Evaluation of surface changes in glass-ceramics submitted to artificial aging by simulated brushing

Avaliação das alterações superficiais de vitrocerâmicas submetidas ao envelhecimento artificial por escovação simulada

Evaluación de los cambios superficiales en vitrocerámicas sometidas a envejecimiento artificial mediante cepillado simulado

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Abstract

The aim of this study was to evaluate the surface changes of glass ceramics, submitted to artificial aging by simulated brushing with different toothpastes, using a brushing machine designed by the authors. Twenty-four specimens were made in the form of an upper right central incisor, in color A1 and made up in color A3, divided into 4 groups with n =6. The mechanical tests were performed by a brushing machine at an ambient temperature of 37°C, with a frequency of 250 strokes per minute, under 250mg brush pressure and simultaneous dripping of the dentifrice solution. Test times were divided into: 0; 25,000; 50,000; 75,000; 100,000; 125,000 and 150,000 strokes and the parameters analyzed by a rugosimeter. The obtained values of surface roughness (SR) were submitted to the Kolmogorov-Smirnov test (p=0.321) to verify the normality of the data distribution and, later, to the Levene test (p=0.076). The results obtained showed that the aging time did not influence the surface roughness of the glass ceramics, there were differences in surface roughness
in some toothpastes. Thus, it can be concluded that the glass ceramics were susceptible to mass loss, but resistant to the action of toothpastes. Dentifrices with anti-plaque and anti-tartar characteristics can change the surface over time.

Keywords: Glass-ceramics; Simulated brushing; Artificial aging.

1. Introduction

With the increase in aesthetic demand and minimally invasive treatments in dentistry, dental ceramics have been a reality present in the daily lives of dentists. Lithium disilicate glass-ceramics meet these two demands, being able to be used in the creation of thin and ultra-thin restorations up to 0.3 mm, different in the conventional layering process, they are obtained by injection (pressing) or machined (CAD/CAM) (Gomes et al., 2008, Li et al., 2014).

The oral cavity undergoes major chemical and mechanical changes, there are reports that ceramics exposed to high concentration of fluoride, both neutral and acidulated, have compromised surface brightness (Kulkarni et al., 2020). The mechanical action of chewing and brushing can compromise the surface, increasing its roughness (Yuan et al., 2018).

The durability of restorative materials and clinical satisfaction are difficult to study in longitudinal studies. However, in vitro research has shown great biostatistical reliability, and its results can be extrapolated to clinical practice, which tests such as accelerated artificial aging are conducted, allowing a dynamism in clinical simulations (Kulkarni et al., 2020).

Therefore, this study demonstrates a low-cost alternative to accelerated aging tests, making it possible to control the frequency and association of toothpastes. In addition, surface roughness tests were performed on lithium disilicate ceramic laminates Empress2 (IPS e.max Press, Ivoclar Vivadent AG), with the aim of observing their surface subjected to artificial aging accelerated by simulated brushing with different toothpastes from Brazilian market.
2. Methodology

The present in vitro study was carried out in the postgraduate technical laboratory of Universidade Cruzeiro do Sul, and was exempted from analysis by the Ethics and Research Committee of the Centro de Pesquisas Odontológicas of São Leopoldo Mandic. The surface roughness of a glass ceramic (IPS e.max Press, Ivoclar Vivadent AG, Schaan / Liechtenstein) was evaluated after aging by brushing with different national toothpastes.

To carry out the sample calculation, the average surface roughness (SR) of the glass-ceramics subjected to aging through the different dentifrices from a pilot was considered (n=3). Thus, considering an effect size of 0.78, and adopting a significance level of 0.05 and a power of 0.80, the number of 6 specimens per group was obtained, which results in 24 specimens produced and randomly numbered.

The specimens were produced in the form of laminated veneers of the upper right central incisor, divided into 4 groups of 6 samples. The fabrication followed the manufacturer's recommendations, therefore, the participants in the fabrication and reading of the parameters obeyed the following recommendations: (a) standardization of the samples, the design of the injection standards were carried out by a highly specialized technician (CNG - Prosthetic Solutions - São Paulo); (b) a denture laboratory that uses Ivoclar Vivadent equipment (dental prosthesis laboratory – Ipiranga/ São Paulo); and parameter reading were performed by a trained operator.

For this study there was a need for a specimen carried out in three different projects, digitally joined together: A standardized platform (P) was created for the mechanical and reading tests. The platforms were fixed in the same position in the brushing simulator to avoid statistical bias (Figures 1 and 2). The exclusion criteria were samples that were loosened during the cycling process, samples that did not reach the desired number of strokes, and the exclusion of brushes that did not reach the number of strokes.

**Figure 1.** Platform design and injection standards.

![Source: Authors.](image-url)
As a clinical simulator, the manikin tooth was scanned and digitally transferred to the center of the platform called the clinical preparation substrate (Su). Subsequently, the injection patterns were digitally reproduced, with photopolymer resins for the future manufacture of laminated veneers, in Empress2, which worked as a specimen (Sp).

A three-dimensional image of tooth 11 was obtained by a benchtop scanner (Unboxing Scanner Neway Open Technologies; YeanDent, Vierzon, France) and the preparation was digitized and transported to the computer program (Asiga Composer; Asiga, Sydney, Australia). The data obtained were transferred to an industrial AutoCAD program (Autodesk Inc., São Rafael, United States of America) for fabrication (P) in the dimensions of 40mm width x 40mm height x 5mm thickness, being digitally joined to the substrate (Su) in the center of the platform with dimensions 9.4 mm and 17 mm and 6 mm.

From the scanning of (P) and (Su) together, the design of 24 identical injection patterns (C) in laminated facet format of upper right central incisor in blue castable polymer (Supercast v3; Asiga, Sydney, Australia) in the Exocad®-Creator program (Align Technology, Darmstadt, Germany), using the Asiga Max printer (Asiga, Sydney, Australia).

For the injection of the ceramic (C), Empress2 ceramics (IPS e.max Press, Ivoclar Vivadent AG) uniformed on the LT A1 color inserts were used. Cementations were performed with aluminum oxide (50 microns) in the cementation zones of (Su) and then the surfaces were washed with air and water spray for 30 seconds.

Prior to cementation, the specimens underwent physical and chemical preparation: the ceramic surface was first cleaned with 70% alcohol and dried; then applied 9% hydrofluoric acid (Porcelain Etch ®; Ultradent Products Inc., Salt Lake City, United States of America) for 20 seconds, followed by washing with water and air spray for 20 seconds, and then application of acid 35% phosphoric acid (Ultra Etch®; Ultradent Products Inc., Salt Lake City, United States of America) for 60 seconds, under agitation and microbrush friction (Magne & Belser, 2003; Callicchio et al., 2015). Then, the pieces were silanized in the cementation areas with Monobond N® (Ivoclar Vivadent AG, Liechtenstein) and oven dried at 100°C for 60 seconds, receiving a thin layer of adhesive system (Tetric® N-Bond, Ivoclar Vivadent AG, Liechtenstein) and a thin layer of Allcem Veneer APS
(FGM Produtos Odontológicas, Joinville, Brazil) and the entire set was light cured with Bluephase®g2 (Ivoclar Vivadent AG, Liechtenstein) for 35 seconds on both sides at maximum power. The specimens were randomly distributed in 4 groups of 6 specimens, being positioned in the brushing simulator machine, being regulated at an ambient temperature of 37°C ambient, with a frequency of 250 strokes per minute, pressure of the brushes on the 250 mg specimen and simultaneous dripping of 1 liter toothpaste solution for every 7 minutes. The times tested were divided into 25,000 strokes for 2.5 years of brushing, as follows: T0 (= prior to the test); T1 (= 2.5 years); T2 (= 5 years); T3 (= 7.5 years); T4 (= 10 years); T5 (= 12.5 years) and T6 (= 15 years) (Sancakli et al., 2015).

The selection of toothpaste followed four separate criteria for each group in marketing parameters such as: market leadership (Colgate total 12® = CT12), high fluoride concentration (Orthogard® = ORG), low relative dentin abrasion (Elmex® = EMX), and high pH index (Colgate Triple Action® = CTA).

The dentifrice solutions were obtained by using a fraction of 1 liter of distilled water for 250 mg of dentifrice and the pH was measured after dilution (Akso Digital pH meter with 3M KCL). To obtain a homogeneous mixture, a magnetic stirrer (1540/A-15; Cienlab, Campinas, Brazil) was used.

Readings were performed in three axes (A, B and C), with a cut-off of 0.001x 3 μin, were performed on the surface of the specimens using a contact roughness meter. The roughness measurement (Rm) for each specimen was given by the average of the values obtained in each axis. In order to keep the specimen in position, it was placed in an acrylic apparatus to standardize the readings.

For the sample calculation, the average surface roughness (SR) of the glass-ceramics subjected to artificial aging from a pilot (n=3) was considered, with CT12= 14.05; CTA=23.96; EMX=11.52; ORG= 17.39. The pilot study determined the abrasiveness level of 0.78 μin for a significance level of 0.05% and a power of 0.80 μin. The final number of bodies with these characteristics was 6 for each group.

3. Results

The values obtained for surface roughness (SR) were initially submitted to the Kolmogorov-Smirnov test (p=0.321) to verify the normality of the data distribution and, later, to the Levene test (p=0.076), in order to verify the homogeneity of variances. The values obtained were then submitted to two-factor analysis of variance with repeated measures (toothpaste and aging time), and Tukey's test for multiple comparisons.

Table 1 presents the averages and standard deviations of surface roughness (SR) for all experimental groups, looking at the behavior of the standard deviation of the groups CT12 and ORG (a) they were similar from the baseline of the tests to until the conclusion of the times. However, this similarity was also seen in the standard deviation of the CTA and EMX groups (b), being different the standard deviation between them. Analysis of variance showed a statistically significant difference in all toothpastes (p < 0.001). The aging time (p = 0.876), as well as the interaction between the toothpaste and aging time did not show a significant difference (p = 0.991). The aging time did not influence the surface roughness of the glass-ceramics, regardless of the toothpaste used. However, the CT12 and ORG dentifrices resulted in similar values of surface roughness, being lower than the other dentifrices. Furthermore, there was no statistically significant difference between CTA toothpaste and EM.
Table 1. Descriptive variables present types of toothpaste. Colgate Total 12 (CT12), Colgate Triple Action (CTA), Elmex (EMX) and Ortagard (ORG). Mean and standard deviation of the surface roughness values (SR) according to the time related to the experimental group.

<table>
<thead>
<tr>
<th>Dentifrice / Aging time</th>
<th>CT12</th>
<th>CTA</th>
<th>EMX</th>
<th>ORG</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>SR-14,27 (±4,05)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>19,08 (±7,51)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>14,94 (±6,35)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>16,54 (±6,79)&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>2,5 years</td>
<td>15,59 (±4,41)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>18,49 (±7,99)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>18,96 (±8,11)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>17,67 (±7,18)&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>5 years</td>
<td>13,41 (±4,33)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>20,60 (±8,25)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>20,87 (±7,53)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>15,19 (±5,74)&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>7,5 years</td>
<td>15,37 (±4,66)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>23,95 (±9,71)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>19,41 (±5,15)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>15,71 (±6,78)&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>10 years</td>
<td>13,19 (±4,53)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>18,49 (±8,55)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>19,54 (±6,30)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>15,56 (±4,88)&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>12,5 years</td>
<td>13,48 (±2,94)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>18,58 (±9,81)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>19,72 (±6,40)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>15,77 (±5,64)&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>15 years</td>
<td>13,36 (±3,85)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>20,40 (±9,28)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>19,05 (±6,01)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>12,92 (±5,49)&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Superscripted letters (a or b) were placed for groups with similar standard deviations when comparing the results with different toothpastes. Source: Authors.

Table 1 shows the means and standard deviations of surface roughness (SR) for all experimental groups, with standard deviation of the CT12 and ORG groups (a) being similar from the baseline of the tests to until the conclusion of the times. However, this similarity was also seen in the standard deviation of the CTA and EMX groups (b), being different the standard deviation between them. Analysis of variance showed a statistically significant difference in all toothpastes (p < 0.001). The aging time (p = 0.876), as well as the interaction between the toothpaste and the aging time, did not show a significant difference (p = 0.991). The aging time did not influence the surface roughness of the glass-ceramics, regardless of the toothpaste used. However, the CT12 and ORG dentifrices resulted in similar values of surface roughness, being lower than the other dentifrices. Furthermore, there was no statistically significant difference between CTA toothpaste and EM.

Graph 1 represents the average surface roughness for each dentifrice tested, according to aging time. It is possible to observe the similarity of the data within each experimental group, regardless of the aging time.
Graph 1. Distribution of surface roughness averages between the different evaluation times for each experimental group.

The graph shows behavior related to roughness over time when compared to different toothpastes: Colgate total 12® = CT12, Colgate Triple Action® = CTA, Elmex® = EMX, Orthogard® = ORG

Graph 1 lists the degrees of surface roughness, according to the toothpaste groups, over a period of 10 years. With the exception of the CTA group, which showed a roughness peak at 7.5 years, the dentifrices acted as an attack on the ceramic surface in the short term of up to 5 years. In addition, the CTA group was also very different from the other groups, showing an increase in roughness at 15 years of age.

The CTA group recorded higher starting roughness at t0, in relation to the other materials, initially showing stabilization and higher peak roughness. The behavior of the CT12 and ORG groups were very similar.

In the medium term, in the EMX group, there was an increase in roughness after 5 years, with a tendency to maintain the roughness after 7.5 to 15 years. The CT12 and ORG groups initially showed an increase in roughness in the short term (2.5 years), and a decrease in roughness in the medium and long term, with the exception of the CTA group, which had a tendency of increasing roughness up to 7.5 years, and stabilization from 10 to 12.5 years.

4. Discussion

The results showed that all groups tested showed loss of mass since the beginning of the tests. The static data indicated that the interaction of simulated brushing time and pH of the dentifrices used did not influence the IPS Empress2 ceramic surface, but the type of abrasives (dentifrices) used.

Justifying the findings in relation to mass loss, Mühlemann and collaborators (2019) described that the degradation of dental ceramics usually occurs due to mechanical forces or chemical attack. In addition to highlighting that chemical exposure to alkaline solutions at pH increases the degradation process. Therefore, common dentifrice substances for clinical use were used in this work, all with alkaline pH, ranging from 9 to 12.9, which corroborates the loss of mass.

Several authors have observed that mechanical degradation from mastication and abrasion by brushing can compromise ceramic surfaces (Nogués et al., 2008; Heintze et al., 2010; Castro et al., 2014; Garza et al., 2016; Yuan et al., 2018; Mühlemann et al., 2019). However, according to Hsu and collaborators (2020) mechanical and chemical degradation must be considered in every process related to corrosion.
Corrosion studies of ceramic surfaces are widely performed for restorative processes in ceramics, these protocols are fundamental for the micoretention of restorative materials. The use of hydrofluoric acid, which causes the removal of the glass phase (glass matrix) and exposure of existing crystals in the composition of the ceramic, provides high adhesiveness (Magne & Belser, 2003; Callicchio et al., 2015). The vitreous matrix is present both in the body of the IPS Empress2 structure and in the fine finishing phase by the glaze, a glass-ceramic substance with little or no crystalline portion, used to seal the porosity of the sintering process of this restorative material. Glaze being the main target of study of corrosion protection and maintenance of surface integrity of restorations (Garza et al., 2016; Flury et al., 2017; Yuan et al., 2018).

Castro et al. (2014) evaluated roughness parameters and observed that the stability of polished ceramics showed differences after 10 years and glazed ceramics began to show roughness after 2 years on average.

The results also showed that there was no statistically significant difference in terms of aging time (p = 0.991), a finding similar to that found by authors who did not observe substantial loss and great durability of ceramics (Heintze et al., 2010; Rosentritt et al., 2015; Garza et al., 2016; Flury et al., 2017; Yuan et al., 2018). Nogués and collaborators (2008) identified a substantial loss, relating the abrasiveness of toothpastes as responsible after artificial aging, similar to our findings that revealed an initial loss at 2.5 years and stability after 7.5 years.

The abrasiveness existing in Brazilian toothpastes serves to enhance brushing. According to ANVISA (RDC n° 04, of 01/30/2014), the degrees of abrasiveness of toothpastes are divided into five groups: antiplaque; anti-tartar; bleach; sensitive teeth and children. According ISO 11609, the acceptable pH can vary between 4.5 and 10.5 (Rios et al., 2014). In this study, the non-significance between pH and abrasiveness, when compared with different toothpastes, is related to another methodological form used in this work. The substance to acidify and basify the pH was applied in the form of a drip, at a temperature of 37ºC. Previous studies evaluated the deterioration of glass ceramics by constant immersion in the solution for pH change. In these studies, the groups showed mass loss at different pHs (Esquivel-Upshaw et al., 2013; Esquivel-Upshaw et al., 2018; Hsu et al., 2020). The present work reveals an initial tendency of mass loss, which is evident in the low-abrasion EMX group, corroborating the findings of Hsu and collaborators (2020), who observed a loss of Si4+, K+, Na+, Al3+, and Ca2+ ions at pH 10, the same EMX toothpaste pH.

Nogués and collaborators (2008) observed the effect of abrasiveness on several materials, pointing out that titanium was the most susceptible, and ceramics the least, a finding similar to those previously described (Heintze et al., 2010; Rosentritt et al., 2015; Garza et al., 2016; Flury et al., 2017; Yuan et al., 2018).

In graph 1, it is possible to observe that CT12 and ORG presented greater abrasiveness, highlighting the abrasive action of the toothpaste in the removal of structural mass, even in the short term (2.5 years), with a tendency to stabilize and decrease the roughness after 7.5 years. In addition, it is possible to observe that the CTA group has a high manufacturing roughness, and had a large increase in its roughness after 7.5 years, with a sharp decrease in its roughness after 10 years of brushing, behaving with an initial alteration and subsequent polishing.

This behavior of decreasing roughness over time is seen in all groups, with the exception of EMX, in which roughness was maintained after its first increase in roughness. The EMX group is a group that used low dentifrice abrasiveness, suggesting that there was an initial chemical attack, and the low abrasiveness did not change the roughness over time.

The importance of the study of roughness in restorative materials can jeopardize the maintenance of health of the surrounding tissues, as reported in previous studies with bacterial adhesion on prosthetic surfaces (Bollen et al., 1997; Glavina et al., 2004; Al-Marzok & Al-Azzawi, 2009). The effects on the opposing dentition were also studied by Fathy & Swain (2018), in in vitro simulated chewing tests with a 1-year projection, and observed in 100x magnification images many prominent wear scars on the enamel tip of the antagonist tooth, highlighting the importance of in-office polishing, due to the exposure of the lens in the form of high-resistance needles.
Previous studies revealed that the breakdown of the silicon network in glass-ceramics and the mobilization of ions is greater when the initial pH is alkaline by immersion, with oscillation in acidified pH (Esquivel-Upshawa et al., 2013; Esquivel-Upshawb et al., 2018). This condition is important because the pH in the oral environment is fluctuating, and there is a tendency for stabilization by the action of saliva, both for acidic and alkaline pH, due to ionic interactions by hydrons in the oral environment. Hsu and collaborators (2020) describe in their studies that at fluctuating pH, the corrosion process is not interrupted, breaking the entire silicon network of the ceramic and more ionic release of ions to the exposed medium.

Fluoride has been questioned about its action on the silicon mesh of ceramics, however, little is said about the pH of these substances, whose topical use is present in the acidulated form. Butler et al. (2004) e Mühlemann et al. (2019).

However, the literature shows us two substances capable of promoting changes in the roughness of ceramics: the very acidic and the very alkaline, thus, the role of acidulated fluorine on these surfaces still raises doubts. If we observe our results with the high fluoride group (ORG), it was not responsible for the aggression of the ceramic surface, its behavior did not show any unusual factor in the tests, on the contrary, it proved to be very similar to conventional toothpastes such as CT12 , which may indicate that acidity is responsible for damage to the ceramic, and not the presence of fluorine.

In parallel, endogenous acidic substances also gain emphasis as responsible for the increase in roughness. Alnasser and collaborators (2019) and Kulkarni et al., (2020) studied the action of the gastric sulcus (acid pH) on glass ceramics, observing changes on surfaces with immediate etching after exposure to hydrochloric acid. Foods were also studied in relation to ceramic roughness, Firouz and collaborators (2019) evaluated the effects of three commonly consumed acidic beverages on the surface roughness of glass-ceramics, highlighting an increase in their roughness.

Previous studies with low and ultra-low fusion porcelain, showed that ceramics deteriorate by: acidulated fluorine and 10% carbamide peroxide at pH 5.76, worsening its acidity in 22% solutions at pH 5.74 (Butler et al., 2004; Silva et al., 2012). This fact indicates the factor of the action of acidity on ceramic roughness, which can extrapolate the data to a clinical view and extend care to our daily lives.

The control of roughness in a clinical environment by the study of final polishing systems on ceramic veneers in a clinical set concluded that the clinician achieves desirable surfaces, in addition to being more durable to abrasion when glazed and polished. (Glavina et al., 2004; Castro et al., 2014; Garza et al., 2016; Flury et al., 2017).

In general, the present in vitro study showed great strength and durability of IPS Empress2 ceramic, polished and glazed under the abrasive action of toothpastes, at a pressure of 250mg for 15 years. However, as a limitation of the study, factors were not considered as the oscillatory action of pH (representing the oral environment), aging by UV, thermal baths in cycles and changes in shade over time, being suggested for future studies.

Thus, it is necessary to implement new studies and new practices for testing restorative materials. However, the major problem is the difficulty of access and the high cost of these technologies, which are always carried out in high standard laboratories.

5. Conclusion

Given the methodology used and the delimitation of the study, the glass ceramic surfaces (IPS e.max Press, Ivoclar Vivadent AG) proved to be resistant in a period of 15 years in relation to simulated brushing, as well as resistant to the action of different dentifrices.

References


