the behavior or these composites when submitted to the abrasion occasioned by toothbrushing,<sup>[1,15]</sup> there are clinical findings indicating that this process occurs and affects adversely the surface characteristics of the restorations..<sup>[11]</sup>

Therefore, dentifrices should promote excellent dental cleaning with minimal abrasive action, with the objective of avoiding damage to the restorations and soft tissues, also avoiding the occurrence of process such as gingival recession, cervical abrasion, and dentinary hypersensitivity.<sup>[3]</sup>

Tests of superficial Ra and simulated toothbrushing have been indicated to evaluate the restorative materials, mainly

regarding to mechanical proprieties. It has been found that simulated toothbrushing may promote an intentional stress on the organic matrix, on the particles, and on their interfaces, allowing, in this way, an analysis of the properties of resistance to the materials.<sup>[16]</sup>

The ISO standard 11,609:2010 for testing dentifrices recognizes one cycle of simulated toothbrushing as a back and forth movement of the brush heads, using a load of 150 g. It is estimated that between 10,000 and 14,600 back and forth brushing cycles in these machines corresponds to 1 year of *in vitro* tooth brushing in a healthy individual.<sup>[15]</sup>

The wear observed in these materials over time might modify the characteristics of surface, compromising the longevity of the restoration.<sup>[17]</sup> In this way, it is essential to perform follow-ups of the restorations made with resin composites, with the objective of avoiding the excessive wear of the material to the point of compromising the properties of the restoration.

In this way, the objective of this study was to evaluate the superficial Ra and the surface of different resin materials, analyzing the behavior of them when submitted to the wear promoted by the process of simulated toothbrushing.

## MATERIALS AND METHODS

On this study, four commercial resins were utilized: Bulk Fill One (3M/ESPE, St. Paul, MN, USA), Bulk Fill Flow (3M/ESPE, St. Paul, MN, USA), Z350 × T (3M/ESPE St. Paul, MN, USA) and Z350 × T Flow (3M/ESPE, St. Paul, MN, USA). The composition of each material is described in Table 1:

### Sample confection and group division

The specimens were made from a silicone matrix (7 mm × 4 mm<sup>2</sup>).<sup>[18]</sup> The matrix was placed over a glass plate and filled with the resin composite, following the manufacturer's recommendations. To the resins Z350 XT and Z350 XT flow, it was followed the incremental technique, with multiple increments of 2 mm, which were measured with the support of a periodontal probe. The resins Bulk Fill One and Bulk Fill Flow were placed on an unique insertion. One polyester strip was placed over the specimen, followed by another glass plate, to obtain a smooth surface.

In sequence, the glass plate was removed, and the specimens were light cured with Rádi Plus (SDI, Australia) for 20 s. The

**Table 1: Description of the materials, composition, and lot number**

Material	Composition	Lot number
Filtek Bulk Fill One 3M ESPE	Silane treated zirconia / silica filler, silane treated with zirconia, ytterbium fluoride, DDDMA, UDMA, ERGP-DMA, water, curing agents, stabilizers and colorants.	1805200490
Filtek Bulk Fill Flow 3M ESPE	Treated silanized ceramics, UDMA, substituted dimethacrylate, BISGMA; benzotriazole, TEGMA and ethyl 4-dimethylaminobenzoate.	1632600450
Filltek Z350XT 3M ESPE	Ceramics treated with silane, BIS-GMA, BIS-EMA, silane-treated silica, silane-treated zirconium silica, dimethacrylate diurethane, TEG-DMA, BHT and pigments.	1733900627
Filltek Z350XT Flow 3M ESPE	Ceramics treated with silane, substituted dimethacrylate, BIS-GMA, silane treated silica, TEG-DMA, ytterbium fluoride, functionalized dimethacrylate polymer and titanium dioxide.	1805000642

surface which was in contact with the polyester strip, was the surface that receive, posteriorly, the simulated toothbrushing with the dentifrice. For each material, 20 specimens were made, totalizing 80 specimens – half of them were submitted to the simulated toothbrushing with 60,000 cycles, while the other half was the control group.<sup>[3]</sup> The division of the groups is described in Table 2:

The specimens remained stored in distilled water at 37°C for 24 h, to complete the process of polymerization and to simulate the conditions of the oral environment.<sup>[10]</sup> Then, it was performed the polishing with the sequence of sandpaper discs Praxis (TDV, Pomerode, SC, Brazil).

### Determination of initial weight

Each specimen was measured each 24 h on an analytical digital balance (GH-252, A and D Company, Limited, Tokyo), with

0.1 mg accuracy. The measurements were repeated until the difference between the last five values were lower than 0.5 mg, proving that the materials achieved constant weight.<sup>[11,18]</sup> On the intervals, the specimens were stored in a plastic recipient, immersed in distilled water, in a stove at 37°C. The simple arithmetic mean of the last five measurements was calculated, and it was considered as the value of initial weight (Wi) of each specimen.<sup>[18]</sup>

### Scanning electronic microscopy

It was chosen, randomly, four samples of each group to be submitted to the analysis of the scanning electronic microscopy (SEM) Hitachi TM3000 (Hitachi, Brazil). The analysis was performed to observe the surface of the samples that did and did not receive the cycles of simulated toothbrushing, on a magnification of  $\times 500$ .<sup>[16]</sup>

### Surface roughness

The surface Ra was measured in two moments: before and after the process of simulated toothbrushing. The readings were taken by rotation the specimens, clockwise at random angles, through a surface profilometer aparelho Surtronic 25 (Taylor Hobson, Leicester, United Kingdom). Five readings were made across the diameter of each sample. The mean Ra parameter was recorded as the average of the five readings.<sup>[11]</sup> The values are described on Table 3.

### Simulated toothbrushing

Ten specimens of each type of resin were submitted to the abrasion test on a simulated toothbrushing machine (MAVTEC,

**Table 2: Division of the groups based in the amount of toothbrushing cycles**

GROUPS (n=10)	Type of material and treatment
ZC	Z350 XT (control)
ZFC	Z350XT Flow (control)
BFC	Bulk Fill One (control)
BFFC	Bulk Fill Flow (control)
Z	Z350 XT + 60,000 cycles
ZF	Z350 XT Flow + 60,000 cycles
BF	Bulk Fill One + 60,000 cycles
BFF	Bulk Fill Flow + 60,000 cycles

**Table 3: Mean and standard deviation of Roughness (Ra) of different composite resins before and after simulated toothbrushing**

Simulated Toothbrushing	Composite Resins			
	Z	ZF	BF	BFF
Before (Ra) initial	0,23 $\pm$ (0,02) <sup>Aa</sup>	0,19 $\pm$ (0,01) <sup>Aa</sup>	0,38 $\pm$ (0,03) <sup>Ba</sup>	0,25 $\pm$ (0,02) <sup>Aa</sup>
After (Ra) final	0,32 $\pm$ (0,09) <sup>Ab</sup>	0,38 $\pm$ (0,06) <sup>Ab</sup>	0,87 $\pm$ (0,08) <sup>Cb</sup>	0,57 $\pm$ (0,08) <sup>Bb</sup>

Same letters represent the absence of significant differences. Uppercase to lines and lowercase to columns

**Table 4: Table 2 – Mean and standard deviation of Initial Weight (Wi) and Final Weight (Wf) of different composite resins before and after simulated toothbrushing**

Simulated Toothbrushing	Composite Resins			
	Z	ZF	BF	BFF
Before (Mi)	0,72 $\pm$ (0,03) <sup>Ba</sup>	0,38 $\pm$ (0,06) <sup>Aa</sup>	0,79 $\pm$ (0,02) <sup>Ba</sup>	0,31 $\pm$ (0,01) <sup>Aa</sup>
After (Mf)	0,69 $\pm$ (0,02) <sup>Ba</sup>	0,33 $\pm$ (0,03) <sup>Aa</sup>	0,73 $\pm$ (0,07) <sup>Ba</sup>	0,27 $\pm$ (0,04) <sup>Aa</sup>

Same letters represent the absence of significant differences. Uppercase to lines and lowercase to columns

**Table 5: Mean and standard pattern of Weight Variation ( $\Delta W$ ) of different composite resins before and after simulated toothbrushing**

Simulated Toothbrushing	Composite Resins			
	Z	ZF	BF	BFF
$\Delta M$	0,03 $\pm$ (0,001) <sup>A</sup>	0,05 $\pm$ (0,002) <sup>A</sup>	0,06 $\pm$ (0,03) <sup>A</sup>	0,04 $\pm$ (0,002) <sup>A</sup>

Same letters represent the absence of significant differences.

Ribeirão Preto, São Paulo, Brazil). Each sample was brushed with a soft, nylon-bristled toothbrush (Colgate Classic, 32 tufts, 60 bristles per tuft), under a brush-head load of 176 g/2N, that is, a constant load of 176 g was applied to promote approximately 2N of force when performed the toothbrushing. This is a typical load utilized in other studies of simulated toothbrushing to resemble the load of 150 g, which is recommended by the ISO pattern.<sup>[3,15]</sup>

The specimens were immersed in slurry of dentifrice (Colgate Total 12, Colgate, São Bernardo do Campo, SP, Brazil) and distilled water (1:2 wt: Wt ratio). In total, 60,000 strokes (complete forward and reverse movement) were performed at a frequency of 4 Hz. This amount of cycles is proportional to the time of 4 years of toothbrushing in real conditions. For each material, the remaining 10 samples were stored at 37°C throughout the study, defining the control groups.<sup>[11]</sup>

### Determination of final weight

After the toothbrushing, the specimens were profusely washed, and in sequence, they were reconditioned to a constant weight as described to the determination of  $W_i$ . The calculus of Final Weight ( $W_f$ ) was made through the mean of the last five measurements (Sousa et al., 2017). The weight variation ( $\Delta W$ ) of each specimen was calculated from the application of the following equation:

$$\Delta W = W_i - W_f$$

In which:

$\Delta W$ : Weight variation

$W_i$ : Initial weight

$W_f$ : Final weight

The data obtained about  $W_i$ ,  $W_f$ , and  $\Delta W$  are described on Tables 4 and 5.

### Statistical analysis

The data of superficial Ra and weight variation were submitted to initial analysis to detection and normal distribution between the values obtained. Then, it was performed the test analysis of variance two factors and Tukey's posttest, with a significance level of 5% to define between which groups occurred significant differences.

## RESULTS

### Scanning electron microscopy

Figure 1 From the analysis performed by SEM, in amplification of  $\times 500$ , it may be observed that, the surface after the polishing (before simulated toothbrushing – left column) was smoother and uniform. On the other hand, after the simulated toothbrushing (right column), it is observed the presence of protuberant particles of medium and small size at the surface of the resinous matrix, being this fact less evident on the resin Z350 flow, that is, ZF group.

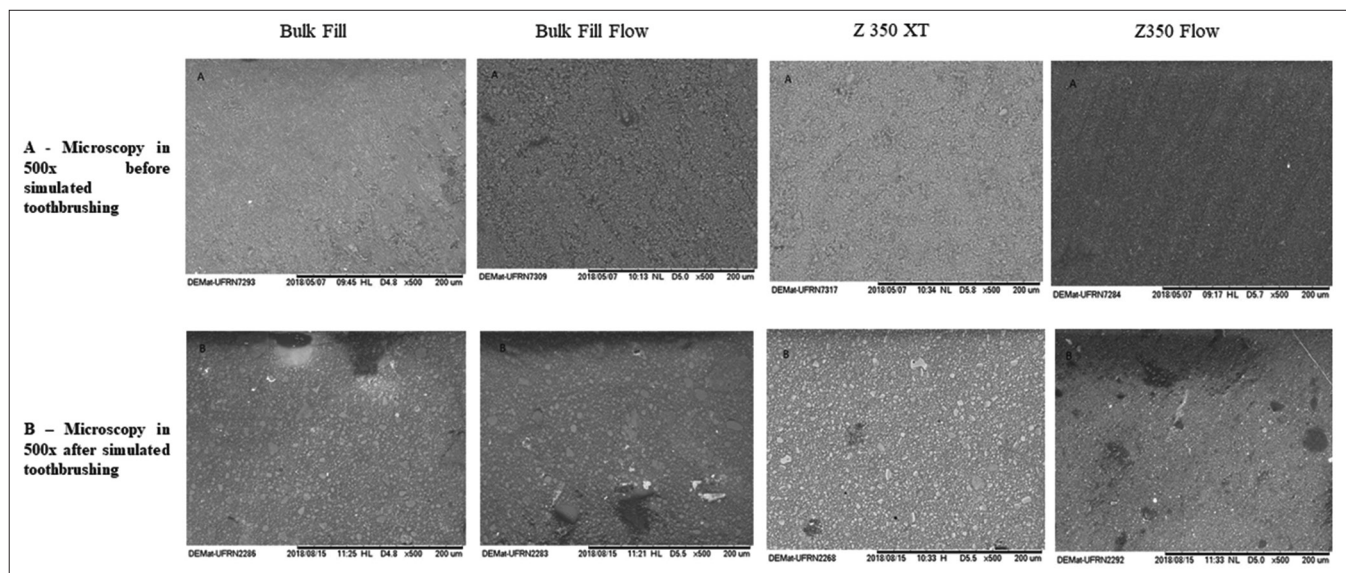
### Surface roughness

Regarding the factor simulated toothbrushing, there was a significant statistical difference in all groups, in which there was an increase on surface Ra after the simulated toothbrushing.

Regarding the factor composite resin, before the toothbrushing, there was a significant difference between the resin BF and the other materials, being the group BF which presented greatest value of initial surface Ra. After the toothbrushing, it was observed a significant statistical difference between the groups Z and ZF with the groups BF and BFF, which presented greater values of final superficial Ra.

### Weight variation

The resins Z350 XT and BF presented greater value of weight (both initial and final). Concerning to the weight



**Figure 1:** Scanning electronic microscopy images before and after simulated toothbrushing



variation ( $\Delta W$ ), it was demonstrated that there was no significant statistical difference.

## DISCUSSION

The processes involved on the wear mechanisms of the resinous composites are too complex and not very elucidated yet.<sup>[14]</sup> On the literature, it is demonstrated that, when present at the oral environment, composite resins are subject to mechanical, thermal, and chemical stimulus, which may lead to damages on the surface of the material, constituting the process of degradation.<sup>[19,20]</sup>

In this way, is observed that, simulated toothbrushing over time promotes the abrasive wear on the surface of the restorations. The intensity of this wear depends on some factors, such as habits of toothbrushing, abrasiveness of the dentifrices utilized, consistency of the bristles of the toothbrush, and some factors related to the properties of the restorative material.<sup>[3,14]</sup>

The dentifrice is influenced by the type and size of the abrasive and the proportion of slurry dentifrice/water; meanwhile, the toothbrush depends on the number, rigidity, and shape of the tufts and bristles. However, since all parameters mentioned were the same to all groups on the present study, the resistance to abrasion of the materials seems to depend on the properties inherent to each one.<sup>[11]</sup>

On this study, it was utilized the dentifrice Colgate Total 12 (Colgate, Brazil), which contains silica in its composition and is considered a dentifrice of low abrasiveness.<sup>[3]</sup> Monteiro and Spohr<sup>[3]</sup> utilized the same dentifrice and resulted in lower values of Ra, when compared to more abrasive dentifrices, for example the dentifrices that contains sodium bicarbonate.

The abrasive process occasioned by simulated toothbrushing is considered a model already consolidated on literature since it is an important factor of wear *in vitro*, which is capable of simulating a clinical condition. According to Sexson and Phillips<sup>21</sup>, to each session of toothbrushing day to day, the patient performs approximately 15 cycles. In this way, by considering the maintenance of the oral hygiene based in two toothbrushings per day, about 10,000–14,600 cycles are completed by the end of 1 year.<sup>[3]</sup> On this study, it was performed 60,000 cycles of simulated toothbrushing, which corresponds to about 4 years of brushing on a healthy individual.<sup>[11]</sup>

As well as on the findings observed on the studies of Suzuki *et al.*<sup>[21]</sup> and Moraes *et al.*,<sup>[11]</sup> regarding the surface analysis performed by SEM, it was noticed that, after simulated toothbrushing, the resin Z350 flow presented a smoother surface, just as it happened on the study of Rigo *et al.*,<sup>[4]</sup> when the resins flow demonstrated smoother surface when compared to conventional resins after the abrasion provided by simulated toothbrushing – fact which may be explained by the minor mean of particles on these materials.

Despite the fact that these composites are not submitted to the resistance provided by the occlusal wear, superficial Ra and

the behavior of these materials after polishing are important characteristics on the proximal areas since at these areas, the materials tend to be exposed to degradation of the oral environment and biofilm accumulation.<sup>[4]</sup> Therefore, justifying the presence of the materials type flow on the present study.

Still concerning to the analysis by SEM, regarding the other resins utilized, it was observed that the surface presented protuberant particles on the resinous matrix. This fact may be caused due to the stress that the process of simulated toothbrushing causes on the matrix, which may lead to loss, fracture, or removal of the matrix, exposing the particles. In this way, the analysis of superficial Ra and wear may establish a comparison of the performance of these materials.<sup>[16]</sup>

Regarding the superficial Ra and the factor simulated toothbrushing, there was significant statistical difference in all resins utilized, in which there was observed an increasing on the superficial Ra after simulated toothbrushing. This result corroborates with other findings on the literature.<sup>[1,14,16]</sup> Garcia *et al.*<sup>[14]</sup> consider that the abrasion mechanism of the composite resins may be explained as the wear of the organic portion of the resin, which leads to the creation of spaces that vary according to the size of the particle, therefore increasing the surface Ra.

According to the studies of Quirynen and Bollen,<sup>[22]</sup> surface Ra should be on a value below 0,2  $\mu\text{m}$ , with the objective of preventing adhesion of biofilm and microorganisms at the surface of the material.<sup>[1,6]</sup> The tongue may detect the Ra of the restoration if the Ra value is superior to 0.5  $\mu\text{m}$ .<sup>[1]</sup> On the present study, only the resin Z350 flow presented Ra value inferior to 0.2  $\mu\text{m}$  before the toothbrushing. After the toothbrushing, the resins Bulk Fill and Bulk Fill flow demonstrated Ra >0.5  $\mu\text{m}$ . It has been observed on the literature<sup>[9]</sup> that surface Ra depends on some factors and is influenced by the resinous matrix, type, size, shape, and distribution of filler particles. Besides that, this same study affirms that, in general, mechanical properties of bulk-fill composites are inferior to the properties of composites micro or nanohybrids.

In view of the above, clinical use of Bulk fill resins is still incipient and long-term studies are restricts yet, which means that, the confirmation of its efficacy and consequent indication as a substitute to conventional composite resins is still pendent.<sup>[7,23]</sup> Nevertheless, the number of *in vitro* studies related to the mechanical performance and light transmission in Bulk fill resins has been growing exponentially on the last few years.<sup>[23]</sup>

Both the changes noticed on the surface and the mass loss observed on the specimens have been reported on the literature.<sup>[11]</sup> Concerning to the mass loss, on this study there was not observed significant statistical differences. Despite this, the group BF was the one that presented greater mass variation. This finding may be related directly to the properties of each material, as mentioned above.

The study of Kanter *et al.*<sup>[24]</sup> demonstrated that the composites with more predisposition of losing weight, were the ones that showed greater increasing of the surface Ra. On this present study, this fact may be observed by analyzing the behavior of the group BF, which presented greater weight variation (despite it did not find any statistical difference), as well as greater increasing on the surface Ra, corroborating with the study mentioned above. Regarding to weight loss, the biggest problem related to this finding would be the consequent increasing on surface Ra, that may point to staining on the restorations, biofilm accumulation, besides irritation on periodontal tissues. Compromising, therefore, the aesthetics and longevity of the restoration.<sup>[14]</sup>

Thus, it is possible to justify the alterations observed on this present study— both related to the mass loss and to the increasing on the surface Ra and to the changes on the surface after the process of simulated toothbrushing. Although, it must be investigated the presence or not of alterations that may occur from the utilization of different dentifrices, as a protocol that be able to soften the wear of the restoration surface over time, with the aim to promote greater longevity and clinical success.

## CONCLUSIONS

From the results of the present study, it can be concluded that:

- All materials demonstrated changes on the deposition of the particles after simulated toothbrushing. The group ZF was the one that presented the smoother surface
- All materials showed increase on the surface Ra after simulated toothbrushing. The group BF presented the rougher surface
- There was no significant statistical difference regarding to weight variation in all materials.

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## Conflicts of interest

There are no conflicts of interest.

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