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Topography and surface roughness of fluid resins used as bioprotectors of mini-implants

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Abstract

Objective: The focus of this study was to test the hypothesis that there is difference between the surface roughness and topography of flow resins used as bioprotective materials of orthodontic mini-implants. Thirty test specimens (5 mm × 3 mm) of flow resins were used, divided into 3 groups (n = 10): Group W (Wave), Group TC (Top Comfort) and Group F (Filtek Z350 XT). Topographic analysis was performed by scanning electron microscopy (SEM) and surface roughness measurement by Atomic Force Microscopy (AFM). One-way analysis of variance ANOVA followed by the Tukey post hoc test were used for statistical evaluation ($p < .05$). By SEM, Group W presented a surface that was not very homogeneous with inorganic particles of up to 5 μm; in a similar manner and with a larger number of particles, Group TC was shown to have particles close to 3 μm in size. Whereas, Group F presented a more homogeneous and regular surface with few inorganic particles of 1 μm. AFM demonstrated that there was a significantly higher degree of surface roughness in Group W, which showed statistically significant difference from Group F ($p = .007$), and no significant difference between Group TC and the other groups ($p > .05$). The hypothesis was partially accepted; it could be affirmed that the flow resin Filtek Z350 presented a lower degree of surface roughness, and had smaller and more uniformly distributed inorganic particles when compared with the Wave and Top Comfort resins.

Keywords: Resins; Surface roughness; Topography

Background

The majority of resins used in orthodontics [1-4] have a smaller quantity of load than the traditional resin composite [5-9]. Recently, fluid dental resins have been used in orthodontics, with the purpose of providing bioprotection over mini-implants to diminish the areas of traumas on the adjacent gingival tissues [10]. There resins present a high level of fluidity and low modulus of elasticity [11], which, theoretically, dissipates the stress generated by thermal and masticatory tensions to a better extent [12]. In spite of the difficulty of handling them, due to their viscosity, they may be applied as a coating material in sites that are difficult to access, because of their fluidity [13,14].

On the other hand, the question has arisen about whether the topography and surface roughness of these resins could favor the retention of food and pathogenic bacteria [10], because of the heterogeneous nature of their resin components, such as the type, size, shape and composition of the load particles, quality and quantity of the organic component, type of bond and polymerization system [11,15,16] which may also generate or aggravate a peri-implant [10] inflammation [17].

Apart from the physical properties of the material, regular tooth brushing may increase the roughness on the surface of these fluid resins, making them more propense to biofilm accumulation [18] and gingival tissue inflammation [11,19]. Taken in conjunction, the physical and mechanical [11,15] properties of flow resins are aligned with the notion that the proximity of mini-implants [10] to the gingiva and oral tissues, make the surface topography and roughness important items to consider in the selection of these resins. Therefore, the focus of this study was to test the hypothesis that there are differences between the surface roughness and topography of flow resins used as bioprotective materials of orthodontic mini-implants.

Methods

Three flow resins were evaluated with regard to surface topography and roughness, and were divided into 3 groups: Group W (Wave), Group TC (Top Comfort) and Group F (Filtek Z350 XT) (Table 1). Thirty specimens (n = 10 for each group) were fabricated using silicone molds measuring 5 mm in diameter and 3 mm high. The material was inserted directly into the mold with the aid of the specific applicators of each resin, as indicated by the manufacturers, thus preventing bubble formation. The specimen surfaces were covered with glass slides under slight finger pressure, in order to flatten the surface of the material on both sides.

All the materials were light polymerized for 40 seconds on each side by a single operator, using a LED appliance (Radii, SDI, Baywater, Victoria, Australia) fixed on a rod to guarantee that the distance between the specimens remained constant, using a light intensity of 1000 mw/cm^2, regularly calibrated with a radiometer (Model 100, Demetron Research Corporation, Danbury, CT, USA).

Surface topography

Fifteen flow resin specimens (n = 5 per group) were evaluated with regard to surface topography, by scanning electron microscopy (SEM) (JSM-6360LV, Jeol, Tokyo, Japan), at different magnifications (10–1000 times) using the same tension of 20 kV in an acquisition time of 100 s for qualitative evaluation of the micromorphological characteristics of the flow resins. Five random fields were captured by SEM in each specimen at magnification of 1000 times in all groups evaluated for the surface microanalysis, as regards the presence and size of inorganic load particles of the resins.

Table 1 Composition of the tested resins

Groups	Resins	Composition	Manufacturer	Lot
F	Filtek Z350 XT	35% by weight of BisGMA, TEGMA, ytterbium fluoride, dimethacrylate-functionalized polymer and 65% by weight of ceramic inorganic particles and silane-treated silica and titanium dioxide.	3 M/Espe, St. Paul, MN, USA	N376841
TC	Top comfort	60% by weight of methacrylate monomers (BisGMA, UDMA and TEGMA), stabilizer, camphorquinone, co-initiator, pigments and 40% by weight of boron-aluminum-silicate glass inorganic particles and nanoparticulate silica.	FGM, Joinville, SC, Brasil	040112
W	Wave	35% by weight multifunctional methacrylate ester and 65% by weight of inorganic particles.	SDI, Bayswater, Vict, Australia	110401 N

Surface roughness

Fifteen flow resin specimens (n = 5 per group) were evaluated with regard to mean roughness (Ra) which was randomly determined three times for each specimen. The Ra represents the arithmetic mean of the absolute values of the digitized surface. Three readouts in distinct areas of each specimen were taken by atomic force microscope (AFM; SPM-9600, Shimadzu, Kyoto, Japan) from each measurement of 1.5 mm of length on the surface. The specimens were fixed to the microscope on a metal support using adhesive tape. The surface morphology of the specimens was probed in "Contact Mode". The images were obtained with standard geometry from a silicon nitride micro-cantilever (radius of curvature < 10 nm) (OMCL-TR, Olympus, Tokyo, Japan) and proved with an elastic constant of 0.15 N/m and resonant frequency of 24 KHz. Images measuring 30 μm × 30 μm, with a resolution of 512 × 512 pixels and point of operation of 1.5 V were collected at a very low rate of digitization in order to obtain details about the structure of the material surfaces, to prevent damaging the tip. By means of Atomic Force Microscopy, quantitative and qualitative surface roughness data were obtained with precise details at a nanometric resolution [20].

Statistical analysis was performed with the statistical program BioEstat (version 5.0, Belém-PA, Brazil). The statistical method was chosen based on the verification of normal distribution and equality of variance evaluated by the Kolmogorov- Smirnov and Levene tests, respectively. One-way analysis of variance ANOVA followed by the Tukey multiple comparisons post hoc test were used, with confidence at a level of 0.05 of statistical significance.

Results

Group W presented an irregular and not very homogeneous surface with inorganic particles of up to 5 μm in size (Figure 1A). A similar aspect could be noted in Group TC which, in spite of the larger number of particles observed, these did not exceed 3 μm in size (Figure 1B). Contrary to Groups W and TC, Group F presented a significantly more regular and homogeneous surface with few visible inorganic particles, with a maximum size of 1 μm (Figure 1C).

The surface roughness shown by Atomic Force Microscopy was of a significantly higher degree in Group W (Figure 2A), which showed statistically significant difference from Group F ($p = .007$) (Figure 2B), and there was no significant difference between Group TC (Figure 2C) and the other groups evaluated (Table 2).

Discussion

The quantity of organic and inorganic particles in flow resins has a direct influence on their physical properties [11]. A resin that has a quantity larger than 80% of the inorganic phase, is more susceptible to compromise of its mechanical and physical properties, depending on the shape, size, chemical composition and distribution of the particles, making the material friable and giving it a rough surface [9], which potentiates biofilm retention [18,19,21] and consequently gingival inflammation [7,17].

In this study, the flow resin Wave demonstrated a more irregular and hardly homogeneous surface with inorganic particles of up to 5 μm in size, which could accentuate the accumulation of microorganisms [18,21] and the roughness could be potentiated by the surface abrasion resulting from tooth brushing [22] and exposure of the inorganic

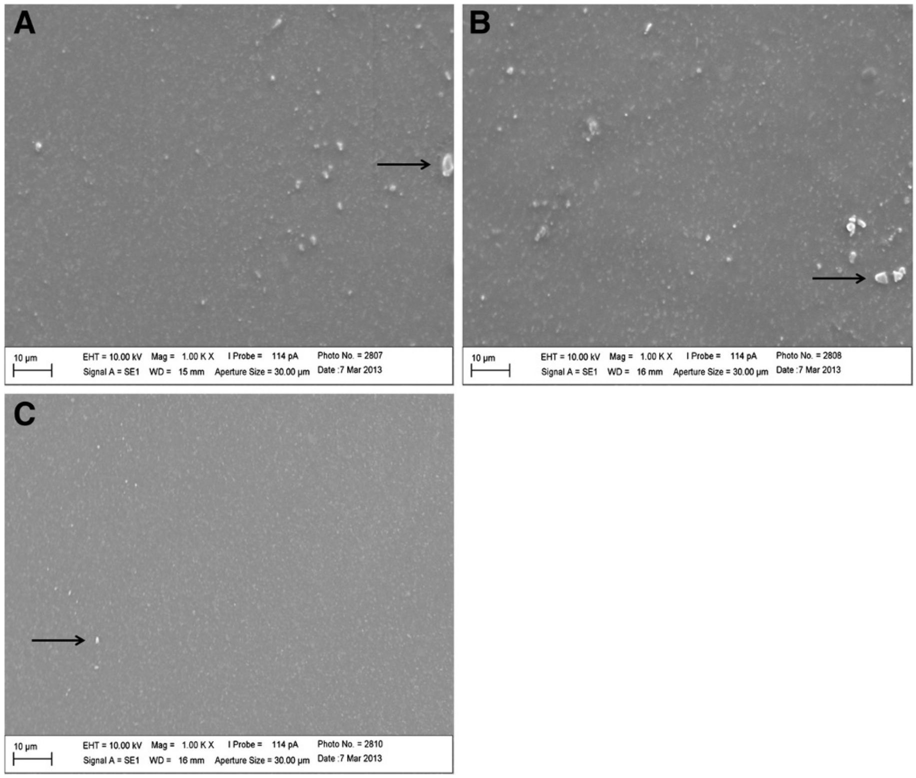

Figure 1 Representative images of surface topography of the resins in scanning electron microscopy (SEM). A) SEM image of the Wave resin showing surface with inorganic particle of 5 µm; **B)** SEM image of the Top Comfort resin showing surface with inorganic particle of 3 µm; **C)** SEM image of the Filtek Z350 resin showing smooth surface with inorganic particles less than 1 µm.

particles over the course of time [11]. The surface roughness of the flow resin Filtek Z350 presented the best results, with inorganic particles smaller than 1 µm in size and a more homogeneous surface. These surface alterations are mainly related to the size and polishing of the inorganic particles of silica in the composition of these resins [11,17,23].

The resin Filtek Z350 has 65% of inorganic particles by weight, and so does the flow resin Wave, however, when observing the size of these inorganic particles by scanning electron microscopy, it was observed that the inorganic particles in resin Filtek Z350 were significantly smaller and more regular in comparison with those of resin Wave. The distribution of the pre-cured silica particles more uniformly distributed within the organic matrix of resin Filtek Z350 may help to reduce the quantity of organic matrix exposed to abrasion [11] during the time of clinical use.

On the other hand, the resin Top Comfort with 40% of inorganic particles by weight, demonstrated particles of an intermediate size (3 µm) among the resins evaluated, although the particles found were smaller than those of resin Wave, the high quantity of organic component of the resin Top Comfort (60%) makes it more propense to clinical wear when compared with the other resins tested. The size of inorganic particles as well as the quantity of organic load may influence the roughness [11,13] which, according to some authors [11] even a less rough surface would not be free of abrasion, and wear of the organic fraction by regular brushing could be potentiated by substances such as mouthwashes [17] and bleaching agents [24,25], due to a probable elution of

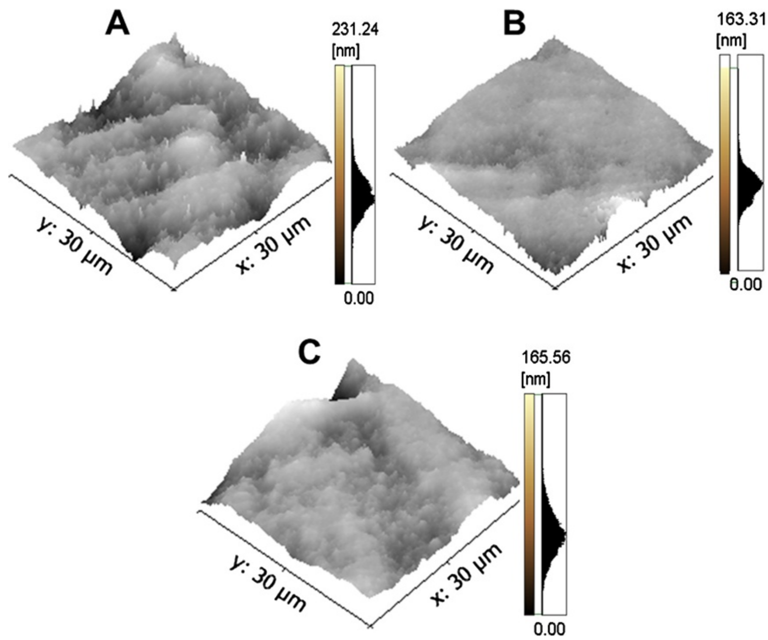

Figure 2 Representative images of surface roughness in Atomic Force Microscopy. A) Group W, **B)** Group F, and **C)** Group TC, showing the highest to the lowest roughness.

non-reacted components, such as residual monomers and the degrading effect on the polymeric chains [17]. This leads to the creation of spaces that vary according to the size, quantity of inorganic particles and inter-particle space [13], thus increasing the surface roughness [11,18].

Fluid resins generally present an inorganic particle size ranging from 0.04-4 μm, so that the higher the percentage of the inorganic component, the greater the resistance to wear [11]. Although no statistical difference was detected between the resin Top Comfort and the other resins, a greater loss of mass appears to be probable of occurring over the time of clinical use, due to the lower concentration of inorganic particle by weight, and higher percentage of organic matrix in comparison with the resins Filtek Z350 and Wave.

The consequence of the difference in surface roughness observed in resin Wave may be the accumulation of biofilm [18,19] and debris, in addition to gingival irritation [10] and pigmentation [19,23], and the same applies to the resin Top Comfort which, due to its large quantity of organic phase, would appear to have a potential for increase in roughness in the long term in the oral environment. Clinical studies [26,27] about resin composites have helped one to understand their behavior in the oral medium, in the

Table 2 Mean surface roughness (RA) of the flow resins

Groups	Mean roughness (nm)	DP*
W	17.67^{A}	3.45
TC	13.58^{AB}	2.36
F	9.83^{B}	1.16
P valor†	0.007	———

*SD = Standard deviation. †P value: One-way ANOVA followed by Tukey multiple-comparison post-hoc test. Different letters express significant difference between groups (p < .05). Letters A, B in the same group indicates no statistical difference with the other groups. (nm) = Nanometre.

same way that clinical trials in orthodontic patients about fluid resins may help one to define the clinical applicability of fluid resins for use as bioprotective materials.

Conclusions

The hypothesis was partially accepted; it could be affirmed that the flow resin Filtek Z350 presented a lower degree of surface roughness, and had smaller and more uniformly distributed inorganic particles when compared with the Wave and Top Comfort resins, which presented a similar roughness, without significant difference between them.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

MFLM prepared the samples and wrote the paper. MMP planned the project and methodology and did the literature review. BASGL and TAP supervised the literature review and performed the topography and surface roughness test. HLC and FGC guided the conception idea, supervised the practical phase. RLS got the idea, the project planning, the methodology, the statistical analysis, and raised funds to do the study. All authors read and approved the final manuscript.

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