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Original Article

In vitro Effect of Erosive Challenge on Surface Microhardness of Orthodontic Composites

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Abstract

Objective: To evaluate *in vitro* the effect of erosive challenge and continuous immersion in cola drink on surface microhardness of orthodontic composites. **Material and Methods:** Forty samples of three composites (Transbond XT, Quick Cure and Ortho Cem) were prepared (4 x 2 mm) and distributed into 4 groups (n=10): erosive challenge, artificial saliva immersion (control 1), continuous cola immersion and artificial saliva immersion (control 2). Erosive challenge was performed 4 times per day (5 min) in cola drink for 2 h in artificial saliva for 7 days. Samples of continuous cola immersion group were soaked in cola drink for 5 weeks and the beverage was renewed every two days. Control samples were immersed in artificial saliva for 7 days (control 1) and 5 weeks (control 2). Vickers microhardness (VHN) measurements were performed before and after erosive challenge and continuous immersion. Data were evaluated by paired Student's T-test, ANOVA one-way and Tukey tests ($\alpha = 0.05$). **Results:** After 7 days of erosive challenge, there was no statistical difference between VHN values before and after erosive challenge and artificial saliva immersion. However, after 5 weeks of cola drink and artificial saliva immersion, significant reduction in VHN values was observed for all composites when compared to baseline values, and specimens immersed in cola drink showed lower VHN compared to those immersed in saliva, regardless of composite composition. **Conclusion:** After 7 days of erosive challenge, there was no alteration in superficial VHN of orthodontic composites. However, after 5 weeks of immersion in cola drink, significant reduction of VHN values was observed for all composites.

Keywords: Tooth erosion; Composite resins; Orthodontic appliances.

Introduction

Dental erosion is the irreversible loss of tooth surface without bacterial involvement [1]. The etiological factors involved in dental erosion are considered extrinsic (acid exposure by diet) and intrinsic (acid reflux, anorexia and bulimia nervosa) [1,2]. Currently, the acids present in foods and drinks are considered the major etiological factors for the development of erosive lesions in enamel [3].

In orthodontic practice, white spot carious lesions are frequently seen around the brackets, especially when hygiene is not satisfactory [4,5]. However, the prevalence of dental caries has decreased in the last years, but an increase in the consumption of soft drinks, sports drinks and wine has been observed, which has led to the appearing of dental erosion [6]. The reduced pH of the oral environmental due to the consumption of acidic beverages can cause demineralization of enamel around brackets, which can interfere in their retention to enamel [6,7].

The most commonly used material for retention of brackets during fixed orthodontic treatment is resin composites. The surface properties of composites can be used to evaluate the surface degradation of the material and to predict its resistance in the oral environment [8,9]. As the exposure to acid can decrease the bond strength of brackets to enamel [6,7], it would be interesting to investigate the surface properties of orthodontic composites after erosive challenge.

Many studies have been conducted to evaluate the surface properties of restorative materials after erosive challenge [8-10], but little information is available in literature about the surface properties of composites for bonding orthodontic brackets to enamel after erosion [6].

Thus, the objective of this study was to evaluate the effect of dynamic erosive challenge and continuous immersion in cola drink on surface microhardness of orthodontic composites.

Material and Methods

Specimen Preparation

Three orthodontic composites were investigated in this study: Transbond XT (3M ESPE, St. Paul, USA), Quick Cure (Reliance Orthodontic Products, IL, USA) and Ortho Cem (FGM, Joinville, SC, Brazil). The composition of materials investigated is described in Table 1.

Table 1. Composition of materials investigated.

Composite/ Batch number	Composition (Provided by the Manufacturer)
Ortho Cem (FGM, Joinville, SC, Brasil)	Bisphenol-A Diglycidyl ether metacrylate (BisGMA) (25-35%), Triethyleneglycoldimethacrylate (TEGDMA) (10-15%), Methacrylated phosphate monomer (>2%), Silane treated silicondioxide (45-60%), Camphorquinone (<1%) and Sodium Fluoride (>1%).
Transbond XT (3M ESPE Dental Products, St. Paul,USA), 481914	Silane Treated Quartz (70-80%), Bisphenol-A Diglycidyl Ether Dimethacrylate (10-20%), Bisphenol-A Bis(2-Hydroxyethyl Ether) Dimethacrylate (5-10%) and Silane Treated Silica (>2%).
Quick Cure, (Reliance Orthodontic Products, Inc.,IL, USA), 123643	Fused Silica (50-75%), Bisphenol-A glycidyl methacrylate (1-5%) and Triethylene glycol dimethacrylate (1-5%).

Forty specimens of each material were prepared according to manufacturer's instructions. Specimens were prepared using silicone molds (4 mm in diameter x 2 mm in height) on a glass plate to obtain cylindrical samples. The surface of each specimen was covered with polyester strip (Probem Ltda, Catanduva, Brazil) to obtain a smooth surface. Then, specimens were polymerized for 40 s with LED curing light (1200 mW/cm² - Radii Cal. SDI, Bayswater, Victoria, Australia) and maintained in 100% relative humidity for 24 h until microhardness test was conducted.

Specimens of each composite were divided into four groups (n = 10) [7,8] according to the experimental erosive model: erosive challenge (7 days), artificial saliva immersion (control 1), continuous cola immersion (5 weeks) and artificial saliva immersion (control 2). In total, twelve groups (n = 10) were formed: Group 1 - Transbond XT/control 1; Group 2 - Transbond XT/erosive challenge; Group 3 - Quick Cure/control 1; Group 4 - Quick Cure/erosive challenge; Group 5 – Ortho Cem/control 1; Group 6 – Ortho Cem/erosive challenge; Group 7 - Transbond XT/control 2; Group 8 - Transbond XT/continuous cola immersion; Group 9 - Quick Cure/control 2; Group 10 - Quick Cure/ continuous cola immersion; Group 11 - Ortho Cem/control 2 and Group 12 - Ortho Cem/ continuous cola immersion.

Surface Vickers Microhardness

Microhardness measurements were performed with a hardness tester (HMV II; Shimadzu Corporation, Kyoto, Japan), using Vickers indenter (VHN) and load of 200 g with dwell time of 15 s [11]. Five indentations were performed in each specimen, at least 100 μm apart, and the mean VHN value was obtained. VHN measurements were conducted before (baseline) and after erosive challenge and continuous cola immersion.

Erosive Challenge and Continuous Cola Immersion

The bottom of all samples was covered with nail polish; only the top surface of specimens was free of nail varnish to be exposed to erosion or artificial saliva.

For dynamic erosive challenge, specimens were immersed in cola drink (Coca-Cola®, SP, Brazil - pH 2.3) at room temperature in individual containers (10 mL/sample) for 5 minutes 4 times/day [12]. Subsequently, specimens were rinsed thoroughly with deionized water and immersed in artificial saliva with pH 7.0 (10 mL/sample) at room temperature for 2 h, both for erosive challenges and overnight [12]. Artificial saliva was developed according to Amaechi, Higham and Edgar [13]. Erosive challenge was repeated for 7 days. Cola drink and artificial saliva were replaced after every cycle. During the acidic cycles, specimens were kept in hermetically sealed containers to prevent loss of carbonation from the cola drink. Specimens in control 1 group were immersed in artificial saliva for 7 days. Artificial saliva was replaced every day.

For continuous cola immersion, each specimen was immersed in 10 mL of cola drink (Coca Cola®, SP, Brazil - pH 2.3) at room temperature in individual containers for 5 weeks [10]. Samples of control 2 group were immersed in 10 mL of artificial saliva for 5 weeks. Cola drink and artificial saliva were replaced every two days. During immersion, samples were kept in hermetically sealed containers to prevent loss of carbonation from the cola drink.

Statistical Analysis

Data were analyzed using GraphPad Instat software, version 2.0 (GraphPad software, La Jolla, CA, USA) at significance level $\alpha=0.05$. Since all variables tested satisfied the assumptions of normal distribution, one-way ANOVA and Tukey's test were performed for statistical comparisons among VHN measurements of composites in each storage solution group. Student's paired t-test was used to compare VHN measurements before and after erosive challenge or continuous cola immersion for the same composite.

Results

After 7 days of erosive challenge, no statistical difference was observed between VHN values before and after erosive challenge (p=0.75) and artificial saliva immersion (control 1) (p=0.82) (Table 2). Quick-cure group showed the highest VHN values, followed by Transbond XT and Ortho Cem (p=0.01) (Table 2), regardless of immersion storage solution group.

Table 2. Vickers hardness (VHN) values of orthodontic composites before and after erosive challenge in cola drink.

	Storage solution (VHN mean ± standard deviation)			
Composite	Baseline	After Saliva	Baseline	After erosion
Transbond XT	$59.9 \pm 0.6^{A**,b*}$	$58.2 \pm 1.3^{A,b}$	$60.3 \pm 1.1^{A,b}$	$58.8 \pm 3.2^{A,b}$
Quick-Cure	$77.4 \pm 0.6^{A,a}$	$78.5 \pm 4.6^{A,a}$	$76.1 \pm 1.8^{A,a}$	$74.5 \pm 4.5^{A,a}$
OrthoCem	$52.6 \pm 0.3^{A,c}$	$51.7 \pm 0.6^{A,c}$	$52.0 \pm 0.4^{A,c}$	$51.2 \pm 1.5^{A,c}$

*Same uppercase letters indicate that there was no significant difference between baseline and values obtained after saliva/ erosion of each composite (paired t test, p > 0.05); **Same lowercase letters indicate that there were no significant differences among composites at baseline or after saliva/ erosion (one-way ANOVA and Tukey's test, p>0.05).

After 5 weeks of continuous immersion in cola drink and artificial saliva, there was a significant decrease in VHN values for all composites when compared to baseline values (p=0.001) (Table 3). When VHN values were compared between saliva immersion and cola immersion after 5 weeks, all composites had significantly lower VHN values after immersion in cola drink (p=0.01) (Table 3).

Table 3. Vickers hardness (VHN) values of orthodontic composites before and after continuous immersion in cola drink.

	Storage solution (VHN mean ± standard deviation)			
Composite	Baseline	After Saliva	Baseline	After erosion
Transbond XT	$63.0 \pm 0.9^{a*}$	$57.4 \pm 1.2^{b,A**}$	62.4 ± 0.7^{a}	$55.5 \pm 1.3^{b,B}$
Quick Cure	76.9 ± 1.4^{a}	$73.8 \pm 1.6^{b,A}$	77.4 ± 1.9^{a}	$68.4 \pm 0.6^{b,B}$
Ortho Cem	55.2 ± 0.7^{a}	$51.9 \pm 0.7^{b,A}$	54.6 ± 1.7^{a}	$45.4 \pm 1.4^{b,B}$

* Same lowercase letters indicate that there was no significant difference between baseline and values obtained after saliva/ erosion of each composite (paired t test, p > 0.05); ** Same uppercase letters indicate that there were no significant differences among composites at baseline or after saliva/ erosion (one-way ANOVA and Tukey's test, p>0.05).

Discussion

The oral cavity is the adequate environment for predicting the degradation and durability of dental materials, but given the complexity of intra-oral conditions, *in vitro* studies are performed to simulate the oral environment. Although *in vitro* studies are generally difficult to extrapolate to *in vivo* conditions, they have the advantage of controlling individual parameters such as erosion time, erosive agent and pH value [14].

Soft-drinks are widely consumed by the world population [15,16] and Coca-Cola® is the most widely consumed [17]. Coca-cola has high erosive potential due to its low pH and contains phosphoric acid and low calcium and fluoride concentrations [6,18]. In the oral cavity, the contact of the enamel with acidic beverage is usually limited to a few seconds before clearance by saliva [19]. Thus, studies using dynamic erosive challenge with cycles of immersion in acid beverage and saliva can better reproduce the oral environmental conditions when compared to continuous immersion of specimens in acid drinks [9]. Due to the use of two *in vitro* methodologies to simulate erosion, the present study investigated the effect of dynamic erosive challenge and continuous immersion in cola drink on surface hardness of orthodontic composites. The microhardness test is the most widely used to assess the surface changes of dental hard tissues and materials after erosion, it can predict the weakening of the surface caused by acids [20].

In the methodology with 7 days of erosive challenge, no statistical difference was observed between VHN values before and after erosive challenge and artificial saliva immersion (Table 2). These results demonstrated that there was no degradation in the surface of composites investigated. Some authors [8] also found no difference in surface hardness of glass ionomer cements, restorative composite and amalgam using the same time and erosion methodology of our study. In the present study, the total time exposure of specimens to acid was two hours and twenty minutes. Possibly, the exposure time to cola drink was not able to promote significant microhardness alterations on the surface of resin composites. However, difference in VHN values was observed among materials (Table 2). Ortho Cem group showed the lowest VHN values (before and after erosion and saliva immersion) compared to the other groups (Table 2). This result is probably related to the lower concentration of inorganic filler (45-60% silica) of Ortho Cem group compared to Quick-Cure (50-75%) and Transbond XT (70-80%) groups (Table 1). As Quick- Cure and Transbond XT have similar concentration of inorganic filler, the difference in VHN values among them can be associated to the different monomers in their composition.

After 5 weeks of continuous immersion of specimens in artificial saliva and cola drink, all groups showed reduction in VHN values (Table 3). This result showed the occurrence of degradation of composite surface in contact with artificial saliva over time. Human whole saliva is a complex mixture of salivary proteins and food residues and salivary proteins have several functions such as digestive, protective, calcium and mineral homeostase, antibacterial and antifungal function [21]. Esterases present in saliva, mainly cholesterol esterase-like and pseudocholinesterase hydrolase, can also be able to degrade composite resin components [22]. Monomers used in composites are mainly

Bis-GMA (Bisphenol-A Diglycidyl ether metacrylate) and the TEGDMA diluent monomer (Triethylene Glycol Dimethacrylate). The ester linkage between the two segments of monomers (Bis-Phenol-A and Triethylene Glycol) is the bond that is highly susceptible to hydrolysis by salivary enzymes [23].

However, the present study used artificial saliva that does not contain natural esterases. Probably, the reduced VHN values after saliva immersion caused degradation in the surface of the composite resin due to the plasticizing effect [24]. The reduction in hardness and other properties of composites caused by the plasticizing effect is a product of the separation of polymer chains by a molecule that does not form primary chemical bonds with the chain, reducing inter-chain interactions and surface properties [24].

Lower VHN values were observed after continuous immersion in cola drink for 5 weeks when compared to artificial saliva immersion (Table 3). In this *in vitro* methodology, the exposure time of specimens to the acid beverage increased in comparison to the total time of 7 days of dynamic erosive challenge. Thus, the increase in the immersion time in acidic beverage caused higher surface degradation of composites. The pH of cola drink can cause softening of Bis-GMA monomer and leaching of diluents such as TEGDMA and lead to the softening of polymer chains and displacement of inorganic fillers [8,12,25]. Probably, this process was the responsible for lower VHN values after continuous cola immersion. Some authors [9] also showed that longer immersion times in cola promoted higher hardness change on the surface of restorative composites.

The effect of 5 weeks of continuous immersion in cola drink on the composite surface could be applied to orthodontic patients who have the habit of consuming soft drinks. As in orthodontic practice, these composites used to bond brackets to enamel may remain in the oral cavity for a long period (two years or more), the erosive effect of acidic drinks could cause changes in the surface properties of composite and cause microleakage, which could interfere in the bracket retention.

The effect of erosive challenge on surface properties of restorative materials has been evaluated [8,9], but there is little evidence about the effect of erosion on orthodontic composite surface. Some researchers [7] investigated the *in vitro* and *in vivo* effect of Coca-Cola® and Sprite® on the resistance of metal brackets to shear forces in enamel. The acidic soft drinks tested showed a negative effect on bracket retention against shearing forces and enamel erosion. Another study [6] evaluated the effect of Coca-Cola® and Lemon Schweppes® on the bond strength and microleakage of brackets to enamel. It was found that the drinks produced erosion in enamel and caused microleakage on the resin/enamel interface, but this result did not affect the bond strength of brackets to the enamel. Thus, further studies are necessary to evaluate the effect of erosion on materials used in orthodontic practice.

The results of this study showed that 7 days of erosive challenge did not cause alterations on surface hardness of orthodontic composites, regardless of their composition. However, longer contact time with cola drink was able to reduce surface hardness. The use of longer periods of dynamic erosive challenge and the evaluation of other surface properties could be useful to complement this study.

Conclusions

1) After 7 days of dynamic erosive challenge in cola drink and saliva immersion, there was no alteration on the hardness values of orthodontic composites, regardless of material composition;
2) After 5 weeks of continuous immersion in cola drink and saliva, there was a decreasing of hardness values for all composites, regardless of material composition. Continuous cola drink immersion caused higher surface degradation compared to saliva immersion.

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