Mechanical and surface properties analysis of restorative materials submitted to erosive challenges *in situ*

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ABSTRACT

Objective: This study aims to evaluate the effect of acidic solutions (AS) on surface roughness (Ra) and microhardness of restorative materials (RM). **Materials and Methods:** Eight volunteers wore intraoral palatal devices (IPD) containing samples of RM: Ketac Nano (KN); Ketac Nano + Biscover LV (KN-B); Esthet-X (EX); Esthet-X + Biscover LV (EX-B); Supreme XT (SXT); Supreme XT + Biscover LV (SXT-B); and bovine enamel. The samples were submitted to three phases: (1) immersion in 0.01M hydrochloric acid (HCl) – 10 min, three times/day (14 days); (2) immersion in soft drink (Sprite®) – 10 min, three times/day (14 days); and (3) keeping in saliva (14 days). Changes in Ra/microhardness were measured before/after the three phases. **Statistical Analysis:** ANOVA ($\alpha = 0.05$) and Fisher's test. **Results:** Materials sealed with Biscover LV (B) presented lowest values in all periods. KN glass ionomer cement showed highest Ra values after exposure in AS. Application of B did not reduce the Ra for the composites studied, except for EX after immersion in HCl. AS promoted changes in Ra/microhardness of RM, except for sealed materials. **Conclusions:** The acids used were able to change the Ra and microhardness of RM, except of the sealed materials. The resin-modified GIC showed the most significant changes after immersion in AS; and the composites sealed with B, even after immersion in acidic solutions (AS), showed the lowest Ra values and the least degradation in microhardness, especially when subjected to low pH solutions.

Key words: Carbonated drink, dental erosion, gastric juice, microhardness

INTRODUCTION

Dental erosion is a new risk for oral health, introduced by today's lifestyles and dietary habits.

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Erosion is a tooth wear attributed to dissolution of the dental-hard tissues by acids, without the involvement of bacteria and may be classified as extrinsic or intrinsic.[1] Extrinsic factors include frequent consumption of acidic foodstuffs or beverages and some medications,[2] while intrinsic factors are related to eating disorders and gastric reflux.[3]

Once in contact with enamel, the acid with hydrogen ion or its chelating capacity begins to dissolve the hydroxyapatite crystal. The nonionized form spreads throughout the enamel's interprismatic areas, dissolving the mineral in the subsurface region.[4] Frequent contact between acids and tooth surfaces causes loss of this structure resulting in a surface susceptible to mechanical abrasion.[5] Furthermore, in severe situations, as in gastroesophageal reflux disease (GERD), a significant loss of tooth structure, vertical dimension, and/or function, hypersensitivity, esthetically unacceptable defects, and pulp exposure could occur.[1]

In the past, patients were left untreated or the rehabilitation was performed with extensive crown and bridge work.[6,7] However, as a result of the improvements in adhesive materials, it has become possible to rehabilitate eroded teeth in a less invasive manner using direct restorative material (RM) such as composite resins and glass ionomer cement (GIC).[6,7] These materials are capable of reestablishing the function and esthetics of tooth structure as well as controlling the hypersensitivity.[1]

It is known that the longevity of dental restorations depend on the durability of the material and its properties such as wear resistance, integrity of the tooth/restoration interface, surface roughness (Ra), and microhardness.[7] To preserve or improve the properties of direct RM, surface sealants were developed. According to the literature,[8,9] this is a dental biomaterial where nanotechnology was applied that is able to improve the properties of existing materials, by filling the cracks, decreasing the porosity, increasing the wear resistance, and maintaining the marginal integrity of restorations.

This study aimed to evaluate *in situ* the effect of different acidic solution (AS) on Ra and microhardness of RM, including a sealed composite. The null hypothesis was that AS cause any effect on the Ra and microhardness of RM.

MATERIALS AND METHODS

Screening of participants
After receiving all relevant information to conduct the research, the volunteers underwent an anamnesis and clinical examination for the inclusion and exclusion criteria checking. The inclusion criteria applied for selection of research volunteers were good general health, normal salivary flow, absence of active caries lesions, no noncarious injuries (e.g., cervical lesions), absence of periodontal disease, absence of prosthesis, absence of orthodontic appliances, and availability to attend the research site. Furthermore, the exclusion criteria were as follows: medical treatment, digestive disorder, use of any drug, pregnancy, and smoking habit. Eight volunteers (four men and four women) with a mean age of 22 years were selected for this research.

Specimen preparation, microhardness, and profilometric measurements
A total of 48 samples of each material (Ketac Nano [KN], Esthet-X [EX] and Supreme XT [SXT]) measuring 4 mm in diameter and 1.5 mm in thickness were made, using a metal die. The die cavity was completely filled with the materials. A polyester strip and a thin glass plate were placed on the material surface to remove the excess and standardize the finishing of the samples. The materials were light polymerized for 40s (Ultralux, Dabi Atlante, Ribeirão Preto, SP, and Brazil) and then stored in distilled water at 37°C for 24 h. After that, the sample's surfaces were ground flat with water-cooled carborundum discs (# 600, 800, and 1200 Grades of Al_2O_3 papers; Extec, Corp., Enfield, CT, USA) and polished with felt paper wet by diamond spray (1 μm; Extec). The samples were cleaned in an ultrasonic cleaning device (Cristófoli, Campo Mourão, SP, Brazil) for 10 min after each disc and after the final polishing.

Half of the samples were etched with 32% phosphoric acid (Bisco Inc., Schaumburg, IL, USA) for 15 s, washed with distilled water, and dried with air spray. On the conditioned surfaces, the surface sealant B was applied (Bisco Inc., Schaumburg, IL, USA) and light polymerized for 30 s.

Twenty-four enamel samples (4 mm × 4 mm × 1.5 mm) were prepared from extracted bovine incisors, which were previously stored in 2% formaldehyde solution (pH 7.0) for 30 days at room temperature. One sample was cut from each crown, using an APL-4 cutting machine (Arotec Ind. Com., Cotia, SP,

Brazil). The enamel surface was also ground flat with water-cooled carborundum discs (# 320, 600, 800, and 1200 Grades of Al_2O_3 papers; Extec) and polished with felt paper wet by diamond spray (1 μm; Extec). The enamel samples were also cleaned in an ultrasonic cleaning device (Cristófoli) for 10 min after each disc and after the final polishing. Bovine enamel was used as an intrinsic control of each group to confirm that the erosive period adopted in this research could lead to the erosion of the specimens.

The surface microhardness was determined by performing five indentations in different regions of the samples (Knoop diamond, 25g, 10 s, HMV-2000; Shimadzu Corporation, Tokyo, Japan). The Ra was determined using a profilometer (SJ-401, Mitutoyo, Kanagawa, Japan). The Ra value was used because it represents the arithmetical mean of roughness of a surface, and it is the most used parameter for this purpose. Each measurement was obtained after turning the specimen 120°, totaling three measurements using a cutoff of 0.25 mm.

Intraoral phase: mouth appliances, tested products, and intervention.

Alginate impressions (hydrogun) were made directly from eight volunteers producing a negative mold, in which dental stone was poured. The intraoral palatal devices (IPD) were manufactured with acrylic resin. Seven holes measuring 4 mm × 4 mm × 2 mm were prepared to fix the specimens. Each one received one sample of each material as well as one enamel sample. The samples were fixed with wax into cavities (4 mm × 4 mm × 2 mm) located at each left and right sides of the IPD. The position of each sample was defined by drawing. The experimental design of the crossover study is showed in Figure 1.

In the first phase of this study, volunteers were instructed to immerse the IPD in 30 mL of HCl at a concentration of 0.01M (Dinâmica®, Química Contemporânea Ltda, Diadema, SP, Brazil), pH 2.0 for 10 min[7] and three times a day during 14 days. The erosive solution was renewed for each erosive challenge. After the immersion, the intraoral devices were washed with water for 30 s and then replaced into the mouth. In the first 12 h of the intraoral phase, samples were not subjected to erosive treatment to allow the formation of a salivary pellicle.[5] After this first phase, the samples were removed and new Ra and microhardness measurements were done in the same manner as described above.

In the second phase, new samples of each material were positioned in the same holes of the intraoral device
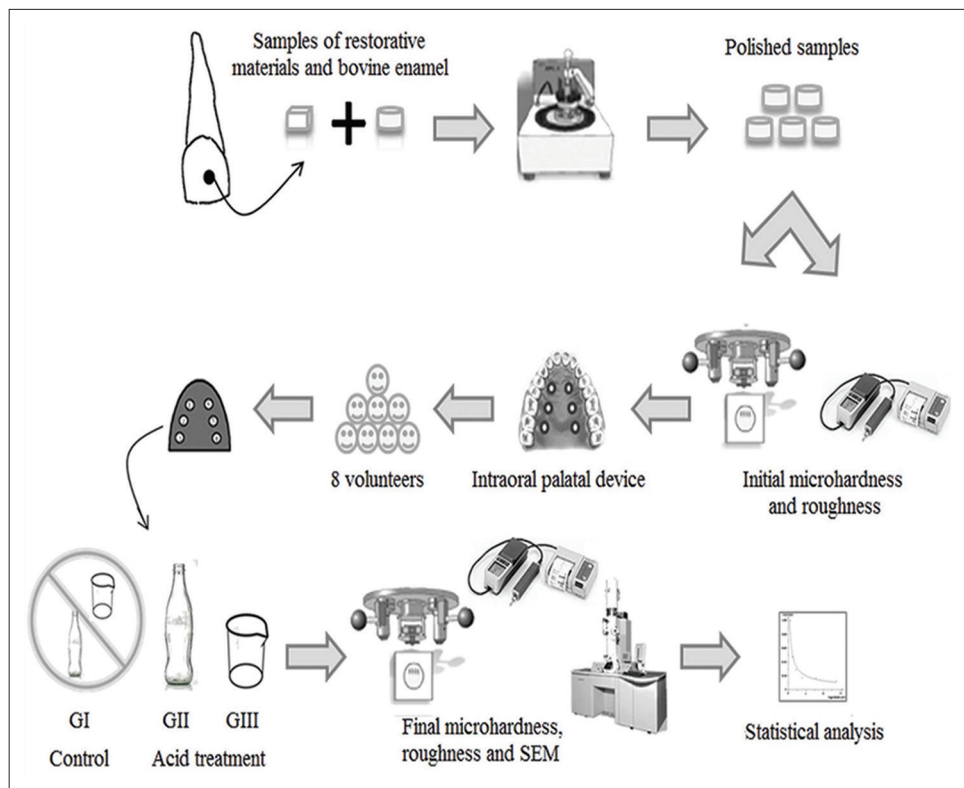

Figure 1: Experimental design used in the study

and the same volunteers were instructed to immerse intraoral devices in 30 mL of soft drink (SD) (Sprite®, Coca-Cola Co, Ribeirão Preto, SP, Brazil), pH 3.6 for 10 min and three times a day during 14 days. The erosive solution was renewed for each erosive challenge. After this second phase, the samples were removed and new Ra and microhardness measurements were done in the same manner as described above.

In the third phase, new samples of each material were positioned in the same holes of the intraoral device and the same volunteers were instructed to keep the samples only in saliva, without submitting them to any extraoral treatment during 14 days. The samples were removed and new Ra and microhardness measurements were done in the same manner as described above.

A washout period of 14 days was performed among the phases. Volunteers received instructions to wear the appliances continuously, but to remove them during meals. After meals, they had to brush their teeth using a soft toothbrush (Colgate Classic Clean, Colgate-Palmolive industrial LTDA, São Bernardo do Campo, SP, Brazil) with a small portion of the same fluoridated toothpaste (Colgate-Palmolive industrial LTDA, São Bernardo do Campo, SP, Brazil). During this period, the appliance was stored in wet gauze. After the brushing process was complete, the appliances were replaced into the mouth. Volunteers received oral and written information to refrain from using any fluoridated mouthwashes.

Postoral phase: microhardness, profilometric, and microscopic analysis.

After Ra and microhardness analysis, representative samples of each group were selected for microscopic examination using scanning electron microscope (SEM) (JSM-5410, JEOL, Tokyo, Japan).

Statistical analysis

The Ra and microhardness data were submitted to repeated-measures ANOVA at 5% significance level and pairwise comparisons were performed using the Fisher's *post-hoc* test (software Statview 5.0.1).

RESULTS

Microhardness results are in Table 1. The aging of the materials in HCl caused decrease in microhardness, with significant difference to other solutions and initial values. Storage in SD caused decrease in microhardness only to SXT and Esthet-X + Biscover LV (EX-B) with significant difference to initial values. For other materials, the SD did not cause significant changes in microhardness. There was no difference between microhardness values, before and after *in situ* test performed only in saliva, for all materials studied.

Comparing the materials, the SXT composite resin (CR) showed highest microhardness average in all periods, followed by EX CR and KN GIC, with significant difference between them. The materials sealed with Biscover LV (B) presented the lowest microhardness compared to materials without sealing, in all periods.

Roughness results are in Table 2. The aging of the materials in HCl caused increase in Ra compared to initial values for all materials studied, except for the sealed ones. In the sealed materials, the difference from baseline and after immersion in saliva, SD, and HCl was not statistically significant. The SD caused increase in the Ra of GIC without sealing. The saliva caused increase in Ra for the composites SXT and EX with significant difference to initial values.

Table 1: Initial and final microhardness values of the studied materials (KHN)

	Baseline	Saliva	Sprite	HCl
SXT	82.7 (3.9) Aa	81.5 (6.1) Aa	76.2 (4.9) Ba	60.7 (13.6) Ca
SXT-B	29.4 (1.6) Ad	27.7 (2.9) ABe	28.4 (3.2) Ade	25.6 (1.7) Bd
EX	59.6 (3.3) Ab	55.5 (2.9) Ab	57.3 (7.6) Ab	40.5 (9.7) Bb
EX-B	30.5 (2.7) Ad	31.4 (2.3) Ad	26.9 (2.8) Be	25.4 (2.0) Bd
KN	45.2 (3.1) ABc	49.6 (2.7) Ac	46.3 (9.6) Ac	30.0 (4.6) Cc
KN-B	32.1 (2.8) Ad	33.7 (3.0) Ad	31.2 (3.4) Ad	26.8 (3.4) Bd

*Distinct capital letter in columns and lower case letter in rows are statistically different (*P*<0.05)

Table 2: Initial and final Ra values of the studied materials (µm)

	Baseline	Saliva	Sprite	HCl
SXT	0.024 (0.005) Cc	0.028 (0.003) Bc	0.027 (0.005) Bb	0.035 (0.005) Ac
SXT-B	0.037 (0.012) Aab	0.043 (0.003) Aab	0.040 (0.016) Ab	0.041 (0.014) Abc
EX	0.028 (0.007) Bc	0.040 (0.015) Aab	0.027 (0.005) Bb	0.051 (0.028) Ab
EX-B	0.033 (0.013) Ab	0.043 (0.003) Aab	0.036 (0.010) Ab	0.032 (0.010) Ac
KN	0.040 (0.008) Ba	0.048 (0.015) Ba	0.076 (0.043) Aa	0.078 (0.016) Aa
KN-B	0.036 (0.008) Aab	0.033 (0.007) Abc	0.038 (0.008) Ab	0.031 (0.007) Ac

*Distinct capital letter in columns and lower case letter in rows are statistically different (*P*<0.05)

Comparing the materials, the KN GIC had the highest Ra after EC. For this material, sealing with B possibly caused minor changes in Ra after aging in saliva, SD, and HCl. The application of the sealant did not reduce Ra values for the two composites studied, except for EX after challenge in HCl.

DISCUSSION

The design and conduct of this study were based on the evidence that after repeated exposure of dental enamel to gastric contents or acidic foods and beverages, a dental erosion typically occurs.[8] Concurrently, with this approach, it is assumed that this acidic exposure could also have effects on RM. Once direct RM as CR and GIC are constantly used to repair eroded tooth surfaces, it was pertinent to investigate the effects of acidity on these materials as well as new techniques to increase the restorations longevity.[7,10] Data analysis revealed that the AS altered the Ra and microhardness of the materials [Tables 1 and 2], rejecting the null hypothesis of the study.

Several studies that evaluated the effect of acids on enamel[3,4,7,8,11,12] and RM[12] under *in vitro* methodologies. However, *in situ* studies simulate a more realistic condition to that found clinically since they are conducted, in part, in the oral environment and suffer the environmental interference, such as saliva,[13] temperatures, microorganisms, and enzymes. In this study, the action of saliva may have contributed to these results since it helps in the neutralization of the acids used in this research.[5]

In this study, the SD was chosen because of its large consumption, high erosive potential, and buffering capacity. According to Larsen and Nyvad,[14] the capability of an SD to erode dental enamel depends not only of the pH of the drink but also of its buffering effect, the ability of the drink to resist a change of pH. The solution of HCl (0.01M, pH 2.0)[7] would simulate a severe condition with high risk for the formation and progression of erosive lesions. In addition, despite that pure acids have a pH between 0.9 and 1.5,[11] the pH in the oral cavity after episodes of vomiting rarely or never is <1.5 because of the buffering effect in the esophagus and dilution with food and drinks.[11] Thus, the use of HCl solution in this concentration simulates a more real condition, making the situation clinically relevant.

The evaluation methods used in this research are very useful to quantify the changes of enamel and RM

subjected to erosive challenges.[15] The profilometer was used to measure the Ra of materials since previous studies[16] have established that the critical threshold for the Ra is 0.2 μm, above which could occur a simultaneous increase in plaque accumulation, thereby increasing the risk for both caries and periodontal inflammation. The evaluation of the surface microhardness is sensitive in determining the erosive lesions at early stages[15] and is considered an important indicator of materials' mechanical properties.[17]

The results of this study showed a significant change in Ra and microhardness after immersion in AS of SD and HCl [Tables 1 and 2]. In this context, Badra *et al*.[12] also reported a decrease in the microhardness of CR and glass ionomer resin modified when immersed in SDs that have low pH. It is believed that a reduction in the surface microhardness of CR soaked in organic acids has been attributed to the softening of bisphenol A glycidyl methacrylate (bis-GMA)-based polymers, which could be caused by leaching of the diluent agents such as triethylene glycol dimethacrylate (TEGDMA).[5] The softening of the resin matrix could promote displacement of the filler particles, contributing to the formation of a rough surface,[12] as observed and illustrated [Figure 2c, f and i] in this study.

The pH, the titratable acidity (the volume of alkali needed to neutralize an acid), and dissociation constant (the ease with which H+ are released from an acid) are factors in the chemistry of acids.[18] It is probable that the titratable acidity or the dissociation constant are more important than the pH in assessing the erosive potential of an acidic solution.[19] In our study, the hydrochloric acidic solution (HCl 0.01M, pH 2.0) caused the largest changes in Ra and microhardness of RM when compared with the SD (Sprite®) as observed in Figure 2b, e, and h. Our results are consistent with studies of Bartlett *et al*.[18] that compared the erosive potential of gastric juice and carbonated drinks, demonstrating the sovereignty of the first one. These findings are a reflection of low pH and acidity of HCl when compared to the SD and confirm the assumptions that the gastric juice has the potential to produce severe erosion patterns in patients with eating disorders and GERD.[4]

It is important to consider the role of saliva in neutralizing acids. It is known that during the intake of acidic drinks, the pH of the oral environment falls below the critical value, the rate of salivary flow increases and the drinks are so diluted by saliva.[5]
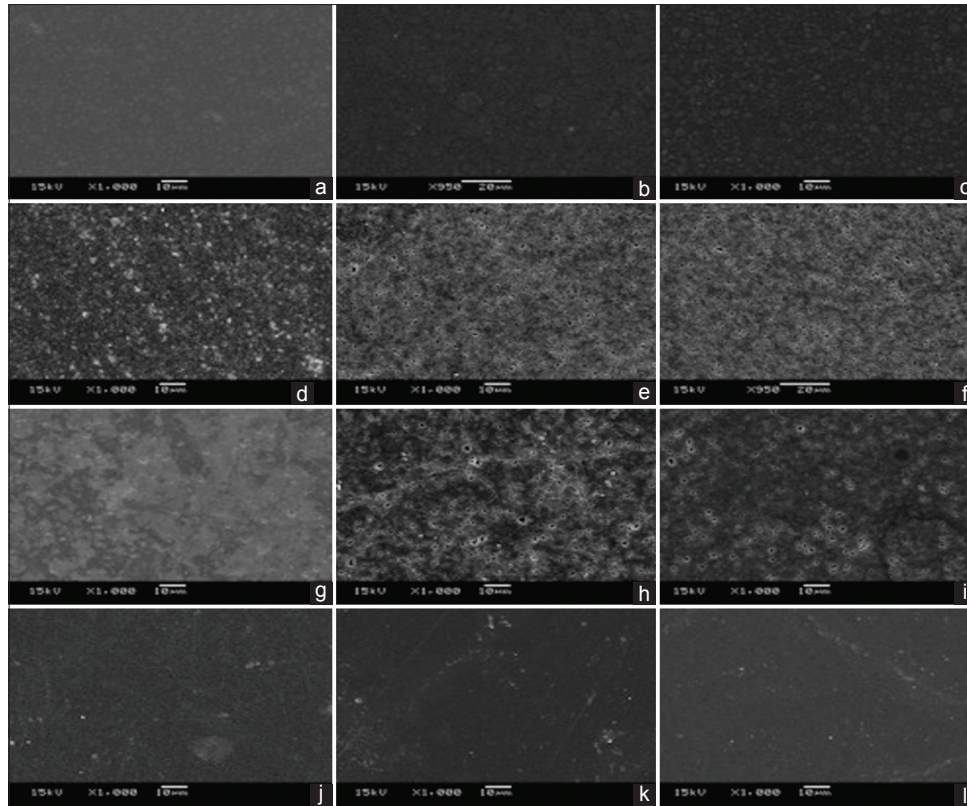
Figure 2: Microscopic images of samples (×1000). (a) Supreme XT after aging in saliva. (b) Supreme XT after aging in HCl. (c) Supreme XT after aging in soft drink (d) Esthet-X after aging in saliva. (e) Esthet-X after aging in HCl. (f) Esthet-X after aging in soft drink. (g) Ketac Nano after aging in saliva. (h) Ketac Nano after aging in HCl. (i) Ketac Nano after aging in soft drink. (j) Sealed materials after aging in saliva; (k) Sealed materials after aging in HCl. (l) Sealed materials after aging in soft drink

This is because esophageal acid clearance occurs in a two-step procedure: peristalsis clearing the volume, followed by saliva neutralizing the acid.[13] However, in this study, the interaction of saliva with the acidic solution may have been impaired because the samples were washed with water after the exposure to the solutions.

Comparing the materials, it was found that the KN glass ionomer was more unstable than the CR. These findings could be explained by matrix dissolution in the periphery of glass particles of glass ionomer, which could result from dissolution of the siliceous hydrogel layer [Figure 2h and i].[5,20] Other factors that could also have contributed to these results are the manipulation and composition of these materials. The presence of glass particles in the composition of resin-modified glass ionomers, such as KN, may reduces its homogeneity and increases its surface roughness. Furthermore, the components have different microhardness, and they are manually handled, which can generate porosity because of the inclusion of visually imperceptible air bubbles [Figure 2g].[21] SEM studies of Abu-bakr *et al.*[22] have shown images of rough surfaces with the presence of

voids and protruding glass particles, which clinically add up to a rough and dull surface, which could explain the higher Ra values of resin-modified glass ionomer materials [Table 2].

On the other hand, under acidic conditions, the composites were more stable due to the formulation of the material and morphology of the filler particles, which are nanosized and regular, allowing the incorporation of a large inorganic volume.[23] This statement explains the higher microhardness values found for the SXT. According to Dos Santos *et al.*,[6] composites with small filler particles are more wear-resistant since they are more homogeneous and their particles are less prominent on the surface, resulting in a lower roughness as showed in Figure 2a. Whereas the type of filler, size, and quantity of the particles influence the properties and quality of polishing of composite resins (for example Figure 2d), the reduction in space between the inorganic nanoclusters[23] is possibly responsible for the superior physical properties of SXT.

In this study, the composite resin sealed with B showed significantly lower microhardness values when

compared with the values of unsealed resins [Table 1]. These differences could be attributed to resin monomers and the ethanol solvent present in the sealant surface. Bertrand *et al.*[24] demonstrated that when examined by SEM, the sealant was shown to reduce the surface microhardness, improving the surface quality of composites because of the disappearance of microcracks and minor surface irregularities. However, according to the results of the present study, the maintenance of lower Ra and microhardness values for the sealed composite after immersion in AS as well as the nonexposure of the particles as illustrated in microscopy images [Figure 2j, k, and l] could be indicative of the sealing material remaining on the composite surface, showing that the material was able to withstand the acidic challenges.

CONCLUSIONS

Data from this study demonstrated some of the changes caused by low pH solutions in direct RM concluding that: (1) The acids used in this study were able to change the Ra and microhardness of RM, except of the sealed materials; (2) The resin modified GIC showed the most significant changes after immersion in AS; and (3) The composites sealed with B, even after immersion in acidic solutions (AS), showed the lowest Ra values and the least degradation in microhardness, especially when subjected to low pH solutions. However, further research is needed to assess simultaneously the effect of other degradation processes such as mechanical abrasion. In addition, to complement the results of this research, a long time erosion could be used to assess differences in the behavior of RM as well as clinical and epidemiological studies.

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Conflicts of interest
There are no conflicts of interest.

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