©Operative Dentistry, 2008, **33-2**, 203-208

Effect of Erosive pH Cycling on Different Restorative Materials and on Enamel Restored with These Materials

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Clinical Relevance

The different types of restorative materials tested were not able to protect adjacent enamel from erosion. Thus, the ability of a restorative material to prevent tooth erosion should not be considered when choosing a material.

SUMMARY

This *in vitro* study evaluated the effect of erosive pH cycling on the percentage of surface microhardness change (%SMHC) and wear of different restorative materials and bovine enamel restored with these materials. Eighty enamel specimens were randomly divided into eight groups according to the restorative materials and immersion media used: GI/GV—resin-modified glass-ionomer, GII/GVI—conventional glass-ionomer, GIII/GVII—resin composite and GIV/GVIII—amalgam. Over a period of seven days, groups GI to GIV were immersed in a cola drink (ERO) for 5 minutes, 3x/day and kept in artificial saliva between erosive cycles. Groups GV to GVIII were immersed in artificial saliva (SAL) throughout the entire experimental period (control). Data were tested for significant differences using ANOVA and Tukey's tests (*p*<0.05). For %SMHC, considering the restorative materials, no significant differences were detected among the materials and immersion media. Mean wear was higher for the resin modified glass ionomer cement when compared to conventional cement, but those materials did not significantly differ from the others. For enamel analyses, erosive pH cycling promoted higher wear and %SMHC compared to saliva. There were no significant differences in wear and %SMHC of enamel around the different restorative materi-

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DOI: 10.2341/07-77

als, regardless of the distance from the restorative material (50, 150 or 300 µm). In conclusion, there were only subtle differences among the materials, and these differences were not able to protect the surrounding enamel from erosion.

INTRODUCTION

Dental erosion is a well recognized problem that has apparently increased among the younger population in the last few decades.[1] Erosion involves a chemical removal of superficial hard tissue from the tooth by acids from soft drinks and fruit juices or from eating disorders and gastric reflux.[2]

Initially, erosive tooth wear is limited to enamel and the teeth are not hypersensitive. Restorations may be inserted because of esthetic needs and/or to prevent further progression. Direct composite coatings should be considered the treatment of choice. These coatings seal the enamel and reestablish tooth contour, thus minimizing further enamel loss by acid exposure.[3] In advanced cases, when dentin becomes exposed, restorative materials, such as glass ionomer cements (resin-modified or conventional) and resin composites, are generally used in daily clinical practice and can be used to reestablish tooth structure, function and aesthetics, as well as to control hypersensitivity.[3]

The longevity of dental restorations depends on the durability of the material and its properties, such as wear resistance, durability of the interface between tooth and restoration and the level of tooth destruction.[3] Under acidic conditions, all dental restorative materials have shown degradation over time.[4-5] However, composite materials have shown higher durability.[3]

On the other hand, fluoride released from glass ionomer cements is taken up by tooth structure,[6-7] and a reduced caries experience has been observed in clinical practice.[8-11] However, when an erosive challenge occurs, there is a lack of knowledge about the influence of the properties of the restorative materials, such as fluoride liberation, on tooth structure adjacent to these restorations.

This study evaluated the effect of erosive pH cycling on different restorative materials and the effect of these materials on surrounding enamel subjected to this erosive challenge by evaluating the percentage of surface microhardness change (%SMHC) and wear (profilometry).

METHODS AND MATERIALS

Experimental Design

The factors studied were restorative materials at four levels, immersion media at two levels and enamel distance from the restoration at three levels. Eighty enamel specimens obtained from bovine teeth were selected and randomly distributed to eight groups according to the restorative materials and immersion media used: GI and GV—resin-modified glass-ionomer; GII and GVI—conventional glass-ionomer; GIII and GVII—resin composite and GIV and GVIII—amalgam (Table 1). Over seven days, Groups GI to GIV were immersed in a cola drink (ERO) for 5 minutes 3x/day and kept in artificial saliva between erosive cycles. Groups GV to GVIII were immersed in artificial saliva (SAL) for the entire experimental period (control). Alterations in the restorative materials and enamel were determined using profilometry (wear) and microhardness (%SMHC) tests.

Specimens Preparation

Enamel specimens (4x4x2.5 mm) were prepared from incisor bovine teeth that were freshly extracted and stored in 2% formaldehyde solution (pH 7.0) for 30 days at room temperature. The teeth were cut using an ISOMET Low Speed Saw cutting machine (Buehler Ltd, Lake Bluff, IL, USA) and two diamond disks (Extec Corp, Enfield, CT, USA), which were separated by a 4 mm diameter spacer. The enamel surface of the specimens was ground flat with water-cooled carborundum discs (320, 600 and 1200 grades of Al_2O_3 papers; Buehler) and polished with felt paper wet by diamond spray (1 µm; Buehler), resulting in the removal of about 100 µm depth of enamel, which was controlled with a micrometer. The surface microhardness determination was performed by five indentations (Knoop diamond, 25 g, 5 seconds, HMV-2000; Shimadzu Corporation, Tokyo, Japan) for selection purposes.

Enamel specimens, with microhardness ranging from 322 to 348 KHN, were randomly distributed into eight

Table 1: *Materials Used in the Different Groups*

Material (Groups)	Category	Manufacturer	City/State, Country	Batch #
Vitremer (GI and GV)	resin-modified and high viscosity glass-ionomer	3M ESPE	Sumaré/SP, Brazil	20050824
Ketac Molar (GII and GVI)	conventional glass-ionomer	3M ESPE	Sumaré/SP, Brazil	182968
Z 250 (GIII and GVII)	resin composite	3M ESPE	Sumaré/SP, Brazil	5BY
Dispersalloy (GIV and GVIII)	amalgam	Dentsply Indústria e Comércio Ltda	Petrópolis-RJ, Brazil	000630

groups, according to the restorative materials and immersion media. Cavities were then prepared in the center of the enamel surface, using drill #2292 (KG SORENSEN, São Paulo, Brazil), which had an automatic stopping device to standardize depth of the preparation (1.5 mm). Each material was handled as outlined by the manufacturers. After placing the material in the prepared cavity, the surface of the restorative materials was covered with a polyester strip and a glass slab under pressure to expel any excess material from the cavity. On the light cured materials, the polymerization procedure was carried out through the polyester strip based on the manufacturers' recommended exposure time, using a light-curing device (Optilux, Demetron, Kerr Corp, Danbury, CT, USA). After seven days of storage at 37°C in 100% relative humidity, in order to maintain reference surfaces for lesion depth determination, the restorations were ground flat in the same manner as described above. The initial surface microhardness of the restorative materials and enamel (SMH) was performed (Knoop diamond, 25 g, 30 and 5 seconds, respectively). To provide a stable, accurate indentation, the indenting time was longer for the restorative materials. For wear references, two layers of nail varnish were applied on half of the surface of enamel and restorative material after microhardness assessment.

Erosive pH-cycling

In Groups I through IV, the specimens were subjected to a pH-cycling model for seven days. Three pH-cycles were performed each day at 8, 14 and 20 hours. The specimens were immersed in a cola drink (Coca-Cola, [pH-2.6, Phosphate-5.43mM Pi, Calcium-0.84 mM Ca^{2+}, Fluoride-0.13 ppm F, titratable acid-40.0 mmol/L OH- to pH 5.5 and 83.6 mmol/L OH- to pH 7.0][12], Spal, Porto Real, RJ, Brazil) for five minutes (30 mL per block) and in artificial saliva (1.5 mmolL^{-1} $Ca[NO_3]_2.4H_2O$, 0.9 mmolL^{-1} $NaH_2PO_4.2\ H_2O$, 150 mmolL^{-1} KCl, 0.1 molL^{-1} Tris buffer, 0.03 ppm F, pH 7.0, 30 mL per block)[13] between erosive cycles, under agitation and at room temperature. During the remaining time, the specimens were maintained in artificial saliva.

Specimens in Groups V through VIII were maintained in artificial saliva for the entire experimental period as a control for erosion and to simulate alterations produced on restorative materials in a moist, *in vivo* environment.

Microhardness and Wear Determinations

Initially, enamel surface microhardness was measured as described in the *Enamel Specimens Preparation* section. Five indentations, 100 µm from each other, were performed in both the enamel and restorative material (SMH). After pH cycling, nail varnish, which was on the surfaces, was carefully cleaned with acetone-soaked cotton wool[13-14] and a final microhardness test (SMH_1) was performed in the middle of the exposed area of the restorative materials and enamel at distances of 50, 150 and 300 µm from the external margin of the restoration. The percentage of surface microhardness change for enamel and the restorative material was calculated as follows: 100(SMH1—SMH)/SMH.

Wear was determined in relation to the reference surface by using a profilometer (Hommel Tester T1000, VS, Schwenningen, Germany).[14] Five readings were performed on each specimen via scanning from the reference to the exposed surface and an average of each group was obtained for enamel and the restorative material (µm).

Statistical Analysis

Assumptions of equality of variance and normal distribution of errors were checked for all variables tested. Since the assumptions were satisfied, ANOVA and Tukey's tests were carried out for statistical comparisons and the significance limit was set at 5%.

RESULTS

Restorative Materials Evaluation

ANOVA revealed a significant effect of the factors material, immersion media and their interaction for variable wear. The Tukey's test showed a higher wear value for resin modified glass ionomer cement when compared to conventional cement, but these materials did not differ from the others. As for the response variable %SMHC, ANOVA did not show a significant effect of the factors under study, as shown in Table 2.

Enamel Evaluation

For both response variables (%SMHC and wear), ANOVA revealed a significant effect only for the factor immersion media. Compared to storage in saliva, pH cycling promoted higher wear and %SMHC of enamel. There were no significant differences in wear and %SMHC of enamel around the different restorative materials at the distinct distances tested (Table 2).

DISCUSSION

The increased risk of dental erosion in the presence of dietary and gastric acids highlights the need for understanding the phenomena of degradation of restorative materials and their interaction with adjacent enamel.

Despite the mouth being the ultimate testing environment for predicting the behavior of restorations[15] due to the complexity and diversity of intra-oral conditions, *in vitro* models are very important for providing insight into the fundamental mechanisms of biodegradation. It is known that, during consumption, food or drink only comes in brief contact with tooth surfaces before it is washed away by saliva.[12,14] However, in previous studies, substrates usually contacted acidic foodstuffs for a prolonged period of time or did not account

Table 2: *Mean Percentage of Superficial Microhardness Change (%SMHC) and Wear for the Experimental Groups*

Groups		Restorative Materials		Bovine Enamel			
					%SMHC*		
		Wear (µm)	% SMHC	Wear (µm)	50 µm	150 µm	300 µm
ERO (erosion in Coca Cola)	Vitremer (GI)	0.333[a]	-9.887[a]	2.351[a]	-67.437[a]	-66.758[a]	-66.372[a]
	Ketac Molar (GII)	0.194[b,c]	-8.734[a]	-2.549[a]	-64.122[a]	-63.021[a]	-62.268[a]
	Z 250 (GIII)	0.248[a,b]	-2.561[a]	2.503[a]	-64.851[a]	-62.876[a]	-62.960[a]
	Dispersalloy (GIV)	0.272[a,b]	-1.147[a]	-2.181[a]	-65.031[a]	-64.813[a]	-62.211[a]
SAL (immersion in saliva)	Vitremer (GV)	0.117[c,d,e]	-3.844[a]	0.108[b]	+1.893[b]	+1.467[b]	+0.877[b]
	Ketac Molar (GVI)	0.095[d,e]	-4.164[a]	0.103[b]	+5.345[b]	+1.834[b]	+4.684[b]
	Z 250 (GVII)	0.147[c,d]	-0.232[a]	0.071[b]	+1.392[b]	+3.080[b]	+4.036[b]
	Dispersalloy (GVIII)	0.049[e]	+3.878[a]	0.092[b]	+1.956[b]	+2.327[b]	+2.004[b]

Values in the same column followed by distinct letters indicate statistical significance ($p<0.05$).
**No significant differences were detected for %SMHC at the distinct distances from the restoration margin.*

for the role of saliva.[16-24] The current study was designed to overlap the above-mentioned limitation of *in vitro* studies by employing a dynamic erosive pH-cycling model.

Another factor of the experimental design that could affect the material's behavior is the specimens' storage before pH cycling. The one-week period chosen was carried out to allow post-irradiation hardening of the composites and stabilization of the acid-base reaction of glass ionomer cements.[17-18,25-26] As the greatest change in hardness has been shown to occur within the first seven days and the hardness of composites is affected by conditioning for seven days, this period of conditioning was selected for this experiment.[19]

Various assessment techniques have been applied to evaluate the degradation of dental materials and loss of dental hard tissue by erosive challenges, such as microhardness,[16-17,19-21] surface roughness,[21,27] weight changes,[22] compressive, biaxial flexure and shear punch strength[18,23] and wear.[22,24] It becomes evident that the complex nature of erosive material changes and mineral loss might not be comprehended by a single technique, thus requiring application of different approaches for a full understanding of the phenomena.[28] Taking this aspect into account, in the current study, microhardness and wear tests were performed.

Based on the results of this study, the wear of restorative materials, when specimens were submitted to erosive challenge, was higher than that observed for the control specimens, which were stored in artificial saliva. However, it is important to consider that the wear values for groups submitted to acidic beverages were low, around 0.3 µm. These data are in agreement with the findings of Shabanian and Richards.[24] These authors found wear values around 0.1 µm for composites, conventional GIC and resin-modified GIC in neutral conditions and higher wear values (0.2–8.1 µm) in an acidic environment (pH 1.2).

After pH cycling, resin modified glass ionomer cement, resin composite and amalgam showed similar wear. Resin modified glass ionomer cement presented higher wear when compared to conventional cement, which did not differ from resin composite and amalgam. This result was not expected, since previous studies have reported higher wear for conventional GICs and lower wear for resin composites.[24] This difference could be attributed to the low wear values found. It should be noted that dissolution of enamel due to acid attack leads to surface roughening of about 0.4 µm. Therefore, reliable detection of minimal losses below 1 µm is generally difficult to accomplish with profilometry.[28]

In relation to %SMHC, all the evaluated materials showed similar values, without statistically significant differences between the experimental (pH cycling) and control groups (storage in saliva). Badra and others[21] also found no significant %SMHC of the restorative materials submitted to an erosive challenge for seven days. The minimal surface alteration in microhardness could be explained by the type of beverage used for pH cycling and the time of acidic exposure. Aliping-McKenzie and others[20] showed a greater reduction in surface hardness for apple and orange juice when compared to colas, the beverage used in this study. Orange and apple juice contain carboxylic acids, which are capable of chelating ions, such as calcium, and forming complexes of reasonable solubility in water. On the other hand, a cola drink contains phosphoric acid, which does not chelate calcium, resulting in less damage to tooth structure and/or dental materials.[20] In previous studies, dental materials were continuously immersed in acidic media for one week.[17-19] However, in the current study, the total time exposure of specimens to acid was one hour and 15 minutes, and possibly this period was not able to promote significant microhardness alterations and differences among the studied materials. Additionally, in this study, a tendency towards numerical differences among the materials was observed, which has been reported in the literature.[16-24] Glass ionomer cements showed the highest microhardness losses, followed by resin composites and amalgam, respectively. These results could be explained by the matrix dissolution peripheral to glass particles of the glass ionomer, which could result from dissolution of the siliceous hydrogel layer.[27,29] On the other hand, acid could also attack the resin (to a lesser extent), since a reduction in the surface hardness of resin composites soaked in organic acids has been reported, due to softening of Bisphenol-A-glycidyl methacrylate (Bis-GMA)-based polymers,[30] which could result from leaching of diluent agents, such as Triethylene glycol dimethacrylate (TEGDMA).[31]

In accordance with the current literature, enamel presented higher wear when compared to restorative materials.[16,24] As expected, the acidic drink promoted significant wear of dental enamel, and storage in saliva did not provide enamel alterations.[14,24]

Despite acids adversely prejudicing the surface integrity of GICs, this erosive loss of material may be accompanied by an increase in the pH of the acid solution by these materials being able to buffer external storage media.[20,27] Such a buffering effect is likely to be beneficial when protecting teeth from the occurrence and evolution of dental erosion. However, the additional preventive effect of these materials on enamel subject to erosion could not be noticed with the response variables used in this study.

This research data suggest that there is little superficial microhardness change and wear of the studied restorative materials, and none of the materials had a preventive effect against erosion on adjacent enamel. However, a longer period of erosive pH cycling and the use of other types of beverages can be useful to complement the results of this study.

CONCLUSIONS

Based on the results of this study, it was concluded that:

1. The wear values of tested materials after pH cycling were discrete, but significantly higher than those obtained for the control regimen;
2. There were no significant differences among materials and between pH cycling and the control regimen for the response variable %SMHC and
3. The restorative materials had no influence on the erosion of adjacent enamel.

Acknowledgements

This study was supported by CAPES–Brazil.

(Received 15 April 2007)

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