In Vitro Evaluation of Surface Roughness and Microhardness of Restorative Materials Submitted to Erosive Challenges

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Clinical Relevance

The effects of dental erosion caused by acidic solutions on the surface of restorative dental materials could be minimized by the application of a surface sealant.

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SUMMARY

The aim of this study was to evaluate the effect of different acidic solutions on the microhardness and surface roughness of restorative materials. The 120 specimens of restorative materials (Fuji II LC, Vitremer, Supreme XT, and Supreme XT + Biscover LV) were randomly divided into three groups according to the immersion media: hydrochloric acid, soft drink, or distilled water. Over a period of five weeks, the groups were immersed in the solutions, which were changed weekly. Data were tested using analysis of variance and the Fisher protected least significant difference test ($p < 0.05$). The results showed that the glass ionomer materials showed the highest surface roughness values (Fuji II LC: 0.111 ± 0.014 µm before and 0.139 ± 0.016 µm after immersion; Vitremer: 0.177 ± 0.012 µm before and $0.084 \pm$

0.012 μm after immersion), whereas the lowest values were found for the resin sealed with Biscover LV before (0.047 ± 0.011 μm) and after exposure in distilled water (0.043 ± 0.007 μm), soft drink (0.040 ± 0.005 μm), and hydrochloric acid (0.045 ± 0.005 μm). The Supreme XT showed the highest microhardness values before (44.96 ± 2.51 KHN) and after the aging process (41.26 ± 1.22 KHN in water, 35.96 ± 0.81 KHN in soft drink, and 34.74 ± 0.97 KHN in HCl), with significant differences from the other materials (p<0.0001). The lowest microhardness values were found for glass ionomer materials. The solutions used in this study decreased the microhardness of all studied materials, whereas the sealed surface suffered minor changes in microhardness and surface roughness after exposure to acidic solutions.

INTRODUCTION

Dental erosion is defined as tooth wear due to dissolution of the dental hard tissues by acids without the involvement of bacteria and may be classified as extrinsic or intrinsic.[1-4] Extrinsic factors include frequent consumption of acidic foodstuffs or beverages and some medications,[5] whereas intrinsic factors are related to eating disorders and gastric reflux.[6] Frequent contact between acids and tooth surfaces cause loss of this structure, resulting in a surface susceptible to the effects of mechanical abrasion.[7-9] Furthermore, in severe situations, such as in gastroesophageal reflux disease, 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 rehabilitation was performed with extensive crown and bridge work.[10,11] 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 materials such as composite resins and glass ionomer cements.[10,11] 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 depends on the durability of the material and its properties, such as wear resistance, integrity of the tooth/restoration interface, hardness, and surface roughness.[11,12] To preserve or improve the properties of direct restorative materials, surface sealants were developed. This material would be able to fill the cracks, decreasing the porosity, increasing the wear resistance, and improving the marginal integrity of restorations.[13–15] Thus, the application of surface sealants is being recommended to increase the longevity of restorations.[14-16]

The aim of this study was to evaluate the effect of different acidic solutions on the microhardness and surface roughness of restorative materials including a sealed composite. The null hypothesis tested was that the acid substances did not cause any effect on the microhardness and surface roughness of restorative materials.

MATERIALS AND METHODS

This study investigated two resin-modified glass ionomer cements, one composite resin, and one surface sealant (Table 1).

A total of 120 samples measuring 6.0 mm in diameter and 1.5 mm thick were made, using a metal die (30 samples for each resin-modified glass ionomer cement and 60 samples for Supreme-XT composite resin). 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 40 seconds (Ultralux, Dabi Atlante, Ribeirão Preto, Brazil) and then stored in distilled water at 37°C for 24 hours. After that, the samples were cleaned in an ultrasonic cleaning device (Cristófoli, Campo Mourão, Brazil) for 10 minutes.

Half of the samples of Supreme-XT composite resin were etched with 32% phosphoric acid for 15 seconds, washed with distilled water, and dried with air spray. On the conditioned surfaces, the surface sealant Biscover LV was applied (Bisco Inc, Schaumburg, IL, USA) and light polymerized for 30 seconds (n=30).

The surface microhardness was determined by performing five indentations in different regions of the samples (Knoop diamond with a 50-g load for 15 seconds, HMV-2000, Shimadzu Corporation, Tokyo, Japan). The surface roughness (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 is the parameter most used for this purpose. Three readings were performed on each specimen in different positions, using a cutoff of 0.25 mm.

After these analyses, the specimens were divided into three groups (n=10):

Table 1: *Materials Used in This Study*

Material	Manufacturer	Composition	Lot
Fuji II LC	GC Corporation, Hasunuma-Cho, Itabashi-Ku, Tokyo, Japan	Distilled water, polyacrylic acid, 2-hydroxyethylmetacrylate, urethane dimethacrylate, camphorquinone, fluoroaluminosilicate filler.	0610171
Vitremer	3M ESPE Dental Products, St Paul, MN, USA	Powder: fluoroaminosilicate glass, potassium persulfate, ascorbic acid. Liquid: aqueous solution of a polycarboxylic acid modified with pendant methacrylate groups, water, HEMA, photoinitiators.	8FJ
Supreme XT	3M ESPE Dental Products, St Paul, MN, USA	Filler: 59.5 vol.% combination of aggregated zirconia/silica cluster filler with primary particle size of 5-20 nm, and a nonagglomerated 20 nm silica filler. Resin: Bis-GMA, Bis-EMA, UDMA, TEGDMA.	7EF
Biscover LV	Bisco Inc, Schaumburg, IL USA	Dipentaerythritol penta-acrylate esters and ethanol.	0700008228

Abbreviations: HEMA – Hydroxyethyl methacrylate; Bis-GMA: Bisphenol A diglycidylether dimethacrylate; Bis-EMA: Bisphenol A polyetheylene glycol diether dimethacrylate; UDMA: Urethane dimethacrylate; TEGDMA: Triethylene glycol dimethracrylate.

- Group I: The samples were individually stored in Eppendorf tubes containing 10 mL of hydrochloric acid (HCl 0.01 M, pH 1.6, Apothicário, Araçatuba, Brazil) for five weeks.
- Group II: The samples were individually stored in Eppendorf tubes containing 10 mL of soft drink with pH 3.6 (Sprite®, Coca-Cola Co, Ribeirão Preto, Brazil) for five weeks.
- Group III: The samples were individually stored in Eppendorf tubes containing 10 mL of distilled water with pH 6.37 for five weeks.

The Eppendorf tubes were sealed and the solutions were changed weekly.

After the immersion in acid solutions, the specimens were submitted to new microhardness and surface roughness measurements in the same manner as described previously.

The surface roughness and microhardness data were submitted to repeated-measures analysis of variance at a 5% significance level and pairwise comparisons were performed using the Fisher protected least significant difference test.

RESULTS

Microhardness

Table 2 shows that the highest changes in the microhardness values occurred in the samples stored in Sprite® soft drink and HCl ($p<0.0001$) for all studied materials. The storage in distilled water decreased the microhardness values for all materials, except for Vitremer ($p=0.1879$). The microhardness of the resin-composite samples sealed with Biscover LV after storage in distilled water or Sprite® soft drink was not affected ($p=0.8422$).

The highest microhardness values were found for the Supreme XT resin-based material ($p<0.0001$). The resin composite sealed with Biscover LV showed intermediate microhardness values in all the storage conditions ($p<0.0001$), whereas the lowest values were found for resin-modified glass ionomer cements. Fuji II LC showed higher microhardness values compared with Vitremer before storage and after immersion in Sprite® soft drink ($p<0.0001$). There was no difference between the resin-modified glass ionomer materials after storage in distilled water ($p=0.7052$) and HCl ($p=0.1307$). These values are shown in Table 2.

Surface Roughness

Table 3 shows that immersion in Sprite® soft drink resulted in a significant increase of surface roughness for Vitremer and Fuji II LC resin-modified glass ionomer cements. Fuji II LC showed the greatest change when immersed in HCl solution ($p<0.0001$). The Sprite® soft drink decreased the surface roughness for Supreme-XT and for samples covered with Biscover LV, with a significant difference when compared with the initial values ($p<0.0001$).

Materials	Before Storage	After Storage		
		Water	Sprite® Soft Drink	Hydrochloric Acid
Fuji II LC	25.00 (3.05) c,a	21.22 (1.90) c,b	11.34 (1.41) c,c	12.58 (1.60) c,c
Vitremer	21.70 (1.57) D,a	20.98 (1.48) c,a	7.22 (1.14) D,c	13.60 (1.50) c,b
Supreme XT	44.96 (2.51) A,a	41.26 (1.22) A,b	35.96 (0.81) A,c	34.74 (0.97) A,c
Supreme XT + Biscover	32.21 (2.17) B,a	30.54 (0.82) B,b	30.38 (1.03) B,b	27.52 (1.72) B,c

Table 2: *Microhardness of the Studied Materials in Knoop Hardness Numbers: Mean (SD)*

Means followed by distinct capital letter in columns and lower case letter in rows are statistically different (p<0.05).

Table 3 also shows that for both initial measurements and after storage in water, the highest values were obtained by Vitremer, with a significant difference from the other materials ($p<0.0001$), whereas the lowest values were obtained for the resin sealed with Biscover LV ($p<0.0001$).

DISCUSSION

The mouth is considered the ideal environment for predicting the behavior of restorative materials. However, due to the complexity and diversity of intraoral conditions, *in vitro* models are very important for providing an insight into the fundamental mechanisms of biodegradation.[11] Thus, this *in vitro* study used the Sprite® soft drink (pH 3.6) and HCl 0.01 M (pH 1.6) to simulate the frequent consumption of acidic beverages and gastric reflux, respectively.

Data analysis revealed that the acid solutions altered the microhardness and surface roughness of the materials (Tables 2 and 3), leading to rejection of the null hypothesis of the study. The highest alterations in microhardness and surface roughness occurred after immersion in Sprite® soft drink and HCl (Tables 2 and 3). Badra and others[17] and Francisconi and others[10] also related a decrease in the microhardness of composite resin and resin-modified glass ionomer after immersion in soft drinks. A reduction in the surface hardness of composite resins 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 (TEG-DMA).[10,11,18] The softening of the resin matrix could promote displacement of the filler particles, contributing to the formation of a rough surface, as observed in this study.[17,19] These results were also confirmed by Abu-Bakr and others in 2000; they showed that alcoholic beverages and soft drinks affect the compressive strengths, microhardness,

Materials	Before Storage	After Storage		
		Water	Sprite® Soft Drink	Hydrochloric Acid
Fuji II LC	0.065 (0.019) B,a	0.062 (0.004) B,a	0.111 (0.014) B,b	0.139 (0.016) A,c
Vitremer	0.079 (0.014) A,a	0.088 (0.012) A,a	0.177 (0.012) A,b	0.084 (0.012) B,a
Supreme XT	0.066 (0.019) B,a	0.055 (0.012) B,ab	0.052 (0.009) C,b	0.057 (0.012) C,ab
Supreme XT + Biscover	0.047 (0.011) C,a	0.043 (0.007) C,ab	0.040 (0.005) D,b	0.045 (0.005) D,ab

Table 3: *Surface Roughness of the Studied Materials in Microns: Mean (SD)*

Means followed by distinct capital letter in columns and lower case letter in rows are statistically different (p<0.05).

solubility, and surface texture of restorative materials.[20] Furthermore, Sales-Peres suggested that the period of time that the teeth are bathed in the acidic environment is more crucial to erosion than the volume of beverage consumed. The erosive effect of carbonated drinks might be exaggerated because while being consumed, these beverages are frequently held in the mouth until all the bubbles have dissipated.[21]

The greater instability of resin-modified glass ionomer cement after immersion in acidic solutions when compared with composite resin could be explained by matrix dissolution in the periphery of the glass particles of glass ionomer, which could result from dissolution of the siliceous hydrogel layer.[11,22,23] Other factors that could also have contributed to these results are the manipulation and composition of these materials. The resin-modified glass-ionomer materials, such as Fuji II LC and Vitremer, present glass particles in their composition that may be responsible for the lower homogeneity and rougher surface. Furthermore, the components have different hardness and they are manually handled, which can generate porosity due to the inclusion of visually imperceptible air bubbles.[24] Scanning electron microscopy (SEM) studies 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[20] that could explain the higher surface roughness values of resin-modified glass ionomer materials (Table 3). Despite acids causing damage to the surface integrity of glass ionomer cements, this erosive loss of material may be accompanied by an increase in the pH of the acid solution resulting from these material degradation products being able to buffer the external storage media. This buffering effect is likely to be beneficial in protecting teeth from the occurrence and development of dental erosion.[22]

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 nano-sized and regular, allowing the incorporation of a large inorganic volume.[25] According to dos Santos and others (2003), composites with small filler particles are more wear-resistant because they are more homogeneous and their particles are less prominent on the surface, resulting in a lower roughness.[14] Whereas the type of filler and size and quantity of the particles influence the properties and quality of polishing of composite resins, the reduction in space between the inorganic

nano-clusters is possibly responsible for their superior physical properties.[25]

In this study, the composite resin sealed with Biscover LV showed significantly lower microhardness values when compared with the values of unsealed resins (Table 2). These differences could be attributed to resin monomers and the ethanol solvent present in the sealant surface (Table 1). Although the surface hardness was lowered by the sealant application, Bertrand and others demonstrated an improvement of the surface quality of composites due to the disappearance of microcracks and minor surface irregularities when examined by SEM.[26] However, according to the results of the present study, the maintenance of lower hardness and surface roughness values for the sealed composite after immersion in acid solutions could be indicative of the sealing material remaining on the composite surface, showing that the material was able to withstand the acid challenges.

This research showed some of the changes caused by low-pH solutions. Nevertheless, further investigations, including *in situ*, clinical, and epidemiological studies, are required. The high occurrence of noncarious lesions in dental tissues, present-day eating habits, dynamics of the oral cavity, and the effects of saliva should be considered in future studies.

According to our results we conclude the following:

1. The acids used in this study were able to change the hardness and surface roughness of restorative materials.
2. The resin-modified glass ionomer cements showed the most significant changes after immersion in acid solutions.
3. The composites sealed with Biscover LV, even after immersion in acid solutions, showed the lowest surface roughness values and the least degradation in hardness, especially when subjected to low-pH solutions.

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