

# Effect of Chemical and Mechanical Degradation on Surface Roughness of Three Glass Ionomers and a Nanofilled Resin Composite

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## Clinical Relevance

The nanofilled glass ionomer provides intermediate resistance to chemical and mechanical degradation among the glass ionomer cements (conventional and resin-modified) and nanofilled composite.

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## SUMMARY

Nanofillers have been incorporated into glass ionomer (GI) restorative materials to improve their mechanical and surface properties. The aim of this present laboratory study was to compare the superficial roughness (*Ra*) of nanofilled GI (Ketac N100) with that of conventional GI (Fuji IX GP), resin-modified GI (Vitremer), and a nanofilled resin composite (Filtek Supreme) after pH cycling and toothbrush abrasion. Ten specimens of each material were made using Teflon molds, which were polished using aluminum-oxide abrasive disks. Three measurements of *Ra* were made of each specimen to serve as baseline values. The specimens were submitted to pH cycling for 10 days in a demineralization solution for six

hours (pH 4.3) and were then stored in remineralization solution for 18 hours (pH 7.0). *Ra* measurements were recorded after the pH cycling. Specimens were then submitted to toothbrush abrasion in a brushing machine with a 200g load for 30,000 cycles at 250 cycles/min. The *Ra* values were then recorded. The surface morphology of specimens from each group was analyzed using a scanning electron microscope. Data were analyzed by analysis of variance, Tukey, and *t*-tests. After toothbrushing, only Fuji IX GP ( $1.10 \pm 0.80$ ) showed *Ra* values that were statistically different from those of the other materials evaluated. Ketac N100 ( $0.68 \pm 0.16$ ) showed intermediate *Ra* values, but it did not differ statistically from the results associated with Vitremer ( $1.04 \pm 0.46$ ) and Filtek ( $0.30 \pm 0.15$ ). Ketac N100 showed intermediate values of superficial roughness among the conventional glass ionomer cement, resin-modified glass ionomer cements, and the nanofilled resin after chemical and mechanical degradation.

## INTRODUCTION

Glass ionomer (GI) restorative materials have been widely used in dentistry, mainly in Class V and primary tooth restorations.<sup>1,2</sup> Chemical adhesion to tooth structure, long-term fluoride release, ability to uptake fluoride, and low coefficients of thermal expansion are characteristics that make GI clinically attractive.<sup>3-5</sup>

The properties of GI have improved in recent years as the result of an increased percentage of filler particle loading, the incorporation of resin monomers into the cement, or both.<sup>6</sup> Resin-modified GI cements (RMGIs), one of the major categories of commercial GIs, were developed to partly overcome the problems associated with conventional GIs, such as poor handling characteristics, sensitivity to moisture during initial setting, and poor physico-mechanical strength.<sup>3,7,8</sup> Recently, a new category of GI restorative materials was introduced for the restoration of primary teeth and small cavities in permanent teeth: the nanofilled RMGIs (Ketac N100 or Ketac Nano Light-Curing; 3M ESPE, St Paul, MN, USA), a paste/paste material.<sup>9</sup> Its primary curing mechanism involves light activation, and no redox reactions or self-curing occur during setting. The major innovation of this material involves the incorporation of nanotechnology, which allows a highly packed filler composition ( $\sim 69\%$ ), of which approximately two-thirds are nanofillers.<sup>10</sup> The

nanofillers improve the mechanical strength and optical properties of the restorative material.<sup>11</sup>

The resistance of dental restorations to superficial degradation has an appreciable influence on their clinical performance.<sup>12</sup> In the oral cavity, the degradation of restorative materials can be caused by low pH (due to cariogenic biofilm and acidic drinks), water sorption, erosion, and wear.<sup>12-14</sup> These factors involve degradation of the resin matrix, filler, or matrix-filler interface on the surface of the materials, leading to increased surface roughness and wear rate.<sup>15,16</sup>

Laboratory studies assess the surface integrity of restorative materials using mechanical degradation tests, such as wear simulation and brushing abrasion, which evaluate the surface texture and wear/abrasion resistance of materials.<sup>17-20</sup> Corrosive wear or mechanical degradation results from the joint action of chemical and mechanical forces and is also associated with the mechanical removal of corroded layers that form on the surface of a material through a reaction with its environment.<sup>21</sup> However, it is also important to determine how the surface of restorative materials performs as a consequence of the chemical degradation that also occurs in the oral cavity. The dynamic variations in the oral pH, due to cariogenic challenges, can develop superficial degradation of restorative materials from the low pH.<sup>16,22</sup> Furthermore, some studies<sup>15,23,24</sup> have demonstrated that materials can be more susceptible to brushing abrasion at lower pH values. The acidic storage medium erodes the surface of conventional GI, for example, and causes hydrolysis and dissolution mainly in the polyalkenoate matrix.<sup>25-27</sup> This degradation has a detrimental and irreversible effect on the cement surface. As a clinical consequence, the low environmental pH, in association with toothbrushing abrasion, can result in a loss of contour and increasing surface roughness, which increase the rate of wear and can be responsible for bacterial biofilm and stain accumulation.<sup>28</sup>

Based on the fact that the incorporation of nanofillers in restorative materials can improve the polish and abrasion resistance of restorations,<sup>11</sup> it seems relevant to evaluate whether a nanofilled, resin-modified GI (Ketac N100, 3M ESPE) has lower superficial degradation after pH cycling and abrasion testing when compared with a conventional or resin-modified GI. Therefore, the null hypothesis tested in this current study was that the nanofilled RMGI subjected to a pH-cycling model (chemical degradation) prior to a three-body abrasion test (mechanical degradation) does not differ in the roughness (*Ra*) and

surface morphology to that of a conventional GI, RMGI, or nanofilled resin composite.

MATERIALS AND METHODS

Materials

Four direct restorative materials, including one conventional GI (Fuji IX GP; GC America Inc, Alsip, IL, USA), one RMGI (Vitremer; 3M ESPE), one nanofilled RMGI (Ketac N100; 3M ESPE), and one nanocomposite resin (Filtek Supreme; 3M ESPE) were evaluated in this current study. Brand names, manufacturers, basic compositions, mean filler size, and batch numbers are indicated in Table 1.

Specimen Preparation

Ten specimens of each restorative material (n=10) were fabricated using Teflon molds (4.0-mm diameter and 2.0 mm thick), according to the manufacturers' instructions. The GI restorative materials were mixed and placed in one increment into the mold. The nanocomposite was also placed into the mold in one increment. The surface of the restorative materials was covered with a polyester strip and a glass slab. A load of 500g was applied for 30 seconds to expel excess material from the matrix. Vitremer, Ketac N100, and Filtek Supreme specimens were polymerized with a light-curing unit (Elipar Trilight;

3M ESPE). The output of the QTH curing light used to polymerize specimens was found to be 690 mW/cm<sup>2</sup> determined with a curing light meter (Hilux Dental Curing Light Meter; Benliglu Dental Inc, Turkey). Fuji IX GP specimens were allowed to set for five minutes, according to the manufacturers' instructions. All specimens were stored in 100% relative humidity at 37°C for 24 hours.

Finishing and polishing were carried out using a sequence of medium, fine, and superfine aluminum-oxide abrasive disks (Sof-Lex Pop On; 3M Dental Products, St Paul, MN, USA). Each disc was applied in a single direction for 15 seconds using air-water spray by a single calibrated operator based on a previous pilot study,<sup>16</sup> which tested the direction and time of oxide abrasive disk application on the surface roughness of each material. A new disc was used for each specimen. After the polishing procedures were completed, the specimens were ultrasonically cleaned (Model T1440D; Odontobrás Ltda, Ribeirão Preto, SP, Brazil) in distilled deionized water for 10 minutes to remove polishing debris and stored in 100% relative humidity.

Surface Roughness Measurements

Before the pH-cycling regime and abrasion testing, the surface roughness of all specimens was analyzed

Table 1: Restorative Materials Evaluated in this Study			
Restorative Material (n=10)	Basic Composition	Mean Filler Size <sup>a</sup>	Batch No.
Fuji IX GP (GC America Inc, Alsip, IL, USA)	Powder: polyacrylic acid, aluminum fluorosilicate glass	4.4 µm	0706061/0706051
	Liquid: polyacrylic acid, tartaric acid and water		
Vitremer (3M ESPE Dental Products, St Paul, MN, USA)	Powder: fluoroaluminosilicate glass; redox system	3.0 µm	6LN/6FC
	Liquid: aqueous solution of a modified polyalkenoic acid, HEMA		
Ketac N100 (3M ESPE Dental Products)	Paste A: silane-treated glass; silane-treated ZrO2 silica; silane-treated silica; PEGDMA; HEMA; Bis-GMA; TEGDMA	1 µm (cluster) 5-25 nm (nanofiller) <3.0 µm (glass)	M3M3
	Paste B: silane-treated ceramic; silane-treated silica; water; HEMA; acrylic/itaconic acid copolymer		
Filtek Supreme (3M ESPE Dental Products)	Bis-GMA, Bis-EMA, UDMA, TEGDMA zirconia/silica cluster filler and a nonagglomerated silica filler	20–75 nm	7CJ
Abbreviations: Bis-EMA, ethoxylated bisphenol-A dimethacrylate; Bis-GMA, bisphenol glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.			
<sup>a</sup> According to the manufacturers' information.			

using a Surfcomer SE1700 surface roughness-measuring instrument (Kosaka Corp, Tokyo, Japan), equipped with a 2- $\mu\text{m}$ -radius diamond needle, which traversed the surface at a constant speed of 0.05 mm/s with a force of 0.7 mN. The cutoff value was set at 0.08 mm (Gauss filter). The surface roughness was characterized by  $Ra$  ( $\mu\text{m}$ ).  $Ra$  is an arithmetic average of the peaks and valleys of the specimen surface, it is recorded as absolute values within the evaluation length, and it is generally expressed in units of height. The roughness parameters were obtained via Surface Analysis (Kosaka Corp), and the mean  $Ra$  values were calculated using the following formula, where the roughness curve is expressed in  $y = f(x)$  and  $L$  is the reference length:

$$Ra = \frac{1}{L} \int_0^L |f(x)| dx$$

Three readings were taken from each specimen, with tracings at three different locations—parallel, perpendicular, and transverse to the finishing and polishing scratch directions. The average of these readings, the mean  $Ra$ , was used as the baseline  $Ra$  value for each sample.

### pH-Cycling Model

The specimens were subjected to a pH-cycling regime, as proposed by Featherstone and others<sup>29</sup> and Silva and others.<sup>22</sup> The demineralization solution contained 2.0 mM calcium and 2.0 mM phosphate in a buffer solution of 74.0 mM acetate at pH 4.3. The remineralization solution consisted of 1.5 mM calcium and 0.9 mM phosphate in a buffer solution of 0.1 mM Tris (hydroxymethyl-aminomethane) at pH 7.0. The specimens were first immersed in 5 mL of demineralization solution for six hours at 37°C, rinsed with distilled deionized water, and then stored in 5 mL of remineralization solution for 18 hours at 37°C.<sup>22</sup> These experimental conditions were repeated for 10 uninterrupted cycles of demineralization and remineralization. The solutions were changed every day.

After the pH-cycling regime, all specimens were submitted to surface roughness testing, as described previously.

### Three-body Abrasion Testing

After pH cycling, the toothbrushing test was conducted in an automatic toothbrushing machine using a 200g load for 30,000 cycles at 250 cycles/min. Colgate Total dentifrice (Colgate Palmolive Ind. e Com. Ltda, SB Campo, São Paulo, Brazil) that had

been diluted in distilled water (1:2) was used as an abrasive third body. This dentifrice was considered to be a moderate abrasive toothpaste, according to the Relative Dentin Abrasion ( $\sim 70$ ) adopted by DIN EN ISO 11609.<sup>30</sup> The specimens were washed in an ultrasonic bath for 10 minutes. The final  $Ra$  readings were taken from each specimen, as described previously, in a direction opposite that of the toothbrushing movement.

### Surface Morphology Assessment

Three specimens of each material at each experimental period (baseline, after pH cycling, and after toothbrushing) were analyzed to determine the superficial morphology using a scanning electron microscope (SEM). The nine specimens of each material were mounted on a holder using double-sided adhesive carbon tape in order to illustrate the effect of pH cycling or/and toothbrushing on the material surfaces. The specimens were sputter-coated with gold in a vacuum appliance (Balzers-SCD 050 Sputter Coater; Liechtenstein, Germany) and examined with a Model JEOL JSM 5600 LV SEM (Tokyo, Japan) operating at 3000 $\times$  magnification.

### Statistical Analysis

The roughness data were evaluated to check the equality of variances and to ensure a normal distribution. After this, the roughness data were submitted to a paired  $t$ -test to analyze the differences in surface roughness before any experimental procedures (baseline), after pH cycling, and after the abrasion test. Two-way analysis of variance (ANOVA) and Tukey test were used to compare the roughness among materials. A level of  $\alpha = 0.05$  was used to demonstrate statistical significance.

## RESULTS

Table 2 presents the comparison of superficial roughness measurements ( $Ra$ ) of each material among the experimental periods (treatment)—baseline, after pH cycling, and after toothbrushing—and among restorative materials in each treatment. When the comparisons of  $Ra$  were made for each material among the treatments, only Fuji IX GP showed a statistically significant increase in surface roughness ( $0.15 \pm 0.10 \mu\text{m}$  at baseline,  $0.27 \pm 0.20 \mu\text{m}$  after pH cycling, and  $1.10 \pm 0.80 \mu\text{m}$  after toothbrushing;  $p=0.005$ ). For Vitremer, Ketac N100, and Filtek Supreme restorative materials there was only a statistically significant increase in surface roughness ( $p<0.001$ ) between the baseline ( $0.20 \pm$



Table 2: Superficial Roughness Measurements (μm) of Restorative Materials Tested After pH-Cycling Regime and Toothbrush Abrasion <sup>ab</sup>			
Material/ Treatment	Baseline	pH-Cycling Regime	Tooth- brushing
Fuji IX GP	0.15 ± 0.10 <sup>a,A</sup>	0.27 ± 0.20 <sup>b,A</sup>	1.10 ± 0.80 <sup>c,A</sup>
Vitremer	0.20 ± 0.16 <sup>a,A</sup>	0.23 ± 0.08 <sup>a,A</sup>	1.04 ± 0.46 <sup>b,A</sup>
Ketac N100	0.14 ± 0.07 <sup>a,A</sup>	0.18 ± 0.12 <sup>a,A</sup>	0.68 ± 0.16 <sup>b,AB</sup>
Filtek Supreme	0.09 ± 0.03 <sup>a,A</sup>	0.11 ± 0.05 <sup>a,A</sup>	0.30 ± 0.15 <sup>b,B</sup>
<sup>a</sup> Values (mean ± standard deviation) expressed in μm. <sup>b</sup> Lowercase letters refer to comparisons of each material among the experimental periods (baseline, after pH-cycling regime, and after toothbrushing). Values followed by the same lowercase letters in each row do not differ statistically (paired t-test, p>0.05). Uppercase letters refer to comparisons among restorative materials in each experimental period. Values followed by the same uppercase letters in the columns do not differ statistically (two-way analysis of variance [ANOVA] and Tukey test, p>0.05).			

0.16 μm, 0.14 ± 0.07 μm, and 0.09 ± 0.03 μm, respectively) and after toothbrushing treatment (1.04 ± 0.46 μm, 0.68 ± 0.16 μm, and 0.30 ± 0.15 μm, respectively). Therefore, the greatest surface roughness values were found after toothbrushing for all materials.

The ANOVA showed a significant interaction between restorative materials and treatment (*p*=0.003). Because the interaction was significant, a comparison of the behavior of the different materials in each treatment was made. There was no statistically significant difference among the roughness values for all of the materials after pH cycling (*p*=0.089). Fuji IX GP (1.10 ± 0.80 μm) and Vitremer (1.04 ± 0.46 μm) had the highest roughness values after toothbrushing; however, no statistically significant difference in surface roughness existed between Fuji IX GP and Vitremer. Ketac N100 (0.68 ± 0.16 μm) showed intermediate roughness values after toothbrushing. This material did not have a statistically significant difference in surface roughness between Fuji IX GP and Vitremer, but it also did not have a statistically significant difference in surface roughness with Filtek Supreme (0.30 ± 0.15 μm). Filtek Supreme showed the lowest superficial roughness values.

The scanning electron micrographs in Figure 1 show details of the surface morphology of the studied materials. The conventional GI and RMGI cements presented a large number of cracks in the micro-

structures, probably caused by dehydration during preparation for SEM analysis (Figure 1B,E). Fuji IX GP, Vitremer, and Ketac N100 showed moderate irregularities in surface morphology after pH cycling (Figure 1B,E,H), and the surface of Filtek Supreme seemed minimally affected (Figure 1K). However, all materials showed a discernible loss of material after toothbrushing, leading to irregular surfaces and protruding filler particles, showing the abrasion aspect of the matrix (Figure 1C,F,I,L). There was a clearly visible difference between the fillers of Ketac N100 (Figure 1I) and the other GIs with regard to shape and size. Ketac N100 has a more regular shape and the lowest size particles when compared with the other GIs. Furthermore, Ketac N100 presents nanoclusters with similar size and shape, which Filtek Supreme does not (Figure 1L).

DISCUSSION

The present study investigated the superficial roughness of a nanofilled GI after a chemical challenge and toothbrushing abrasion and compared those data with conventional GI, RMGI, and nanofilled composite. The pH-cycling regime used in the current study was an *in vitro* chemical degradation method that simulated alterations in the pH of the oral cavity and mimics clinical conditions whereby there is always a dynamic between demineralization and remineralization.<sup>22</sup> The method of acid challenge by the dynamic pH-cycling model used in the current study was proposed by Featherstone and others.<sup>29</sup> This method involves six hours of acid challenge and 18 hours of remineralization per day, which is a reasonable estimate for subjects who frequently consume sugar.<sup>29</sup> Moreover, Silva and others<sup>22</sup> showed that the surface properties of restorative materials were influenced by pH variation in the demineralizing solution of pH cycling. The acidic pH led to a greater alteration in hardness and surface morphology of GI cements.<sup>22</sup>

In this present study, the dynamic pH cycling did not affect the superficial roughness of RMGI, nanofilled GI, and nanofilled resin; only the conventional GI showed an increase in superficial roughness after pH cycling (Table 2). Since the conventional GI did not have resin monomers in its composition, an acid-base reaction occurs after the formation of a calcium and aluminum polyalkenoate matrix, in which the filler particles are embedded and surrounded by a silica hydrogel.<sup>31</sup> While resin-based materials undergo the cleavage of polymer chains to form oligomers and monomers, the ionomeric cements present a complex process of absorption, disintegra-

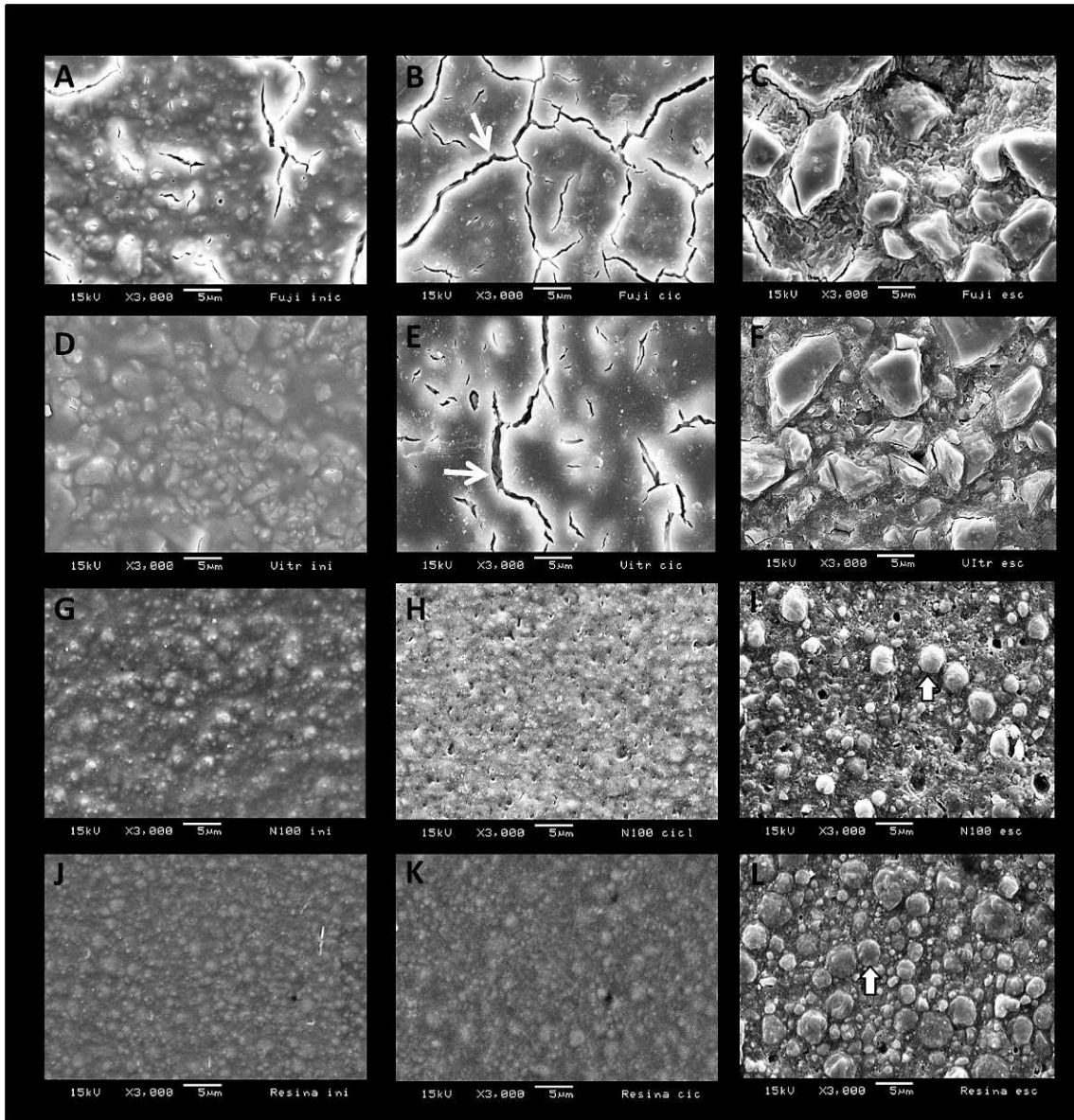


Figure 1. Scanning electron micrographs of the surface morphology of Fuji IX GP (A-C), Vitremer (D-F), Ketac N100 (G-I), and Filtek Supreme (J-L). The first column shows the surface morphology of restorative materials without treatment (control). The second column represents the surface morphology of restorative materials after pH cycling with a large number of cracks on the Fuji IX and Vitremer surfaces (→) and undetectable fillers (B and E). Filtek Supreme shows minimal alteration in surface morphology (K), and Ketac N100 shows minimal degradation of the matrix. The third column represents the surface morphology of restorative materials after toothbrushing; an irregular surface and protruding filler particles were visualized for all materials (C, F, I, and L). Open arrows indicate nanoclusters and nanofillers of Ketac N100 and Filtek Supreme (I and L).

tion, and outward transportation of ions.<sup>12</sup> The acidic degradation begins with water absorption that diffuses internally through the matrix, filler interfaces, pores, and other defects, accelerated by a low pH and causing filler-matrix debonding or even hydrolytic degradation of the filler-matrix interface.<sup>32,33</sup>

No statistically significant difference was found among the roughness values of all materials tested

after the pH cycling (Table 2). It is likely that there was no statistically significant difference among the roughness values as a result of the large standard deviations obtained, mainly for Fuji IX GP and Ketac N100. Moreover, Filtek Supreme showed minimal alterations in surface morphology after pH cycling, as observed by SEM (Figure 1K), while the GIs showed few irregularities with minimal degradation of the matrix (Figure 1H) and a surface layer with



undetectable fillers (Figure 1B,E). While other studies<sup>15,24</sup> have shown that a pH-cycling regime similar to the one used in the current study (six hours of demineralization/18 hours of remineralization for 10 uninterrupted days) affected the surface roughness of restorative materials, it is possible that the period of pH cycling (10 days) was not long enough to cause significant hydrolytic and corrosive degradation among the GI restorative materials and nanocomposite, regardless of their different compositions. It is possible that if the period of the pH-cycling regime was increased, differences in the roughness and morphology could be found among the GIs and composite.

The design of the toothbrush abrasion used in this current study, 30,000 strokes, is equivalent to three years of brushing in the clinical situation.<sup>34</sup> However, the abrasion regimen used in this study certainly does not correspond to 10 episodes of variation in pH in the clinical situation. This extensive number of brushing strokes was performed in order to develop grooves to analyze the surface roughness. After toothbrushing abrasion, GIs and composite resins behaved differently with regard to their surface roughness and morphology (Table 2; Figure 1). Possible explanations for these differences are the inherent resistance of the major constituents (polymer matrix and glass filler particles), size and shape of the filler particles, adhesion between the particles and matrix, and the setting reaction of all materials.<sup>35</sup>

While the pH cycling did not present a roughness difference between RMGI and conventional GI (Table 2), it was not possible to find any studies regarding superficial roughness analyses between conventional GI and RMGI after pH-cycling and abrasion tests. In this present study, the conventional GI and RMGI did not show significant differences in roughness values after toothbrushing abrasion. As both GIs present larger and irregular glass filler particles with similar mean size (around 3–4  $\mu\text{m}$ ) (Table 1), the toothbrushing abrasion provided a discernible loss of material, causing these filler particles to be lost or to protrude from the surface (Figure 1C,F), while differences in roughness values were not found between them. Moreover, the difference between the abrasion of the matrix and filler particles might have been large enough to provide an anisotropic degradation, which gradually slowed down as the filler became exposed to the surface.<sup>15</sup> Thus, the differences of composition between the matrix of conventional GIs and RMGIs—conventional GI has a polyalkenoate ma-

trix and RMGI has a polyalkenoate and poly-2-hydroxyethyl methacrylate (HEMA) matrix—were minimized, and the superficial roughness after abrasion was similar.

Although the roughness value of the nanofilled GI Ketac N100 did not statistically differ from those of Vitremer and Fuji IX GP, it behaved like an intermediate material between a GI and a nanocomposite resin when abrasion resistance was considered (Table 2). A reasonable explanation for this result is the composition of Ketac N100, which includes other resin monomers in addition to HEMA, is similar in composition to RMGI, with bisphenol glycidyl methacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA). Additionally, this cement represents a blend of fluoraluminosilicate technology (40%) and nanotechnology (60%), including silica cluster fillers, nanoagglomerated silica fillers, and acid-reactive glass fillers that are smaller than those of resin-modified glass ionomers (Ketac™ N100-Technical Product Profile).<sup>9</sup> Filtek Supreme nanocomposite presents nanofillers and nanoclusters and the resin matrix had other resin monomers, such as the ethoxylated version of Bis-GMA (Bis-EMA) and urethane dimethacrylate (UDMA).<sup>36</sup> Thus, Ketac N100 had an intermediate composition between RMGIs and nanocomposites, presenting intermediate superficial characteristics and abrasion resistance. Ketac N100 presented an appearance more like a polyacid-modified resin composite based on resin components and structure; however, further studies must be conducted to investigate this fact.

The incorporation of nanofillers in restorative materials improves the abrasive resistance because it promotes a higher filler loading with smaller particle size and provides a reduction in the interparticle spacing, which effectively protects the softer matrix, reduces the incidence of filler exfoliation, and enhances the overall resistance of the material to abrasion.<sup>37</sup> Ketac N100 has a highly packed filler composition (~69%), of which approximately two-thirds are nanofillers.<sup>10</sup> Figure 1I and L show the nanoclusters of nanofilled GIC and nanocomposite. According to the manufacturer information, the aggregated “nanoclusters” in Ketac N100 are in the 1- $\mu\text{m}$  size range but are composed of 5- to 20-nm spherical particles that have been lightly sintered together to form a porous structure interpenetrated with resin monomers (Ketac™ N100-Technical Product Profile).<sup>9</sup> As the surface of the “nanocluster”/resin combination is subjected to stress and toothbrushing abrasion, the smaller

nanosized particles, which make up the clusters, tend to break apart rather than the entire particle being plucked from the resin matrix, leaving the surfaces with defects smaller than the wavelength of light and providing satisfactory polish and optical properties.<sup>11</sup> In this current study, these characteristics contributed to the abrasion resistance of Ketac N100, which was not statistically significantly different from that of the nanocomposite (Table 2). Similar results were found by de Paula and others,<sup>17</sup> in whose study the surface roughness of Ketac N100 did not differ from that of a nanocomposite after biomechanical degradation. However, the larger and irregular filler particles present in Vitremer and Fuji IX GP (Figure 1C,F) made it easier to “pluck out” a whole filler particle from the resin matrix, which could act as an additional abrasive agent once it has detached from the surface and is held against the specimen.<sup>38</sup> In addition to the filler size, the hydrophilic property of the Vitremer polymer matrix, as a result of the hydroxyl groups of HEMA,<sup>39</sup> and the insufficient coherent entanglement between the ionic cross-linked polyalkenoate network and the polymeric chains<sup>3,35</sup> could have provided a greater chemical and mechanical superficial degradation than was associated with Ketac N100 and Filtek Supreme.

The nanocomposite Filtek Supreme showed lower roughness values than did the conventional GI and RMGI. The presence of nanofillers and nanoclusters, as explained previously, contributes to this result. In addition, the presence of Bis-EMA can promote hydrolytic and biochemical stability as a result of the hydrophobicity of this monomer when compared to Bis-GMA.<sup>13</sup> The greater part of TEGDMA of Filtek Supreme was replaced with a blend of UDMA and Bis-EMA. The absence of a phenol ring in the monomer chain of UDMA leads to greater flexibility and toughness when compared with Bis-GMA, making the UDMA resins more reactive, with higher conversion and cross-link density than are associated with the Bis-GMA polymers, contributing to the matrix being more resistant to abrasion.<sup>36</sup>

Based on the results of this current study, the hypothesis that a nanofilled RMGI subjected to a pH-cycling model prior to a three-body abrasion test would not differ in surface roughness and micromorphology from a conventional GI, RMGI, and nanofilled resin composite was rejected. Ketac N100 showed statistically significant intermediate roughness values between GI and a nanocomposite resin after pH cycling and toothbrushing abrasion.

## CONCLUSION

Concerning the surface roughness and surface morphology after chemical and mechanical degradation, the nanofilled GI restorative material, Ketac N100, behaved as an intermediate material among GI restorative materials (conventional and resin-modified) and a nanocomposite.

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## Conflict of Interest Declaration

The Authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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## REFERENCES

1. Tyas MJ, & Burrow MF (2002) Clinical evaluation of a resin-modified glass ionomer adhesive system: Results at five years *Operative Dentistry* **27**(5) 438-441.
2. Krämer N, Lohbauer U, & Frankenberger R (2007) Restorative materials in the primary dentition of polycaries patients *European Archives of Paediatric Dentistry* **8**(1) 29-35.
3. Gladys S, Van Meerbeek B, Braem M, Lambrechts P, & Vanherle G (1997) Comparative physico-mechanical characterization of new hybrid restorative materials with conventional glass-ionomer and resin composite restorative materials *Journal of Dental Research* **76**(4) 883-894.
4. Wiegand A, Buchalla W, & Attin T (2007) Review on fluoride-releasing restorative materials—Fluoride release and uptake characteristics, antibacterial activity and influence on caries formation *Dental Materials* **23**(3) 343-362.
5. Markovic D, Petrovic BB, & Peric TO (2008) Fluoride content and recharge ability of five glass ionomer dental materials *BMC Oral Health* **8**(1) 21-28.
6. Mitra SB (1991) Adhesion to dentin and physical properties of a lightcured glass ionomer liner/base *Journal of Dental Research* **70**(1) 72-74.
7. Xie D, Brantley WA, Culbertson BM, & Wang G (2000) Mechanical properties and microstructures of glass-ionomer cements *Dental Materials* **16**(2) 129-138.
8. Ellakuria J, Triana R, Mínguez N, Soler I, Ibaseta G, Maza J, & Garcia-Godoy F (2003) Effect of one-year water storage on the surface microhardness of resin-modified versus conventional glass-ionomer cements *Dental Materials* **19**(4) 286-290.
9. Ketac™ N100 Light Curing Nano-Ionomer Restorative Technical product profile, retrieved online February 10, 2010 from: <http://www.3m.com>
10. Coutinho E, Cardoso MV, De Munck J, Neves AA, Van Landuyt KL, Poitevin A, Peumans M, Lambrechts P, &



- Van Meerbeek B (2009) Bonding effectiveness and interfacial characterization of a nano-filled resin-modified glass-ionomer *Dental Materials* **25**(11) 1347-1357.
11. Mitra SB, Wu D, & Holmes BN (2003) An application of nanotechnology in advanced dental materials *Journal of the American Dental Association* **134**(10) 1382-1390.
12. Oilo G (1992) Biodegradation of dental composites/glass-ionomer cements *Advances in Dental Research* **6** 50-54.
13. Yap AU, Low JS, & Ong LFKL (2000) Effect of food-simulating liquids on surface characteristics of composite and polyacid-modified composite restoratives *Operative Dentistry* **25**(3) 170-176.
14. Fúcio SB, Carvalho FG, Sobrinho LC, Sinhoreti MA, & Puppini-Rontani RM (2008) The influence of 30-day-old *Streptococcus mutans* biofilm on the surface of esthetic restorative materials—An in vitro study *Journal of Dentistry* **36**(10) 833-839.
15. Turssi CP, de Magalhães CS, Serra MC, & Rodrigues Júnior AL (2001) Surface roughness assessment of resin-based materials during brushing preceded by pH-cycling simulations *Operative Dentistry* **26**(6) 576-584.
16. Turssi CP, Hara AT, de Magalhães CS, Serra MC, & Rodrigues AL Jr (2003) Influence of storage regime prior to abrasion on surface topography of restorative materials *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **65**(2) 227-232.
17. de Paula A, Fucio S, Ambrosano G, Alonso R, Sardi J, & Puppini-Rontani R (2011) Biodegradation and abrasive wear of nano restorative materials *Operative Dentistry* doi:10.2341/10-221-L.
18. Dyer D, Addy M, & Newcombe RG (2000) Studies in vitro of abrasion by different manual toothbrush heads and a standard toothpaste *Journal of Clinical Periodontology* **27**(2) 99-103.
19. Yap AUJ, Teoh SH, & Tan KB (2000) Influence of water exposure on three-body wear of composite restoratives *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **53**(5) 547-553.
20. Carvalho FG, Fucio SB, Paula AB, Correr GM, Sinhoreti MA, & Puppini-Rontani RM (2008) Child toothbrush abrasion effect on ionomeric materials *Journal of Dentistry for Children* **75**(2) 112-116.
21. Sarkar NK (2000) Internal corrosion in dental composite wear *Journal of Biomedical Materials Research* **53**(4) 371-380.
22. Silva KG, Pedrini D, Delbem AC, & Cannon M (2007) Effect of pH variations in a cycling model on the properties of restorative materials *Operative Dentistry* **32**(4) 328-335.
23. Buchalla W, Attin T, & Hellwig E (2000) Brushing abrasion of luting cements under neutral and acidic conditions *Operative Dentistry* **25**(6) 482-487.
24. Turssi CP, Hara AT, Serra MC, & Rodrigues AL Jr (2002) Effect of storage media upon the surface micromorphology of resin-based restorative materials *Journal of Oral Rehabilitation* **29**(9) 864-871.
25. Hotta M, Hirukawa H, & Aono M (1995) The effect of glaze on restorative glass-ionomer cements: Evaluation of environmental durability in lactic acid solution *Journal of Oral Rehabilitation* **22**(9) 685-689.
26. Fano L, Fano V, Ma W, Wang X, & Zhu F (2004) Hydrolytic degradation and cracks in resin-modified glass-ionomer cements *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **69**(1) 87-93.
27. Hengtrakool C, Pearson GJ, & Wilson M (2006) Interaction between GIC and *S. sanguis* biofilms: Antibacterial properties and changes of surface hardness *Journal of Dentistry* **34**(8) 588-597.
28. Teughels W, Van Assche N, Sliepen I, & Quirynen M (2006) Effect of material characteristics and/or surface topography on biofilm development *Clinical Oral Implants Research* **17**(2) 68-81.
29. Featherstone JDB, O'Reilly MM, Shariati M, & Brugler S (1986) Enhancement of remineralization in vitro and in vivo In: Leach SA (ed) *Factors Relating to Demineralization and Remineralization of the Teeth* IRL, Oxford University Press, USA 23-34.
30. ISO11609 (1995) ISO 1995 International Standard: Dentistry Toothpaste Requirements, Test Methods and Marking Switzerland: International Organization for Standardization.
31. Wasson EA, & Nicholson JW (1993) New aspects of the setting of glass-ionomer cements *Journal of Dental Research* **72**(2) 481-483.
32. Toledano M, Osorio R, Osorio E, Fuentes V, Prati C, & Garcia-Godoy F (2003) Sorption and solubility of resin-based restorative dental materials *Journal of Dentistry* **3**(1) 43-50.
33. Ferracane JL (2006) Hygroscopic and hydrolytic effects in dental polymer networks *Dental Materials* **22**(3) 211-222.
34. Goldstein GR, & Lerner T (1991) The effect of toothbrushing on a hybrid resin composite *Journal of Prosthetic Dentistry* **66**(4) 498-500.
35. Peutzfeldt A, García-Godoy F, & Asmussen E (1997) Surface hardness and wear of glass ionomers and compomers *American Journal of Dentistry* **10**(1) 15-17.
36. Floyd CJ, & Dickens SH (2006) Network structure of Bis-GMA- and UDMA-based resin systems *Dental Materials* **22**(12) 1143-1149.
37. Söderholm KJ, & Richards ND (1998) Wear resistance of composite: A solved problem? *General Dentistry* **46**(3) 256-263.
38. Correr GM, Alonso RCB, Sobrinho LC, Puppini-Rontani RM, & Ferracane JL (2006) In vitro wear of resin-based materials—Simultaneous corrosive and abrasive wear *Journal of Biomedical Materials Research: Part B, Applied Biomaterials* **78**(1) 105-114.
39. Kugiyamiya A, & Takeuchi T (1999) Effects of 2-hydroxyethyl methacrylate on polymer network and interaction in hydrophilic molecularly imprinted polymers *Analytical Sciences* **15**(1) 29-33.