Impact of filler size and distribution on roughness and wear of composite resin after simulated toothbrushing

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ABSTRACT

Objectives: Nanofilled composite resins are claimed to provide superior mechanical properties compared with microhybrid resins. Thus, the aim of this study was to compare nanofilled with microhybrid composite resins. The null hypothesis was that the size and the distribution of fillers do not influence the mechanical properties of surface roughness and wear after simulated toothbrushing test. Material and methods: Ten rectangular specimens (15 mm x 5 mm x 4 mm) of Filtek Z250 (FZ2), Admira (A), TPH3 (T),Esthet-X (EX), Estelite Sigma (ES), Concept Advanced (C), Grandio (G) and Filtek Z350 (F) were prepared according to manufacturer's instructions. Half of each top surface was protected with nail polish as control surface (not brushed) while the other half was assessed with five random readings using a roughness tester (Ra). Following, the specimens were abraded by simulated toothbrushing with soft toothbrushes and slurry comprised of 2:1 water and dentifrice (w/w). 100,000 strokes were performed and the brushed surfaces were re-analyzed. Nail polish layers were removed from the specimens so that the roughness (Ra) and the wear could be assessed with three random readings (μm). Data were analyzed by ANOVA and Tukey's multiple-comparison test (α=0.05). Results: Overall outcomes indicated that composite resins showed a significant increase in roughness after simulated toothbrushing, except for Grandio, which presented a smoother surface. Generally, wear of nanofilled resins was significantly lower compared with microhybrid resins. Conclusions: As restorative materials suffer alterations under mechanical challenges, such as toothbrushing, the use of nanofilled materials seem to be more resistant than microhybrid composite resins, being less prone to be rougher and worn.

Key words: Composite resins. Toothbrushing. Roughness. Wear.

INTRODUCTION

Material properties and technical approaches are two essential factors that have developed together over time in order to allow for a better clinical performance of composite resin restorations[2,10,14,18].

Composite resins are so far the most esthetic restorative material applied in direct restorations[2,14,18]. Filler and organic matrix have been modified in an attempt to offer satisfactory mechanical and esthetic characteristics, thus allowing its indication to posterior teeth[2,7,10,11,12,14,18,20-22]. In this sense, nanotechnology has been applied to Dentistry, permitting the incorporation of a larger amount of small-sized filler particles in a more homogeneous

distribution into organic matrix[5,6,15].

The decreasing filler size over the years resulted in some changes in commercial composites, resulting in different implications to their adequate clinical use and perspectives of success. Since the introduction of nanotechnology, these resins (0.1-100 μm) are classified as Nanohybrid, NanoMicrohybrid, Microybrid, Microfilled[10,19]. This classification varies extensively according to manufacturers and cannot be precisely identified[5,6,10,15]. This scenario pointed out to the need for investigating optical and mechanical properties of this new generation of composite resins. As fillers play a major role to reflect the irradiated light, enhanced esthetic properties were previously verified[13,15]. A perspective of improved material is also expected as high mechanical properties are attributed to filler load[23]. However, it should be highlighted that the particles are not arranged in the same pattern to all nanotechnology materials. There are composites with nanofillers, nanoclusters and/or microhybrid particles that are combined in different structures[5,6,10,15]. Additionally, a larger surface area of particles with reduced filler size result in a material more prone to water uptake, which can affect negatively its mechanical properties by degradation[8-10]. It is believed that as silane-based fillers are susceptible to hydrolytic degradation, it may affect adversely their dynamic mechanical properties over time[9,22].

Surface roughness and wear tests after simulated toothbrushing have been indicated to assess the mechanical features of restorative materials[3,4,8,20,25]. Simulated toothbrushing can intentionally provoke a stress in the organic matrix, fillers and their interfaces, and adhere to an assessment of their resistance properties[16].

As fillers are incorporated into the organic matrix by a chemical treatment of their surfaces, this interface can be stressed and be loosened from the matrix in different patterns[17]. Through a three-body abrasive action, toothbrushing provokes a mechanical challenge. In consequence, these particles can be loosed, fractured or the organic matrix can be removed, exposing the particles. Thus, roughness and wear readings can establish a comparison of the performance of these materials.

The aim of this study was to compare mechanical performance of nanotechnology high-density composite resins, according to their dimensions and distribution. The null hypothesis is that there is no difference of their performance on surface roughness and wear assessments after simulated toothbrushing testing.

MATERIAL AND METHODS

Experimental design

This *in vitro* study was performed involving two factors: material (in eight levels) and time (in two levels). The quantitative response variables were the surface roughness and wear analyzed by profilometry (μm). Information about materials under investigation is presented in Figure 1.

Ten specimens of each material were prepared using a previously lubricated steel stainless mold of 15 mm length x 4 mm in width x 5 mm in height placed over a glass slab and polyester strip (TDV Dental LTDA, Pomerode, SC, Brazil). Four individual increments were inserted which were light-cured according to manufacturers' recommendations, using a halogen lamp VIP (VIP, Bisco Inc, Schaumburg, IL, USA), with irradiance of 600 mW/cm^2, as measured with a curing radiometer (Curing Radiometer - Model 100P/N-150503, Demetron Research Corp., Danbury, CT, USA). The final increment was pressed with a polyester strip (TDV Dental LTDA, Pomerode, SC, Brazil) and glass slab under a constant axial load of 500 g for 30 s. Regardless of the recommended time, the last increment of all composite resins was light-cured for 40 s to standardize the surface. The specimens were removed from the molds and the excess was cut off with a #12 Bard-Parker scalpel blade.

Thereafter, the specimens were subjected to mechanical polishing in a metallographic polishing machine (Arotec - APL 4, Arotec SA Ind. Com., Cotia, SP, Brazil) using the sequence of #600-, #800- and #1200-grit silicon-carbide abrasive papers under water-cooling. Polishing was performed under a load of 172 g for 20 s for each granulation. All specimens were ultrasonically cleaned in deionized water for 10 min and were identified and stored at 37°C for 1 week.

In order to allow wear analysis, each specimen had half of its surface protected with two layers of nail polish that served as control (with no abrasion).

Baseline surface roughness of the specimens was analyzed by a profilometer (Hommel Tester T1000, Hommelwerke GmbH, Villingen-Schwenningen, Germany) accurate to 0.01 μm and was expressed in μm as a Ra value. Five records of each specimen were randomly assessed. To record roughness measurements of the surfaces a device containing a diamond needle affixed to the profilometer was used. The average of five randomized transversal readings was established as the baseline roughness value. Ra range was previously adjusted at 0.01 to 0.8 μm at a cut-off of 0.25 mm. Readings were obtained from 4.8-mm-long measurements.

A specially designed toothbrushing machine was used for the test. It allowed controlled performance using soft nylon bristle toothbrush heads (Colgate Classic, Colgate-Palmolive Ind. Com. Ltda, São Paulo, SP, Brazil) under a load of 300 g and temperature of 37°C. Slurry was prepared according to ISO specification #14569-1, mixing 2:1 of deionized water and dentifrice (Colgate Total 12, Colgate-Palmolive Ind. Com. Ltda, São Paulo, SP, Brazil) and 0.4 mL amount was injected periodically to renew for fresh slurry. For each specimen, a total of 100,000 strokes were performed and toothbrushes were replaced after 50,000 strokes. At the end of the test, each specimen was rinsed under running water and cleaning was completed by sonicating in deionized water for 10 min.

Final roughness was analyzed in the same way as described for baseline. Differences between initial and final readings were registered. For wear assessment, the same profilometer was used; allowing a needle to run from the protected half (control) to the abraded half. Parameters were adjusted to tolerances from 0 to 40 μm, length of assessment at 4.8 mm and cut off of 0.25 mm. The mean values among three readings were registered for each specimen.

Random samples of each tested groups, before and after toothbrushing, were selected for microscopic examination to illustrate possible events. These specimens were prepared and mounted on metal stubs, sputter coated with

Material	Manufacturer	Classification	Monomer/Filler	% w/v
Filtek Z250	3M ESPE, St Paul, MN, USA	Microhybrid	Monomers: Bis-GMA, UDMA, Bis-EMA Filler: Zircon and SiO_2 - 0.6 μm (0.01 - 3.5 μm)	84.5/60
Admira	Voco, GmbH, Cuxhaven, Germany	Microhybrid	Monomers: Ormocer, Additive aliphatic and aromatic dimethacrylate Filler: Glass ceramic SiO_2 - (mean of 0.7 μm)	78/56
TPH3	Dentsply, York, PA, USA	Nanohybrid	Monomers: Bis-GMA; BisEMA Filler: Barium aluminium borosilicate glass, Fluoro-aluminium borosilicate glass, Silica - (0.02 - 1 μm)	75/*
Estelite Sigma	Tokuyama Dental Corporation, Japan	NanoMicrohybrid	Monomers: Bis-GMA/ TEGDMA Filler: Spherical silica/zirconia submicron filler - 0.2 μm (0.1 μm - 0.3 μm)	82/71
Esthet-X	Dentsply, York, PA ,USA	Microhybrid	Monomers: Bis-GMA, BisEMA, TEGDMA Filler: Silanized Fluoro-aluminium borosilicate glass, silanized barium (1 μm) and colloidal silica (0.04 μm)	77/60
Concept Advanced	Vigodent S/A Produtos e Comércio, Rio de Janeiro, RJ, Brazil	Nanofilled	Monomers: Bis-GMA/UDMA Filler: Alluminium and barium silicate - 0.04mm (0.001 - 2 μm)	77.5/*
Grandio	Voco, GmbH, Cuxhaven, Germany	Nano-hybrid	Monomers: Bis-GMA, TEGMA Filler: glass ceramic filler (1 μm) and SiO_2 (20 - 60 nm)	87/71.4
Filtek Z350	3M ESPE, St Paul, MN, USA	Nanofilled	Monomers: Bis-GMA, UDMA, TEGDMA, Bis-EMA Filler: aggregated zirconia (0.6 - 1.4 μm) and SiO_2 (20 nm)	78.5/59.5

All informations were supplied by the manufacturers

*Not informed by manufactures

Bis-GMA=bisphenol-A-glycidyl methacrylate; Bis-EMA=bisphenol-A-ethoxylate glycidyl methacrylate; Bis-PMA=bisphenol-A-polyethylene glycoldiether dimethacrylate; TEGDMA=triethylene glycol dimethacrylate; UDMA=urethane dimethacrylate

Figure 1- Information of tested composite resins

gold, and examined under a scanning electron microscope (JSM T220A, JEOL Ltd., Peabody, MA, USA) at 500x magnification.

The assumptions of equality of variances and normal distribution of errors were checked for the tested response variables. Since the assumptions were satisfied, data were subjected to one-way ANOVA and Tukey's *post-hoc* test for the comparison of initial and final roughness and wear among the materials. Paired t-test was applied for roughness analysis considering a two-time evaluation ($p<0.05$).

RESULTS

Comparative roughness assessments and wear values after simulated toothbrushing are presented in Table 1. For all composite resins, initial and final roughness was statistically different ($p<0.05$).

Comparison among the tested materials regarding their initial roughness, showed that the nanofilled resins, Filtek Z350 and Concept Advanced, as well as the microhybrid resins, Admira and Estelite Sigma, presented smoother surfaces while Filtek Z250, TPH3, and Esthet X presented rougher surfaces.

According to the final condition, roughness outcomes revealed a great variability of performance after simulated toothbrushing. Admira, Grandio, Filtek Z350 and TPH3 were less rougher than the other composite resins. Concept and Esthet X were more susceptible to abrasion, consequently, presented significantly higher roughness. Intermediate values were found for Filtek Z250 and Estelite Sigma. According to the presented results, the qualitative analysis of the SEM micrographs of the resins, after the abrasion test, showed more polished surface of the nanofilled and the nanohybrid resins than the microhybrid resins, as seen in Figure 2.

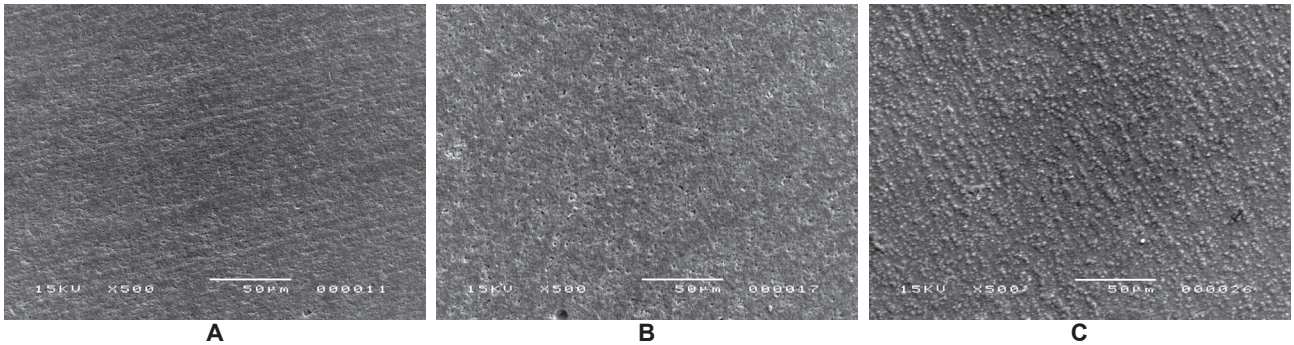
Comparing each system before and after toothbrushing simulation, except for Grandio, all materials became significantly rougher than their initial condition ($p<0.05$).

The wear assessment values revealed that Admira, Grandio and Filtek Z350 were less susceptible to wear after toothbrushing simulation, followed by TPH3, Estelite Sigma and Esthet X. Concept and Filtek Z250 presented the least resistance to wear ($p<0.05$).

Table 1- Mean and standard deviation of initial surface roughness (Ra), final surface roughness (Ra) and wear after simulated toothbrushing (μm)

Material	Initial roughess	Final roughness	Wear
FZ2	0.08 ± 0.01^{Ac}	0.19 ± 0.04^{Bbc}	14.6 ± 4.39^{d}
A	0.05 ± 0.00^{Aa}	0.06 ± 0.02^{Ba}	3.17 ± 1.16^{a}
T	0.08 ± 0.01^{Ac}	0.18 ± 0.10^{Bab}	8.02 ± 2.51^{bc}
ES	0.05 ± 0.01^{Aa}	0.30 ± 0.08^{Bbc}	11.75 ± 3.32^{cd}
EX	0.06 ± 0.01^{Ab}	0.47 ± 0.15^{Bd}	12.61 ± 4.26^{cd}
C	0.05 ± 0.01^{Aab}	0.51 ± 0.17^{Bd}	13.26 ± 5.29^{d}
G	0.08 ± 0.01^{Ac}	0.07 ± 0.01^{Ba}	4.27 ± 1.80^{ab}
FZ3	0.04 ± 0.06^{Aa}	0.13 ± 0.05^{Bab}	5.58 ± 1.46^{ab}

Different capital letters indicate differences between columns
Different lower case letters indicate differences between rows



A B C

Figure 2- Qualitative analyzis of different composite resins after abrasion test. A - Nanofilled (Esthet X); B – Nanohybrid (TPH3); C – Microhybrid (Z250)

DISCUSSION

Enamel and dentin are directly affected by caries disease. When these tissues are compromised, teeth lose the ability to absorb the load from mechanical impact. Dentin presents a mechanical property from a complex arrangement of collagen type-I-fibrils reinforced with a nanocrystalline apatite mineral in the extra and intrafibrilar spaces[1]. However, when caries affects dentin, it results in a disorganized structure. Thus, when this natural complex is changed, the restorative material needs to present properties that are able to recover it an appropriate manner. In order to reach a satisfactory clinical performance, the composite resin is indicated as a hybrid material composed mainly by fillers and organic matrix. Thus, mechanical properties are of great interest to allow composite resins to be well indicated[10,19].

Filler particles play an important role in this mechanism. They are responsible for the strength of the material and also protect organic matrix from wear[8,17,25]. Nanotechnology provides incorporation of well-distributed and larger amount of fillers compared with other categories[5,6,15]. Consequently, a high mechanical resistance is expected. This is essential in posterior restorations[2,14,18].

Organic matrix is the second point of interest to be focused. There have been several investigations with the purpose to promote modifications to reach better properties[10]. This balance of organic matrix and fillers is responsible for the determination of long-term clinical use[2,14].

Therefore, the comparison of the performance of resin-based materials is an essential parameter to aid clinical indication. Roughness is well accepted as a comparative feature. Basically, it quantifies surface texture by means of randomized readings of the amplitudes in mm, established as Ra (arithmetical roughness)[3,8,20,25].

According to the results of the present study, simulated toothbrushing was a mechanical process able to modify the balance between organic matrix and filler since all composite resins showed rougher surface after the abrasion challenge as shown in Table 1.

Initial roughness is essential to establish a parameter of comparison. Filtek Z350 and Concept Advanced presented the smoothest surfaces. This was somehow expected as they are categorized as nanofilled resins. Nanofilled materials have the ability to provide more volume of filler in homogeneous distribution, which enables it to protect organic matrix wear[3,23].

Admira and Estelite were significantly rougher than the nanofilled composite resins. The possible explanation relies on the fact that Admira, even classified as a microhybrid composite, is composed differently than other tested resins as its monomers are based on Ormocer, which is considered a resistant organic matrix[10]. Long-term clinical studies have shown the superiority of this matrix regardless of the size of filler[2,14]. In occlusal stress-bearing cavities, the Ormocer-based composite materials tested performed comparably to conventional microhybrid Bis-GMA-based composites. This study reveals that this organic matrix itself is more resistant than conventional monomers. On the other hand, the manufacturer of Estelite classifies it as nanomicrohybrid material. Since the variability between nano-sized materials also includes their distribution, it might affect their performance in a not well-clarified manner. It requires more investigation.

Filtek Z250, TPH3 and Grandio were the materials that exhibited rougher initial values. Filtek Z250 is a microhybrid resin while the manufacturers classify TPH3 and Grandio as nanohybrid. Also, it is should be highlight that even Filtek Z250 and Esthet X are both microhybrid, they differed statistically from each other. Despite this same categorization, it might vary in different levels that are not possible to precise, as their manufacturers do not supply this information in details. In comparison with the other tested materials, the distribution of the particles in hybrid resins is less homogeneous, which allows this condition. Additionally, the range of fillers of nanohybrid resins is large.

When final roughness was analyzed, distinct performances were observed after simulated toothbrush testing. Except for Grandio, all resins became rougher after toothbrushing, but Admira, Grandio, Filtek Z350 and TPH3 were less prone to be rougher. Once again, the organic matrix composition of Admira seemed to determine a good performance. Filtek Z350 as nanofilled material attended the expectation of this technology, confirming previous results[9]. Likely, toothbrushing abrasion caused a polishing effect on the surface, allowing smother surface compared with other resins, even rougher than its initial condition. Concept and Esthet X were more susceptible to abrasion in terms of roughness as they presented the greatest values after the test. Concept is categorized as a nanofilled composite resin and its organic matrix is based on conventional BisGMA. Limited information is supplied by its manufacturer, and so, with the limitations of this study, we cannot confirm precisely the reason of this poor performance. Esthet X, on the other hand, as a microhybrid resin, was rougher as expected, compared with nanosized resins. Intermediate final roughness values were detected in Filtek Z250, TPH3 and Estelite Sigma. As these materials are classified as micro or nanohybrid materials, their

performances are similar according to a previous study, which stated that they are both resistant materials[10].

According to wear values, Admira was the least susceptible to wear after toothbrushing simulation. The specific performance of this Ormocer-based material seems to confirm the relevance of organic matrix as well as fillers.

Grandio and Filtek Z350 did not differ significantly from Admira. Once again, nanofilled materials also attest to the relevance of a combination of reduced size with homogeneous distribution of filler to reach satisfactory performance. In sequence, TPH3, Estelite Sigma and Esthet X showed moderate resistance to wear. Concept and Filtek Z250 were the resins that had the high level of wear. As also stated, the poor results and lack of information about the organic matrix composition of Concept makes this resin less reliable.

Manufacturers have produced composites with different filler sizes (ranging from 5 to 100 nm) and distributions, in order to enhance performance. The mechanical properties like high flexural strength, low abrasion, low polymerization shrinkage and resistance to fracture are attributed to the high-filler load of these materials because of the small size the fillers possess[21]. An explanation for the improvement of the wear resistance with the smaller particles is that the mean distance between neighboring particles was smaller than that with the coarsest filler particles[24]. This size and distribution is favorable to protect organic matrix against wear, resulting in greater durability. From the observations of Admira, it can be stated that the Ormocer-based composite is relevant to resist wearing and roughness changes, which is comparable to the performance of the nanofilled composite resins compared with the other tested materials.

CONCLUSIONS

With the limitations of this study, the null hypothesis is rejected. Comparison of different categories of direct composite resins revealed that all materials became rougher after simulated toothbrushing. Different levels of wear occurred according to filler size and distribution. In general, nanofilled systems and the Ormocer-based resin showed better performance than the microhybrid and conventional organic matrix composites. This comparison can be helpful to predict the performance of these materials under clinical service.

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