Effect of Water Storage and Surface Treatments on the Tensile Bond Strength of IPS Empress 2 Ceramic

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<u>Purpose</u>: The aim of this study was to evaluate the effect of water storage (24 hours and 1 year) on the tensile bond strength between the IPS Empress 2 ceramic and Variolink II resin cement under different superficial treatments.

<u>Materials and Methods</u>: One hundred and eighty disks with diameters of 5.3 mm at the top and 7.0 mm at the bottom, and a thickness of 2.5 mm were made, embedded in resin, and randomly divided into six groups: Groups 1 and 4 = 10% hydrofluoric acid for 20 seconds; Groups 2 and 5 = sandblasting for 5 seconds with 50 μ m aluminum oxide; and Groups 3 and 6 = sandblasting for 5 seconds with 100 μ m aluminum oxide. Silane was applied on the treated ceramic surfaces, and the disks were bonded into pairs with adhesive resin cement. The samples of Groups 1 to 3 were stored in distilled water at 37° C for 24 hours, and Groups 4 to 6 were stored for 1 year. The samples were subjected to a tensile strength test in an Instron universal testing machine at a crosshead speed of 1.0 mm/min, until failure. The data were submitted to analysis of variance and Tukey's test (5%).

<u>Results</u>: The means of the tensile bond strength of Groups 1, 2, and 3 (15.54 ± 4.53, 10.60 ± 3.32, and 7.87 ± 2.26 MPa) for 24-hour storage time were significantly higher than those observed for the 1-year storage (Groups 4, 5, and 6: 10.10 ± 3.17, 6.34 ± 1.06, and 2.60 ± 0.41 MPa). The surface treatments with 10% hydrofluoric acid (15.54 ± 4.53 and 10.10 ± 3.17 MPa) showed statistically higher tensile bond strengths compared with sandblasting with 50 μ m (10.60 ± 3.32 and 6.34 ± 1.06 MPa) and 100 μ m (7.87 ± 2.26 and 2.60 ± 0.41 MPa) aluminum oxide for the storage time 24 hours and 1 year.

<u>Conclusions</u>: Storage time significantly decreased the tensile bond strength for both ceramic surface treatments. The application of 10% hydrofluoric acid resulted in stronger tensile bond strength values than those achieved with aluminum oxide.

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INDEX WORDS: ceramic, surface treatments, water storage

C ERAMICS ARE ROUTINELY USED for dental restorations. The ceramic acid etch technique allows adhesive ceramic restora-

tions to be made on anterior teeth, and the use of this restorative material has increased substantially since the 1980s.¹ The use of ceramic

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Accepted January 5, 2006.

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This work was presented at the International Association for Dental Research meeting, Honolulu, HI, March 11, 2004. Copyright © 2007 by The American College of Prosthodontists 1059-941X/07

doi: 10.1111/j.1532-849X.2006.00171.x

as a restorative material substitution for metal– ceramic has increased substantially, because the translucent properties of ceramics can be affected by the metal core. The clinical success of the ceramic restoration depends on a number of factors, including the cementation procedure.

One of these new ceramic systems is IPS Empress 2. This material is a multiphase glass ceramic with a high degree of crystallinity, which improves mechanical properties and allows the fabrication of crowns and three-unit fixed partial dentures.²⁻⁴ The framework IPS Empress 2 may be conventionally or adhesively cemented.⁵ An adhesive luting agent is desirable when the retentive area preparation is small and retention may be inadequate.⁶

Techniques for bonding to ceramic take advantage of the formation of chemical bonds and micromechanical interlocking at the resin–ceramic surface. Etching procedures with hydrofluoric acid are used to create a rough surface in the ceramic bonding area to enhance the bonding between ceramic and resin. Surface roughness promotes mechanical retention of the composite luting agent, which penetrates into these irregularities maximizing the bond strength between etched ceramic and resin cements. The etched ceramic surface must be coated with suitable silane.^{5,7} The applications of silane help maintain the bond strength between the ceramic surface and resin luting cements.

Variolink II is a dual-curing luting composite resin with the presence of BisGMA, UDMA, TGDMA, and fillers with 73.4 wt% for the base paste and 71.2 wt% for the catalyst paste, pigments, stabilizers, and catalysts.8 Fonseca et al9 evaluated the influence of chemical activation compared with dual-curing activation on cement hardness of four dual-curing resin cements. Variolink II showed that to the time of 24 hours, chemical activation alone was unable to promote hardness similar to dual-curing chemical and light activation; however, the durability of the bonding strength between resin cement and the ceramic surface may be influenced by time and storage conditions in the oral environment.¹⁰⁻¹² According to Appeldoorn et al¹³ and Örtengren et al,¹² there is a decrease in the bond strength of resin cements when stored in water. Kern and Thompson¹⁴ showed, after 150 days of storage, a significant decrease in bond strength between resin cement and ceramic surface. Roulet at al⁷ reported that

samples sandblasted with 50 μ m Al₂O₃ demonstrated a 50% decrease in bond strength after 1 year in water storage. With respect to IPS Empress 2, knowledge of whether the durability of the ceramic–resin cement bond strength may decrease during storage in water is important.

The purpose of this study was to evaluate the effect of water storage conditions (24 hours and 1 year) on the tensile bond strength between the IPS Empress 2 ceramic framework and Variolink II resin cement under different ceramic surface treatments.

Materials and Methods

Wax (Renfert, Hilzingen, Germany) patterns were fabricated using a stainless steel mold, 5.3 mm in diameter at the top and 7.0 mm at the bottom. The height of pattern was 2.5 mm. The larger diameter was used for attaching the specimens for tensile test. The wax patterns were sprued and attached to a muffle base with a surrounding paper cylinder (Ivoclar Vivadent, Shaan, Liechtenstein), invested with IPS Empress 2 speed investment (Ivoclar Vivadent), and the wax eliminated in a Vulcan A-550 burnout furnace (Degussa-Ney, Yucaipa, CA). One hundred and eighty samples were pressed using IPS Empress 2 ingots (Ivoclar Vivadent, shade A2) in an automatic press furnace (EP 600, Ivoclar Vivadent) at 5-bar pressure, using the manufacturer's instructions. After cooling, the samples were divested using glass beads (100 μ m) at 1-bar pressure. Sprues were removed using a diamond disk (KG Sorensen, Barueri, SP, Brazil) and ground, and samples were cleaned in Invex Liquid (Ivoclar Vivadent) for 10 minutes in an ultrasonic unit (Odontobras, Piracicaba, SP, Brazil). All steps in the fabrication and divesting of IPS Empress 2 specimens were according to manufacturer's instructions.

The alignment of the ceramic disk within the PVC tube (Tigre, São Paulo, SP, Brazil) was achieved using a metallic adaptor. The ceramic disk was attached to the tube with acrylic resin (Clássico, Artigos Odontológicos, São Paulo, SP, Brazil), leaving 1 mm of the ceramic surface exposed (Fig 1), and positioned in a stainless steel holder and wet ground with 400- and 600-grit SiC paper (Carborundum Abrasivos Ltda, Recife, PE, Brazil) to obtain a flat area with a diameter of 5.5 mm, which was controlled with the use of a digital caliper rule (Mitutoyo Corp., Kanogawa, Japan). All ceramic disks were then cleaned ultrasonically (Odontobras) in water for 20 seconds. The samples were randomly paired for cementation and divided into six groups, and each group was submitted to a different surface treatment:⁵



Figure 1. Specimen preparation for tensile bond strength. (A) Metallic adaptor; (B) ceramic disk; (C) PVC tube; (D) ceramic disk positioned into metallic adaptor; (E) PVC tube adjusted in the metallic adaptor and embedded in acrylic resin; (F) specimen positioned in a stainless steel holder for polishing with SiC paper; (G) ceramic disks joined in pairs. Measurements in mm.

-Groups 1 and 4: etching with 10% hydrofluoric acid (Dentsply, Petrópolis, RJ, Brazil) for 20 seconds, followed by distilled water rinsing for 1 minute. Samples were then cleaned ultrasonically with water for 20 minutes and dried with compressed air.

–Groups 2 and 5: sandblasting with 50 μ m Al₂O₃ particles (Bioart, São Carlos, SP, Brazil) for 5 seconds under a pressure of 2 bars, with a sandblasting device (Microetch, Bioart) held 10 mm from the ceramic surface. Samples were then rinsed, cleaned ultrasonically in distilled water for 20 minutes, and dried with compressed air.

–Groups 3 and 6: sandblasting with 100 μm Al_2O_3 particles (Bioart) for 5 seconds as described for groups 2 and 5.

One coat of Monobond-S (Ivoclar Vivadent) was applied to all samples and allowed to air dry for 2 minutes, and one coat of Heliobond adhesive (Ivoclar Vivadent) was then applied. A resin luting cement, Variolink II (Ivoclar Vivadent), was manipulated according to the manufacturer's instruction and applied to the ceramic surface. Randomly paired ceramic disks were then joined, and a 500-g static load was applied for 1 minute. The excess cement was removed with a brush before light curing for four 40-second periods at right angles to each other using an XL 2500 curing unit (3M ESPE, St Paul, MN) with 650 mW/cm² of power density. The samples of Groups 1 to 3 were stored in distilled water at 37°C for 24 hours, and those of Groups 4 to 6 were stored for 1 year. The distilled water was changed once a week.

Tensile strength testing was performed using an Instron universal testing machine (Canton, MA) at a crosshead speed of 1.0 mm/min until failure occurred



Table 1. Mean Tensile Bond Strength (MPa) and Standard Deviation (SD) of IPS Empress 2 Ceramic Treated with Different Surface Treatments, Following 24 Hours and 1 Year of Storage

	Tensile Bond Strength (MPa)			
Surface Treatments	24 Hours	SD	1 Year	SD
10% hydrofluoric acid 50 μ m Sandblasting 100 μ m Sandblasting	15.54 ^{aA} 10.60 ^{bA} 7.87 ^{cA}	4.53 3.32 2.26	10.10^{aB} 6.34^{bB} 2.60^{cB}	3.17 1.06 0.41

Means followed by different lower case letters in each column and upper case letters in each row differ significantly at a 5% significance level according to the Tukey test.

Stereomicroscopic examination demonstrated that the majority of failures were *mixed* in the surface treatment with 10% hydrofluoric acid for both storage times and *adhesive* for the surface treatment with 50 and 100 μ m Al₂O₃ sandblasting (Table 2). Figures 3 to 6 show SEM sample images of the surface morphology of IPS Empress 2 ceramic after different surface treatments.

Discussion

In this study, the effects of three ceramic surface treatments, as well as the effect of water storage conditions (24 hours and 1 year), on the tensile bond strength between IPS Empress 2 ceramic and resin luting agent were evaluated.

Morphology modifications on the ceramic surface may be performed to promote better bond strength. In this study, three treatments were carried out: 10% hydrofluoric acid etching and sandblasting with 50 and 100 μ m aluminum oxide particles.

Etching with hydrofluoric acid was the most effective procedure for enhancing and retaining bond strength.⁷ In this study, the etching

Table 2. Failure Mode Analysis of the Debonded Specimens

Surface Treatment	Adhesive	Mixed	Cohesive
100 μ m sandblasting (24 hours)	9	6	_
$50 \ \mu m$ sandblasting (1 year) $50 \ \mu m$ sandblasting (24 hours)	13 7	2 8	_
50 μ m sandblasting (1 year) 10% hydrofluoric acid (24 hours)	10 1	5 12	$\frac{-}{2}$
10% hydrofluoric acid (1 year)	3	11	1

Figure 2. Tensile test.

(Fig 2). The tensile bond strength was calculated by dividing the maximum load by the cross-sectional area under the test to give results in MPa. Fifteen samples were made and tested for each group. The results were subjected to analysis of variance and Tukey's test at the 95% significance level.

A stereomicroscope (Olympus Corp, Tokyo, Japan) at $\times 20$ was used to visualize the fractured surfaces of the samples and to classify the type of failure that occurred during the debonding procedure. Failure was classified as *adhesive* when the fracture occurred at one of the ceramic interfaces, *cohesive* when the resin cement was fractured, and *mixed* with a combination of adhesive and cohesive failures. The surfaces treated by sandblasting with 100 and 50 μ m Al₂O₃ particles and etching with 10% hydrofluoric acid were coated in gold and examined by scanning electron microscopy (SEM; LEO 435 VP, Cambridge, England).

Results

The samples surface treated with 10% hydrofluoric acid demonstrated significantly higher bond strengths than those that were sandblasted with 50 and 100 μ m Al₂O₃ (p < 0.05). Sandblasting with 50 μ m Al₂O₃ produced significantly higher tensile bond strengths than sandblasting with 100 μ m Al₂O₃ (p < 0.05) for the storage times of 24 hours and 1 year (Table 1). The values of the tensile bond strength for the 24-hour storage time for the three surface treatments were significantly higher than for the 1-year storage time (p < 0.05) (Table 1).



Figure 3. SEM morphological aspect of ceramic surface treated with 10% hydrofluoric acid etching for 20 seconds.

procedure with 10% hydrofluoric acid resulted in a statistically higher tensile bond when compared with sandblasting with 50 and 100 μ m Al₂O₃.

The difference in bond strength can be explained on the basis of differences in morphology between etched and sandblasted samples. Acid etching of surfaces promotes dissolution in the glassy matrix of the samples to the depth of a few microns; as a result, the lithium disilicate crystal protrudes from the glassy matrix (Figs 3 and 4). The change in surface morphology increased the surface area and favored infiltration and retention of adhesive, making the ceramic surface more retentive. Etching with hydrofluoric acid has also



Figure 5. SEM of morphological aspect of ceramic surface treated with sandblasting with 50 μ m Al₂O₃ particles.

been noted as an efficient surface treatment for other ceramics. $^{5,7,15-18}$

Our results indicate that air abrasion with 50 and 100 μ m aluminum oxide particles cannot provide a mechanically retentive surface as satisfactorily as etching with hydrofluoric acid can. The lowest mean bond strength was obtained for samples sandblasted with 100 μ m Al₂O₃ (Fig 6). Sandblasting with 100 μ m Al₂O₃ is the customary method used in dental laboratories to remove the refractory investment. This promotes morphologic alteration of the ceramic surface, resulting in an increase in the number of potential retention areas.¹⁸ SEM showed evidence of a roughness that contains a large number of microscopic undercuts.



Figure 4. Higher magnification of specimen in Figure 3 of ceramic surface treated with 10% hydrofluoric acid etching for 20 seconds.



Figure 6. SEM of morphological aspect of ceramic surface treated with sandblasting with 100 μ m Al₂O₃ particles.

Sandblasting with 50 μ m increased the ceramic surface roughness (Fig 5), which promoted a significant increase in bond strength compared with sandblasting with 100 μ m Al₂O₃; however, neither of the procedures produced a surface as retentive as that obtained with etching.

After ceramic surface treatments, the silane agent was applied on the ceramic surface followed by the Variolink II resin luting agent. Both of these applications are necessary to maintain the bond strength between ceramic and resin luting cements.^{16,19-22}

Silane coupling agents are usually monomeric species in which silicon is linked to reactive organic radicals and hydrolyzable ester groups. The reactive organic groups become chemically bonded to the resin molecules, such as BisGMA and TEGDMA, found in the Heliobond adhesive, as well as in the Variolink II resin cement. Hydrolyzable monovalent groups bond chemically to silicon contained in the glassy matrix and lithium disilicate crystal in the IPS Empress 2.⁵ Another important factor is the capacity of silane to improve surface wettability,²³ causing better contact and infiltration of the adhesive into irregularities caused on ceramic surface by the sandblasting or etching.²⁴

The durability of the bond between the silinated ceramic surface and the resin luting agent²⁵ decreased with water storage and thermocycling. Depending on silane type used, thermocycling might have a significant effect on the bond strength reduction between the resin-ceramic interface.^{13,26-29} Degree of hydrolysis is responsible for the difference between silane products and their efficacy-the higher the degree of hydrolysis, the better the bond provided by the silane agent.³⁰ Silane permeability is hydrolysis of the Si-O bonds at the porcelain-silane interface by the water absorption. This fact may also be responsible for the degradation level of the bond strength between the ceramic-resin interface during water storage.³¹ Spohr et al⁵ reported that the Scotchbond Ceramic Primer followed by Single Bond adhesive system and Rely X resin luting cement was effective in the thermocycling procedure, since the standard failure mode was mixed or cohesive at the resin cement and not at the cementceramic interface. The Monobond-S silane used in this study was shown to be partially efficient in water storage conditions, since the mean bond strength decreased after 1 year, and the failure

was mainly adhesive for the ceramic sandblasted with 50 or 100 μ m aluminum oxide particles. However, the failure modes were mainly mixed for acid-etched samples, which showed a decrease in bond strength.

In the present study (Table 1), the data showed the lowest bond strength in specimens stored in distilled water for 1 year. Kern and Thompson¹⁴ showed no statistical difference after 30 days of storage in isotonic artificial saliva solution and thermocycling; however, after 150 days of storage, a significant decrease in bond strength was observed. Shahverdi et al¹⁷ showed that storage period and thermocycling decreased the bond strength of ceramic surface samples treated with different methods such as hydrofluoric acid etching, roughening with stones, and sandblasting with aluminum oxide particles. Roulet et al⁷ reported that when three different ceramic surface treatments were used (hydrofluoric acid etching, sandblasting with aluminum oxide, and ground with 600-grit SiC abrasive) and fixed with dual resin cement followed by silane agent, bond strength to both sandblasted and ground specimens decreased by 50% to 75% after 1 year of storage in water, while no difference was found for the etching acid.

Another factor is the reduction in the mechanical properties of composite resins in water storage. Some studies have shown that the reduction in the mechanical properties of composite resins aged in water may occur within 2 to 6 months.³²⁻³⁴ Carrilho et al³⁵ observed significant reduction in the mechanical properties of the composite resins after 1 year of storage in water. The reduction of its properties is probably a result of a continuous action of water on the structure of the material. The mechanism of water transport and its effects on the mechanical properties of polymers depend on several factors.^{36,37} Composition and monomer ratio varies according to the specific applications and manufacturer's goals,³⁸ and variability will define the chemical stability of resin in a specific environment.³⁹ Increasing the ratio of TGDMA and UDMA to BisGMA caused an increase susceptible to water sorption.^{40,41} Besides, the sensitivity of resin-based materials to water also depends on the degree of monomer conversion,⁴² presence of fillers, volume fraction of intrinsic nanometersized pores, and degree of polymer cross-linking.³⁶ Perhaps the presence of the UDMA and TGDMA in the resin cement used in this study may have contributed to the acceleration of water sorption

and affected the mechanical properties of the resin cement after 1 year of storage in water.

The present investigation evaluated one silane agent and one resin cement. Future study could include various other available cements and silane agents to compare materials and tensile bond strength over time.

Conclusions

Within the limitations of the present in vitro study, it can be concluded that:

- (1) Tensile bond strengths for specimens surface treated with 10% hydrofluoric acid and specimens sandblasted with 50 μm and 100 μm aluminum oxide particles, following storage for 24 hours, were significantly higher than following storage for 1 year.
- (2) The surface treatment with 10% hydrofluoric acid resulted in significantly stronger tensile bond strength values than those specimens treated by sandblasting with 50 and 100 μ m aluminum oxide particles for both storage times tested (24 hours and 1 year).

Acknowledgment

The authors would like to thank FAPESP (Fundação de Amparo à Pesquisa do Estado de São Paulo) process 01/07687-4 for financial support for this project.

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