

# Effect of surface treatment and storage on the bond strength of different ceramic systems

Fernando Carlos Hueb de Menezes<sup>1</sup>, Gilberto Antônio Borges<sup>1</sup>, Thiago Assunção Valentino<sup>1</sup>,  
Maria Angélica Hueb de Menezes Oliveira<sup>1</sup>, Cecília Pedroso Turssi<sup>1</sup>, Lourenço Correr-Sobrinho<sup>2</sup>

<sup>1</sup>DDS, MSc, PhD, Associate Professor, Department of Dental Materials, University of Uberaba, Brazil

<sup>2</sup>DDS, MSc, PhD, Full Professor, Dental Materials Area, Piracicaba Dental School, State University of Campinas, Brazil

## Abstract

**Aim:** The aim of this study was to evaluate the micro shear bond strength of different ceramic systems - IPS Empress 2, Cergogold, In-Ceram Alumina and Cercon - and a dual luting agent. **Methods:** Twelve specimens of each ceramic were fabricated and divided according different surface treatments: Group 1: No additional treatment was applied to the ceramic surface; Group 2: Ceramics were etched with 9.5% hydrofluoric acid; Group 3: specimens treated with airborne particle abrasion for each ceramic system in accordance with manufacturer's instructions (n=20). The tests were performed after 24 h or after water storage for 6 months. Data were then assessed statistically using the 3-way ANOVA and the Tukey's test (P<0.05). **Results:** For Cergogold and IPS Empress 2 systems, the treatments performed with airborne particle abrasion and hydrofluoric acid showed no significant differences from each other, and both were superior to the groups without treatment. For Cercon and In-Ceram ceramics, no differences were found among the groups (P<0.05). When the surface was treated with hydrofluoric acid, the highest bond strength was found to IPS Empress 2 in the 6-month storage period (P<0.05). **Conclusion:** Lower bond strength values were only observed with IPS Empress 2 ceramic for the control group in the 6-month storage (P<0.05).

**Keywords:** ceramics, cementation, surface treatment, micro shear, bond strength.

## Introduction

Ceramic has been used in Dentistry as a restorative material since the 18th century. Their clinical use has oscillated throughout history, being widely used in some periods and almost abandoned in others<sup>1</sup>.

The association with a metallic substructure assured ceramic success, combining metal resistance with the excellent esthetics of the ceramic material<sup>2</sup>. However, Dentistry has always sought for eliminating the use of metal to improve esthetics and this esthetic demand has stimulated the research with new ceramic systems and mechanisms to increase their attachment to the luting agent and tooth structure<sup>3</sup>.

With the development of adhesive dentistry and improvement of the resins, the use of metal-free restorations has increased. In the recent past decades, the development of ceramic materials has increased significantly and their use has been more and more frequent. This material presents features, such as translucence, fluorescence, thermal-linear expansion coefficient close to dental structure, biological compatibility, chemical stability and compression and abrasion resistance. These properties enable it for being used as a substitute of natural tooth<sup>3</sup>.

The bonding between ceramics and dental structure is a relevant factor for the longevity of restorations and, depending on the ceramic material used, the cementation can be carried out by conventional or adhesive technique. Either glass ionomer or zinc phosphate cements can be used for the conventional luting, although adequate frictional area is necessary to provide retention<sup>4</sup>. However, when retentive areas are small or even absent, friction may be inadequate and a resin based luting agent is needed<sup>3</sup>. When using adhesive technique, both dental and

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### Correspondence to:

Fernando Carlos Hueb de Menezes,  
Departament of Dental Materials, Faculty of  
Dentistry of Uberaba, University of Uberaba,  
Av. Nené Sabino, 1801, Uberaba, MG,  
38055-500, Brazil.

Phone: +55-34-3319-8884.

E-mail: fernando.menezes@uniube.br

ceramic surfaces must be treated. Acid etching is performed on enamel and dentin, followed by hydrophilic adhesive application<sup>4</sup>. Polymerization of monomers at the demineralized regions provides a micromechanical bonding and the formation of the hybrid adhesive layer.

Likewise, inner surfaces of ceramic restorations must be susceptible to treatments that provide micromechanical retentions between the ceramic and the resin-based material. The frequently applied technique for feldspathic ceramic has been hydrofluoric acid etching, which provides irregular surface formation by removing the vitreous and crystalline phase<sup>5</sup>. Another pre bonding treatment for ceramic surfaces is airborne aluminum oxide particle abrasion and the airborne abrasion changes the microstructure of ceramic, similarly. In addition, the use of chemical substances such as silane, allows for a chemical bonding between the inorganic phase of ceramic and the organic phase of the resin material, since ceramic presents components that are susceptible to be bonded to silane<sup>5</sup>.

However, the surface of some ceramics is not likely to be modified by hydrofluoric acid etching. Thus, restorations with usual etching procedures and silanization, used for silicate-based ceramic, are not efficient for all types of ceramic materials<sup>6</sup>. The treatment using airborne particle abrasion and silane agent application has shown to be effective for ceramic restorations reinforced with aluminum and zirconia<sup>7-8</sup>.

The literature is controversial with relation to the type of treatment for the different metal-free ceramic systems<sup>3,7-9</sup> in order to obtain an effective and long-term bonding with the luting agents used. Furthermore, the hydrolytic degradation of adhesive systems should be considered. Therefore, the present study aimed to evaluate the bond strength of different ceramic systems, according to the types of surface treatments applied and the time of restoration storage. The null hypothesis was that the ceramic surface inner treatments and the time of storage are not influence the bond strength of the restorations.

## Material and methods

Compositions of the resin cement, the ceramic systems, and the porcelain primer are listed in table 1.

Twelve rectangular ceramic specimens were fabricated for each ceramic system in accordance with manufacturer's instructions, as follows: a) IPS Empress 2 (Ivoclar-Vivadent, Schaan, Liechtenstein): Wax patterns of 15 mm in length, 10 mm in width, and 1 mm in thickness were sprued and invested in IPS Empress 2 Speed investment. The wax was eliminated in a burnout furnace (700-5P; EDG Equipments Ltda, São Carlos, Brazil). Following, the investment, plunger, and 2 ingots of IPS Empress 2 (shade 300) were transferred to a furnace (EP 500; Ivoclar-Vivadent) and automatically pressed in accordance with manufacture's instructions. After cooling to room temperature, the specimens were divested with 50-mm glass beads at 2-bar pressure, ultrasonically cleaned in a special liquid (Invex liquid; Ivoclar-Vivadent), washed in running water, and dried. They were then treated with airborne particle abrasion with 100-mm aluminum oxide at 1-bar pressure. b) Cergogold (Degussa Dental, Hanau, Germany): Wax patterns 15 mm in length, 10 mm in width, and 1 mm in thickness were invested (Cergofit investment; Degussa Dental) and allowed to set. It was then placed in a burnout furnace to eliminate the wax. The Cergogold ingots (shade A3) were pressed in an automatic press

furnace (Cerampress Qex, Ney Dental Inc, Bloomfield, Conn.). After cooling, the specimens were divested using 50-mm glass beads at 4-bar pressure, followed by airborne particle abrasion with 100-mm aluminum oxide at 2-bar pressure, to remove the refractory material. The specimens were then treated with airborne abrasion with 100-mm aluminum oxide at 1-bar pressure. c) In-Ceram Alumina: (Vita Zahnfabrik, Seefeld, Germany) a model of stainless steel (30 x 20 x 5 mm) with a rectangular central depression (15 x 10 x 1 mm) was obtained. An impression of this model was made with polyvinyl siloxane, and then duplicated in a plaster (Special plaster; Vita Zahnfabrik). The aluminum oxide powder was mixed with a special liquid as instructed by the manufacturer. The slurry mixture was then painted into the depression in the special plaster die and fired at 1120° C in the furnace (Inceramat II; Vita Zahnfabrik) for 10 h. Glass infiltration was achieved by coating the aluminum oxide framework with glass powder (silicate-aluminum-lanthanum) mixed with distilled water, and fired for 4 h at 1100° C. The excess glass was removed by use of a fine-grained diamond (Renfert, Hilzingen, Germany) followed by airborne particle-abraded with 100-mm aluminum oxide at a pressure of 3-bar. d) Cercon (DeguDent): Wax patterns of 15 mm in length, 10 mm in width, and 1 mm in thickness were obtained. The wax model was placed in the Cercon brain unit for scanning. The confocal laser system measured the wax to an absolute precision of 10 mm and reproducibility of < 2 mm, scanning was accomplished in 4 min. A Cercon base blank of presintered zirconia was milled and then sintered to fully dense structure in the Cercon heat at 1350° C for 6 h. The specimens were finished by use of a fine-grained diamond (Renfert, Hilzingen, Germany) under refrigeration, followed by airborne particle-abrasion with 100-mm aluminum oxide at a pressure of 3-bar.

The tablets of ceramic system were ground with Al<sub>2</sub>O<sub>3</sub> sandpapers with decreasing granulation of 320, 400 and 600. They were then randomly divided into 3 groups, according to the surface treatments: Group 1: No additional treatment was applied to the ceramic surface after the treatment with Al<sub>2</sub>O<sub>3</sub> sandpapers; Group 2: Ceramics were etched with 9.5% hydrofluoric acid (Ultradent, South Jordan, Utah). The etching time protocol was considered for each ceramic type (20 seconds for IPS Empress 2, 60 seconds for Cergogold, and 2 min for In-Ceram Alumina and Cercon). After etching, ceramics were washed in tap water for 1 min and cleaned ultrasonically for 10 min; Group 3: specimens treated with airborne particle abrasion according to described for each ceramics systems previously (1-bar for IPS and Cergogold; 3-bar for In-Ceram and Cercon). The distance of the tip from the ceramic surface was approximately 4 mm. These specimens were washed under running tap water for 1 minute, ultrasonically cleaned in a water bath for 10 min, and air-dried.

For Panavia F (Kuraray Co., Osaka, Japan) (Table 1), the ceramic surface was first treated with a primer-acid mixture and silane agent (Clearfil Porcelain Bond activator, Kuraray Co., Osaka, Japan) for 20s and then the adhesive system was applied (ClearFil SEBond; Kuraray Co.), being light-cured for 20s. The same amount of the cement universal and catalyst pastes were mixed for 10 s. The cement mixed was used to fill the plastic microtubule (TYG-030, Small Parts Inc., Miami Lakes, FL) with inner diameter of 0.75 mm and 0.50 mm in height, which were bonded at ten different locations of the ceramic tablet surface (n=20) and light-cured for 40 s. Test specimens were left at room temperature (23° ± 2° C) for 1 h before the plastic microtubule removal. Later, half the specimens was stored in distilled water at 37° C during 24 h, and the other half stored in distilled water

at 37° C for 6 months.

After the storage, the test specimens were subjected to the microshearing bonding test. Before the test, all of the specimens were verified under optical microscope at a 20x magnitude in order to check for bonding interface. Microtubules showed interfacial opening formation; bubble inclusion or any other relevant defects were excluded from the study and replaced. The ceramic tablet was bonded to a metallic device treated with aluminum oxide sandblasting of 120 micrometers (especially developed) with cyanoacrylate-based adhesive (Superbond, Loctite, São Paulo, Brazil). This set was positioned into a test machine (EMIC DL3000, São José dos Pinhais, PR, Brazil) so that the microshearing test could be performed. A thin steel blade was gently placed in the interface ceramic/resin. The load was applied upon each test specimen at a speed of 0.5 mm/min until the failure occurred. The test interface, the blade and the load cell were gently aligned to assure the test force direction.

Additionally, after the micro shear bond strength was carried out, samples were examined with a scanning electron microscope (LEO 435 VP; Cambridge, England) at 100X magnification to assess the fracture pattern and at 1000X to obtain better visualization of the most characteristic regions of the fracture patterns. The bonding interface fractures were ranked according to the predominance of the surface observed as Mixed, Cohesive in the Resin Cement, Cohesive in the Ceramic, and Adhesive<sup>10</sup>.

Data were analyzed by a 3-way ANOVA to check significant effect of factors under study and their interactions. Tukey's test was applied to run the post-hoc comparisons. Significance level was set at  $\alpha=0.05$ .

## Results

ANOVA revealed interactions among ceramics x treatment ( $P<0.0001$ ), ceramics X storage ( $P=0.0120$ ), and treatment x storage ( $P=0.0156$ ) were observed, followed by Tukey's test (Table 2).

The Cergogold and IPS Empress 2 ceramics groups, that received either etched with hydrofluoric acid or airborne particle abrasion, presented higher bond strength values when compared to the control groups, for both immediate and after 6-month storage. With regard to Cercon and In-Ceram ceramics systems, for bond strength between surface treatments, no differences were found among the groups (Figure 1).

Considering the treatment groups, no differences were found between the control and treated with airborne particle abrasion groups. However, when the surface was treated with hydrofluoric acid, the highest bond strength was found for IPS Empress 2 in the 6-month

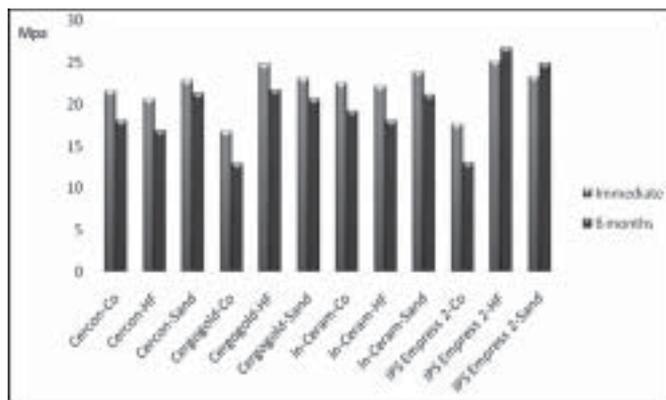


Fig 1. Shear bond strength (MPa) of ceramics in accordance with surface treatment and storage time.

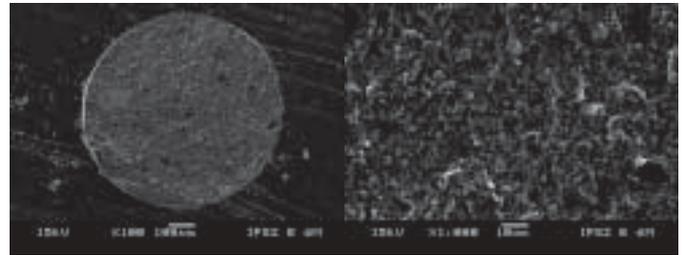


Fig 2. SEM of IPS Empress 2: Cohesive fracture pattern (arrow) (A- Original magnification X 100; B- Original magnification X 1000).

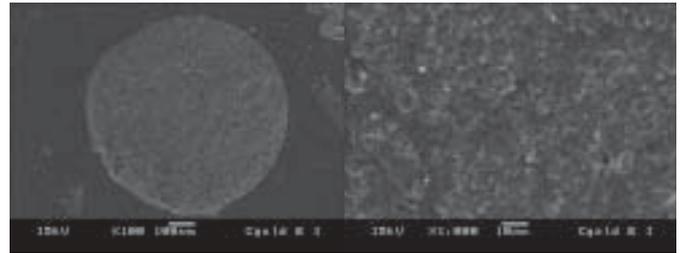


Fig 3. SEM of Cergogold: Cohesive fracture pattern (arrow) (A- Original magnification X 100; B- Original magnification X 1000).

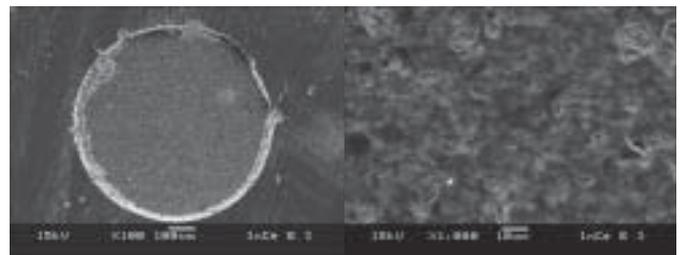


Fig 4. SEM of In-Ceram Alumina: Adhesive fracture pattern (arrow) (A- Original magnification X 100; B- Original magnification X 1000).

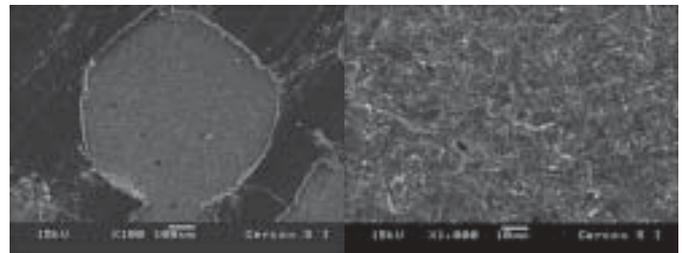


Fig 5. (a) SEM of Cercon: Adhesive fracture pattern (arrow) (A- Original magnification X 100; B- Original magnification X 1000).

storage period. The Cercon ceramic system surface treated with hydrofluoric acid showed lower bond strength values in comparison to the IPS Empress ceramic system for immediate storage group. For the 6-month storage period, decrease of the bond strength was only observed in IPS Empress 2 ceramic for the control group (Figure 1).

Predominance of the cohesive fracture pattern was observed in the SEM analysis of the bonding interface, i.e., rupture of the bonding interface between the resin cement and the ceramic systems IPS Empress 2 (68%) and Cergogold (73%) (Figs. 2A, 2B, 3A and 3B). For In-Ceram and Cercon, adhesive fracture pattern was foremost (75% and 78% respectively) (Figs. 4A, 4B, 5A and 5B). The SEM analysis also showed that the treatment with airborne particle abrasion and hydrofluoric acid modified the surface topography, increasing the irregularities of Cergogold and IPS Empress 2 ceramics.

## Discussion

The null hypothesis that both the surface treatments and storage time do not interfere in the ceramic bond strength was rejected. The results showed that bond strength can be modified, influenced not only by surface treatment, but also by storage time and composition of the ceramic used.

When a comparative study was carried out, it was observed that there are variables that can be used in the methodology in order to reach the objective formerly proposed in the investigation. Nevertheless, it is sometimes difficult to compare results obtained due to the lack of standardization of the techniques and materials used in the literature. Within the limits of the present study, the tested specimens were all treated and cemented by the same investigator in an attempt to standardize the procedures. Thus, considering that the methodology used to standardize the size of tested specimens is quite sensitive, results obtained can provide important information for the application of the materials studied here.

Micro shear bond strength test was used in this present study, through which the surface area is significantly reduced; hence, it leads to a safer and more accurate assessment of the bonding interface<sup>11</sup>. Although several investigations have used a myriad of bond strength methods, microshearing test has been found to be rather popular, providing satisfactory results<sup>12-13</sup>. It is believed that the tensions caused by the shearing test are important for the occurrence of restorative systems bonding failure<sup>14</sup>. In the present study, some bond strength values obtained with micro shear bond test showed to be comparable to the results obtained by Shimada et al.<sup>15</sup>, demonstrating coherence in the methodology used. Nevertheless, several difficulties were found, mainly during the insertion of the cement in the microtubules, as well as in controlling the overflow of the material on its base.

Bond failures between ceramics and resin cements may lead to premature loss of restorations. Considering this statement, several papers have been conducted in order to investigate the relationship between ceramic materials and composites<sup>16-17</sup>. The cementation technique is vital for the success of ceramic restorations, which depending on the clinical situation and the composition of the ceramic; it is possible to use cements that do not bond micromechanically to the ceramic restoration and to the tooth, such as zinc phosphate cement. However, if preparations without frictional retention are used, a closer relationship among cement, restoration, and dental structure is necessary. This relationship is provided by the formation of a hybrid layer between the resinous material and dental structure by means of an adhesive bond system<sup>7</sup>. On the other hand, the ceramic material also needs to have micro-retention and an excellent relationship with the cementation material<sup>16</sup>.

According to the results obtained in the present study, treatment with airborne particle abrasion with 50- $\mu$ m aluminum oxide caused morphological change that favored the material retention in IPS Empress 2 and Cergogold ceramic systems. Results are in accordance with those found by Kamada et al.<sup>18</sup>. Treatment with 50- $\mu$ m aluminum oxide airborne particle abrasion produced morphological conditions with surface aspect susceptible to mechanical retention through the formation of irregularities with uniform distribution in these ceramics systems<sup>8</sup>. However, for Cercon and In-ceram systems, the airborne particle abrasion altered the surface but did not increase the bond strength. These results disagree with a previous study that used the same treatment<sup>19</sup>.

The present investigation verified the efficiency of Panavia F bonding

agent in the adhesion of ceramics treated with airborne particle abrasion, which was already observed in a former study<sup>3</sup>. The hydrofluoric acid etching changed significantly the surface morphology of IPS Empress and Cergogold ceramics. This process can be explained by the preferential reaction of the hydrofluoric acid with the silica phase of the feldspathic ceramic to form hexa-fluorosilicates<sup>8</sup>. These silicates are removed by rinsing with water. The final result is a surface rich in irregularities for micromechanical retention<sup>18</sup>. However, for Cercon and In-ceram, the etching treatment did not interfere, probably due to the absence of glass phase (SiO<sub>2</sub>) in these systems, which did not influence the results of bond strength, as demonstrated by Borges et al.<sup>19</sup>. Although there are similarities between the results of bond strength, it is also important to observe the fracture pattern occurred. For Cercon and In-ceram, the pattern of fracture was predominantly adhesive, which features more weakness at the interface. As for the IPS and Cergogold, the predominant pattern was cohesive in the cement, suggesting a greater bonding strength at the interface and greater weakness in the bulk of the material.

Storage time can also be considered an influence factor in adhesive restorations bond strength. Recent publications showed that the bonding interface degradation is an ordinary phenomenon when the clinician uses composite materials<sup>20-21</sup>. However, in the present study, lower bond strength values were observed only for IPS Empress 2 without treatment, after 6 months of storage. It could be suggested that the interface degradation during the storage is more intense with less surface area interaction between the luting agent and the ceramic<sup>19</sup>.

Due to the different ceramics available in the market, as well as to the different luting agents and surface treatments, the present paper aimed to reach the best relationship among these materials, minimizing failures of the restorative system. Former and traditional procedures in cementation techniques have been questioned with the availability of state-of-the-art adhesive products, which provide promising perspectives, regarding that the professional has basic knowledge about them<sup>22</sup>.

Within the limitations of the present investigation, it may be concluded that the bond strength of Cergogold and IPS Empress 2 ceramics was superior when the systems were treated with airborne particle abrasion with 50- $\mu$ m aluminum oxide or etched with hydrofluoric acid. However these treatments did not influence in the bond strength of In-Ceram and Cercon systems. Storage for 6 months only interfered on the IPS Empress 2 when this system was tested without treatment.

The literature is controversial regarding to the durability of the ceramic/resin restorative system clinically. The surface treatment of ceramics is dependent on their composition and dictates the relationship between the ceramic and the cement system. Therefore, the knowledge of the ceramic material composition is vital for the correct application of the ideal surface treatment and obtains an appropriate adhesive cementation to achieve a better longevity.

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