The Influence of Internal Surface Treatments on Tensile Bond Strength for Two Ceramic Systems

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Clinical Relevance

Appropriate surface treatment in ceramic restorations is an important step in the cementation process and should be conducted for each type of ceramic.

SUMMARY

Statement of the Problem: The ceramic composition and surface microstructure of all-ceramic restorations are important components of an effective bonding substrate. Hydrofluoric acid and sandblasting are well-known procedures for

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surface treatment; however, surface treatment for high alumina-containing and lithium disilicate ceramics have not been fully investigated.

Purpose: This *in vitro* study evaluated the tensile bond strength of resin cement to two types of ceramic systems with different surface treatments.

Methods and Materials: Thirty specimens of each ceramic system were made according to the manufacturer's instructions and embedded in polyester resin. Specimens of In-Ceram Alumina [I] and IPS Empress 2 [E] were distributed to three groups with differing surface treatments (n=10): sandblasting with 50 µm aluminum oxide (APA); sandblasting with 110 µm aluminum oxide modified with silica particles (ROCATEC System-RS); a combination of sandblasting with APA and 10% hydrofluoric acid etching (HA) for two minutes on In-Ceram and for 20 seconds for IPS Empress 2. After the respective surface treatments, all the specimens were silanated, and Rely-X resin cement was injected onto the ceramic surface and light polymerized. The specimens were stored in distilled water at 37°C for 24 hours and thermally cycled 1,100 times (5°C/55°C). The

tensile bond strength test was performed in a universal testing machine at a 0.5 mm/minute crosshead speed.

Results: The mean bond strength values (MPa) for IPS Empress 2 were 12.01 ± 5.93 (EAPA), 10.34 ± 1.77 (ERS) and 14.49 ± 3.04 (EHA). The mean bond strength values for In-Ceram Alumina were 9.87 ± 2.40 (IAPA) and 20.40 ± 6.27 (IRS). All In-Ceram specimens treated with 10% hydrofluoric acid failed during thermal cycling.

Conclusion: The Rocatec system was the most effective surface treatment for In-Ceram Alumina ceramics; whereas, the combination of aluminum oxide sandblasting and hydrofluoric acid etching for 20 seconds worked more effectively for Empress 2 ceramics.

INTRODUCTION

The search for esthetic restorations has become a challenge, because patients have higher expectations from esthetic treatments.¹⁻² The introduction of the enamel etching technique, combined with effective adhesion to dentin, allowed resin composite to be use directly on the dental structure.³⁻⁴ However, since resin composites have mechanical limitations, ceramic systems that are more resistant to masticatory forces have been developed and have allowed all-ceramic restorations, without metal infrastructure, to be fabricated.⁵⁻⁶ These allceramic restorations have been indicated for inlays, onlays, crowns and fixed partial dentures.⁷ One of the technical modifications for improving ceramic strength has been to increase the amount of crystals in relation to the glass phase.^{5,8-9}

Many types of high strength ceramics that provide superior esthetic appearance have been developed since 1980, including In-Ceram Alumina (In-Ceram; Vita Zahnfabrik, Bad Säckingen, Germany) and IPS Empress 2 (Ivoclar/Vivadent, Schaan, Liechtenstein). The In-Ceram Alumina ceramic was developed in France by Mickäel Saudon and consisted of an alumina infrastructure fabricated by slip casting or by CAD/CAM. The alumina infrastructure was then infiltrated with glass. Over this infrastructure, a veneering feldspathic ceramic was applied to provide form, function and esthetics. The 0.5 to 3.5 um size alumina particles were interspersed with lantanium and amino-silicate, with a low quantity of sodium and calcium in its glass phase. After glass infiltration in sinterized alumina, ceramic flexural strength was reported to be 236.15 Mpa.^{5,10} Due to its high strength, In-Ceram Alumina was suggested for posterior restorations and for threeunit fixed partial dentures. 11-13 This development of reinforcing ceramics with alumina and quartz crystals enabled ceramics to be used as a metal-free infrastructure for crowns and fixed partial dentures because of their improved mechanical properties.¹⁴

Another system is IPS Empress 2, a lithium dissilicate glass ceramic with a flexural strength of approximately 350 Mpa. ¹⁵ According to the manufacturer, the IPS Empress 2 ceramic system is composed of 60% lithium dissilicate by volume, embedded in a glass matrix.

The cementation process is vital for the clinical success of all-ceramic restorations. Ceramics reinforced with a high crystal content, such as In-Ceram Alumina, can be luted by conventional techniques using zinc phosphate or glass ionomer cements. When mechanical retention and stability of the dental preparation requires adhesive cementation with resin cements, the internal surface of the ceramic should be treated to provide mechanical retention. However, increasing the mechanical strength by increasing the crystalline content and decreasing the glass content results in an acid resistant ceramic in which any type of acid treatment produces insufficient surface changes for adequate bonding to resin.

For the In-Ceram system, aluminum oxide sandblasting has been suggested to promote surface changes;18-19 however, conventional surface treatments used for feldspathic ceramics, such as hydrofluoric acid etching and silane application, are considered to be not as effective due to the high alumina and minimal glass phase content within its composition. All-ceramic coping materials, such as alumina and zirconia ceramic, are not sufficiently roughened by airborne-particle abrasion or hydrofluoric acid etching and do not sufficiently react with a silane coupling agent due to their low silica content. 6,8,20 Different surface treatments, such as the Rocatec system, 8,11 Silicoater MD and PyrosilPen, seem to improve the bond strength between aluminous ceramic and resin cement. This silica coating process is based on similar adhesive principles and appears to be a promising method for treating high crystalline ceram-

Other surface treatments have been suggested for In-Ceram Alumina ceramics, such as the application of a sintered refractive powder suspension on the ceramic surface,²¹ the Bateman Etch Retention System (BEARS) application¹⁸ and synthetic diamond particle sandblasting.²² These methods provide greater bond strength values and could, therefore, solve the In-Ceram Alumina surface treatment problem.^{8,11,20}

IPS Empress 2 has been shown to benefit from hydrofluoric acid treatment;²³ however, little information has focused on the specific surface treatment for lithiumdisilicate ceramics.

Therefore, this *in vitro* study evaluated the bond strength between two ceramic systems (In-Ceram and IPS Empress 2) and a resin cement with different surface treatments, namely, sandblasting with 50 µm aluminum oxide, silicate coating using the Rocatec system and hydrofluoric acid etching, to test the hypothesis

that higher bond strength values can be achieved with the Rocatec System for In-Ceram Alumina ceramics and the association of hydrofluoric acid and sandblasting with $50~\mu m$ aluminum oxide for the IPS Empress 2 ceramics

METHODS AND MATERIALS

The entire preparation sequence for specimens is shown in Figure 1. For each ceramic type (In-Ceram Alumina and IPS Empress 2), 30 conical specimens were fabricated (4 mm thick, 5 mm in diameter at one end and 6 mm in diameter on the opposing end surface) in a die stone master cast, following the manufacturer's instructions (Figure 2). For the In-Ceram specimens, three layers of die spacer (Interspace Varnish, Vita Zahnfabrik, Bad Säckingen, Germany) were applied over the stone die. An impression was made with impression material (Vita Duplication Material, Vita Zahnfabrik) and the dies were duplicated in plaster. The aluminum oxide powder was mixed with a liquid as instructed by the manufacturer. The slurry mixture was then painted over the special plaster die (Special Plaster, Vita Zahnfabrik) and fired at 1120°C for 10 hours in a furnace. Glass infiltration was obtained by coating the aluminum oxide framework with a glass powder- (silicate-aluminum-lantanium, Vita In-Ceram, Vita Zahnfabrik) distilled water mixture and firing in the furnace for four hours at 1100°C.

For IPS Empress 2, two 0.8 mm thick layers of die spacer were applied over the stone die and wax conical copings were prepared. The wax patterns were invested in IPS investment and eliminated in a burnout furnace (EP 500, Ivoclar, Vivadent) pre-heated to 920°C, heating the refractory die at the same time as the IPS Empress 2 ingots and the alumina plunger at 3°C per minute to 850°C and held for 90 minutes. Next, the investment, plunger and ingot were transferred to a furnace that increased the temperature to 1180°C and pressed the melted ingot into the mold. After pressing and cooling to room temperature, the specimens were divested with 50 µm glass beads at 2-bar pressure, ultrasonically cleaned in a special liquid (Invex liquid, Ivoclar-Vivadent) and dried.

The ceramic cones were fixed, with the smaller base directed towards the surface and a cylindrical matrix positioned around the ceramic cone to allow polyester resin (Buehler, Lake Bluff, IL, USA) to be poured into the matrix and flow around the specimen until a height of 10 mm was achieved around the specimen (Figure 3). The cylindrical matrix was made of plastic and was not deformable. After 24 hours of storage, the resin-ceramic specimens were removed from the matrix, and the ceramic surfaces were polished with 600 grit carbide paper for 15 seconds in a mechanical grinder. The specimens were cleaned in an ultrasonic cleaner for five minutes with distilled water and dried. Subsequently,

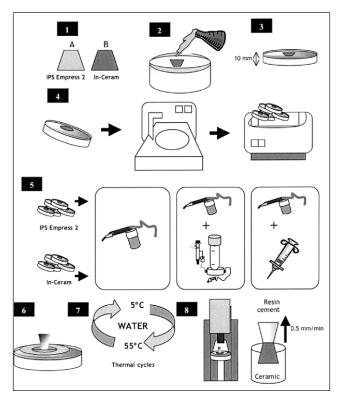


Figure 1: 1) Conical specimens of In-Ceram and IPS Empress 2 were fabricated (4x5x6 mm); 2) Ceramic cones were fixed with the smaller base towards the surface and a cylindrical matrix was positioned around the ceramic cone; 3) Resin-ceramic specimens were removed from the matrix; 4) The surface of the ceramics was polished with 600 grit carbide paper in a mechanical grinder. The specimens were cleaned in an ultrasonic cleaner; 5) The specimens were randomly assigned to experimental groups: I) 50 μm aluminum oxide sandblasting; II) 50 μm aluminum oxide sandblasting + 110 μm silica oxide particle sandblasting (Rocatec Plus); III) 50 µm aluminum oxide sandblasting + 10% hydrofluoric acid; 6) After surface treatment, all the specimens received a silane application, and a Teflon matrix with a conical internal shape was placed over each ceramic block in which resin cement was inserted and polymerized; 7) 1100 thermal cycles of one minute dwell time at 5°C and at 55°C was performed; 8) Tensile bond strength testing was carried out using a universal testing machine.

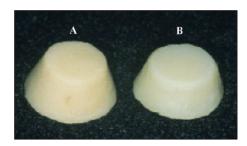


Figure 2: The ceramic cones (A: In-Ceram/ B: IPS Empress 2).

the specimens were kept in air-tight containers at 37°C for 48 hours. The specimens were randomly assigned to the experimental groups as shown in Table 1.

In group IAPA, the In-Ceram specimens received 50 um of aluminum oxide sandblasting for 10 seconds under a pressure of 2.8 bars with a Sandblaster Micro Etcher (Danville Engineering Co, San Ramon, CA, USA), with the nozzle positioned 10 mm from the surface.^{8,24} The specimens were then washed with water for 20 seconds and air dried with absorbent papers for five seconds. In the IRS group, after aluminum oxide sandblasting, as described in the IAPA group, the In-Ceram specimen surfaces received 110-um silica oxide particle sandblasting (Rocatec Plus, 3M ESPE, St Paul, MN, USA) for 10 seconds and 10 mm from the surface under 2.8 bars of pressure according to the manufacturer's instructions. The excess particles were removed by air blasting for five seconds. In the IHA group, the In-Ceram specimens were sandblasted with aluminum oxide followed by 10% hydrofluoric acid etching (Dentsply In de Com Ltda, Petropolis, RJ, Brazil) for two minutes, following the manufacturer's and published recommendations.2 Then, all the specimens were washed with water for 20 seconds and air-dried for 10 seconds. Groups EAPA, ERS and EHA consisted of IPS Empress 2 specimens and underwent the same surface treatments as the IAPA, IRS and IHA group, respectively, except that, in Group EHA, the IPS Empress blocks were etched for 20 seconds instead of two minutes, according to the manufacturer's instructions.

After surface treatment, all the specimens received a silane application (Rely-X Ceramic Primer, 3M ESPE) and five-second gentle air spray. A Teflon matrix with a conical internal shape was placed over each ceramic block and resin cement (Rely-X, 3M ESPE) was inserted into the inverted cone with a syringe (Centrix Inc, Shelton, CT, USA).²² These specimens were polymerized for 40 seconds using an XL 1500 (3M ESPE) light polymerization unit at 560mW/cm², verified by a radiometer (Demetron Research, Kerr, USA, Orange, CA, USA), with the light directed at approximately a 45° degree angle to the intersection of the ceramic bonding sites and the resin cement. Four 40-second polymerization sequences, divided equally around the circumference of the matrix, were completed. The Teflon matrix was then removed and another light polymerization was performed for 40 seconds directly on the specimen and towards the resin cement (Figure 4). The specimens were stored in distilled water at 37°C for 24 hours, followed by 1100 thermal cycles of one minute dwell time

at 5°C and at 55°C.²⁵ Thermal cycling was performed to simulate oral temperature and humidity. This is an important step when evaluating bond strength with *in vitro* studies.²⁵⁻²⁷

The specimens (ceramic cones embedded in polyester resin + resin cement cones) were mount-



Figure 3: The ceramic cone positioned after removal of the cylindrical matrix.



Figure 4: Resin cement cones made with a Teflon matrix.

ed on the bottom fixture of a universal testing machine (Instron, Model 4442, Canton, MA, USA), and the bonded resin cement was gripped by the top apparatus and subjected to a tensile force at a crosshead speed of 0.5 mm/minute until fracture occurred (Figure 5). Tensile bond strengths were calculated and recorded using the formula: σ =P/A, where σ is the tensile bond strength (MPa), P is the force (N) and A is the interfacial area (mm²). The data were analyzed with the Kruskal-Wallis test due to non-normally distributed data and the Mann-Whitney test to compare groups 2 by 2, with the level of significance being 5%.

Table 1: Group Surface Treatment and Ceramic Type						
Groups	Ceramic Type	Treatment				
IAPA	In-Ceram Alumina	50 μm sandblasting				
IRS	In-Ceram Alumina	50 μm sandblasting + Rocatec System				
IHA	In-Ceram Alumina	50 μm sandblasting + 10% hydrofluoric acid				
EAPA	IPS Empress 2	50 μm sandblasting				
ERS	IPS Empress 2	50 μm sandblasting + Rocatec System				
EHA	IPS Empress 2	50 μm sandblasting + 10% hydrofluoric acid				

To illustrate the work, four additional blocks of each ceramic type were made, and the surface topography of the internal treatment of the two types of ceramics was examined by a scanning electron microscope (SEM). The blocks were carbon sputtered and observed by SEM

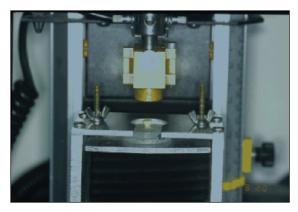


Figure 5: Specimen in position in the Universal testing machine for the tensile bond strength test.

Table 2: Tensile Bond Strength Mean and Median Values							
Groups	ps Mean (MPa) Median (MPa)		Standard Deviation				
IAPA	9.87	9.63	2.40				
IRS	20.40	20.45	6.27				
EAPA	12.01	11.85	5.93				
ERS	10.34	10.10	1.77				
EHA	14.49	13.65	3.04				

*Group IHA was eliminated from the statistical analysis due to few specimens being obtained

Table 3: Kruskal-Wallis Test Analysis						
Groups	N	Median	Mean Post	Z statistics		
APA	10	9.63	14.50	-2.67		
IRS	10	20.45	41.00	3.76		
EAPA	10	11.85	23.40	-0.51		
ERS	10	10.10	16.50	-2.19		
EHA	10	13.65	32.20	1.61		
Geral	50		25.5			
Descriptive level				0.000		
*Group IHA was eliminated from the statistical analysis due to few specimens being obtained.						

Table 4: Mann-Whitney Test to Compare Groups Two by Two* **IPS Empress 2** In-Ceram Ceramic **Treatment APA** RS HA **APA** RS 0.5205 0.0006 In-Ceram **APA** 0.2413 0.0036 RS 0.0102 0.0006 0.0343 0.0006 IPS Empress 2 APA 0.4057 0.3075 0.2413 0.0102 RS 0.0017 0.4057 0.5205 0.0006 HA 0.3075 0.0017 0.0036 0.0343 *Values below 0.0051 show significant differences.

(Phillips XL-30, FEI Phillips, São Paulo, Brazil) at 200x and 1000x magnification

RESULTS

All In-Ceram blocks treated with hydrofluoric acid failed during thermal cycling, even after the twominute exposure time recommended by the manufacturer, which was strictly followed. Therefore, no statistical analysis was made of the specimens of the IHA group. Tensile bond strength values for the different surface treatments are described in Table 2. In order to assess differences among the groups, it was necessary to use the non-parametric Kruskal-Wallis test (Table 3), due to non-normally-distributed variables and the impossibility of transforming the data. The Kruskal-Wallis test showed significant differences among all groups (p<0.005). The Mann-Whitney test was performed to compare groups 2 by 2, as shown in Table 4.

With regards to the IPS Empress 2 ceramics, there was no statistical significance between the EAPA (sandblasting) and ERS (Rocatec System) groups; however, the bond strength values for the hydrofluoric acid

> group (EHA) showed statistical significance when compared with the other groups. For the In-Ceram ceramics, statistical significances were shown between the two groups, and the Rocatec group presented higher bond strength values when compared with sandblasting.

SEM analysis showed differences in the ceramic surfaces after the different surface treatments. Figure 6A-D shows the

ceramic surfaces in In-Ceram specimens and Figure 7A-D shows the representative micrographs of IPS Empress 2 ceramics. It can be noted that the IPS Empress 2 surfaces presented fewer rough surfaces than the In-Ceram specimens. The micrographs showed that, after the Rocatec System, In-Ceram presented the roughest surface, and no change could be detected after airborne particle abrasion with alu-

> minum oxide and acid etching when compared with the control group (without treatment). The IPS Empress 2 specimens changed after surface treatments, thus obtaining a retentive pattern after hydrofluoric acid etching with elongated crystals and shallow irregularities.

^{*}Group IHA was eliminated from the statistical analysis due to few specimens being obtained.

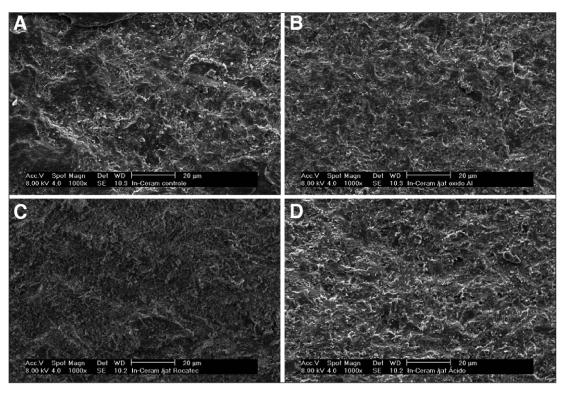


Figure 6: A- Photomicrograph of In-Ceram Alumina without surface treatment (I), B- sandblasted with aluminum oxide (IAPA), C- sandblasted with Rocatec System (IRS), D- Alumina etched with 10% hydrofluoric acid (IHA) (original magnification 1000x).

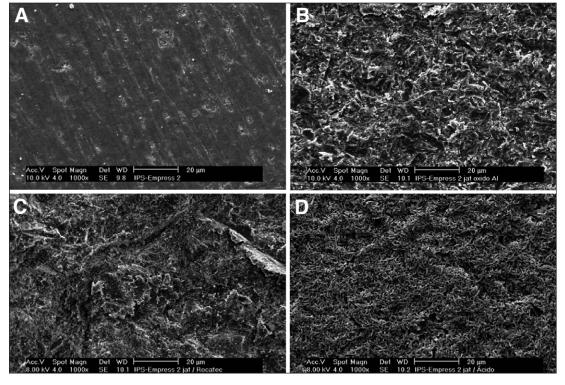


Figure 7: A- Photomicrograph of IPS Empress 2 without surface treatment (E), B- sandblasted with aluminum oxide (EAPA), C- sandblasted with Rocatec System (ERS), D- etched with 10% hydrofluoric acid (EHA) (original magnification 1000x).

DISCUSSION

Research has focused on the bond strength between ceramic restorations and resin cement; however, little information has been provided on the correct use of surface treatments in different types of ceramics.^{2,19} The results of the current study confirmed the hypothesis that sandblasting In-Ceram ceramics with aluminum oxide modified with silica particles (Rocatec System) was able to increase the bond strength to a resin luting agent. Kern and Thompson⁸ observed surface irregularities on the surface of In-Ceram ceramics associated with an increase from 15.8% to 19.7% in silica content after treatwith ment Rocatec Plus system and its ability to interact with the silane treatment, suggesting that this surface treatment could improve the bond strength to resin, which was later reported by other $studies^{\scriptscriptstyle 12,19\text{-}20,28\text{-}29}$ confirmed in the current work.

The sandblasting method has indeed been widely used to promote micromechanical retention in several types of ceramics, and it is the conventional procedure performed by commercial laboratories. However, due to an increase in the

crystalline content and a decrease in glass content, an acid resistance can be seen in ceramics, so that any type of acid treatment produces insufficient surface changes for adequate bonding to resin.¹⁷

Ceramics reinforced with a high crystal content, such as In-Ceram Alumina, can be luted by conventional techniques using zinc phosphate or glass ionomer cements. He has a mechanical retention and stability of the dental preparation requires adhesive cementation with resin cements, the internal surface of the ceramic should be treated to provide mechanical retention. It is suggested that silicatization and silanization procedures are essential in promoting bonding between resin and In-Ceram ceramic.

Hydrofluoric acid etching is a procedure that creates micro roughness on the internal surface of ceramic restorations12,18 and could improve a cement bond. Many authors have shown that the use of hydrofluoric acid etching, with a silane application, can improve some ceramic interaction with resin cements. 25,30-33 Silane has the ability to wet and, consequently, contributes to covalent bonding between the siloxane and ceramic SiO₂ group. Silane is considered a bi-functional coupling agent. This means that each molecule reacts with two different surfaces: the inorganic phase of the ceramic material and the organic component of the resin luting agent. In view of the scientific literature supporting the use of silane agents, the silane application was used in all tested groups in this study. Research involving hydrofluoric acid etching and silane application has shown an increase in bond strength values in ceramics that incorporate silica particles. $^{25,30\cdot31,33\cdot34}$ Ceramics with a high Al_2O_3 content are resistant to acids and, therefore, hydrofluoric acid, which successfully etches feldspathic ceramics and has no effect on aluminous ceramic surfaces. 35 Hydrofluoric acid etching, followed by silane application, is accepted as not providing a strong bond in In-Ceram ceramics, 20-21 as demonstrated in the current study results, due to its minimal glass phase and high aluminum oxide content, which renders it acid resistant.20

Madani and others¹⁹ verified that an increase in hydrofluoric acid concentration decreased bond strength between In-Ceram Alumina and Panavia 21 cement, demonstrating its inefficiency. These findings are in agreement with the current study, in which the In-Ceram Alumina acid group failed during thermo cycling, thus presenting adhesive failures between ceramic and resin cement. This can be explained by the association between thermal cycling and partial silica removal from the ceramic glass matrix during etching, which results in an ineffective silane action.

As the interaction between silane and ceramics depends on the presence of SiO₂,³⁶ some studies suggest a silicoating process for aluminous ceramics, such

as the Rocatec System, the Silicoater MD (Heraeus Kulzer, Hanau, Germany), the PyrosilPen Technology (SurA Instruments, Jena, Germany), airborne abrasion with diamond particles,²⁴ surface grinding with diamond burs³⁷ and irradiation with Nd:YAG laser¹⁶ to improve bonding to resin cements.

According to Jedynakiewicz and Martin,36 silane coupling-agents are recognized as a critical component of resin-based composites in which they enable bonding of the resin matrix to glass filler particles. Silane coupling-agents may be used to enhance ceramic restoration bonding to resin-based composite luting agents. The chemical coupling is achieved by the reaction of gamma-methacryloxypropyl trimethoxysilane to the silicon oxide phase in the ceramic. Silanes are not a substitute for micro-mechanical resin bonding to the etched ceramic surface; instead, they are a way of strengthening this union with a chemical bond. Following application of silane to the ceramic, the methacrylate group of silane is available for cross-linking with the methacrylate group in the resin adhesive. The adhesive will, in turn, cross-link with the methacrylate groups in the luting composite. In this way, a very strong bond is formed, which will resist the forces of polymerization shrinkage and avoid microleakage.

The Rocatec System uses sandblasting of the ceramic surface with alumina silica to coat the ceramic surface with a $0.1~\mu m$ silica layer. This study showed that the Rocate System group of In-Ceram ceramics had greater bond strength than the sandblasting group, which is in agreement with previous studies. 16,20,29

With regards to IPS Empress 2, which contains lithium dissilicate, there are few studies on surface treatments. According to the manufacturer's recommendation for IPS Empress 2 ceramics, 20 seconds of hydrofluoric acid etching is indicated, followed by silane application, while excessive exposure time to hydrofluoric acid would cause the ceramic to weaken.

In the current study, IPS Empress 2 etched with hydrofluoric acid had a mean bond strength of 14.49 MPa. This IPS Empress 2 acid group presented statistically significant differences compared to the silica group (Rocatec Plus). Due to possible ceramic weakness caused by excessive acid etching, it was imperative in this study to subject the specimens to 20 seconds of acid etching. The IPS Empress 2 acid group, and the same ceramic group treated with aluminum oxide sandblasting, did not show any statistical differences, suggesting that the standard surface treatment with aluminum oxide sandblasting is effective for the IPS Empress 2 ceramic.

Morphological analyses of the acid treated IPS Empress 2 ceramic showed an irregular surface as a result of etching the glassy component of the material,

making the surface more retentive. Aluminum oxide sandblasting and Rocatec surface treatment showed a similar morphological surface; however, with irregularities not presenting a retentive pattern. The similarity of surface roughness promoted by these types of particles was also reported by Borges and others.² These results show the importance of silica deposition for chemical bonds to increase the interaction between In-Ceram and resin cement.

In a study by Kim and others,⁶ alumina and zirconia ceramic specimens treated with a silica coating technique, and lithium disilicate ceramic (IPS Empress 2) specimens treated with airborne-particle abrasion and acid etching, yielded the highest tensile bond strength values to a resin composite for the materials tested.

Future studies should examine the effect of silica and alumina coating on the bond strength to other ceramics. The differences in composition and microstructure of all-ceramic restorations might be an important factor in obtaining effective bond strengths between the ceramic and resin-luting agent.

CONCLUSIONS

In this study, the Rocatec system provided the most effective surface treatment for In-Ceram Alumina ceramics. However, this bonding system did not appear to benefit IPS Empress 2 ceramics, which attained higher bond strength values with aluminum oxide sandblasting and hydrofluoric acid etching for 20 seconds.

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References

- Christensen GJ (1999) Porcelain-fused-to-metal vs nonmetal crowns Journal of the American Dental Association 130(3) 409-411.
- Borges GA, Spohr AM, De Góes MF, Sobrinho LC & Chan DCN (2003) Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics *Journal of Prosthetic Dentistry* 89(5) 479-488.
- 3. Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34** (6) 849-853.
- Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials Research* 16(3) 265-273.

 McLean JW & Hughes TH (1965) The reinforcement of dental porcelain with ceramic oxides British Dental Journal 119(6) 251-267.

- Kim BK, Bae HE, Shim JS & Lee KW (2005) The influence of ceramic surface treatments on the tensile bond strength of composite resin to all-ceramic coping materials *Journal of Prosthetic Dentistry* 94(4) 357-362.
- Anusavice KJ (1993) Recent developments in restorative dental ceramic Journal of the American Dental Association 124(2) 72-74.
- Kern M & Thompson VP (1994) Sandblasting and silica coating of a glass-infiltrated alumina ceramic: Volume loss, morphology, and changes in the surface composition *Journal of Prosthetic Dentistry* 71(5) 453-461.
- Valandro LF, Della Bona A, Antonio Bottino M & Neisser MP (2005) The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic *Journal of Prosthetic Dentistry* 93(3) 253-259.
- Chong KH, Chai J, Takahashi Y & Wozniak W (2002)
 Flexural strength of the In-Ceram alumina and In-Ceram zirconia core materials International Journal of Prosthodontics 15(2) 183-188.
- 11. Kern M & Strub Jr (1998) Bonding to alumina ceramic in restorative dentistry: Clinical results over up to 5 years *Journal of Dentistry* **26(3)** 245-249.
- Kern M, Knode H & Strubb JR (1991) The all-porcelain, resin-bonded bridge Quintessence International 22(4) 257-262
- 13. Pospiech P, Rammelsberg P & Unsold F (1996) A new design for all-ceramic resin-bonded fixed partial dentures *Quintessence International* **27(11)** 753-758.
- 14. McLean JW (1995) New dental ceramics and esthetics Journal of Esthetic Dentistry 7(4) 141-149.
- 15. Nagai T, Kawamoto Y, Kakehashi Y & Matsumura H (2005) Adhesive bonding of a lithium disilicate ceramic material with resin-based luting agents *Journal of Oral Rehabilitation* **32(8)** 598-605.
- 16. Da Silveira BL, Paglia A, Burnett LH, Shinkai RS, Eduardo CP & Spohr AM (2005) Micro-tensile bond strength between a resin cement and an aluminous ceramic treated with Nd:YAG Laser, Rocatec System, or aluminum oxide sand-blasting *Photomedicine and Laser Surgery* 23(6) 543-548.
- 17. Della Bona A & Anusavice KJ (2002) Microstructure, composition, and etching topography of dental ceramics *International Journal of Prosthodontics* **15(2)** 159-167.
- Wood DJ, Bubb NL, Millar BJ & Dunne SM (1997)
 Preliminary investigation of a novel retentive system for
 hydrofluoric acid etch-resistant dental ceramics Journal of
 Prosthetic Dentistry 78(3) 275-280.
- Madani M, Chu FC, McDonald AV & Smales RJ (2000) Effects of surface treatments on shear bond strengths between a resin cement and an alumina core *Journal of Prosthetic Dentistry* 83(6) 644-647.
- Kern M & Thompson VP (1995) Bonding to glass infiltrated alumina ceramic: Adhesive methods and their durability Journal of Prosthetic Dentistry 73(3) 240-249.
- Sadoun M & Asmussen E (1994) Bonding of resin cements to an aluminous ceramic: A new surface treatment *Dental Materials* 10(3) 185-189.

- Wolf DM, Powers JM & O'Keefe KL (1993) Bond strength of composite to etched and a sandblasted porcelain American Journal of Dentistry 6(3) 155-158.
- Zawta C (2001) Fixed partial dentures with an all-ceramic system: A case report Quintessence International 32(5) 351-358.
- Sen D, Poyrazoglu E, Tuncelli B & Goller G (2000) Shear bond strength of resin luting cement to glass-infiltrated porous aluminum oxide cores *Journal of Prosthetic Dentistry* 83(2) 210-215.
- Chang JC, Nguyen T, Duong JH & Ladd GD (1998) Tensile bond strengths of dual-cured cements between a glass-ceramic and enamel *Journal of Prosthetic Dentistry* 79(5) 503-507.
- Kern M, Fechtig T & Strub JR (1994) Influence of water storage and thermal cycling on the fracture strength of all-porcelain, resin-bonded fixed partial dentures *Journal of Prosthetic Dentistry* 71(3) 251-256.
- Diaz-Arnold AM & Aquilino SA (1989) An evaluation of the bond strengths of four organosilane materials in response to thermal stress *Journal of Prosthetic Dentistry* 62(3) 257-260.
- Amaral R, Özcan M, Bottino MA & Valandro LF (2006) Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: The effect of surface conditioning *Dental Materials* 22(3) 283-290.
- Özcan M, Alkumru HN & Gemalmaz D (2001) The effect of surface treatment on the shear bond strength of luting cement to a glass-infiltrated alumina ceramic *International* Journal of Prosthodontics 14(4) 335-339.

- 30. Horn HR (1983) Porcelain laminate veneers bonded to etched enamel *Dental Clinics of North America* **27(4)** 671-684.
- 31. Lu R, Harcourt JK, Tyas MJ & Alexander B (1992) An investigation of the composite resin/porcelain interface *Australian Dental Journal* **37(1)** 12-19.
- 32. Hayakawa T, Horie K, Aida M, Kanaya H, Kobayashi T & Murata Y (1992) The influence of surface conditions and silane agents on the bond of resin to dental porcelain *Dental Materials* 8(4) 238-240.
- Jardel V, Degrange M, Picard B & Derrien G (1999) Surface energy of etched ceramic International Journal of Prosthodontics 12(5) 415-418.
- 34. Barghi N (2000) To silanate or not to silanate: Making a clinical decision *Compendium of Continuing Education in Dentistry* **21(8)** 659-662.
- 35. Pape FW, Pfeiffer P & Marx R (1991) [Haftfestigkeit von geätztem] In-Ceram® and Zahnschmelz Zahnärztl Welt 100(7) 450-453.
- 36. Jedynakiewicz NM & Martin N (2001) The effect of surface coating on the bond strength of machinable ceramics *Biomaterials* **22(7)** 749-752.
- 37. Saygili G & Sahmali S (2003) Effect of ceramic surface treatment on the shear bond strengths of two resin luting agents to all-ceramic materials *Journal of Oral Rehabilitation* **30(7)** 758-764.