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EFEITO DO REFLUXO GASTROESOFÁGICO SIMULADO EM MATERIAIS RESTAURADORES

EFFECT OF SIMULATED GASTROESOPHAGEAL REFLUX ON RESTORATIVE MATERIALS

PIRACICABA

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Trabalho de Conclusão de Curso apresentado à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para obtenção do título de Cirurgião Dentista.

Undergraduate final work presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Dental Surgeon

Orientador: Prof. Dr. Marcelo Giannini

Coorientadora: Doutoranda Amanda Endres Willers

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RESUMO

Este estudo avaliou o efeito do refluxo gastroesofágico simulado (RGES) nas superfícies de 5 materiais restauradores (Admira Fusion, Activa BioActive-Restorative, Charisma, EQUIA Forte HT Fil/EF e Filtek Universal Restorative/FU). Dez amostras em formato de placas (6 x 6 x 1 mm) foram preparadas para cada material. Metade da superfície das amostras foi protegida com fita adesiva, criando uma área controle e outra submetida ao RGES (5 mL de HCl 0,06M, pH 1,2, a 37ºC, por 30 horas). As amostras foram analisadas quanto à Rugosidade de Superfície (Sa), ao Perfil de Rugosidade (Rv) e à Perda de Volume (SL) em Microscopia Confocal (n=10). A Microdureza (MI) e o Brilho de Superfície (SG) foram determinados por indentador Knoop e aparelho medidor de brilho (n=10). A topografia foi avaliada por MEV e a identificação dos elementos químicos por EDX (n=5). Os dados foram analisados por ANOVA e Teste de Tukey (α=0,05). A maioria dos materiais não foram afetados pelo RGES. FU apresentou menores valores de Sa e Rv, e maiores valores de SG após o RGES. Contrariamente, EF mostrou maiores valores de Sa, Rv e SL, e o menor SG. A MI dos materiais não foi alterada com RGES. Alterações morfológicas e dos elementos químicos foram observados após RGES apenas para o material ionomérico (EF). Os materiais restauradores resinosos apresentaram mínimas alterações superficiais após RGES, que não foi observado para o material ionomérico (EF). FU apresentou a melhor performance segundo os parâmetros avaliados.

Palavras-chave: Dentes - Erosão. Resina composta. Cimento de ionômero de vidro.

ABSTRACT

This study evaluated the effect of simulated gastroesophageal reflux (SGER) on the surfaces of 5 restorative materials (Admira Fusion, Activa BioActive-Restorative, Charisma, Equia Forte HT Fil/EF, Filtek Universal Restorative/FU). Ten samples in the shape of plates (6 x 6 x 1 mm) were obtained for each material. Half of the plate surfaces were covered with adhesive tape, creating a control area, and the other side was submitted to the SGER (0.06 M HCl; pH 1.2; at 37ºC; for 30 hours). The samples were analyzed regarding the Surface Roughness (Sa), Roughness Profile (Rv) and Surface Loss (SL) using a confocal microscope (n=10). The surface microhardness (MI) and gloss (SG) were determined through Knoop and a glossmeter (n=10). Surface morphology was analyzed by SEM and chemical elements were identified by EDX (n=5). The data was analyzed by ANOVA and Tukey's Test (α=0.05). Most materials were not affected by SGER. FU showed the lowest Sa and Rv, and the highest SG after SGER. On the other hand, EF presented the highest Sa, Rv and SL, and the lowest SG. The MI of the materials was not changed after SGER. Topographical and chemical element alterations were observed after SGER only for EF. The resin-based restorative materials showed minor surface changes after SGER, which was not observed for the glass ionomer cement (EF). FU presented the better performance regarding the parameters evaluated.

Key words: Teeth - Erosion. Composite resin. Glass ionomer cement.

SUMÁRIO

1 INTRODUÇÃO

Quando um procedimento restaurador é realizado na cavidade oral, devemos levar em consideração as situações às quais a restauração será exposta. Uma dessas situações a ser considerada é o processo erosivo, ou seja, dissolução ácida da estrutura dental sem o envolvimento de bactérias (Roque et al., 2015). Esses ácidos presentes no meio oral podem ter duas origens: extrínseca, vinda de alimentos e bebidas ácidas (ácido cítrico principalmente); ou intrínseca, originária de distúrbios alimentares e refluxos gastroesofágicos (ácido clorídrico) (Tantanuch et al., 2016).

O problema relacionado aos indivíduos que sofrem com refluxo gastroesofágico (regurgitação involuntária e repetitiva) está no contato com o ácido clorídrico, oriundo do trato gastroesofágico, o qual permanece na cavidade oral por um longo período, gerando uma grave perda na estrutura dental por meio da dissolução ácida (Hengtrakool et al., 2011). Quando regularmente expostos a regurgitação, um padrão é observado na cavidade oral desses indivíduos, haja que, há erosão nas superfícies palatinas dos dentes anteriores superiores e/ou nas superfícies oclusais dos dentes posteriores (Backer, et al., 2017).

Em situações em que a perda da estrutura dentária atinge uma gravidade funcional elevada, a reabilitação dos dentes se torna indispensável. A escolha do material restaurador adequado desempenha um papel extremamente relevante na longevidade da restauração, pois da mesma forma que os ácidos prejudicam a superfície dos dentes, eles também podem afetar os compósitos restauradores (Guler, et al., 2018). Resinas compostas fornecem excelentes propriedades para restaurações diretas, incluindo resistência ao desgaste e estética, no entanto, a erosão pode danificar as propriedades físicas e químicas dos compósitos, levando à degradação da matriz orgânica e à exposição do conteúdo inorgânico (Correr et al., 2012). Estas alterações aumentam a rugosidade superficial, a qual reduz a longevidade da restauração e afeta o seu desempenho a longo prazo (Roque et al., 2015; Backer, et al., 2017). A severa degradação dos materiais restauradores é devido ao baixo pH (1-3) dos ácidos endógenos (ácido clorídrico) presentes no ambiente oral de indivíduos com distúrbios alimentares e refluxos gastresofágicos (Backer, et al., 2017).

Os compósitos restauradores diretos contemporâneos apresentam composições variadas frente ao desenvolvimento de diferentes novos monômeros que compõem a matriz resinosa, além dos diferentes tipos de partículas de carga, os quais são resultados da evolução deste tipo material restaurador odontológico nos últimos anos. A composição monomérica básica é aquela que apresenta Bis-GMA, TEG-DMA, UDMA e outros di- e metacrilatos. Outros compósitos apresentam monômeros chamados de “alternativos” ou de “nova geração”, os quais representam os novos compósitos comerciais de “baixa contração”, do tipo “bulkfill”, à base de Ormocer, “Universais” e outros. Com relação aos tipos de partículas, elas podem variar com relação ao tamanho, ao formato e à composição. A sílica, e os vidros de bário, alumínio, boro e estrôncio são as partículas de carga mais comuns, entretanto fluorosilicato, trifluoreto de itérbio, óxidos como zircônio combinado com sílica, fibras de vidro e pré-polímeros também podem ser encontrados nos produtos comerciais (Yamasaki et al., 2013; Bacchi et al., 2015; Fronza et al., 2015; Kruly et al., 2018).

Frente a esta grande variabilidade de materiais, é possível esperar diferentes comportamentos quando expostos ao ácido gástrico. Considerando o assunto atual sobre o refluxo gastroesofágico e a variabilidade de compósitos restauradores, o atual projeto de pesquisa avaliou o impacto do desafio erosivo intrínseco simulado sobre a superfície de 5 materiais restauradores de diferentes composições monoméricas e de partículas de carga, sendo um deles o material de composição regular que serviu como “controle” reproduzindo aquilo que ocorre com os materiais tradicionais e os demais materiais que foram comparados ao controle apresentam características composicionais diferentes, representando materiais restauradores contemporâneos e de composição “alternativa”. Foram avaliados quanto à rugosidade de superfície (Sa), perda de volume (PV), perfil de rugosidade (Rv), microdureza (KN), brilho de superfície (BS), topografia de superfície (TS) e composição elementar (EDX).

2 ARTIGO: EFFECT OF SIMULATED GASTROESOPHAGEAL REFLUX ON RESTORATIVE MATERIALS.

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ABSTRACT

Objective: To evaluate the effect of simulated gastroesophageal reflux disease (GERD) on the surface of restorative materials.

Material and Methods: Ten plates (6 mm x 6mm x 1 mm / plates) of five restorative materials (Admira Fusion, Activa BioActive-Restorative, Charisma, Equia Forte HT Fil/EF, Filtek Universal Restorative/FU) were obtained. Half of the plate surfaces was covered with an adhesive tape, creating a Control area, and the other side was submitted to the GERD (0.06 M HCl; pH 1.2; at 37ºC; for 30 hours). Plates with Control and HCl-treated areas were analyzed regarding the Surface Roughness (Sa), Roughness Profile (Rv) and Surface Loss (SL) using a confocal microscope (n=10). The surface microhardness (MI) and gloss (GL) were determined for Control and HCl-treated areas, using Knoop indenter and glossmeter, respectively (n=10). Surface morphology was analyzed by SEM and chemical elements were identified by EDX (n=5). The obtained data were evaluated by ANOVA and Tukey's Test (α=0.05).

Results: Most restorative materials were not affected by GERD. FU showed the lowest Sa and Rv, and the highest GL after GERD. On the other hand, EF presented the highest Sa, Rv and SL, and the lowest GL. The MI of materials was not changed after GERD. Topographical and chemical element alterations were observed after GERD only for EF.

Conclusions: The resin-based restorative materials showed minor surface changes after GERD, which was not observed for the glass ionomer cement (EF). FU presented the better performance regarding the parameters evaluated.

CLINICAL SIGNIFICANCE:

The effects of simulated erosive challenge with HCl on regular restorative composites were minimal, while the glass ionomer cements might be not indicated as restorative material for patients with gastroesophageal reflux disease.

KEYWORDS

Reflux. Erosion. Gastric Acid. Composite. Compomer. Glass Ionomer.

INTRODUCTION

Recent studies have described the gastroesophageal reflux disease (GERD) [1-3]. An isolate episode of acid reflux into the oral cavity does not lead to a pathological condition; however, if reflux episodes occur on a regular basis over a long period, they are characterized as gastroesophageal reflux disease [4]. This repetitive gastroesophageal reflux can generate the intrinsic dental erosion because the gastric acid (hydrochloric acid and pepsin) [5] has a low pH, around 1.2 [6].

A previous study has compared the erosive potential of gastric acid with a soft drink, observing that gastric acid has a greater potential for erosion than carbonated drinks [7]. Thus, the hydrochloric acid from the gastroesophageal reflux may be responsible for a severe mineralized dental tissues loss when it remains in contact with the oral cavity for a long time [8]. If tooth structure loss reaches a high severity, functional and esthetic rehabilitation of the teeth are required [9].

Thus, the choice of an adequate restorative material plays an extremely relevant role in the durability of the restoration [10]. Composite resin provides excellent properties for restoration [11]; however, erosive challenge can damage the physical and mechanical properties of them [12], leading to organic matrix degradation and exposure of the inorganic filler particles [13]. These changes can lead to morphological changes and increase of surface roughness, which are responsible for the decrease in restoration durability [11,14]. It is essential to choose a material suitable for restoring the teeth of patients with gastroesophageal reflux in order to ensure longevity of the restorations [10].

The bisphenol A-glycidyl methacrylate (Bis-GMA) is the resin monomer commonly used in dental resin composites [15]. However, new types of organic matrix for composites have been developed, such as the Ormocer (organically modified ceramic) [16]. Besides the organic matrix, it is known that the size, shape, and concentration of the filler particles greatly affect the final surface of the restorations, in terms of roughness and gloss [10]. Different types of filler particles are found in the commercial dental composites such as the traditional glasses, nanofillers and nanoceramics, which guarantee the possibility to obtain high-quality restoration surface [17].

Besides the restorative composites, the glass ionomer cement and hybrid materials can be also used. Glass ionomer cements are fluoride releasing materials and some manufacturers claim that this material can be used in load-bearing class II restorations in addition to class I and V restorations. It has been reported that new glass ionomer cements exhibit increased flexural strength and resistance to wear and acid erosion [18]. Moreover, hybrid materials were also introduced to overcome the problem associated with conventional glass ionomers and composite resins, maintaining their clinical advantages [19]. Bioactive restorative materials are examples of hybrid ones and present a specific biological response at the dentin-resin interface [20]. These materials are expected to release some ions, such as calcium, phosphate, and fluoride and seem to be more bioactive than glass ionomers and traditional resin-modified glass ionomer [21].

Concerning the rehabilitation of patients with GERD, the aim of this study was to evaluate the effect of simulated gastric acid on restorative materials with different compositions, regarding the surface roughness (Sa), roughness profile (Rv), surface loss (SL), microhardness (MI), gloss (GL), surface topography (ST) and chemical elementary composition. The tested hypothesis is that the simulated GERD would impact differently the studied properties of materials of varied compositions.

MATERIAL AND METHODS

A total of ten standardized plates (6 mm length x 6 mm wide x 2 mm thick) were obtained for each restorative material (Table 1). Resin-based materials were placed into silicon molds and were light activated by a polywave LED-curing unit (Valo, Ultradent Product Inc., South Jordan, UT, USA) for 20 seconds (886 mW/cm^2 irradiance measured by Demetron LED Radiometers, Kerr Corp., Brea, CA, USA). For the glass ionomer material (EQUIA Forte HT Fil / EF), the capsule was mixed for 10 seconds, inserted into applier and the material was placed and filled into the silicon molds. The EF surface was finished by applying EQUIA Forte

Coat that was light activated for 20 seconds. The hybrid material (Activa BioActive-Restorative / AB) was inserted with a mixing tip on the automix syringe into the mold silicone.

Table 1. Restorative Materials used in this study.

Material / Abbreviation	Composition	Manufacturer	Lot Number
Admira Fusion (AF)	Filler particles (84 wt%): barium, aluminum, organically modified silicic acid (10-25%), silicon oxide. Matrix: ORMOCER (Organically Modified Ceramic).	Voco GmbH, Germany	1805413
Activa BioActive-Restorative (AB)	Powder: silanated bioactive glass and calcium, silanated silica, sodium fluoride. Liquid: Urethane dimethacrylate (UDMA) modified by the insertion of a hydrogenated polybutadiene and other methacrylate monomers, modified polyacrylic acid.	Pulpdent, MA, USA	190523
Charisma (CH)	Filler particles (81 wt% / 64 vol%): Barium, aluminum fluoride glass, silicon oxide. Matrix: Bisphenol A glycidyl methacrylate (Bis-GMA)	Kulzer, Germany	K010723
EQUIA Forte HT Fil (EF)	Powder: Strontium fluoroaluminosilicate glass, polyacrylic acid, iron oxide. Liquid: Polybasic carboxylic acid, water.	GC Corp., Tokyo, Japan	1905271
Filtek Universal Restorative (FU)	Filler particles (76.5% wt% / 58.4 vol%): Silica (20 nm), zirconia (4 to 11 nm), zirconia/silica compound, ytterbium trifluoride composed of agglomerated particles (100 nm). Matrix: Aromatic urethane dimethacrylate (AUDMA), addition-fragmentation monomer (AFM), 1,12-dodecane-DMA.	3M Oral Care, MN, USA	NA20159

All plates were flattened and polished using a polishing machine (Arotec Ind. Com., São Paulo, SP, Brazil) with silicon carbide papers (600, 1000, and 1200-grit, Norton, Vinhedo, SP, Brazil) under 220 g axial load and water-cooling for 20 seconds for each sandpaper, and ultrasonic cleaned (USC 1400, Unique Ind. Com. Prod. Eletr. Ltda, Indaiatuba, SP, Brazil) with distilled water for five minutes.

A three millimeters wide strip of polyvinyl chloride adhesive tape (Graphic Tape; Chartpack Inc., Leeds, MA, USA) was adhered to one side of the plates' surface, creating a Control area, which corresponded to half of the plate and protected it against the erosion challenge.

To simulate the erosive challenge, a protocol was performed with a 0.06 M solution of hydrochloric acid (aqueous solution of 0.113% HCl, pH 1.2). Each plate was immersed in 5 mL HCl for 30 hours in an incubator at 37 ºC with the test surface facing upwards. Afterwards, plates were washed with distilled water for one minute, the adhesive tape was removed, and the plates were ultrasonically cleaned for five minutes with distilled water.

Surface Roughness (Sa), Roughness Profile (Rv) and Surface Loss (SL)

Sa, Rv and SL were analyzed using a Confocal Microscope (LEXT 3D Measuring Laser Microscope OLS4000, Olympus Corp., Tokyo, Japan) (n = 10). It was used a 5x objective lens (1x Zoom) to obtain images (1024 x 1024 pixels, XYZ fast scan), with a 405 nm laser (Gaussian Filter). Additionally, representative 3D images were obtained to compare the control and treated areas (submitted to HCl) of the plates.

For Sa, that assesses the average arithmetic mean deviation of the roughness profile of an area of the plate, images of the control and treated areas were obtained separately in each plate. Rv was determined by measuring the largest valley depth deviation from the mean line within a plate length (10 readings on each image were performed). For SL, a reference flat at the top of the control area was defined and then the software calculated the loss located bellow this control area.

Microhardness Determination

Surface MI (n = 10) was analyzed in the control and treated areas, with a microhardness machine (HMV-2000; Shimadzu Corp., Tokyo, Japan) using a Knoop indenter (load of 50 grams for 10 seconds). Three equidistant indentations, 100 micrometers from the center of each area of the plates, were performed and the MI mean of these values were obtained.

Gloss Evaluation

The gloss unit (n = 10) was analyzed with a Glossmeter (Novo-Curve, Rhopoint Instruments) under 60º-illumination angle. The device had a 4.5 mm aperture that was calibrated (gloss unit: 93.3) before using. Three equidistant measurements were performed on each area of the plate (Control and treated one) and the mean of these measurements were obtained.

Surface Topography and Chemical Elementary Composition Evaluations

Plates (n = 5) were covered by carbon (MED 010 Baltec, Balzers, Liechtenstein) and then analyzed by a scanning electron microscopy (SEM) (JEOL, JSM-5600LV, Tokyo, Japan) to characterize the surface topography, with a voltage of 15 kV, beam width of 25 to 30 nm, working distance of 10 to 15 mm and magnifications of 500x. Representative images were obtained for each material (Control and tested areas) and were used for qualitative analysis of the surface.

To identify the elementary chemical composition of the restorative materials, an X-ray scattering spectroscopy (EDX) (Vantage, NORAN Instruments, Middleton, WI, USA) coupled to the SEM was used. Each EDX spectrum was acquired for 100 s (voltage 15 kV, dead time 20-25%, working distance 20 mm) and images of each area of the plates were obtained to identify the chemical elements, with their relative concentration.

Statistical Analysis

Data were submitted to exploratory analysis (SPSS Statistics Version 23, IBM Corp, Armonk, New York, USA), presenting normal distribution and homoscedasticity (Shapiro-Wilk Test). The different evaluations were assessed with the following parametric tests: Sa and gloss with two-way analysis of variance (ANOVA, “Treatment” and “Restorative Material” factors) repeated measures and Tukey’s post-hoc Test ($p<0.05$). Rv, MI and SL with one-way ANOVA ("Restorative Material" factor) and Tukey's post-hoc Test (p<0.05). Images obtained by SEM and EDX were qualitatively analyzed.

RESULTS

Surface Roughness (Sa), Roughness Profile (Rv) and Surface Loss (SL)

Statistical results indicated that both Treatment (p<0.001) and Restorative Material (p<0.001) factors significantly influenced Sa results, with significant interaction between them (p<0.001). For most restorative materials, the treatment with HCl did not change the Sa, except for the EF, which showed significant increase in Sa. Comparing the materials, FU presented the lowest Sa values (0.19 ± 0.08 μm^2 and 0.17 ± 0.06 μm^2 for Control and treated areas, respectively), while EF the highest one (2.14 ± 0.29 μm^2 and 3.44 ± 0.37 μm^2 for Control and treated areas, respectively), regardless the HCl treatment. Other materials (AF, AB and CH) showed intermediate results that ranged from 0.53 ± 0.08 μm^2 to 1.01 ± 0.03 μm^2 (Table 2).

Table 2. Means and standard deviation of Sa values (μm^2) obtained for each material.

Restorative Material	Surface Area	
	Control	HCl-treated
AF	0.53 ± 0.08 Da	0.53 ± 0.13 Da
AB	1.38 ± 0.21 Ba	1.24 ± 0.17 Ba
CH	1.01 ± 0.03 Ca	1.02 ± 0.05 Ca
EF	2.14 ± 0.29 Aa	3.44 ± 0.37 Ab
FU	0.19 ± 0.08 Ea	0.17 ± 0.06 Ea

Means followed by different uppercase letters (column: comparing materials within the same surface area) and different lowercase letters (row: comparing surface areas for the same the material) present statistically significant difference (p < 0.05).

For Rv, ANOVA detected that the results were significantly influenced by the Restorative Material factor (p<0.001), with EF showing the highest Rv value (21.8 ± 2.6 μm) and FU the lowest one (0.3 ± 0.1 μm). Other materials (AF, AB and CH) showed intermediate results that ranged from 0.53 to 1.01 μm (Table 3).

Table 3. Means and standard deviation of Rv values (μm) obtained for each material.

Restorative Material	Roughness Profile
AF	1.5 ± 0.4 C
AB	4.3 ± 0.5 B
CH	3.5 ± 0.3 B
EF	21.8 ± 2.6 A
FU	0.3 ± 0.1 D

Means followed by different uppercase letters present statistically significant difference among restorative materials ($p < 0.05$).

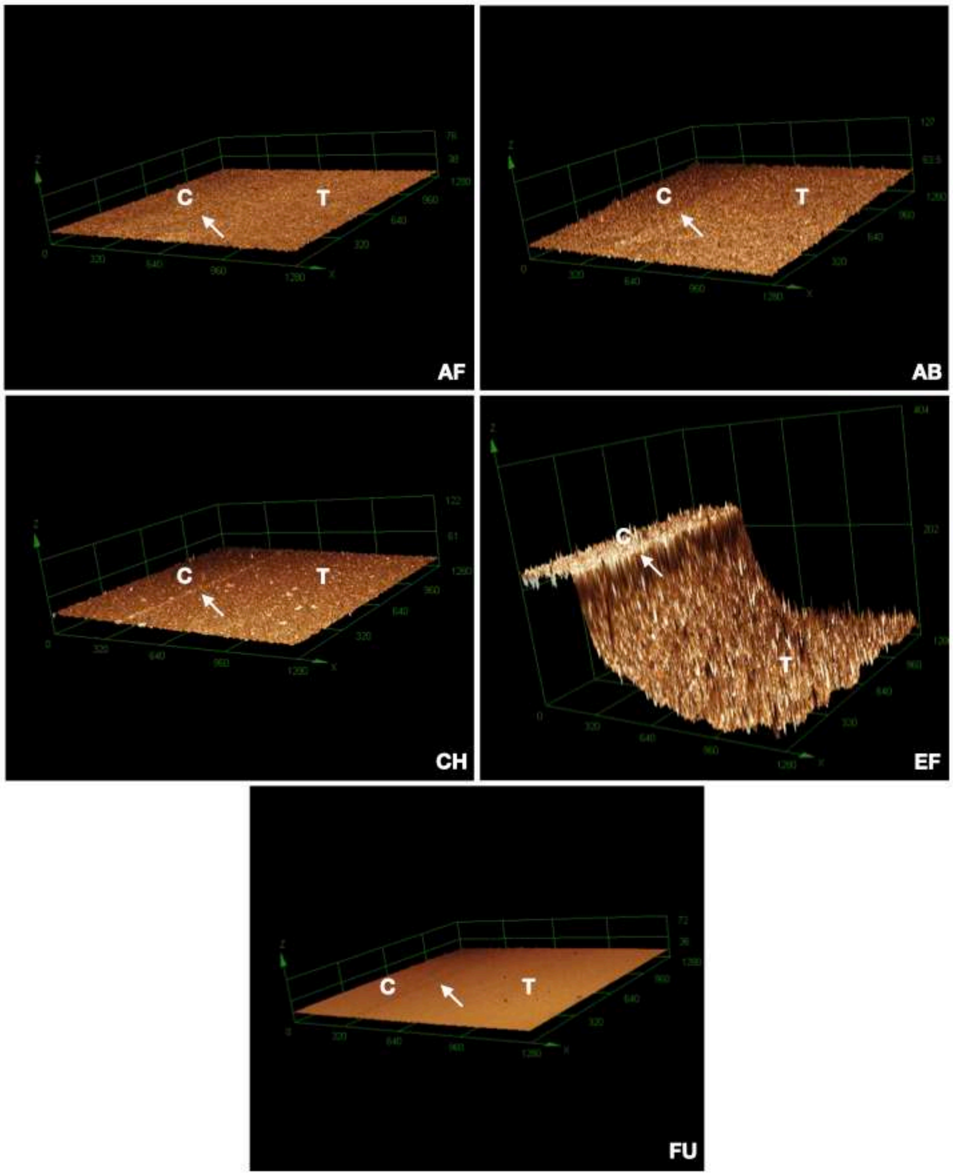
For SL, the Restorative Material factor ($p<0.001$) influenced the results, with most materials did not differ among them, except for EF, which presented the highest SL (0.2250 ± 0.0245 mm^3). Other restorative materials showed lower SL results that ranged from 0.0006 ± 0.0001 mm^3 to 0.0042 ± 0.0006 mm^3 (Table 4). Representative 3D images of the Control and HCl-treated surfaces of all tested materials obtained with the confocal microscope are shown in Figure 1.

Table 4. Means and standard deviation of SL values (mm^3) obtained for each material.

Restorative Material	Surface Loss
AF	0.0038 ± 0.0007 B
AB	0.0027 ± 0.0006 B
CH	0.0042 ± 0.0006 B
EF	0.2250 ± 0.0245 A
FU	0.0006 ± 0.0001 B

Means followed by different uppercase letters present statistically significant difference among restorative materials ($p < 0.05$).

Image 1. Three-dimensional confocal images showing Control (left side) and HCl-treated (right side) areas of restorative materials.



Abbreviations: C: Control Area, T: HCl-Treated Area, AF: Admira Fusion, AB: Activa BioActive-Restorative, CH: Charisma, EF: Equia Forte HT Fil, FU: Filtek Universal Restorative

* Arrow: Indicates the border between Control and Treated areas.

Microhardness Determination

Statistical results indicated that Treatment factor ($p<0.001$) did not significantly influence MI results, that is, the MI was not altered by HCl treatment that simulated gastroesophageal reflux (Table 5). The comparison among materials was not investigated, because of the variety among the compositions of materials. Also, the MI for ionomeric material was not determined, because of its composition with large particles was incompatible with the MI methodology.

Table 5. Means and standard deviation of Microhardness values (KHN) obtained for each material.

Restorative Material	Surface Area	
	Control	HCl-treated
AF	55.2 (7.4) A	54.2 (6.1) A
AB	18.7 (1.4) A	19.7 (1.7) A
CH	46.1 (4.5) A	45.3 (3.7) A
FU	57.9 (3.8) A	57.2 (4.4) A

Means followed by the same uppercase letters in row showed that Control and HCl-treated areas did not differ between them (*p* < 0.05).

Gloss Evaluation

Statistical results indicated that both Treatment (p<0.001) and Restorative Material (p<0.001) factors significantly influenced on the gloss results, with significant interaction between the factors (p<0.001). The treatment with HCl did not significantly change the gloss for most restorative materials, except for EF, which showed significant gloss decrease when exposed to the simulated erosive challenge (from 2.9 ± 0.6 GU to 1.4 ± 0.1 GU). FU presented the highest gloss (64.6 ± 7.2 GU), while EF showed the lowest one. AF, AB, and CH restorative materials showed intermediate results that ranged from 23.4 ± 5.5 GU for CH/Control to 6.6 ± 2.8 GU for AF/Control (Table 6).

Table 6. Means and standard deviation of Gloss (GU) values obtained for each material.

Restorative Material	Surface Area	
	Control	HCl-treated
AF	6.6 ± 2.8 Ca	7.0 ± 2.5 Ca
AB	7.5 ± 2.1 Ca	7.5 ± 1.8 Ca

CH	23.4 ± 5.5 Ba	20.5 ± 3.5 Ba
EF	2.9 ± 0.6 Da	1.4 ± 0.1 Db
FU	64.6 ± 7.2 Aa	63.5 ± 7.7 Aa

Means followed by different uppercase letters (column: comparing materials within the same surface area) and different lowercase letters (row: comparing surface areas for same the material) present statistically significant difference ($p < 0.05$)

Surface Topography and Chemical Elementary Composition Evaluations

Figures 2 to 6 show the SEM and EDX mapping images for the Control and HCL-treated areas of all restorative materials. Topography changes after HCl treatment were detected only for EF, which showed an irregular surface as a result of the erosion process (Figure 5D).

Figure 2. Representative SEM and EDX mapping images showing the Control (A and B, respectively) and HCl-treated (C and D, respectively) areas for AF restorative material.

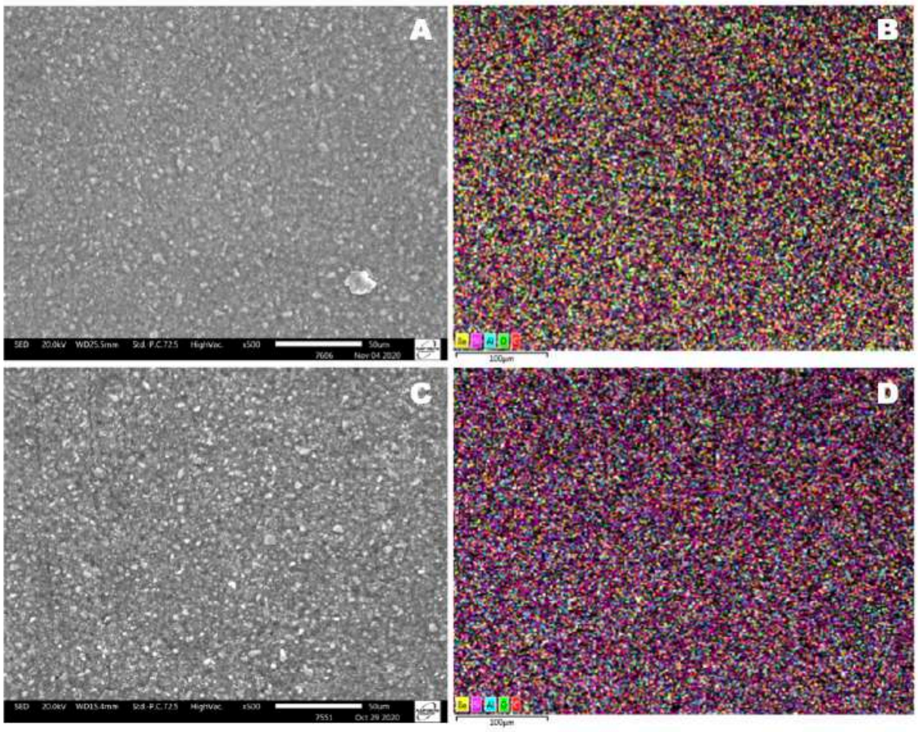


Figure 3. Representative SEM and EDX mapping images showing the Control (A and B, respectively) and HCl-treated (C and D, respectively) areas for AB restorative material.

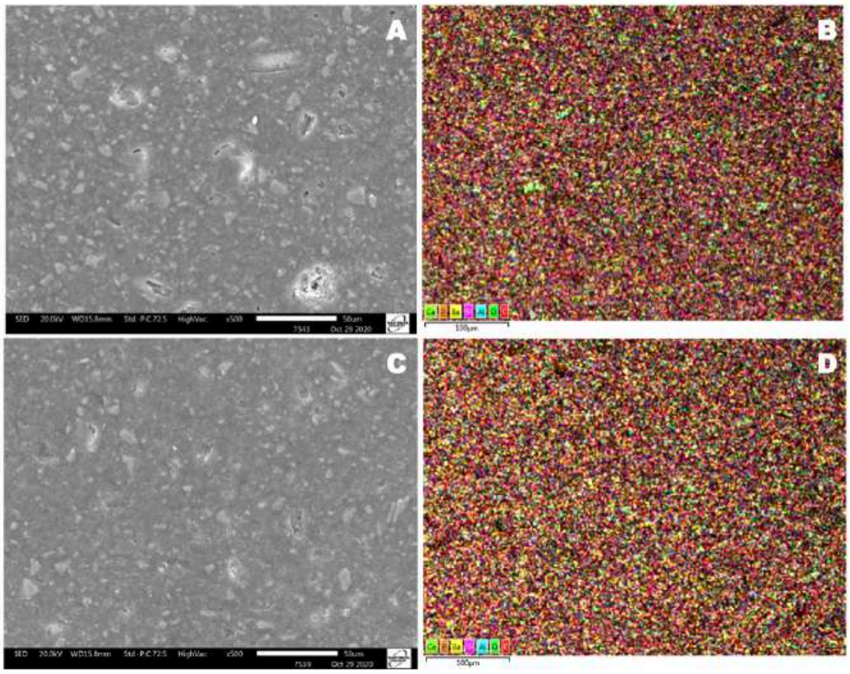


Figure 4. Representative SEM and EDX mapping images showing the Control (A and B, respectively) and HCl-treated (C and D, respectively) areas for CH restorative material.

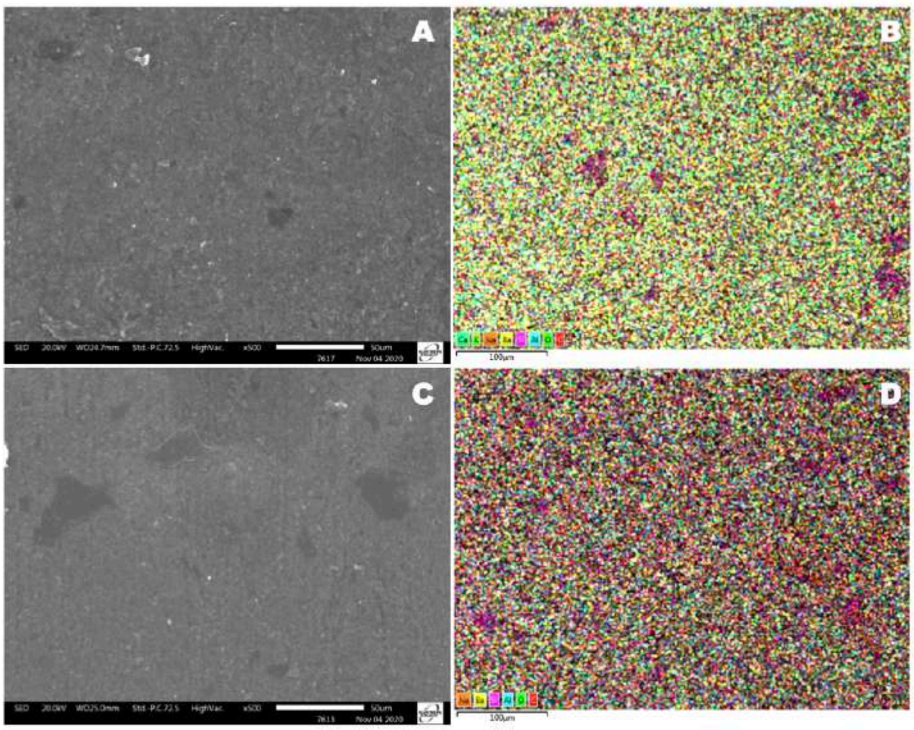


Figure 5. Representative SEM and EDX mapping images showing the Control (A and B, respectively) and HCl-treated (C and D, respectively) areas for EF restorative material.

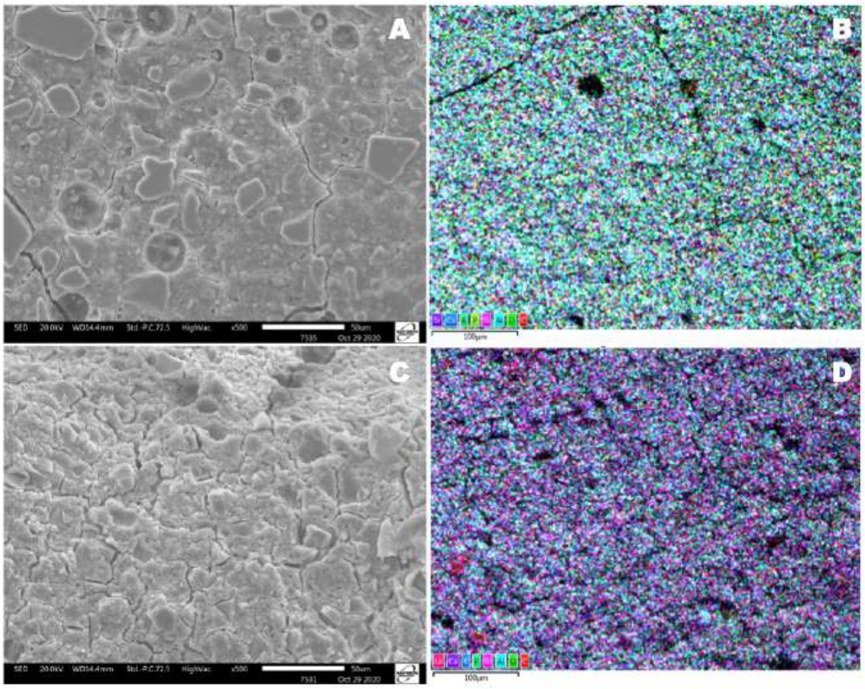
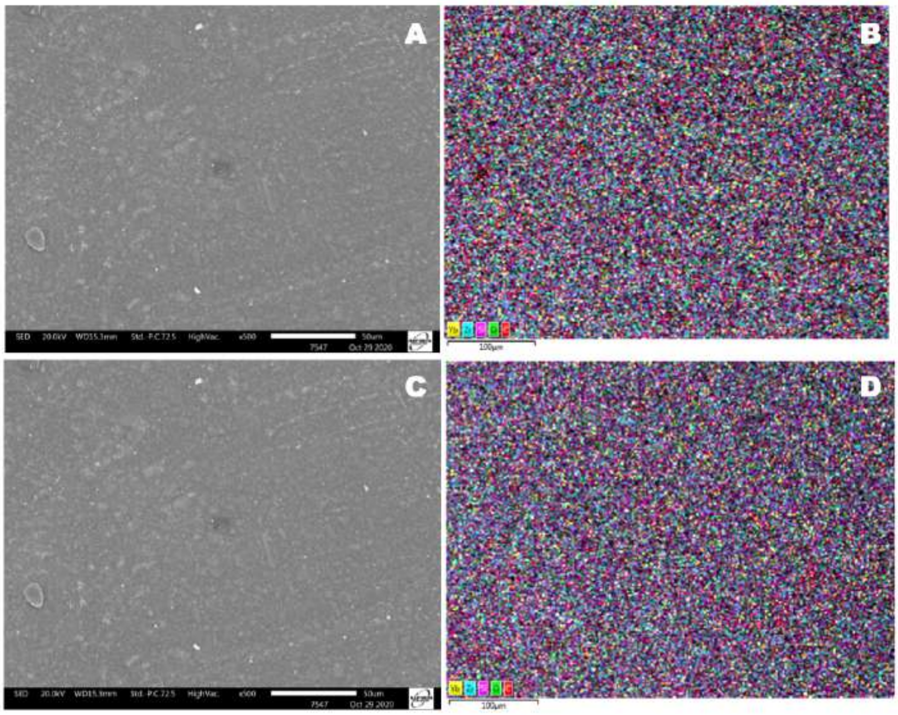


Figure 6. Representative SEM and EDX mapping images showing the Control (A and B, respectively) and HCl-treated (C and D, respectively) areas for FU restorative material.



EDX evaluations detected that the all restorative materials contained silicium (Si) and for resin-based materials (AF, AB, CH and FU) Si was the main element identified. Barium (Ba) and aluminum (Al) were also found for AF, AB and CH resin-based materials (Figures 7 to 11). AB bioactive material showed the presence of calcium (Ca) and phosphorus (P) (Figure 8), while FU exhibited the zirconium (Zr) and ytterbium (Yb) chemical elements (Figure 11). The glass ionomer material (EF) presented a complex chemical composition that contained Al, Si, F (fluorine), Sr (strontium), La (lanthanum), P, K (potassium), Ca and Na (sodium) elements (Figure 10).

The HCl treatment promoted minor changes in the chemical elementary composition for AF, AB, CH and FU resin-based materials (Figures 7, 8, 9 and 11). On the other hand, the simulated erosive treatment promoted the reduction of most chemical elements for EF, such as Si, Al, F, Sr and Na (Figure 10).

Figure 7. EDX graphs showing the elemental composition of filler particles of AF for Control (A) and HCl-treated areas (B).

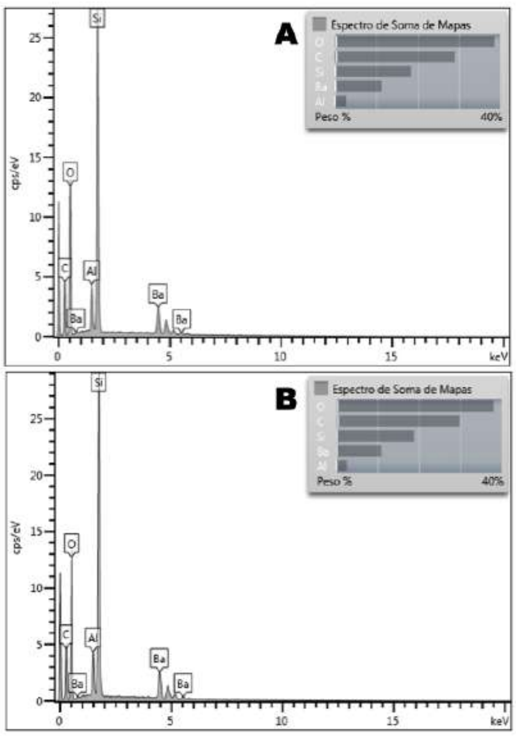


Figure 8. EDX graphs showing the elemental composition of filler particles of AB for Control (A) and HCl-treated areas (B).

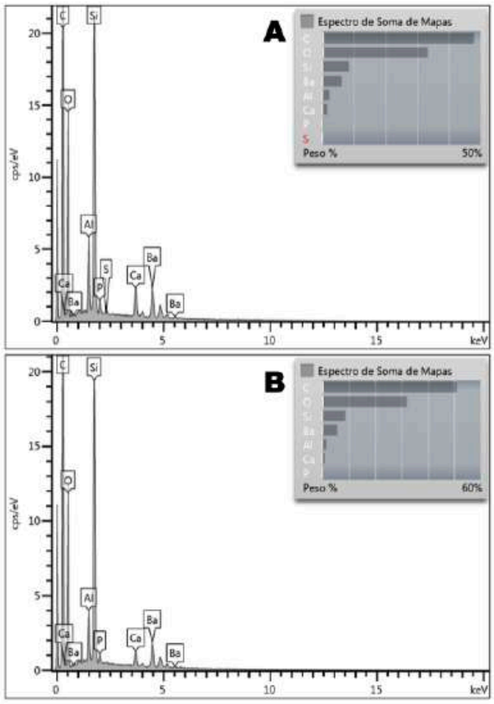


Figure 9. EDX graphs showing the elemental composition of filler particles of CH for Control (A) and HCl-treated areas (B).

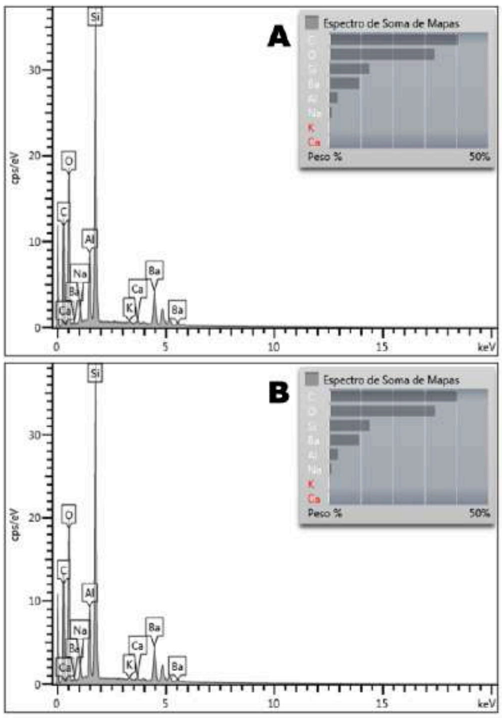


Figure 10. EDX graphs showing the elemental composition of filler particles of EF for Control (A) and HCl-treated areas (B).

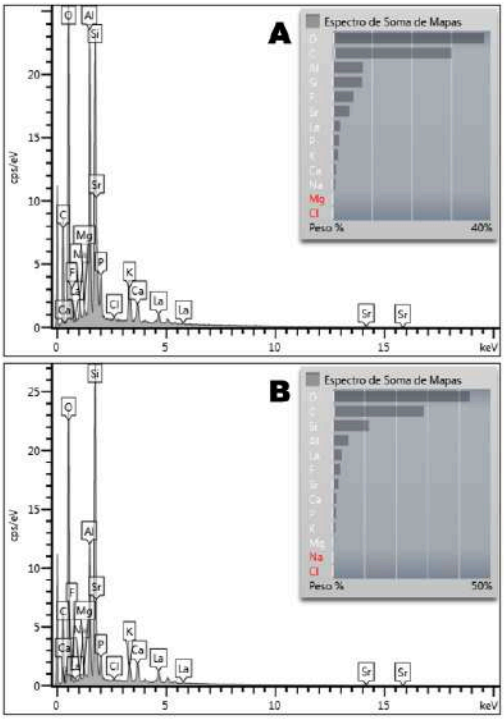
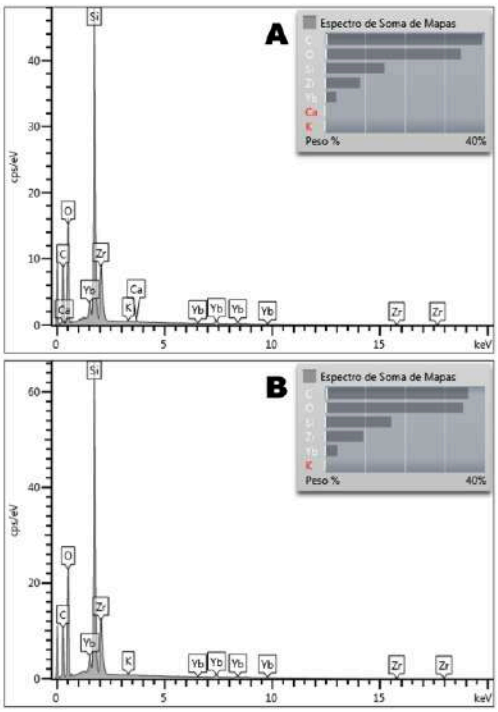


Figure 11. EDX graphs showing the elemental composition of filler particles of FU for Control (A) and HCl-treated areas (B).



DISCUSSION

The pH found within the oral cavity was shown to have a direct influence on the degradation process of restorative materials [22]. It is reasonable to assume that an acidic solution can increase the rate of hydrolysis of methacrylate ester bonds within the resin matrix of polymer-based materials [23]. These processes have been known to relate to swelling of the organic matrix, thus producing pores and intermolecular spaces within the material from which the fillers can be released, resulting fast degradation of the polymer network and a reduction of physical properties [24]. This scenario becomes more important when the restorative treatment is conducted in a patient that presents GERD [4]. Studies showed that the intrinsic erosion, caused by the gastric liquid has high titratable acidity (acid’s impact) and dissociation constant (the ease with which H^+ are released from an acid) [25-27], presenting a huge capability to damage the restorative materials. In this study, the erosive protocol used (0.06 M HCl; pH 1.2; at 37ºC; for 30 hours) aimed to simulate three years of the in vivo condition of GERD [28,29].

Despite the previous literature about the vulnerability of the traditional restorative materials under acidic environment, most the restorative materials tested in this presented study (except the EF, the ionomeric material) showed a satisfactory ability to resist against the erosive degradation, probably due to the high polymerization level reached light-activation process that formed a rigid cross-linked structure [30]. Therefore, the tested hypothesis, that “the simulated GERD would impact differently the studied properties of materials of varied compositions” was accepted, because the erosive challenge with HCl altered the topography and chemical elements of glass ionomer cement (EF), as well as yielded higher Sa, Rv, SL and the lowest GL among the materials.

Conversely, FU presented significantly lower Sa and Rv values, and greater gloss than those obtained for other materials, both in Control and HCl-treated areas. Its microhardness did not change after the HCl challenge, and the SL did not differ from other resin-based restorative materials. FU is a nanofilled composite that presents novel matrix monomers, with scarce or no literature about, such as AUDMA, AFM and 1,12-dodecane-DMA. These monomers have the intention to improve the mechanical and physical characteristics of this composites. AUDMA is an improvement of the traditional UDMA [31]. Kerby et al produced an aromatic and aliphatic UDMA monomers with pendant ethyl groups, discovering that the novel aromatic UDMA had lower water sorption characteristics [32], turning the composite more stable against oral solutions, and probably is an important factor against erosive challenges. AFM is a novel monomer that has the capability of fragmentation during the polymerization process, and these fragments can then re-polymerize in a lower stress state. The 1,12-dodecane-DMA is a larger monomer with ability to form a rigid crosslinked structure, providing better stability to the composite.

Besides the novel matrix technology presented by FU, it is important to notice that nanofilled composite resins can have better mechanical properties with high esthetic features than the other traditional composites [33]. It presents 76.5% by weight or 58.4 by volume of filler particles and the Si, Yb and Zr chemical elements were identified by EDX analysis. Yb is used as a radiopacifier agent, while Si and Zr are related to main filler of this composite (silica-zirconia) that is a nano scale (20 nm and 4 to 11 nm size) and agglomerated in clusters. These characteristics of organic matrix and inorganic filler particles that constitute FU can explain the excellent outcomes reached in this study.

AF, CH, and AB restorative materials presented intermediate outcomes, with adequate stability regarding the simulated erosive challenge. AF presented the second better Sa and Rv outcomes. It is a Bis-GMA-, TEGDMA- and HEMA-free monomer and considered an ormocer-based material [16]. Ormocer materials contain inorganic-organic (Si–O–Si) oligomeric resin (co-polymers) in addition to the inorganic silanated glass filler particles of Ba and Al [34,35]. All these chemical elements were identified by EDX in this study. According to previous studies, ormocer is a highly functionalized molecule with a dense network, denser than dimethacrylate polymers, which can turn this material more stable against oral chemical challenges [36,37].

On the other hand, other authors found that nanoparticles and glass fillers could neither be sufficiently silanized nor perfectly integrated within the ormocer matrix, contributing to a reduced degree of conversion and higher susceptibility to sorption [38,39]. Despite this, it was found in the present study that AF is stable in the same degree of traditional monomers. Besides that, AF presented better outcomes than CH for surface roughness, contradicting a previous study stating that ormocer does not show comparable performance to conventional Bis-GMA-containing resin composites [16]. However, AF presented poor gloss results for both Control and HCl-treated surfaces areas. The gloss was significantly lower than FU and CH, but did not differ from AB. The low gloss can be due to the filler particles that are in high concentration (84% by weight) and exposed at the Control and HCl-treated surfaces, according to the Figures 2A and 2C, respectively.

The CH restorative material is a traditional microhybrid composite with Bis-GMA organic matrix [11]. Monomers such as Bis-GMA and UDMA have been incorporated in the materials' matrix since the beginning of the manufacturing process [40]. However, while UDMA is considered mainly hydrophobic, Bis-GMA is expected to present a higher water sorption character [31]. Therefore, alterations in CH are expected when immersed in aqueous acid solution, causing the softening of Bis-GMA polymers, swelling and plasticization of the resin composite [41]. Its filler content was also conventional, with silicon dioxide, Ba and Al glasses that were identified by EDX. Despite that, CH showed intermediate results of Sa, Rv and gloss. Also, MI, topography and chemical element were not changed following the HCl challenge.

This study aimed to compare different types of restorative materials, such as a bioactive material (AB) and a novel glass ionomer cement (EF). A previous study reported that resin composites are more resistant material than glass ionomer and compomer materials when immersed in different acid solutions [42]. The explanation is that proper cured resin composites are more stable and less soluble material due to the formation of polymeric structures filled with different inorganic particles that are less affected by acidic conditions [43,44]. As AB has resinous features and is a polymerizable material, with di- and methacrylates [21,45], it presented Rv and SL similar to other resin-based materials (AF and CH) and its MI was not affected by HCl. AB contains the varied particles, such as bioactive glass, Ca, silica, and sodium fluoride, but the last chemical element were not identified by EDX analysis. However, a study demonstrated that AB is able to release fluoride ions as well as the glass ionomer cements [46].

EF contains ultrafine and highly reactive glass dispersed in the structure of the glass powder. In addition, the molecular weight of the polyacrylic acid has been optimized. Based on these modifications, this new type of ionomeric material exhibits enhanced mechanical properties, improved wear and acid erosion resistance in comparison with the traditional glass ionomer cements [18]. However, in this study EF showed the highest Sa, Rv and SL after simulated gastric acid among restorative materials. The image taken at confocal microscope shows the significant SL of EF after HCL treatment (Figure 1D). It presented the lowest gloss values in both Control and HCL-treated areas, with significant reduction after HCl challenge. The SEM images showed that its surfaces were not smooth due to the large particles and that the roughness increased with HCl challenge (Figures 5A and 5C).

Because of the size of fillers of EF, the MI evaluation for this material was not performed. EF presents large and hard particles that compromise the MI readings, because the size of Knoop indenter is smaller than particles. Also, after the exposure to acidic solution, plates of EF suffered ruptures and cracks during the indentation, making the MI reading unfeasible. Studies using SEM reported that traditional glass ionomer cements are vulnerable to severe damage in patients experiencing strong citric or gastric acid [42,47]. Moreover, it was reported that ionomeric materials can be dissolved after 6 months of storage in fruit juices of pH 2.5–3.4 [48,49]. Since the present study used a methodology that aimed to simulate three years in vivo of GERD, the surface of EF was compromised following the HCl challenge.

EDX analysis of EF showed reduction of Al, F, Sr and Na elements, which were susceptible to the HCl challenge, while Si seemed to be resistant, remaining in the surface. It was reported that the F release from glass ionomer cements is increased under acidic conditions in the long term [50], causing the reduction of its presence at HCl-treated surface, as observed in this study. Also, glass ionomers tend to buffer the storage media, reacting its components with storage solution and this could explain their major susceptibility to HCl challenge [51].

DISCLOSURE STATEMENT AND ACKNOWLEDGEMENTS

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CONCLUSIONS

Within the limitation of the present *in vitro* study, it was possible to conclude that:

- The traditional resin composites (Admira Fusion, Charisma and Filtek Universal Restorative) and the bioactive material (Activa BioActive-Restorative) were not affected by the simulated GERD.
- Filtek Universal Restorative presented better outcomes for surface roughness, surface loss, and surface gloss after simulated GERD.
- The glass ionomer cement (EQUIA Forte HT Fil) had superficial changes following the HCl treatment (simulated GERD). Thus, it might be not indicated for this clinical situation.

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3 CONCLUSÃO

A partir dos resultados obtidos é possível concluir que:

a) A maioria dos materiais restauradores apresentaram estabilidade após exposição ao desafio erosivo simulado, uma vez que mantiveram resultados semelhantes no grupo controle e no grupo tratado, com exceção do material EQUIA Forte, o qual teve uma performance inferior aos demais materiais, com maior rugosidade de superfície, perda de volume e perfil de rugosidade, além de menor valor de brilho.

b) O material Filtek Universal Restorative apresentou a melhor performance entre os materiais restauradores estudados, com menores valores de rugosidade de superfície, maior microdureza e maior brilho.

c) Os materiais restauradores apresentaram perdas de volume semelhantes, com exceção do material EQUIA Forte, o qual apresentou uma perda de volume significativamente maior que os demais.

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* De acordo com as normas da UNICAMP/FOP, baseadas na padronização do International Committee of Medical Journal Editors - Vancouver Group. Abreviatura dos periódicos em conformidade com o PubMed.

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ANEXOS

Anexo 1 – Verificação de originalidade e prevenção de plágio

EFEITO DO REFLUXO GASTROESOFÁGICO SIMULADO EM MATERIAIS RESTAURADORES

RELATÓRIO DE ORIGINALIDADE

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1	Ana Carolina Cabral Roque, Lauren Oliveira Lima Bohner, Ana Paula Terossi de Godoi, Vivian Colucci, Silmara Aparecida Milori Corona, Alma Blásida Concepción Elizaur Benitez Catirse. "Surface Roughness of Composite Resins Subjected to Hydrochloric Acid", Brazilian Dental Journal, 2015 Publicação	2%
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Anexo 2 – Iniciação Científica

FAPESP SAGe

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Situação	Em Execução
Vigência	01/12/2019 a 30/11/2020
Beneficiário	Thaís Bulzoni Branco
Responsável	Marcelo Giannini
Vínculo Institucional do Processo	Faculdade de Odontologia de Piracicaba/FOP/UNICAMP
Título	"Efeito do desafio erosivo sobre três resinas compostas de diferentes composições monoméricas e conteúdo de carga"

Folha de Despacho

Datas do Despacho

Emitido em :	08/03/2021

Objetos de análise

Objeto de análise	Data de Submissão	Resultado
Relatório Científico 2	09/12/2020	Aprovado

Observações / Transcrições / Frases

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Atenciosamente,
Luiz Eugênio A. M. Mello
Diretor Científico da FAPESP

Frases para o Beneficiário

Não há frases associadas.

Transcrição de Parecer para o Beneficiário

Não há transcrição associada.

Frases para Termo de Outorga

Não há frases associadas.

Relatório Científico 2 (Aprovado)

Compromisso	10/12/2020
Período Relacionado	10/05/2020 a 30/11/2020
Situação	Atendido

Anexo 3 – Comprovante de submissão do artigo no Periódico Internacional Journal of Dentistry, na data de 14/10/2021

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