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Laboratory Research

Self-etching Primers vs Acid Conditioning: Impact on Bond Strength Between Ceramics and Resin Cement

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Clinical Relevance

A conditioning protocol of dental ceramics with a less hazardous material and a reduction of clinical steps, making the resin cement bonding procedure safer and easier, is an alternative to hydrofluoric acid conditioning.

SUMMARY

This study tested whether a self-etching surface agent and the conventional hydrofluoric acid (HF) would provide the same bonding capacity between resin cement and feldspathic (Fd) and lithium disilicate (Ld) ceramics. Ceramic blocks were cut with a low-speed diamond saw with water cooling (Isomet 1000, Buehler, Lake Bluff, IL, USA) into 20 blocks of 5 × 7 × 4 mm, which were ground flat in a polishing machine (EcoMet/AutoMet 250, Buehler) under water cooling. The blocks were randomly divided into eight groups (n=5), according to ceramic type (Ld or Fd), surface conditioning (HF + Monobond Plus or Etch and Prime), and aging by thermocycling (TC or absence-baseline). After 24 hours in 37°C distilled water, blocks were embedded into acrylic resin and 1-mm^2 cross-section beams composed of ceramic/cement/composite were obtained. The microtensile test was performed in a universal testing machine (DL-1000, EMIC, São José dos Campos, Brazil; 0.5 mm.min^{-1}, 50 kgf load cell). Bond strength (MPa) was calcu-

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DOI: 10.2341/16-348-L

lated by dividing the load at failure (in N) by the bonded area (mm²). The fractured specimens were examined under stereomicroscopy, and one representative sample of each group was randomly selected before the cementation and was further used for analysis using scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS). The self-etching agent showed the highest bond strength for Fd (24.66±4.5) and Ld (24.73±6.9) ceramics and a decrease in surface wettability. SEM and EDS showed the presence of similar components in the tested materials with different topographies for both. Therefore, the self-etching primer was able to deliver even higher bonding than HF+silane to a resin cement.

INTRODUCTION

The use of feldspathic (Fd) porcelain as an indirect restorative material is common in modern dentistry because of its esthetic and functional capacities.[1] Hydrofluoric acid (HF) etching is often used in preparing the material, but bonding in regions that require large masticatory effort can result in failures.[2] Lithium disilicate (Ld)–based ceramics represent attractive alternatives because of their esthetic and resistance properties; these materials are also amenable to HF conditioning. Thus, these ceramics are used in various indications, such as onlays, inlays, veneers, and even fixed partial dentures as far posterior as premolars.[2-4]

Working with acid-sensitive ceramics and their component materials requires a comprehensive understanding of their response to HF exposure, including concentration and time, to properly condition the material surface for adhesion to resin cement.[5-7]

Etching of ceramic with HF selectively dissolves the glassy phase and makes the surface porous to allow resin cement penetration.[2-5] This porous surface includes hydroxyl groups that have great interaction with silane-coupling agents.[8]

Self-etching materials, which could replace the current standard treatment of HF and silanization, would offer reduced occupational hazards (i.e., occupational and patient exposure to potential risks and biological damage from acid contact with living tissue),[9] as well as a decrease in clinical steps. Monobond Etch and Prime (Ivoclar Vivadent AG, Schaan, Liechtenstein), composed of ammonium polyfluoride, trimethoxypropyl methacrylate, alcohols, and water, is one such self-conditioning silane that, according to the manufacturer, replaces HF etching without compromising bond strength.

According to the manufacturer, this material allows for superficial etching of the ceramic restoration through ammonium polyfluoride and silanization with trimethoxypropyl methacrylate. Moreover, according to the manufacturer, the roughened surface is less pronounced than the surface formed after conditioning with HF but allows for adequate adhesion of the restoration.

Beyond the evaluation of the initial adhesion, the maintenance of the bond after continued use is paramount for significant adoption. When adhesive interfaces are submitted to aging, they tend to degrade and drastically compromise the bonding between resin cement and glass ceramic.[10]

In this study, the bond strength between Ld and Fd ceramics to a resin cement via HF etching followed by silanization or self-etching silanes was evaluated in the absence or presence of aging by thermocycling. The hypothesis was that the simplified self-etching agent would provide bond strengths as high and durable as HF for both ceramics.

METHODS AND MATERIALS

Specimen Preparation

The materials used in this study, as well as the respective manufacturers, compositions, and batch numbers, are described in Table 1.

Ld and Fd ceramic blocks were cut with a low-speed diamond saw with water cooling (Isomet 1000, Buehler, Lake Bluff, IL, USA) into 20 (5 × 7 × 4 mm) blocks of each ceramic type. The surfaces of the blocks were ground flat with decreasing granulation SIC paper (600, 800, and 1200 grit) using a polishing machine (EcoMet/AutoMet 250, Buehler) under water cooling.

Ld ceramic blocks were crystallized following the instructions of the manufacturers. The blocks were randomly divided into eight groups (n=5), according to ceramic type (Ld or Fd), silanization technique (Plus or EP), and the presence of aging by thermocycling (TC or absence/baseline).

Prior to the conventional silanization technique using Monobond Plus (Plus), "LdPlus" and "LdPlusTC" blocks were etched with 10% HF (Condacporcelana, FGM, Joinville, Brazil) for 20 seconds, rinsed with tap water for the same time, and dried with an oil-free air jet; "FdPlus" and "FdPlusTC" blocks were etched with 10% HF (Condacporcelana,

Table 1: *Materials, Trademarks, Manufacturers, Composition, and Batch Numbers of Materials Used in the Present Study*

Material	Trademark	Manufacturer	Composition
Lithium dissilicate ceramic (Ld)	Emax CAD	Ivoclar Vivadent, AG, Schaan, Liechtenstein	SiO, Li_2O, K_2O, MgO, Al_2O_3, P_2O_5
Feldsphatic ceramic (Fd)	VITA Mark II	Vitablocs Mark II, Vita Zahnfabrik, Bad Säckingen, Germany	Al_2O_3, Na_2O, K_2O, TiO_2, SiO_2, CaO
Silane (Plus)	Monobond Plus	Ivoclar Vivadent, AG, Schaan, Liechtenstein	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, sulphide methacrylate
Self-etching silane (EP)	Monobond Etch and Prime	Ivoclar Vivadent, AG, Schaan, Liechtenstein	Ammonium polyfluoride, trimethoxypropyl methacrylate, alcohols, water
Resin Cement	Variolink II	Ivoclar Vivadent, AG, Schaan, Liechtenstein	BisGMA, UDMA, TEGDMA, DMA, bariumsulfate, Ba-Al-F-glass, silica benzoperoxy glycerol.
Composite	IPS Empress Direct	Ivoclar Vivadent, AG, Schaan, Liechtenstein	Dimethacrylates, barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide copolymer

FGM) for 60 seconds, rinsed, and dried. Monobond Plus was then applied on the surface, and the time for volatilization of the solvent was observed before cementing.

Ceramic blocks in the Ld subgroup were not acid etched and received a single-step surface treatment using Monobond Etch and Prime (EP). Blocks from LdEP, LdEPTC, FdEP, and FdEPTC groups received an active application of EP for 20 seconds, followed by 30 seconds of setting. The samples were then washed with running tap water and dried with an oil-free air jet. The protocols used herein are those indicated by the manufacturers. The materials have different microstructures requiring different etching times. However, the groups treated with EP have the same time because this material is indicated to be used with this same protocol no matter the ceramic.

A silicone matrix with a $5 \times 7 \times 4$ mm cavity was prepared. With it, composite blocks (IPS Empress) were prepared using the layering technique (maximum 1-mm-thick layers). The freshly polymerized composite blocks were cemented onto ceramic blocks using a resin cement (Variolink II). A 750-g weight was used to keep blocks in position during the removal of excess cement, using a microbrush and photoactivation for 40 seconds on each surface.

Microtensile Bond Strength Test

After 24 hours of storage in 37°C distilled water, the blocks were immersed into acrylic resin, and 1-mm^2 cross-section beams composed of ceramic/cement/composite were obtained by means of a cutting machine (Isomet 1000, Buehler) under constant cooling. The external beams of each block were demarcated and excluded.

Baseline beams were immediately tested, while beams from the TC subgroups were used to analyze the effect of aging on the bond strength resulting from each silanization technique on different ceramic materials.[11] TC samples were subjected to 5000 thermal cycles in water at temperatures of 5 and 55°C, with 30 seconds of immersion and 5 seconds of transition (521-6D, Ethik Technology, Vargem Grande, São Paulo, Brazil).

The dimensions of the specimens were measured with a digital caliper, and the specimens were glued to the testing device (OG01, Odeme, Lucerne, Brazil) with cyanoacrylate (Superbonder, Loctite, Lucerne, SC, Brazil). Microtensile bond strength (μTBS) testing was performed on at least 15 beams from each block in a universal testing machine (DL-1000, EMIC, São José dos Campos, Brazil; 0.5 $mm.min^{-1}$, 50 kgf load cell), and the bond strength (MPa) was calculated by dividing the load at failure (N) by the adhesive area (mm^2). For the pretest failures, the lowest load at failure (N) for the group was assigned.

Failure Analysis

The fractured specimens were examined by stereomicroscopy (Stereo Discovery V20, Zeiss, Göttingen, Germany), and the failure modes were classified as cohesive failure of composite resin (COHES res), adhesive failure between composite resin/resin cement (ADHES cim/res), a mixed adhesive/cohesive failure (MIXED), adhesive failure between ceramic/

resin cement (ADHES cer/cim), or cohesive failure of the ceramic (COHES cer).

Goniometry

Ld and Fd blocks of 3 × 3 × 2 mm were used for the wettability evaluation (n=3). The samples were prepared by Plus or EP treatment as described above. One additional sample of each ceramic was only polished and used as a control surface.

The wettability was analyzed by the sessile drop technique using an optical tensiometer (TL 1000, Theta Lite Attention, Lichfield, Staffordshire, UK). First, a syringe (No. 1001 Gastight Syringes, 1 mL, Hamilton, Reno, NV, USA) deposited a drop of distilled water on the sample surface with a mean volume of 8.3 μL. After 20 seconds of drop settling,[12] a series of 60 images per second was recorded by the equipment for 20 seconds. OneAttension (Biolin Scientific, Lichfield, Staffordshire, UK) software was used for calculation of the contact angle mean value from the images obtained in 10 different areas for each sample.

Scanning Electron Microscopy

One representative sample from each group was randomly selected before the cementation and was further used for analysis using scanning electron microscopy (SEM; Inspect S50, FEI, Czech Republic). The specimens were sputter coated with gold for 180 seconds at 40 mA, creating a 30-nm-thick layer and examined under different standard SEM magnifications operated at 20 KV using secondary electron detection by a single operator. One sample from each group was irradiated using x-ray fluorescence (EDX) spectroscopy.

Data Analysis

Data were tabulated and the assumptions of normality were confirmed. Blocks were the experimental units for statistical analysis of the bond strength results, and cohesive failures were removed from statistical analysis. μTBS results were statistically analyzed by three-way analysis of variance (ANOVA; ceramic×silanization technique×aging), and differences were detected by Tukey test (both α=0.05). Contact angle values were analyzed by two-way ANOVA (ceramic×silanization technique) and Tukey test (both α=0.05). EDX and SEM data were qualitatively analyzed.

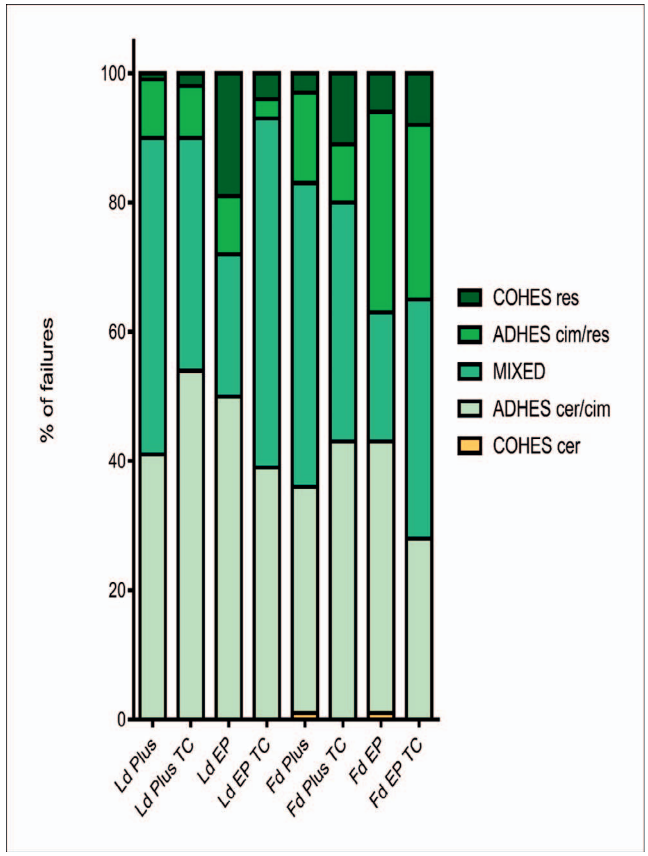

Figure 1. *Column graph of the analysis of different failure profiles between the groups.*

RESULTS

The mean values of μTBS ranged between 16.05 and 24.73 MPa (Table 2). ANOVA showed that the "aging" factor significantly influenced the bond strength results (p<0.0001); thermocycling signifi-

Table 2: *Means (in MPa) and SD of the μTBS Test*[a]

	Lithium Disilicate		Feldspathic	
	Plus	EP	Plus	EP
Baseline	22.60 ± 3.0^{A}	24.73 ± 6.9^{A}	21.35 ± 2.5^{A}	24.66 ± 4.5^{A}
TC	16.05 ± 4.0^{B}	16.08 ± 5.4^{B}	18.26 ± 3.3^{B}	19.53 ± 2.3^{B}

[a] *Same capital letters indicate absence of significant differences.*

Table 3: *Mean (in °) and SD of the Goniometry Test*

	Ceramic	
	Lithium Disilicate	Feldspathic
Polished samples	27.28 ± 6.17^A	31.56 ± 4.51^A
Plus	19.23 ± 4.13^B	12.47 ± 3.41^B
EP	89.31 ± 9.30^C	88.19 ± 4.07^C

[a] *Same capital letters indicate absence of significant differences, α=5%.*

cantly reduced the bonding between ceramics and resin cement.

Mixed failures (association of adhesive and cohesive failures) were present in all groups, being predominant only for LdEPTC. For the other groups, adhesive failures were mostly seen, with more failures in the interface between ceramic and resin cement than composite resin and resin cement. Cohesive failures of composite resin were present in all groups, and cohesive failures of ceramic were present only for Fd ceramic without thermocycling regardless of surface treatment (Fd Plus and Fd EP), presenting the lowest frequency of occurrence in these groups compared with the other types of failures. All failures are summarized in Figure 1.

The wettability results showed a statistically significant difference between groups, with a decrease in the surface wettability caused by the EP surface agent (Table 3).

Figure 2 presents the representative micrographs of tested samples. Polished samples of Ld and Fd showed smooth and homogeneous surfaces without any porosity as a result of polishing. The etched surfaces showed changes in the surface microstructure with the presence of irregularities including numerous microporosities, grooves, and striations as a result of the dissolution of the glassy phase. The Ld etched for 20 seconds predominantly showed a glassy phase and small, isolated pores, while the Fd specimens showed large cavitations. EDX spectroscopy showed the presence of similar components in the tested materials. Ld-based ceramic showed peaks attributed to aluminum, silicon, phosphorus, potassium, and lightweight elements that could not be resolved in the analysis. The Fd samples were composed of sodium, aluminum, silicon, and potassium. The samples treated with Plus also presented sulfur.

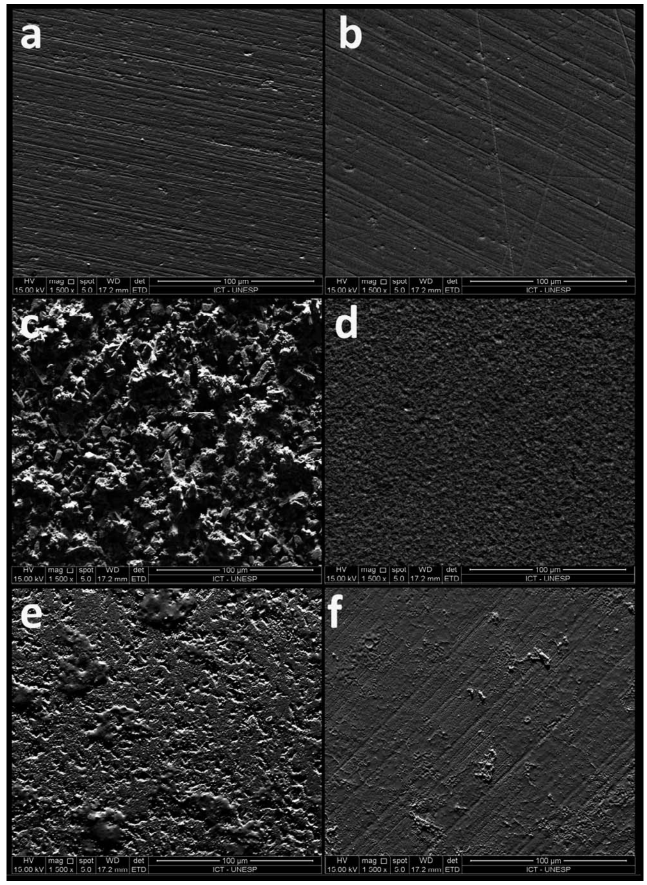

Figure 2. *Representative micrographs. 1500×. Polished samples of (a) lithium disilicate and (b) feldspathic ceramics. (c) LdPlus. (d) FdPlus. (e) LdEP. (f) FdEP.*

DISCUSSION

The hypothesis that the simplified self-etching agent would provide bond strengths as high and durable as HF for both ceramics was accepted. The use of a chemical agent capable of performing as both surface etchant and adhesive between the inorganic matrix of the ceramic and the organic matrix of the resin cement is attractive to dentists because of the streamlined bonding process. This simplification of clinical steps during the surface preparation of both the tooth and substrate was obtained with self-etching dental adhesives,[13] self-adhesive resin cements,[14] and, more recently, with self-etching primers for glass ceramics.

The use of self-conditioning primers in this study resulted in similar bonding strengths as those of the conventional acid-etched group. Because glass ceramics are acid sensitive, there was no need to significantly change the surface treatment protocol.[3,14,15] The results revealed that self-conditioning primers do not present disadvantages related to bond integrity while reducing clinical steps and minimizing risks related to HF etching.[9] The protocol of self-etching primers also appears to be more predictable than others tested in the literature, such as mechanical conditioning, which damages the material surface with and without silica coating,[15,16] and silane heating before its use.[17]

Kimmich and Stappert[18] mentioned some approaches for repair of fractured ceramic restorations in the oral environment. Among the options were repair with resin or with the ceramic fragment. To do that, the restoration must be properly prepared in the intraoral environment, which includes surface conditioning with HF to achieve adequate adhesive strength. However, the authors emphasized the need for total control of the technique because of the toxicity of HF. Intraoral conditioning with EP has not yet been reported in the literature, and its effects on tissues have yet to be studied, but this technique requires fewer clinical steps and is possibly not as harmful as HF.

In this study, both ceramics presented similar contact angles and wettability for HF-treated surfaces. This indicates that the removal of the glass matrix and exposure of chemical ligands was similar in both tested ceramics; any overtreatment would increase the number of pores and consequently influence the surface wettability.[6,19] These modifications can be observed in SEM micrographs (Figure 2c, d). Fd and Ld displayed decreased wettability after surface treatment with the self-etching silane, suggesting that the resulting microstructural changes (Figure 2e, f) possibly led to the formation of a layer containing debris, making the surface less susceptible to wetting.[6,20]

Topographic change is essential to the maintenance of adhesive strength, as an increased number of pores of various sizes and widths allows for better micromechanical bonding area.[19] This increase in surface roughness also affects the wettability of the ceramic surface for the application of silane-coupling agents and resin composites.[6,21] An adequately porous surface is vital for the durable cementation of both Ld[6] and Fd ceramic[4,7] indirect restorations. In addition to roughness, other factors that contribute to the effectiveness of bonding between the materials are clean surfaces, suitable wetting, and the use of low-viscosity adhesives and cements.[20] Unfortunately, the presence of fluoride in EP materials decreases the wettability of the substrate,[20] but this does not appear to have affected the ceramics in this study.

Ramakrishnaiah and others[6] demonstrated that wettability is directly proportional to surface irregularities, so that increasing the HF action time on a ceramic surface causes increased wettability of the ceramic. Despite that, the authors counterindicated a prolonged conditioning of the ceramic surface, because although wettability increases, it does not lead to increased bond strength.[22] On the other hand, self-etching silane cannot dissolve the glassy phase as profoundly as HF (Figure 2), but silane will spread more because of active application. This justifies a higher contact angle in the EP groups, although bonding was efficiently attained after the silane was actively applied on the ceramic surface. Moreover, fewer surface defects that could weaken the ceramic as a result of overetching were created when using a self-etching silane.

Aging by thermocycling had a negative effect on bond strength in this study. Samples in groups without cycling had an average bond strength of 25.2 MPa, while the aged groups showed lower mean values (18 MPa). Most samples withstood the thermal cycles, and the low percentage of predominantly adhesive failure between the resin cement and ceramics showed that surface treatment in a single step ensures a stable bond, similar to the conventional treatment of ceramic and cement for both glass ceramics used.[23] Moreover, the EDX analysis did not show the presence of fluoride, likely because of the small concentration and the low atomic weight of that element. Thermocycling instead of long-term water storage was chosen because both protocols show similar behaviors with regard to bond degradation.[24]

Microtensile testing of composite-ceramic bonding to evaluate ceramic processing minimizes the effects of dental substrate variation originating from different diameters of exposed dentinal tubules.[6,18,25] Most beams showed adhesive failure between resin cement and ceramics, which is corroborated with studies that analyzed conventional silanization.[6,25] Regardless of the material present at the adhesive interface, all specimens were vulnerable to temperature changes in the water, resulting in a significant decrease in bond strength.

Through SEM, it was observed that the Monobond EP caused morphological changes compared with the control groups for both materials, producing an increase in surface roughness as well as the formation of a discrete residual layer.

For the Monobond Plus group, there was an even more remarkable morphological change: the use of HF resulted in a significantly rougher surface, as previously described in the literature.[6,18,25] However, this morphological difference changed only the wettability and did not affect the bond strength.

CONCLUSIONS

Considering the positive results obtained for the surface treatments and the potential risks associated with HF procedures, it is suggested that the self-etching system is ideal for maintaining adhesive bonds to glass ceramics. Further studies are needed to explore the time required for conditioning of ceramics due to the observed structural changes caused by the self-etching primer.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 30 October 2017)

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