

# Effect of Surface Pretreatments on the Microtensile Bond Strength of Lithium-Disilicate Ceramic Repaired with Composite Resin

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The aim of this study was to evaluate the influence of ceramic surface treatments and silane drying temperature on the microtensile bond strength ( $\mu$ TBS) of a resin composite to a lithium disilicate ceramic. Twenty blocks (7x7x5 mm) of lithium disilicate-based hot-pressed ceramic were fabricated and randomly divided into 4 groups: G1: acid etching with 9.5% hydrofluoric acid for 20 s and drying silane with room-temperature air; G2: acid etching with 9.5% hydrofluoric acid for 20 s and drying silane with  $45 \pm 5$  °C warm air; G3: airborne-particle abrasion with 50  $\mu$ m aluminum oxide particles and drying silane with  $45 \pm 5$  °C warm air; G4: airborne-particle abrasion with 50  $\mu$ m aluminum oxide particles and drying silane with air at room-temperature. After treatments, an adhesive system (Single Bond 2) was applied, light-cured and direct restorations were built up with a resin composite (Filtek Z250). Each specimen was stored in distilled water at 37 °C for 24 h and cut into ceramic-composite beams with 1 mm<sup>2</sup> of cross-sectional area for  $\mu$ TBS testing. Statistical analysis was performed with one-way ANOVA and Student-Newman-Keuls test ( $\alpha=0.05$ ).  $\mu$ TBS means (S.D.) in MPa were: G1: 32.14 (7.98), G2: 35.00 (7.77) and G3: 18.36 (6.17). All specimens of G4 failed during the cutting. G1 and G2 presented significantly higher  $\mu$ TBS than G3 ( $p<0.05$ ). There was no statistically significant difference between G1 and G2 ( $p>0.05$ ). As far as the bond strength is concerned, surface pretreatment of lithium-disilicate ceramic with hydrofluoric acid and silane application can be used as an alternative to repair ceramic restorations with composite resin, while surface pretreatment with sandblasting should be avoided.

Key Words: surface treatment, lithium disilicate ceramic, bond strength.

## Introduction

The increasing demand for esthetics in restorative dentistry has led to the development of all-ceramic restorations using different resin-based adhesive systems, since they provide excellent esthetic metal-free characteristics (1,2). However, the physical and bonding properties of ceramics are still problematic (2).

The establishment of a durable and reliable bond between dental ceramic and resin composite is important in dental practice because of its extensive use. This bond is usually created via two mechanisms: micro-mechanical attachment by hydrofluoric acid (HF) etching and/or sandblasting, and chemical bonding by a silane coupling agent (3). Effective etching of the ceramic surface is considered an essential step for the clinical success of indirect ceramic-bonded restorations and direct ceramic repair procedures (4). Acid etching increases the surface area and the wettability of ceramics, changing their surface energy and the bonding potential of ceramic to resin (5,6).

The bonding properties between resin-based adhesives and ceramics are mainly associated with the presence of

silica on the surface that provides chemical compatibility mediated by silane-coupling agents (7). The incorporated silica of acid-sensitive ceramics such as lithium disilicate ceramics is well-exposed by HF acid gel to give increased surface roughness, which is available for mechanical interlocking (4,6,7).

The use of silane is recommended for glasses and porcelains in order to form a siloxane network with the silica in the ceramic surface, to improve the bond strength between the ceramic and luting material (8,9). Silane-coupling agents, because of their chemical compatibility and the micro-roughness on the acid-etched surface, allow for sufficient bonding properties by mediating between the ceramics and resin-based adhesives (10).

To accelerate the mechanism of chemical interaction of silane, the reaction is usually catalyzed by acid or heat (11). The heat treatment can also evaporate solvent and volatile reaction products are formed during condensation of silanol groups (12). It is also possible that heating of silane-coated ceramic

might facilitate vaporization and elimination of excess silane from the surface (5).

The lithium disilicate based E-max (IPSe.max CAD; Ivoclar Vivadent, Schaan, Liechtenstein) is a new ceramic material and there is not enough information about the effects of pretreatment methods on the bond strength of ceramic repairs with composite. The purpose of this *in vitro* study was to evaluate the influence of ceramic surface treatments and the silane drying temperature on the microtensile bond strength ( $\mu$ TBS) resin composite to a lithium disilicate ceramic. The hypothesis tested was that the use of warm-air after silane application could increase the tensile bond strength of composite to disilicate based ceramic.

## Material and Methods

Twenty hot-pressed ceramic blocks (7x7x5 mm) were fabricated from partially crystallized lithium disilicate-based glass blocks (IPSe.max CAD; Ivoclar Vivadent). The ceramic blocks were mechanically polished using silicone rubbers impregnated with diamond (Exa-Cerapol; Edenta AG, AU/SG, Switzerland) at low speed. After polishing, all ceramic blocks were ultrasonically cleaned in distilled water for 10 min and dried with oil-/water-free air for 60 s.

The specimens were randomly allocated in 4 groups (n=5) by the Excel software (Excel 2003; Microsoft Corporation, One Microsoft Way, Redmond, WA, USA), according to the following surface treatments and silanization protocols: G1: 9.5% hydrofluoric acid was applied for 20 s and silanated surface was dried at room temperature (23 °C); G2: 9.5% hydrofluoric acid was applied for 20 s and silanated surface was dried with warm air at 45 ± 5 °C; G3: sandblasting with 50  $\mu$ m aluminum oxide particles (Bio Art, São Carlos, SP, Brazil) for 5 s, applied perpendicularly to the surface at a pressure of 30 psi from a distance of approximately 10 mm and silanated surface was dried with warm air at 45 ± 5 °C; G4: sandblasting with 50  $\mu$ m aluminum oxide particles (Model Standard; Bio Art) for 5 s, applied perpendicularly to the surface at a pressure of 30 psi and from a distance of approximately 10 mm and silanated surface were dried with room-temperature air.

After the surface treatments, the blocks were rinsed with air-water spray for 30 s, dried with oil-water-free air blast for 30 s, and silane (Rely-X Ceramic Primer; 3M-ESPE Dental Products, St. Paul, MN, USA) was applied with a brush, left undisturbed for 1 min, and the surface was subjected to one of the following drying procedures: G1 and G4 ceramic blocks were dried at room-temperature air (23 °C), allowing the silane to penetrate into the surface irregularities for 1 min, then gently blowing off the excess with an air blast at approximately 20 cm from the surface. G2 and G3 ceramic

blocks were dried with a stream of warm air (45 ± 5 °C) generated from a blow dryer (model RV456ABG; Revlon, El Paso, TX, USA) for 15 s to approximately 15 cm from the surface. The air temperature was measured by digital portable thermometer (model MT 600; Minipa, São Paulo, SP, Brazil) with a resolution of 0.1 °C, a basic precision of 0.1 ± 0.7%, and with a temperature range from 0.1 to 200 °C. An etch-and-rinse adhesive system (Adper Single Bond 2; 3M ESPE) was applied according to manufacturer's instructions: apply two coats of adhesive, air-drying for 10 s at 20 cm and light-curing for 10 s. After light curing of the adhesive system, 5 increments of 1 mm thickness of composite resin (Filtek Z250; 3M ESPE) were build-up. Each increment was light-cured with a halogen light-curing unit (VIP Junior; Bisco Inc., Schaumburg, IL, USA) for 40 s, with power density of 500 mW/cm<sup>2</sup>. The bonded ceramic blocks were stored in distilled water at 37 °C for 24 h.

The ceramic-resin blocks were longitudinally sectioned in both the "x" and "y" directions across the bonded interface using a diamond saw in a Labcut 1010 machine (Extac Corp., Enfield, CT, USA) under water cooling to obtain bonded sticks with a cross-sectional area approximately 1.0 mm<sup>2</sup>. The cross-sectional area of each stick was measured with the digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) to the nearest 0.01 mm and recorded for subsequent calculation of the  $\mu$ TBS. In all blocks, the first slice was discarded because results could be influenced by excess or lack of adhesive at the interface.

For  $\mu$ TBS testing, each specimen was attached with cyanoacrylate glue (Zapit; Dental Venture of America Inc., Corona, CA, USA) to a modified Bencor Mult-T testing apparatus (Danville Engineering Co., Danville, CA, USA) and subjected to a tensile force at 1.0 mm/min crosshead speed in a universal testing machine (Instron 4484; Instron Inc., Canton, MA, USA). The load at fracture was used to calculate bond strength and expressed in MPa.

After the  $\mu$ TBS testing, the fractured surfaces of both ceramic side and resin side were investigated using a stereoscope (StereoZoom® Leica S8 APO; Leica Microsystems, Wetzlar, Germany) at 80x magnification. The failure modes were classified as adhesive failure (1) when occurred at the ceramic/resin interface; cohesive failure (1) when fractured exclusively within ceramic or resin composite; or mixed failure (3) when two modes of failure happened simultaneously.

Bond strength values were analyzed statistically with one-way ANOVA and Student-Newman-Keuls test at 5% significance level. Statistical procedures were performed using the STATA SE 9.1 for Windows statistical program software (STATA; College Station, TX, USA). Stick was used as statistical unit and the number of prematurely debonded specimens was recorded but was not included in the analysis.

## Results

The results were influenced by surface treatment among the groups. G1 and G2 showed no significant differences between them ( $p>0.05$ ) and values of bond strength were significantly higher than those recorded in G3 ( $p<0.01$ ) (Table 1). All G4 specimens failed during the procedure of cutting and were eliminated.

The prevalent fracture pattern in all groups was the mixed failure (Table 2).

## Discussion

The clinical success of either a repaired ceramic restoration or a resin cemented ceramic restoration depends on the quality and durability of the bond between the ceramic and the resin. To obtain reliable bond strength between resin composite and ceramic materials, mechanical retention performed with surface roughening and chemical retention with silane agent is essential (13).

The application of hydrofluoric acid in ceramic surface promotes the reaction with the glass matrix that contains silica and forms hexafluorosilicates. This glass matrix is selectively removed and the crystalline structure is exposed. As a result, the surface of the ceramic becomes rough, which is expected for micromechanical retentions (6). This roughly etched surface also helps to provide more surface energy prior to combining with the silane solution (14). Some studies have demonstrated that roughening the ceramic surface by etching has been considered the most effective procedure of retaining the bond integrity (12,14) and for the clinical success of indirect ceramic bonded restorations and direct-repaired ceramic prostheses (15-21). This data corroborate the results of the present study (Table 1).

Heating of silane on the surface of ceramics can promote better adhesion between this material and resin composite (3,5,11,22). The heating of silane on the surface of ceramics can eliminate water, alcohol and byproducts of the reaction and help to complete the condensation reaction of silane-silica promoting the formation of siloxane (22). Evaporation of alcohol or acetic acid can increase the density of local connections available for the solution of silane react with the ceramic (22). However, in the present study no statistical differences were found between samples treated with

hydrofluoric acid and single-bottle silane dried with air at room temperature or heated at  $45 \pm 5$  °C, rejecting the hypothesis. Barghi et al. (23) demonstrated that different kinds of silanes yield different bond strength values. These authors also found differences in bond strengths following thermal treatment of silanated porcelain using two-bottle silanes, whereas prehydrolyzed single-bottle silanes were not affected by this procedure. Different silanes may present in their composition different solvents which will influence their reactivity and stability in various ways (20).

On the other hand, sandblasting as a surface pretreatment reduced the bond strength (Table 1). According to Chen et al. (20), sandblasting should be avoided due to the possibility of volume loss and changes in the morphology of ceramics. However, in the present study, the specimens could be tested when heated silane was used, unlike in G4. Probably, in this case, the heating promoted the condensation reaction within silane that would reduce the thickness of the silane and prevent it from incorporation with the adhesive resin applied later (24). Without the heat treatment, the silane on the ceramic surface could blend with the adhesive resin, forming a continuous layer of adhesive between the ceramic and the composite. A study has shown that the high concentration of silane in the solution (greater than 5%) has a negative effect on the bond strength values (2).

The prevalent fracture pattern of G1, G2 and G3 was the mixed failure (Table 2). These findings suggest that the bond between single-bottle silane and the porcelain are a proportional mixing of chemical and mechanical bonds. These data were in accordance with those of other studies (12,23,25).

The present study was limited to the investigation of the effect of post silanization with warm-air drying to seek an alternative treatment modality of clinically applicable heat treatment of silane. Further laboratory tests need to be conducted, using a similar methodology but with thermocycling and another silane composition, to investigate how this could influence the bond strength of lithium-disilicate ceramics to resin composite.

In conclusion, the findings of this study showed that the surface pretreatment of lithium-disilicate ceramic

Table 1. Microtensile bond strength according to the surface treatments

Group	Microtensile bond strength
G1	32.14 $\pm$ 7.98 <sup>A</sup> (n=33)
G2	35.00 $\pm$ 7.77 <sup>A</sup> (n=32)
G3	18.36 $\pm$ 6.17 <sup>B</sup> (n=20)

Same letters indicate no statistically significant difference ( $p>0.05$ ). Results are given in MPa  $\pm$  SD (n).

Table 2. Distribution of fracture pattern according to groups. Relative percentage is in parentheses

Group	Premature failure	Adhesive failure	Cohesive failure	Mixed failure
G1	0 (0%)	2 (6.5%)	1 (3.2%)	30 (90.3%)
G2	0 (0%)	1 (3.2%)	0 (0)	31 (96.8%)
G3	0 (0%)	0 (0%)	0 (0%)	20 (100%)

with hydrofluoric acid and the application of silane can be used as an alternative to repair ceramic restorations with composite resin. On the other hand, surface pretreatment with sandblasting should be avoided.

## Resumo

O objetivo deste estudo foi avaliar a influência dos tratamentos de superfícies e a temperatura de secagem do silano na resistência de união de resina composta à cerâmica de disilicato de lítio. Vinte blocos (7x7x5 mm) de cerâmica à base de disilicato de lítio foram fabricados e aleatoriamente divididos em quatro grupos: G1: condicionamento com ácido fluorídrico a 9,5% por 20 s e secagem do silano com ar à temperatura ambiente; G2: condicionamento com ácido fluorídrico a 9,5% por 20 s e secagem do silano com ar aquecido a  $45 \pm 5$  °C; G3: jateamento com partículas de óxido de alumínio de 50 µm e secagem do silano com ar aquecido a  $45 \pm 5$  °C; G4: jateamento com partículas de óxido de alumínio de 50 µm e secagem do silano com ar à temperatura ambiente. Após os tratamentos, o adesivo Adper Single Bond 2 foi aplicado, fotoativado e foi construído um platô de resina composta (Filtek Z250). Cada espécime foi armazenado em água destilada a 37 °C por 24 h e cortados em forma de palito de área adesiva de 1 mm<sup>2</sup> para a realização do teste de microtração. Os dados foram submetidos à ANOVA e ao teste de Student-Newman-Keuls ( $\alpha=0,05$ ). As médias (D.P.) dos valores de resistência de união em MPa foram: G1: 32,14 (7,98), G2: 35,00 (7,77) e G3: 18,36 (6,17). G1 e G2 apresentaram maiores valores de resistência de união que G3 ( $p<0,05$ ). Não houve diferença estatística entre as médias dos valores dos grupos G1 e G2 ( $p>0,05$ ). Levando-se em consideração a resistência de união, o pré-tratamento de cerâmica de disilicato de lítio com ácido fluorídrico e aplicação de silano pode ser uma alternativa para o reparo de restaurações de cerâmicas com resinas compostas, enquanto que o pré-tratamento de superfície com o jateamento deve ser evitado.

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