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Effect of Acid Neutralization and Mechanical Cycling on the Microtensile Bond Strength of Glass-ceramic Inlays

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Clinical Relevance

Acid neutralization of the ceramic surface etched with hydrofluoric acid appears to be unnecessary.

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SUMMARY

Objectives: To evaluate the hypothesis that a process of hydrofluoric acid precipitate neutralization and fatigue load cycling performed on human premolars restored with ceramic inlays had an influence on microtensile bond strength results (MTBS). Methods: MOD inlay preparations were performed in 40 premolars (with their roots embedded in acrylic resin). Forty ceramic restorations were prepared using glass-ceramic (IPS Empress). The inner surfaces of all the restorations were etched with 10% hydrofluoric acid for 60 seconds, rinsed with water and dried. The specimens were divided into two groups

(N=20): 1–without neutralization; 2–with neutralization. All the restorations were silanized and adhesively cemented (self-curing and self-etching luting composite system, Multilink). Ten premolars from each group were submitted to mechanical cycling (1,400,000 cycles, 50N, 37°C). After cycling, the samples were sectioned to produce non-trimmed beam specimens (vestibular dentin—restoration—lingual dentin set), which were submitted to microtensile testing. Results: Bond strength was significantly affected by the surface treatment (*p*<0.0001) (no neutralization > neutralization) and mechanical cycling (*p*<0.0001) (control > cycling) (2-way ANOVA and Tukey test, alpha=.05). Conclusion: Hydrofluoric acid precipitate neutralization appears to significantly damage the resin bond to glass-ceramic and should not be recommended. The clinical simulation of the specimens, by using mechanical cycling, is important when evaluating the ceramic-dentin bond.

INTRODUCTION

Adhesive dentistry has changed restorative practice by providing less invasive procedures that preserve the greatest amount of tooth structure. The search for greater longevity of these restorations has been a decisive factor in stimulating the development of new restorative materials. In all-ceramic restorations made with feldspar materials, the bond to resin contributes to the clinical success of the restorative approach.[1-4] Therefore, the bond between dental structure and ceramic should be optimal.

During cementation of all-ceramic restorations, it is necessary to use adhesive systems and resin cements.[1-4] The bond strength between resin cement and ceramic occurs by acid etching the ceramic surface, enabling mechanical interlocking and the use of an organic functional molecule, known as silane, thus promoting a bond between the inorganic substrates and organic polymers.[5-7] Some additional procedures can enhance or aid this bond, such as eliminating excess acid[8] and acid precipitates from the ceramic surface.[8-10] Canay and others[11] observed that there was formation of fluorosilicate precipitates of Na, K, Ca and Al after etching the glass-ceramic surface with hydrofluoric acid, which could be eliminated with an ultrasonic bath. Thus, the topographic pattern of the surface changes and, consequently, the resin bond, is enhanced. Saavedra[8] showed similar results, using microtensile bond strength and SEM analysis.

Despite the limitations of *in vitro* studies, mechanical fatigue tests conducted in a humid environment seem to be the best method for predicting the clinical performance of different materials and restorative techniques.[12-14] A mechanical fatigue test may lead to fracture of a structure after a repeated load. The fracture may be explained as the result of the spread of microscopic cracks from areas of force concentration, usually in areas presenting with macroscopic or molecular structural defects. Normally, fatigue tests are conducted in a humid environment, contributing to the degradation of the physical and mechanical properties of the restorative materials.[15-16] Ceramics are materials susceptible to fatigue, in which the accumulation of microstructural damage during mastication could lead to fracture. The majority of failures in ceramic restorations studied with quantitative fractrographic analyses presented cracks on the internal surface of the occlusal region, where the greatest stress was applied during the masticatory cycle.[17-20]

Based on the knowledge of the authors' of the current study, there have been no studies that have investigated the effect on bond between dentin and ceramic with mechanical cycling of premolars adhesively restored with MOD inlay glass-ceramic, whether or not they were submitted to acid neutralization. Therefore, the current study evaluated the influence on microtensile bond strength results of a process of hydrofluoric acid precipitate neutralization (neutralization) and mechanical fatigue testing performed on premolars restored with resin-bonded ceramic inlays.

METHODS AND MATERIALS

Forty human maxillary premolars were selected according to the inclusion criteria of there being no cracks in the tooth. According to its vestibular-lingual dimensions, the specimens were homogeneously divided into four groups (N=10).

The roots of each specimen were embedded in a plastic cylinder filled with chemically-cured acrylic resin (Dencrilay, Dencril, Caieiras, Brazil) up to 2 mm from the cervical line in the apical direction. To accomplish positioning of the teeth in resin, the occlusal surface was glued to an adapted surveyor, with the root perpendicular to the y-axis (ground); the acrylic resin was prepared and poured inside the cylinder up to half of the root (Ø=12 mm; h=20 mm) and the tooth was inserted into the resin to the appropriate dimensions (Figure 1).

Standardized cavity preparations (inlay type) were performed on all teeth using a conical trunk diamond bur with rounded angles (KG Sorensen 3131, Barueri, Brazil) mounted in a high speed handpiece and fixed to a modified optic microscope, which enabled reductions to be obtained as parallel as possible to the long axis of the tooth. The preparations had the following dimensions: vestibular-lingual width of 3 mm; 4 mm depth of the occlusal box, rounded internal line angles (Figures 2 and 3).

Impressions of the prepared teeth were created using polyvinyl siloxane (Elite, Zhermack, Badia Polesine, Italy). Master dies were produced and 40 pressed all-ceramic restorations were made from leucite-feldspar material (IPS Empress Esthetic, Lot: E53039, Ivoclar-Vivadent, Schaan, Liechtenstein) using the injection molding technique. All of the instructions recommended by the manufacturer were followed.

Ceramic Surface Conditioning and Cementation

Before cementation, all of the restorations were tried-in.

Initially, the inner surfaces of all restorations (N=40) were etched with 5% hydrofluoric acid (IPS Ceramic Etching, Ivoclar Vivadent, Lot: H22436) for 60 seconds, rinsed with water and dried. They were then divided into two groups (n=20) according to the acid neutralization process: 1–without neutralization; 2–precipitated hydrofluoric acid neutralization (IPS Ceramic Neutralizing powder, Ivoclar Vivadent, Lot: H35971).

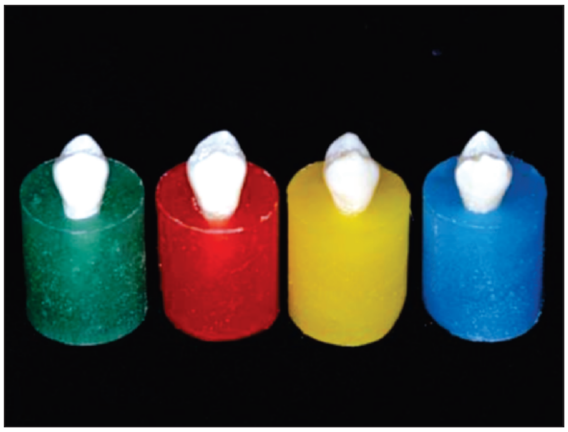

Figure 1: *Teeth embedded in acrylic resin (each color indicated a testing group).*

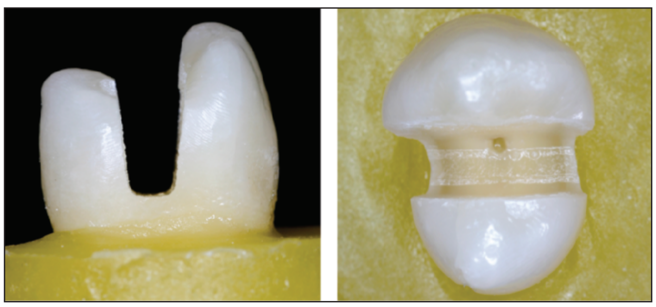

Figure 2: *Inlay preparations.*

Next, the treated surfaces were silanized (Monobond S, Ivoclar Vivadent, Lot: H22436). A self-curing and self-etching luting composite system (Multilink, Ivoclar Vivadent, Lot: H22436) was applied for cementation of the restorations, following the manufacturer's recommendations: 1) the dental substrate was treated with a self-etching adhesive system [mixing Multilink Primer A and Primer B, application of the mixed primers on the enamel and dentin]; 2) mixing the Multilink cement and application on the restoration; 3) placement of the restoration and removal of excessive cement.

A vertical load of 750g was applied on the restoration using an adapted surveyor.

Mechanical Cycling Testing

Ten specimens from each conditioning method were submitted to mechanical cycling, which was carried out in a machine designed and produced by Baldissara.[21] The specimens were placed in a metallic base at a 90° angle, so that a cylinder at the upper rod of the cycling machine could induce load pulses from 0 N to 50 N at a frequency of 8 Hz on the cusps only (the restoration was not loaded). During cycling, the specimens were irrigated with water at 37 ± 1°C, which was regulated by a thermostat.[12,15-16] The specimens were cycled 1,400,000 times, simulating approximately five years of clinic service.[12,22]

Based on the two factors in this study ("acid neutralization" in two levels and "mechanical cycling regimens" in two levels), four groups were examined (N=10) (Table 1).

Microtensile Bond Strength Test

The specimens were fixed with cyanoacrylate (Super-Bonder Gel, Loctite, São Paulo, Brazil) to a cylindrical metal base coupled to a cutting machine (Figure 3). The crown was sectioned in the x and y axes to produce bar specimens characterized with a non-trimmed interface and bar-specimens composed of vestibular dentin, with ceramic in the middle and lingual dentin, comprising a bonded area of 1 mm^2.

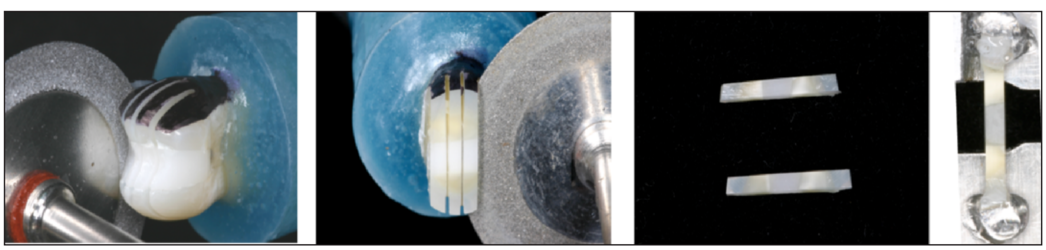

Figure 3: *Production of non-trimmed bar specimens.*

The bar-specimens were glued to the adapted device and submitted to the microtensile bond strength test (Emic DL-2000, Emic, São Jose dos Pinhais, Brazil) at a speed of 0.5 mm/minute until the sample fractured (Figure 3). The data were analyzed

Table 1: *Testing Groups*

	Acid Neutralization	Mechanical Cycling (n=10)
40 teeth	Without (N=20)	Without cycling (G-1) With cycling (G-3)
	With (N=20)	Without cycling (G-2) With cycling (G-4)

Table 2: *Two-way ANOVA of Bond Strength Data*

Source	*df*	SS	MS	F	*P*
Mechanical cycling	1	87.853	87.853	11.25	0.0019
Neutralization	1	49.863	49.8629	6.38	0.0160
Between	1	12.41	12.41	1.59	0.2156
Within	36	281.156	7.8099		
Total	39	431.281			

Table 3: *Means and Standard Deviations of the Bond Strength Data*

Mechanical Cycling	Neutralization		Total
	Without	With	
No cycling	G1: 10.2 ± 2.4[a]	G2: 6.8 ± 1.9[ab]	8.5 ± 2.7
Cycling	G3: 6.1 ± 3.1[b]	G4: 5 ± 3.5[b]	5.5 ± 3.2
Total	8.1 ± 3.4	5.9 ± 2.9	

The different letters mean statistical differences; equal letters indicate statistically similar data (Tukey's test, p<.05).

using two-way ANOVA and Tukey test (α=.05). For statistical analysis, the mean values of the tested bar-specimens were calculated from each tooth (n=10).

RESULTS

Bond strength was significantly affected by the neutralization procedure (p=0.0160) (no neutralization> neutralization) and mechanical cycling (p=0.0019) (no cycling > cycling). The interaction was not statistically significant (p=0.2156) (two-way ANOVA) (Table 2).

Aging significantly damaged the bond strength values of the non-neutralized group, while the group with neutralization was not damaged. After mechanical fatigue testing, the non-neutralization and neutralization groups had statistically similar bond strength values (Tukey test, Table 3).

DISCUSSION

The success and longevity of the restorations made with ceramic materials is intimately related to the surface treatment applied before cementation and at the time of cementation itself.[1-4]

IPS Empress is an acid-sensitive ceramic (silica-based) and presents surface degradation when exposed to some acids.[5-7] Hydrofluoric acid attacks the glassy phase of these ceramics (SiO_2), exposing silica oxides and yielding topographic changes for increased micromechanical retention and chemical bonding with the silane coupling agent. Previous studies have demonstrated that HF acid provides high bond strength to resin materials.[5-7] In addition to providing a favorable micromorphology for micromechanical retention, hydrofluoric acid significantly reduces the surface contact angle when compared with APF, thus increasing the surface-free energy and wettability of the cement.[23-24] Silanes have been indicated for bonding to glass-ceramic.[5-7,25-27] Silanes with a general chemical formula of X-(CH_2) Si-(OR) are able to provide chemical bonding with organic surfaces, such as resin materials and polymers, and inorganic surfaces, such as silica-based ceramics.[23] Silanes based on 3-methacryloxypropyltrimethoxysilane (MPS) present molecules that react with water, forming silanol groups (–Si–OH) from the methacryloxy groups (–Si–O–CH_3). Silanol groups react to form a siloxane network (–Si–O–Si–O–), with the silica oxide present in a material. Silica oxide is present in feldspathic ceramics or oxides artificially deposited on acid-resistant ceramics; they serve as a good basis for the silane coupling agents to react.[5,25-27] The monomeric ends of silane then react with the methacrylate groups of resin material. Thus, the bonding process between the ceramic surface and resin cement occurs through the chemical process between silica oxides and the silane bonding agent.

However, after acid conditioning and before silanization, the neutralization of hydrofluoric acid can be performed. After rinsing the acid and its neutralizing, precipitates are created on the ceramic surface, which can damage the resin bond strength.[8] Acid precipitation is created by the reaction between the hydrofluoric acid and a salt for neutralization, producing a sodium fluoride and unstable carbonic acid ($NaHCO_3$ + HF ⇔ NaF + <H2CO$_3$>). According to Canay and others,[11] the precipitates remain on the ceramic surface, preventing penetration of the resinous materials into ceramic to create micro-retention. The type of precipitate produced depends on the microstructure and composition of the glass-ceramic.[28] Findings from the current study corroborated these statements, since the group with neutralization had lower microtensile bond strength than the non-neutralization group. Saavedra[8] found results similar to the current findings.

Clinically, performing ultrasonic cleaning after neutralization, which may remove precipitates and positively contribute to resin bond, is recommended.[8]

However, the manufacturer of the hydrofluoric acid examined in the current study does not recommend ultrasonic cleaning after application of the neutralizer. In the current authors' opinion, the clinician should not neutralize the acid, but if clinicians choose to neutralize, it is mandatory that the restoration be cleaned using an ultrasonic device.

The aging of specimens using fatigue resistance testing is important if one is basing a clinical procedure for resin-bonded restorations on analysis of the performance of a bonded surface.[12-16] Abdalla and others[16] observed that fatigue loading resulted in a significant reduction in bond strength for Hybrid Bond (Sun Medical Inc, Tokyo, Japan) when compared with unloaded restorations. Toledano and others[15] also noted a reduction in the bond strength of an adhesive system to dentin after mechanical cycling. In the current study, the microtensile bond strength results were significantly affected by mechanical cycling. The application of load cycling on the cusps of premolars restored with inlay restorations allows for the bonded interfaces (ceramic—resin cement—dentin) to be stressed, simulating clinical loading.[29]

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. A process of hydrofluoric acid precipitate neutralization seems to cause reduced bond strength between dentin and glass ceramic.
2. Mechanical cycling reduced the bond strength of teeth restored with ceramic inlays when compared with non-cycled groups.

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