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Original Article

Influence of pH-cycling and abrasion wear on the mechanical properties of conventional and bulk fill resin composites

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Aim: The objective of this study was to evaluate the effect of abrasion wear on surface roughness and microhardness of different commercially available resin composites simulating pH-challenges of the oral cavity. **Methods:** Three resin composites (RC) were used in this study: one conventional: Z250; and two bulk fill resin composites (BRC): Tetric N-Ceram (TNC) and Sonic Fill (SF). The RC was inserted in a prefabricated mold (15mm wide x 4mm thickness) in two layers, or in a single layer for BRC. Thirty samples were prepared and surface roughness (Ra) and Knoop microhardness (KHN) test were performed at three different time-points of evaluation: baseline (24h after sample preparation); partial (after pH cycling); and final (after simulated toothbrushing procedure). Two samples of each group were selected after different treatments and analyzed descriptively on a scanning electron microscopy (SEM). Data from Ra and KHN were analyzed by two-way repeated-measures ANOVA and Bonferroni's *post-hoc* test with a significance level set at 5%. **Results:** Ra increased for all groups ($p<0.001$), at the final time-point, Z250 and TNC groups present the highest values. Oppositely, KHN decreased for all groups ($p<0.001$), Z250 group showed the highest KHN values for all time-points ($p<0.001$). The SEM imagens showed a regular surface for samples cycled and irregular with inorganic particles exposed for samples toothbrushed. **Conclusion:** pH-cycling and simulated toothbrushing affected the superficial properties (roughness and Knoop microhardness), as observed at SEM imagens, with irregular surface with inorganic particles exposure.

Keywords: Composite resins. Surface properties. Toothbrushing.

Introduction

Resin composites (RC) are the most useful material for direct restorations due to the esthetic properties and ease of handling, but also, because they do not require an invasive tooth preparation[1]. When this material is used, the restorative procedure is conducted thru the layering technique, where multiple 2 mm thick layers are inserted, and separately light cured until the tooth cavity is filled[1,2]. However, multiple exposures to photoactivation can lead to a high polymerization shrinkage stress, besides the demand of time of this sensitive technique[3]. In order to overcome the time-consuming incremental cavity filling technique, bulk-fill resin composites (BRC) have been developed[2,3].

BRC allow for a single layer of up to 4-5mm thickness due to different types of photoinitiators, and due to reinforcement by different sizes, shapes, and types of filler particles[3]. Recently, a 3-year clinical trial of posterior restorations showed similar performances between BRC with RC[4]. Also, Engelhardt et al.[5] reported that the abrasion resistance of bulk-fill composites was not superior to that of conventional composites. However, during the abrasion wear, the chemical challenges in the oral cavity must be considered, such as the composition of saliva and the low pH of the de/remineralization process, influencing the sorption and solubility of the material. The acid challenge promoted in the resin-based materials can be explained due to acid hydrolysis by the disruption of the polymer molecule, and dissolution or swelling of the resin composite matrix by sorption of liquids[6]. Besides that, smaller size and shape of filler particles contained in the inorganic matrix are able to control the wear performance of the RC[7].

The most important method of oral hygiene is brushing associated with a toothpaste. However, studies indicate that brushing movements with the abrasive components present in toothpastes can cause wear not only on the teeth, but also on restorative materials. Therefore, an in vitro brushing test was used to evaluate the wear and resistance of composite resins[8].

The abrasion resistance can be correlated with surface roughness, that is determined by the inorganic filler size[9]. The larger the size of fillers lost in the process of abrasion wear, the more the surface roughness increases[2]. Thus, Sonic Fill resin composite (SF- Kerr) was developed with different sizes of particles, and in order to optimize the insertion of the material, this BRC uses sonic energy[10]. However, this technology may alter the mechanical properties of the material and should be evaluated[10]. Also, surface roughness can be correlated with the degradation of the organic matrix[11]. The organic matrix of most dental composites presents the methacrylate-based monomer Bis-GMA (bisphenol-A diglycidyl dimethacrylate), characterized by its high molecular weight, along with low molecular weight diluents, usually ethylene glycol derivatives, such as triethylene glycol dimethacrylate (TEGDMA)[12].

However, Tetric N-Ceram Bulk fill (TNC – Ivoclar Vivadent) is composed by an Orcomer® hybrid polymer (organic polymer linked with the inorganic matrix), instead of being based on methacrylates[13]. Due to the many other monomers that

can be used in composite formulations, the mechanical properties of these materials can be expected to present variations[1,14-16]. Thereby, Knoop microhardness is a simple test that can provide the influence of different organic and inorganic matrix on the hardness and indirectly provide the degree of conversion/cure depth of the material[6,13,14].

Thus, considering that the inorganic particles are approximately 60-80% of the total volume of the material and the resin matrix can influence the mechanical properties, it is clinically relevant to investigate the effect of toothbrushing on the surface roughness and microhardness of different commercially available RBCs simulating the pH-challenge of the oral cavity[12,15,16]. The null hypotheses of this study were that: 1) surface roughness and microhardness would not be affected by pH-cycling and abrasion wear, and 2) surface roughness and Knoop microhardness would not be different between RC and BRC after pH-cycling and/or abrasion wear.

Materials and methods

Three resin composites were used in this study: one conventional (Z250: Filtek Z250 XT - 3M ESPE, St Paul, MN, USA) and two bulk fill resin composites (TNC: Tetric N-Ceram bulk fill - Ivoclar Vivadent AG, Schaan, Liechtenstein and SF: Sonic Fill – Kerr Corporation, Orange, CA, USA). Table 1 shows the manufactures specifications. The Surface Roughness and KHN of the materials were tested at three different time-points: baseline (after the specimen preparation), after the pH-cycling and after pH-cycling associated with abrasion wear.

Table 1. Manufactures specification of each resin composite.

RESIN COMPOSITE AND MANUFACTUR (#LOT NUMBER)	COMPOSITION	INCREMENTAL THICKNESS (mm)
Filtek Z250 XT 3M ESPE, St Paulo, Minnesota, USA (#761671)	**Matrix:** bis-GMA, UDMA, bis-EMA **Filler type:** Zirconia/silica without silane treatment **Filler loading (volume%):** 60%	2
Tetric N-Ceram Bulk Fill Ivoclar Vivadent, Bendererstrasse, Schaan, Germany (#W83652)	**Matrix:** bis-GMA, UDMA, bis-EMA **Filler type:** Barium aluminum silicate glass with two different mean particle sizes, an "Isofiller," ytterbium fluoride and spherical mixed oxide. **Filler loading (volume%):** 61%	4
SonicFill Bulk Fill Kerr Corporation, Orange, California, USA (#6617848)	**Matrix:** bis-GMA, EBADMA, TEGDMA **Filler type:** Silica, barium glass, ytterbium fluoride, mixed oxides. **Filler loading (volume%):** 81.35%	4

Abbreviations: bisphenol A-glycidyl methacrylate (bis-GMA); urethane dimethacrylate (UDMA); bisphenol A diglycidyl methacrylate ethoxylated (bis-EMA); ethoxylated bisphenol A dimethacrylate (EBADMA); triethylene glycol dimethacrylate; (TEGDMA).

Specimen preparation

Sixty samples were prepared according to the resin composite technique. Two layers with 2mm of RC-Z250 were insert in a prefabricated mold (15mm wide x 4mm thickness) and each layer was light-cured (wavelength of 1,200 mW / cm^2 - VALO,

Ultradent Product Inc., South Jordan, UT, USA) separately for 20s according to the manufacture's instruction. For the BRC, one single increment was inserted into the prefabricated mold and light cured for 20s. Afterwards, the specimens were polished under water irrigation using grit-sic papers (#400, 600 and 1,200-grift) and washed in an ultrasound bath (Marconi, Piracicaba, SP Brazil) for 5 minutes to remove possible abrasive granules. Finally, the samples were stored for 24h at 37°C in distilled water.

Measurement of surface roughness (Ra)

The surface roughness test (n=10) was performed at three different time-points of evaluation: baseline (24h after sample preparation); partial (after pH cycling); and final (after simulated toothbrushing procedure). For the surface roughness test, each sample was individually fixed with utility wax on an acrylic base and planned. The measuring tip of the rugosimeter (Surftest 211; Mitutoyo Corp., Tokyo, Japan) was positioned on the top surface of the sample and the Ra values (arithmetic mean of surface roughness) were measured using a cut-off of 0.25 mm at a speed of 0.05 mm/s. Three readings were taken at different positions after 120° rotation of the sample. Afterwards, the specimens were individually stored in 5 ml of deionized water, kept at 37° C in absolute humidity[9,12].

Measurement of Knoop microhardness (KHN)

As the roughness measurement, the Knoop microhardness test (n=10) was performed at three different time-points of evaluation: baseline, partial and final. For the KHN, the top of the specimens was positioned on a microdurometer (Shimadzu, Kyoto, Japan) and three indentations were performed (left, middle and right of the sample) with a load of 25 g for 5 s. Afterwards, the specimens were individually stored in 5 ml of deionized water, kept at 37° C in absolute humidity[13].

pH-cycling

For the pH-cycling, the groups were immersed in demineralizing solution for 6 hours, consisting of 2.0 mM calcium and 2.0 mM phosphate in a 75 mM acetate buffer solution pH 4.3 with 0.02% NaN3, followed by 18 hours immersed in 1.5 mM Ca; 0.9 mM PO4; 150 mM KCl in 20 mM Tris buffer pH 7.0 with 0.02% NaN3. The protocol was repeated during 10 days, and the solutions were monitored by a pH-meter and afterwards, the KHN and Ra were measured again as described[7,16].

Abrasion Procedure

The abrasion procedure was performed after the pH-cycling. The samples were brushed for 50,000 reciprocal strokes (corresponding to 4.5 years of in vivo toothbrushing) using a Colgate Classic soft bristle toothbrush, with a brushing speed of 2.5 cm/s. A toothpaste (Colgate Optic White, Colgate Palmolive Canada Inc) solution (50 g of toothpaste to 80 mL of deionized water) was used to brush the samples with a 180-g force as recommended by ISO[17]. After brushing, the samples were thoroughly washed and air dried. Finally, the KHN and Ra were measured again as described[14].

Scanning Electron Microscope

Two specimen samples of each group were select after different treatments (baseline, pH-cycling and pH-cycling associated with abrasion wear). The samples were sputter-coated with gold (MED 010, Balzers, Liechtenstein) and observed under a scanning electron microscope (SEM - JEOL-JSM, 6460LV, Tokyo, Japan) in a 1,000x magnification[12].

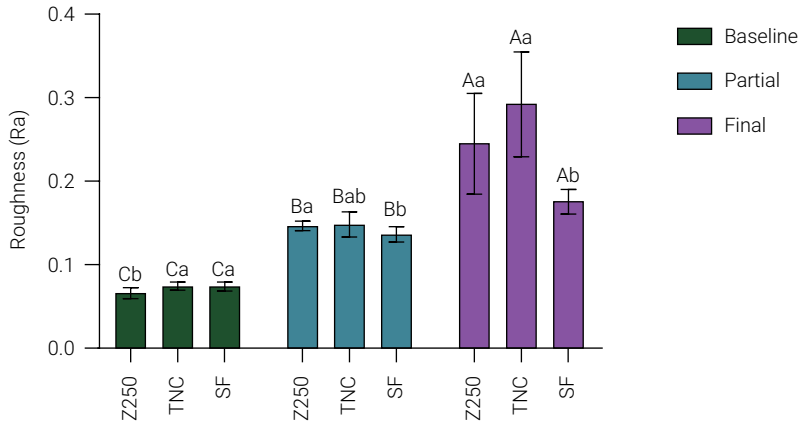
Statistical Analyzes

Data from Ra and KHN tests were evaluated for normal distribution (Shapiro-Wilk). Two-way repeated-measures ANOVA and Bonferroni's *post-hoc* test was used to compare the resin composites not cycled from those that were cycled, besides the different time-points of evaluation (baseline, partial and final). SPSS 21.0 (SPSS, Chicago, IL, USA) was used to perform the statistical analysis with a significance level set at 5%. SEM images were analyzed descriptively.

Results

Surface roughness

Figure 1 shows the mean (SD) values of Ra. For all groups, the Ra increased over the evaluations, with at final time results statistically different between baseline and partial times ($p < 0.001$). At baseline, TNC and SF presented the highest Ra values compared to Z250 ($p < 0.041$). However, at the partial time-point, Z250 was statistically different from SF ($p = 0.017$), and TNC was not statistically different ($p > 0.05$). And at the final time-point, Z250 and TNC presented the highest Ra values compared to SF ($p < 0.006$).

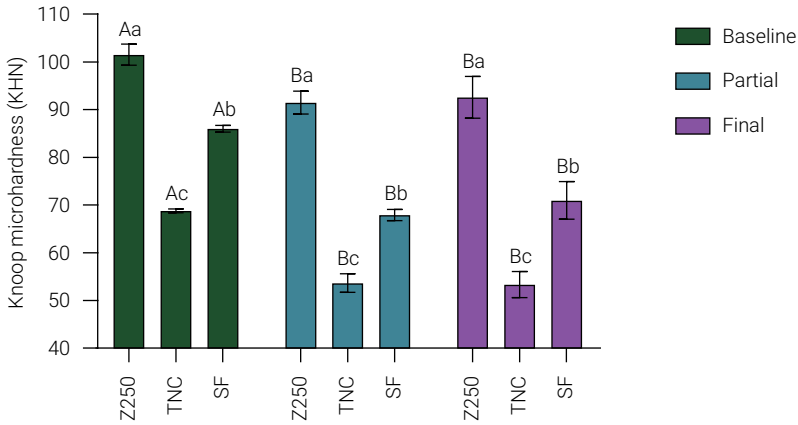


Legend: Mean values followed by distinct letters differ statistically at 5%, according to two-way repeated measures ANOVA and Bonferroni *post-hoc* test. Uppercase letters compare the times of evaluation (baseline, partial and final) within each resin composite. Lowercase letters compare the resin composite groups cycled and not cycled within each evaluation time-points.

Figure 1. Mean and standard deviation (SD) of baseline, partial and final time-points surface roughness (Ra) values.

Knoop Microhardness

Figure 2 shows the mean (SD) values of KHN. For all groups, the KHN decreased from baseline to partial and final times ($p < 0.001$), where the partial and final times were not statistically different ($p > 0.05$). For all time-points, Z250 presented the highest KHN values compared to TNC and SF ($p < 0.001$), as well when comparing SF to TNC ($p < 0.001$).



Legend: Mean values followed by distinct letters differ statistically at 5%, according to two-way repeated measures ANOVA and Bonferroni *post-hoc* test. Uppercase letters compare the times of evaluation (baseline, partial and final) within each resin composite. Lowercase letters compare the resin composite groups cycled and not cycled within each evaluation time-points.
Figure 2. Mean and standard deviation (SD) of baseline, partial and final Knoop microhardness (KHN) values.

Scanning Electron Microscope

SEM representative images of resin composite surfaces are shown in Figure 3. Accordingly, figures 3B, 3E and 3H that represent the samples cycled, show a regular surface with clean aspect in relation to the figures 3A, 3D and 3G, that represent the baseline samples. Besides that, the figures 3C, 3F and 3I present an irregular surface with inorganic particles exposed, caused by the toothbrushing action.

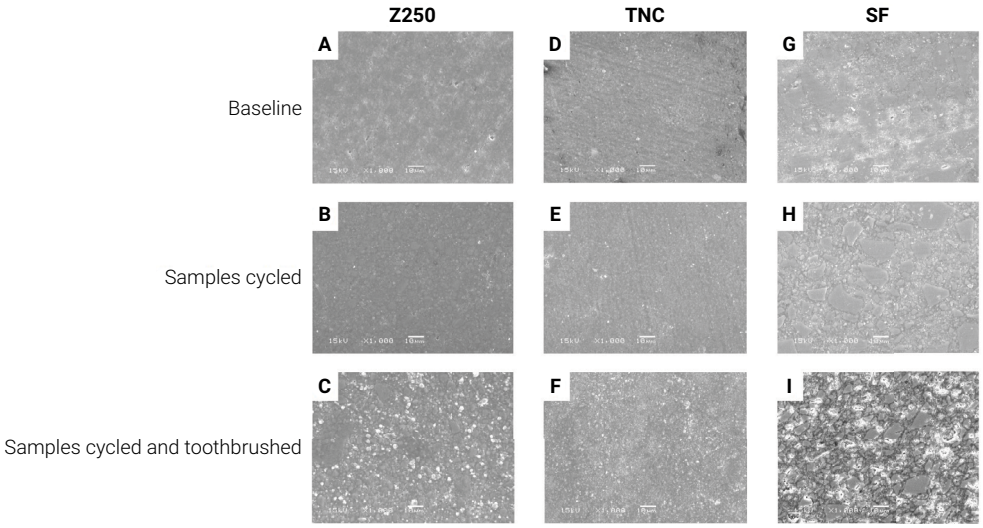


Figure 3. Representative images of the resin composites submitted to pH cycling and toothbrushing. A- Z250 sample at baseline; B- Z250 sample cycled; C- Z250 sample cycled and toothbrushed; D- TNC sample at baseline; E- TNC sample cycled; F- TNC sample cycled and toothbrushed; G- SF sample at baseline; H- SF sample cycled; I- SF sample cycled and toothbrushed.

Discussion

Resin composites have become clinician's choice for direct restorations, yet a variation of mechanical properties has been reported due to the different compositions[1,7,9,11,13,14]. Thus, it is important to understand some material characteristics that are essential for a successful long term restoration procedure, such as a smooth surface to reduce plaque retention and avoid recurrent caries and discoloration[18]. Furthermore, it must be considered that some other factors may influence the mechanical behavior of the resin composite.

Turssi et al.[19] showed higher surface roughness of resin composites subjected to pH cycling than those stored in artificial saliva and deionized water. Thus, surface roughness and microhardness after simulated de/remineralization process thru pH-cycling and toothbrush wear, is an important characteristic to consider when determining which resin composite can be indicated[2]. According to this study, similar results were found because the Ra increased over the evaluated time-points, with final time results statistically different than baseline and partial time-points, regardless the resin composite, therefore, the first null hypotheses that the surface roughness and microhardness would not be affect by the pH-cycling and abrasion wear cannot be accepted. This result can be correlated with the degradation of the resin matrix and formation of cavities on the composite surface due to acid attacks[16].

The degradation of the resin matrix can be associated with some specific organic monomers[20]. The bis-GMA monomer is presented in the composition of all groups, and this monomer has a high capacity of water sorption and consequently hydrolysis[2]. Nevertheless, the monomer TEGDMA can also play a role in surface roughness,

however, it is present only in the BRF-SF composition. Thus, due to the low molecular weight of TEGDMA and the higher water susceptibility compared to bis-GMA and bis-EMA, a higher Ra value for BRF-SF compared to the other groups was expected, however, this resin composite showed the lowest value at the final time-point. Therefore, it can be assumed that the concentration of the monomers and the interaction between the polymers during the carbon double bonds reaction can influence the final surface roughness[2,20]. However, the amount of each content is not reported by the manufactures due to patent protection, and it is a limitation to discuss these findings.

Yet, it is possible to associate the results with the filler volume presented in the composition. According to the manufacture's information, the BRC-TNC and RC-Z250 have approximately 60% of filler volume, consequently, this study showed, according to roughness results, that these groups did not statistically differ at all evaluation time-points, however, the BRC-SF that has 81.35% of filler volume in the composition, presented the lowest Ra values. Thus, it can be assumed that despites the type and amount of the monomers composition of the resin matrix, the quantity of inorganic particles may improve the wear behavior of the resin composites. However, the inorganic filler particles can vary from shape and size and this characteristic can influence the hardness of the material, therefore, the second null hypothesis needs to be rejected.

This study showed that the highest microhardness value was at the baseline time-point regardless of the resin composite. After being submitted to pH cycling and abrasion wear, the values decreased for all resin composites as well. The results can be correlated with the fact that the surface abrasion has removed the resin matrix in between the filler particles, leaving a particle-free resin layer that can be easily abraded and may initiate cracks, influencing the hardness values[12,19]. Also, according to several authors, large and irregular filler particles are the main reason for low microhardness values[12,18,19,21]. Figure 2 showed that after the samples were cycled and toothbrushed, the particles became more apparent in the resin matrix, and it can be noticed that RC-Z250 and BRC-TNC contained smaller and round-shaped particles compared to BRC-SF, thus, higher values of microhardness for these groups could be expected because it can be assumed that the combination of both sizes of particles can promote a great adaptation into the resin matrix[21,22]. However, for all times, RC-Z250 presented the highest KHN values compared to BRC-TNC and BRC-SF, and this results can be associated with the capacity of the degree of conversion of each resin composite evaluated.

Bulk-fill resin composites are usually more translucent, to enable light to pass through deeper layers (4 to 5mm), providing a more uniform monomer conversion[7,13]. A more translucent resin composite can be achieved through reduction in the filler content, small particles, or the interaction between the fillers of both sizes and organic matrix refractive indexes besides the initiator system[13,23]. However, as a disadvantage, the translucency of BRC can be observed in clinical situations, in which there is a necessity of a capping layer for both mechanical and esthetical properties[13,23,24]. Thus, despite the advantage of easy application of BRC, it is important to evaluate and understand the mechanical properties that could help in the selection of the material.

Conclusion

Considering the limitations of this *in vitro* study, both pH-cycling and simulated toothbrushing affected the superficial properties (increasing surface roughness and decreasing Knoop microhardness). pH cycling was deleterious to resin-based materials, and simulated toothbrushing promoted greater morphological changes, with irregular surface and inorganic particles exposure.

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Conflits of interest

The authors declare that they have no conflict of interest. The authors do not have any financial interest in the companies whose materials are included in this article.

Author contributions

Conceptualization, L.D.O.; methodology, L.D.O. and R.B.E.L.; formal analysis, R.B.E.L. and M.R.S.; investigation, L.D.O.; resources, L.R.M.M.; writing—original draft preparation, L.D.O.; writing—review and editing, R.B.E.L., M.R.S. and L.R.M.M.; supervision, L.R.M.M. All authors have read and agreed to the published version of the manuscript.

Data availability

Datasets related to this article will be available upon request to the corresponding author.

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