The purpose of this study was to determine the influence of hydrochloric acid on surface roughness of composite resins subjected to brushing. Sixty samples measuring 2 mm thick x 6 mm diameter were prepared and used as experimental units. The study presented a 3x2 factorial design, in which the factors were composite resin (n=20), at 3 levels: microhybrid composite (Z100), nanofilled composite (Filtek™ Supreme), nanohybrid composite (Ice), and acid challenge (n=10) at 2 levels: absence and presence. Acid challenge was performed by immersion of specimens in hydrochloric acid (pH 1.2) for 1 min, 4 times per day for 7 days. The specimens not subjected to acid challenge were stored in 15 mL of artificial saliva at 37 °C. Afterwards, all specimens were submitted to abrasive challenge by a brushing cycle performed with a 200 g weight at a speed of 356 rpm, totaling 17.8 cycles. Surface roughness measurements (Ra) were performed and analyzed by ANOVA and Tukey test (p≤0.05). Surface roughness values were higher in the presence (1.07±0.24) as compared with the absence of hydrochloric acid (0.72±0.04). Surface roughness values were higher for microhybrid (1.01±0.27) compared with nanofilled (0.68±0.09) and nanohybrid (0.48±0.15) composites when the specimens were not subjected to acid challenge. In the presence of hydrochloric acid, microhybrid (1.26±0.28) and nanofilled (1.18±0.30) composites present higher surface roughness values compared with nanohybrid (0.77±0.15). The hydrochloric acid affected the surface roughness of composite resin subjected to brushing.

Introduction

Dental erosion is one of the main factors for tooth wear (1) that occurs without bacterial involvement by chemical dissolution of the dental structure (1-3) by acids (1). These acids are derived from the dietary and endogenous factors, such as eating disorders and gastroesophageal reflux (1,4).

The hydrochloric acid released by gastroesophageal reflux may be responsible for a severe tooth wear, when it remains in contact with the oral cavity for a long time (5). If tooth structure loss reaches a high severity, functional and esthetic rehabilitation of the teeth becomes indispensable (1). The choice of an adequate restorative material plays an extremely relevant role in the durability of the restoration.

Composite resin provides excellent properties for restoration, including wear resistance (6). However, dental erosion can damage the physical and mechanical properties of the composite (3), leading to organic matrix degradation and exposure of the inorganic filaments (7). These changes lead to increase of surface softening and roughness, which are responsible for the decrease in restoration durability (8).

Likewise, the resin matrix may be eroded by tooth brushing, leading to irregularities on the material’s surface (9). The abrasion resulting from brushing acts synergistically with the erosion phenomenon, leading to product degradation over the course of time (3).

Recently, a composite resin containing nanofillers, which has better mechanical properties than those of microhybrid resin, was introduced on the market. The insertion of a large quantity of small, homogeneously distributed fillers provides the organic matrix greater protection against wear, thereby enhancing the material resistance to degradation (10).

It is essential to choose material suitable for restoring the teeth of patients with gastroesophageal reflux in order to ensure longevity of the procedure, because contact with hydrochloric acid may be cause for superficial alteration in composite resin. This may especially occur simultaneously with brushing. Thus, the aim of this study was to evaluate the effect of hydrochloric acid on the surface roughness of composite resins submitted to brushing. The tested null hypotheses were: 1) hydrochloric acid does not affect the surface roughness of composite resins submitted to brushing; 2) The composition of material has no influence on the wear resistance of composite resin.

Material and Methods

Experimental Design

The experimental design was factorial, with the evaluated factors composite resin at three levels: microhybrid composite (Z100), nanofilled composite...
(Filtek Supreme) and nanohybrid composite (Ice); and acid challenge, at two levels: absence and presence. The response variable was surface roughness (μm). The materials used in this study are described in Tables 1 and 2.

**Specimen Preparation**

Twenty specimens of each material were prepared, totaling 60 samples. The composite resin filaments were inserted in a cylindrical stainless steel matrix with a 6 mm diameter and 2 mm deep. After the matrix was filled, a polyester strip was pressed onto the surface, by a glass slab and a 500 g weight. After 30 s the weight was removed and the composite resin was light-polymerized using a LED light-curing unit with 1200 mW/cm² power output (Dabi Atlante, Ribeirão Preto, SP, Brazil), according to the manufacturer’s instructions.

The specimens were stored in 15 mL of artificial saliva at 37 °C for 24 h. The specimens were polished with Sof-Lex discs (3M/ESPE, Sumaré, SP, Brazil) with different granulations for 20 s each.

**Acid Challenge**

For each material, half of the composite cylinders were subjected to acid challenge, which was done by immersing each specimen in 15 mL of hydrochloric acid (Quality Ind. Chemicals and Cleaning Ltda-EPP, pH=1.2) for 1 min, 4 times per day for 7 days. The immersion cycles were performed each day at 8 am, 2 pm, 6 pm and 8 pm, according to a modification of the original protocol proposed by Honório et al. (11). Between immersions each specimen was stored in distilled water at 37 °C. The specimens not submitted to acid challenge were stored in 15 mL of artificial saliva at 37 °C.

**Abrasive Challenge**

All the specimens were subjected to abrasive challenge by a tooth brushing machine (Pepsodent - MAVTEC, Ribeirão Preto, SP, Brazil) fitted with soft toothbrushes (Condor S/A Ind. Com. Ltda., São Bento do Sul, SC, Brazil).

Brushing was performed at a speed of 356 rpm, with dentifrice slurry ratio 1:1 covering a 3.8 cm track with 200 g load on the specimen. The specimens were brushed for cycles with a 50 min duration each, for 7 days (17.8 cycles). The first cycle was performed after the second daily acid challenge (7).

**Surface Roughness Analysis**

The surface roughness analysis was performed before the experiment began (T0) and at the end of the abrasive challenges (T1). For each analysis, three roughness measurements (Ra) were taken by a rugosimeter (Mitutoyo Co, Kawasaki, Japan).

**Statistical Analysis**

The mean and standard deviation of roughness (ΔRa) were calculated and analyzed by two-way ANOVA and Tukey tests (p≤0.05). The calculations were performed with the GMC software (www.forp.usp.br/restauradora/gmc/gmc.html#gmc).

**Results**

Surface roughness values were statistically higher for the specimens subjected to acid challenge and brushing (1.07±0.24) in comparison with specimens subjected to brushing only (0.72±0.04).

There was significant interaction (p<0.05)

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**Table 1. Composite resins utilized on this study**

<table>
<thead>
<tr>
<th>Composite resin</th>
<th>Composition</th>
<th>Particle size</th>
<th>Shade</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Z100</td>
<td>Bis-GMA and TEGDMA</td>
<td>0.6 μm</td>
<td>A2</td>
<td>3M/ESPE, St. Paul, MN, USA</td>
</tr>
<tr>
<td>Filtek Supreme</td>
<td>BIS-GMA, BIS-EMA, UDMA and TEGDMA (72.5%)</td>
<td>75 nm</td>
<td>A2</td>
<td>3M/ESPE, St. Paul, MN, USA</td>
</tr>
<tr>
<td>Ice</td>
<td>61% of inorganic matrix and particles of strontium alumino-silicate</td>
<td>0.01-1.3 μm</td>
<td>A2</td>
<td>SDI, São Paulo, SP, Brazil</td>
</tr>
</tbody>
</table>

**Table 2. Materials used in this study**

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Artificial saliva</td>
<td>Na2HPO4 (0.26 g/L), NaCl (6.7 g/L), KSCN (0.33 g/L), KH2PO4 (0.2 g/L), NaHCO3 (1.5 g/L), KCl (1.2 g/L)</td>
<td>School of Pharmaceutical Sciences of Ribeirão Preto, University of São Paulo, Brazil</td>
</tr>
<tr>
<td>Hydrochloric acid</td>
<td>30% HCl, H2O</td>
<td>Quality Ind. Chemicals and Cleaning Ltda-EPP, São Paulo, SP, Brazil</td>
</tr>
<tr>
<td>Sof-Lex disc</td>
<td>Mylar™ metal center, aluminum oxide</td>
<td>3M/ESPE, St. Paul, MN, USA</td>
</tr>
<tr>
<td>Dentifrice</td>
<td>Water, sorbitol, hydrated silica, sodium laurel sulfate, PVM / MA copolymer, carrageenan, sodium hydroxide, sodium fluoride, triclosan, sodium saccharin, titanium dioxide</td>
<td>Colgate-Palmolive Industrial LTDA, São Paulo, SP, Brazil</td>
</tr>
</tbody>
</table>
between acid challenge × composite resin. The microhybrid composite showed higher surface roughness than the nanofilled and nanohybrid resins for the specimens not subjected to acid challenge. However, in presence of hydrochloric acid, there was no significant difference between the microhybrid resin and the nanofilled composite, while the nanohybrid composite showed the lowest surface roughness. The means and standard deviations of surface roughness (Ra) are presented in Table 3.

Table 3. Surface roughness mean and standard deviation (Ra) to composite resin × acid challenge

<table>
<thead>
<tr>
<th>Composite resin</th>
<th>Absence</th>
<th>Presence</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microhybrid</td>
<td>1.01±0.27&lt;sup&gt;aA&lt;/sup&gt;</td>
<td>1.26±0.28&lt;sup&gt;bB&lt;/sup&gt;</td>
</tr>
<tr>
<td>Nanofilled</td>
<td>0.68±0.09&lt;sup&gt;aA&lt;/sup&gt;</td>
<td>1.18±0.30&lt;sup&gt;bB&lt;/sup&gt;</td>
</tr>
<tr>
<td>Nanohybrid</td>
<td>0.48±0.15&lt;sup&gt;aA&lt;/sup&gt;</td>
<td>0.77±0.15&lt;sup&gt;bB&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Different lowercase letters in columns and uppercase letters in rows indicate statistically significant difference at 5%.

Discussion

In the present study, the association between gastroesophageal reflux and brushing was simulated, whereas other external factors that could contribute to tooth and restorative material erosion were eliminated. Thus, despite the limitations of an in vitro study, this was advantageous, because it allowed control of the erosion time, pH and the applied erosive agents.

The first null hypothesis was rejected, since surface roughness value was higher for specimens immersed in hydrochloric acid compared with those that were not subjected to acid challenge. The main explanation for this is that restorative materials tend to undergo degradation after subjected to acid conditions (2), due to polymeric matrix wear, leading to the exposure of filaments and loss of filamentous particles (9).

In addition, abrasion plays an important role in restorative material wear (7,18), causing the chemical dissolution of composite resin (7). Toothbrush abrasion modifies the balance between the organic matrix and filamentous particles, damages the bond between components, leading to loss of the inorganic portion and exposure of particles (10). Oliveira et al. (2011) showed the high surface roughness of restorative material after brushing. Moreover, studies have reported that abrasion and erosion act synergistically in restorative material wear (3). However, as the present study did not evaluate the brushing effect alone, it is not possible to affirm that brushing is responsible for material surface alterations.

With regards to the material, the second null hypothesis was rejected, since the results suggest that its composition plays an important role in wear resistance. Since previous studies have shown that surface roughness is determined by hardness, size and quantity of filler particles immersed in the matrix (19,20), it is supposed that the findings are mostly related to the inorganic components of composite resin.

Nanofilled composite showed a lower surface roughness compared with microhybrid resin. However, the composites presented similar results when immersed in hydrochloric acid. The smaller the particles, the higher will be the wear resistance of material, since it will present greater homogeneity and less prominent particles on surface (21). Thus, microhybrid composite may have undergone a higher degree of degradation by brushing due to its large particles, which protrude more easily through the surface (22). Nevertheless, acid challenge resulted in a higher wear in both composites during the first time interval, leading to fillers being pulled out and greater susceptibility to degradation by brushing.

Furthermore, the properties of composite resin will be influenced by type and volume of the filler particles (23). With regards to the volume and arrangement of inorganic particles in a composite containing a high volume of fillers, agglomeration of particles may occur with subsequent deterioration of wear resistance (24).

In the present study, the results showed that the nanohybrid composite presented the lowest surface roughness both in the absence and presence of acid challenge. As the composite has smaller volume of fillers compared with the other composites, it may be assumed that there was a uniform dispersion of particles, which leads to an improvement in composite properties.

An important factor to consider with regards to erosion is the role of saliva protecting the restorative material surfaces from erosion, since it dilutes and neutralizes the acid solution (1,4,16). The specimens were stored in artificial saliva at 37 °C, allowing protection of the restorative material.

The specimens were immersed in acid for 1 min during each cycle. However, saliva neutralizes the acid up to 3 min after its permanence in the oral cavity (3). Similarly, the 50-min abrasive cycle is equivalent to 1 year of toothbrushing, considering that under clinical conditions the material would be in contact with the toothbrush for 10 s during each brushing session (3). Considering the short period of the acid contact with the surface of composite material in a clinical situation, the present study was designed with a long period of tooth erosion followed by brushing.

Although restorative material usually displays a greater resistance to dental erosion in comparison with the tooth (6), a short period was sufficient to change the composite resin surface. Intrinsic factors may cause dental erosion according to how frequently they will occur (17). Although
each cycle was responsible only for a short contact between the hydrochloric acid and the composite resin, the high number of cycles was shown to play a significant role in dental erosion.

In summary, the smaller the particles, the higher will be the wear resistance of the material, since it will present greater homogeneity and less prominent particles on the surface (21). Nanotechnology allows a smaller contraction, higher resistance and less polishing (25). Thus, considering that nanoparticle composite resin has smaller particles, it might be preferable when performing procedures in patients with gastroesophageal reflux.

Within the limitations of an in vitro study, it was possible to conclude that a high frequency of exposure to hydrochloric acid promotes increase in surface roughness of composite resin subjected to brushing. Nanoparticle resins may be preferable to use in the treatment of patients with gastroesophageal reflux due to their higher wear resistance.

Acknowledgements
The authors are acknowledge CNPq (grant #032/2012).

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Received October 11, 2014
Accepted January 21, 2015