Journal section: Clinical and Experimental Dentistry *doi:10.4317/medoral.15.e95*
Publication Types: Research

In vitro experimental study of bonding between aluminium oxide ceramics and resin cements

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Received: 12/05/2009
Accepted: 02/08/2009

Román-Rodríguez JL, Roig-Vanaclocha A, Fons-Font A, Granell-Ruiz M, Solá-Ruiz MF, Amigó-Borrás V, Busquets-Mataix D, Vicente-Escuder A. In vitro experimental study of bonding between aluminium oxide ceramics and resin cements. Med Oral Patol Oral Cir Bucal. 2010 Jan 1;15 (1):e95-100.
http://www.medicinaoral.com/medoralfree01/v15i1/medoralv15i1p95.pdf

Article Number: 2895 http://www.medicinaoral.com/
© Medicina Oral S. L. C.I.F. B 96689336 - pISSN 1698-4447 - eISSN: 1698-6946
eMail: medicina@medicinaoral.com
Indexed in:
-SCI EXPANDED
-JOURNAL CITATION REPORTS
-Index Medicus / MEDLINE / PubMed
-EMBASE, Excerpta Medica
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-Indice Médico Español

Abstract
To evaluate bond strengths of different resin cements to two aluminum oxide-based ceramics. Methods: One hundred ten ceramic cylinders were produced and given four different surface treatments. Resin cement cylinders were then bonded to the ceramic cylinders using different resin cements and the bond strength was determined by shear testing to the breaking point. We were thus able to obtain results for the different combinations of porcelain, surface treatments and cements. All data was analyzed using the Kruskal-Wallis test for more than two independent samples and the Bonferroni correction applied (a=0.01). An optical microscopy study was carried out to analyze the type of failure, and an electronic microscopy examination was carried out in order to evaluate the changes produced in the ceramic by the different surface treatments. Results: The best values corresponded to the control group, composed of silicate ceramics combined with Variolink II resin cement. In-Ceram Alumina showed no significant differences with respect to the type of cement applied. Procera AllCeram obtained the best values when silica coated using the CoJet System and applying Variolink II, or when sandblasted and applying Clearfil SE Bond + Porcelain Bond Activator and Panavia F cement. Significance: Surface treatment modifies the ceramic surface and influences the bond strength, as does the type of cement used. Silica coating is recommended to improve adhesion to Procera AllCeram, applying Variolink II, or sandblasting plus resin cement containing MDP (Panavia F).

Key words: *Bond strength, silica coating, Procera AllCeram, In Ceram Alumina, Empress ceramic.*

Introduction

A variety of materials can be used to cement these restorations to the tooth, although maximum bond strength between tooth and ceramic is currently obtained with resin cements (1-3). Said adhesion improves the marginal sealing of the restoration, thus minimizing microleakage that may cause secondary caries or changes in color of the restoration by staining the abutment. It also avoids or delays marginal staining (4). Likewise, this adhesion is crucial to avoid debonding in cases of non-retentive abutments. Furthermore, in the case of porcelain laminate veneers, reliable adhesion is essential since these restorations have no frictional component and only remain fixed in the mouth through the adhesion obtained. Finally, it has been concluded that this adhesion improves the fracture strength of both the ceramic restorations and the tooth itself (5). Therefore, although cementing with resins is complex and takes longer, their use is recommended to obtain the best possible bond strengths (4).

Traditionally, silicate ceramics are etched with hydrofluoric acid to create microporosities on the internal surface of the ceramic, followed be silane application, obtaining a very reliable micromechanical and chemical bond (6-8). It has been described that this bond is stronger than that of the resin cement and tooth interface (9).

However, HF etching is not the most suitable treatment for oxide ceramics as these contain practically no silica (4,8,10). Therefore, mechanical techniques such as sandblasting with aluminum oxide particles are used to create microporosities that trap the cement and improve retention (2,4).

The objectives were to study by shear testing the bond strength between two oxide ceramics and different composite cements, using different porcelain surface treatments. These results are compared with those obtained with proven silicate ceramic and resin cement.

Likewise, optical and electron microscopy studies were carried out, to describe the effects of the different surface treatments on the ceramic itself.

Materials and Methods

One hundred ten porcelain cylinders were made for the study: 10 IPS-Empress II® (Ivoclar Vivadent, Schann, Liechtenstein) (lithium-disilicate glass-ceramic as control group), 20 In-Ceram Alumina® ceramic cylinders (Vita Zahnfabrick, Bad Säckingen, Germany) (lanthanum-glass-infiltrated 85% aluminum-oxide ceramic) and 80 Procera AllCeram ceramic cylinders (99% aluminum-oxide ceramic).

Depending on the ceramic, the various sample groups received four different surface treatments.

The IPS-Empress II silicate ceramic samples were etched with hydrofluoric acid (Porcelain Etch Ultradent®, Salt Lake City, USA) at 9.5% for two minutes. The In-Ceram Alumina oxide ceramic samples were sandblasted with 80 µm aluminum oxide particles, at a pressure of 3 bars for 10 seconds. The Procera AllCeram oxide ceramic samples were either sandblasted with 80 µm aluminum oxide particles or silica coated using the CoJet System® (3M Espe, St. Paul, Minn., USA), as shown in Table 1, including one group that received both surface treatments (Group 11 ASCVA). All samples except Group 6 (ASNOSILPA) were silanated, those treated with the CoJet System were silanated for 5 minutes according to the manufacturer's recommendations with the silane included with the system (3-methacryloxypropyl-trimethoxysilane) (Espe- Sil®; 3M Espe), the remainder were silanated for one minute with Monobond S (Ivoclar®).

To complete the study, 110 composite cement cylinders were made, 50 Variolink II (Ivoclar), 40 Panavia F® (Kuraray Co, Osaka, Japan), 10 Rely X Unicem (3M Espe) and 10 Multilink® (Ivoclar).

A resin cement cylinder was bonded to each of the previously-treated ceramic cylinders using the corresponding adhesive and resin cement or each group, polymerizing the cement or placing Vaseline insulation according to each case.

Thus eleven test groups comprising ten samples in each group in order to obtain reliable statistical data were formed (Table 1).

The samples were maintained at 37º C in a humid environment for 24 hours in a J.P Selecta model 210 stove. The shear test was carried out using an Instron 4804 universal test machine, with a load of 1 kN and a of crosshead speed of 0.5 mm/min, applying the load 0.5 mm from the bond.

The results were analyzed using the Kruskall-Wallis test for more than two independent samples. Given the large number of combinations a Bonferroni correction was applied (α=0.01).

The fracture surfaces were examined using a Nikon® SMZ-10A Stereozoom microscope to evaluate exactly where the ceramic-cement failure occurred, and a Jeol® JSM 6300 scanning electron microscope (SEM) with an EDX microanalysis system (Oxford Instruments). In addition, transmission electron microscope (TEM) images were obtained with a Philips CM10 instrument at 100 Kv/Kw in samples prepared on a Fischione Ion mill.

Results

The best bond strength values for the test corresponded to the control group, that is, the combination of IPS Empress II silicate ceramic and Variolink II resin cement. In contrast, the worst values were found for the sandblasted and silanated Procera AllCeram together with Panavia F. This same combination, but without silane, obtained notably improved results with bond strength

Table 1. Test group specifications.

Group	Ceramic	Surface treatment	Silane	Adhesive	Cement
1.EHVA	IPS-Em-press II	HF	Yes	Excite	Variolink II
2.ISVA	In-Ceram Alumina	Sandblasting	Yes	Excite	Variolink II
3.ISPA	In-Ceram Alumina	Sandblasting	Yes	Primer A+B	Panavia F
4.ASVA	AllCeram	Sandblasting	Yes	Excite	Variolink II
5.ASPA	AllCeram	Sandblasting	Yes	Primer A+B	Panavia F
6.ASNOSILPA	AllCeram	Sandblasting	No	Clearfil SE Bond+Porcelain Activator	Panavia F
7.ACVA	AllCeram	Cojet Sand	Yes	Excite	Variolink II
8.ACPA	AllCeram	Cojet Sand	Yes	Primer A+B	Panavia F
9.ACRE	AllCeram	Cojet Sand	Yes	No	Rely X Unicem
10.ACMU	AllCeram	Cojet Sand	Yes	No	Multilink
11.ASCVA	AllCeram	Sandblasting + Ultra-sound bathing in 96% alcohol + Cojet Sand	Yes	Excite	Variolink II

Table 2. Combinations joined with continuous vertical shade are not statistically different. Rankings are from lower (top) to highest (bottom) mean shear bond strength.

Group	Mean (MPa)	SD (MPa)	Groupings
ASPA	3.28	1.82	
ASCVA	6.59	2.72	
ACMU	6.65	2.35	
ISPA	8.64	1.76	
ASVA	8.81	2.25	
ACPA	9.08	2.15	
ACRE	10.07	3.56	
ISVA	10.18	3.01	
ASNOSILPA	12.12	3.31	
ACVA	13.72	3.51	
EHVA	15.27	4.00	

values from 3.28 to 12.12 MPa. However, the best combination for Procera AllCeram was obtained using silica coating and Variolink II. There were no statistically significant differences between groups 2, 3, 4, 8, and 9; although there were significant differences between these groups and the control group.

Regarding oxide ceramic surface treatment, statistically significant differences were found in favor of the CoJet System, except with group 6 ASNOSILPA (Table 2). The results are shown according to the scheme of Blatz et al. (11).

3.2 Optical microscopy:

An optical microscopy examination was made of the samples after shear testing. All the IPS-Empress II samples suffered cohesive failure, while all failures in the oxide ceramic were adhesive.

3.3 Electronic microscopy:

In a polished state the IPS-Empress II sample reveals a very regular homogeneous surface. Once etched with HFA at 9.5% a quite rough, uneven, highly porous surface appears, created by elimination of the glassy matrix while conserving the crystalline structure (Fig. 1).

The Procera AllCeram presents a wholly contiguous crystalline structure formed of polygonal aluminum oxide crystals between 2 and 5 microns in size. Once silica coated the surface appears eroded with notable silica deposits (Figs. 2 and 3), and confirmed by subsequent spectrometric analysis. A spectrometric analysis revealed the silica deposits on the surface of the silica-coated Procera AllCeram oxide ceramics.

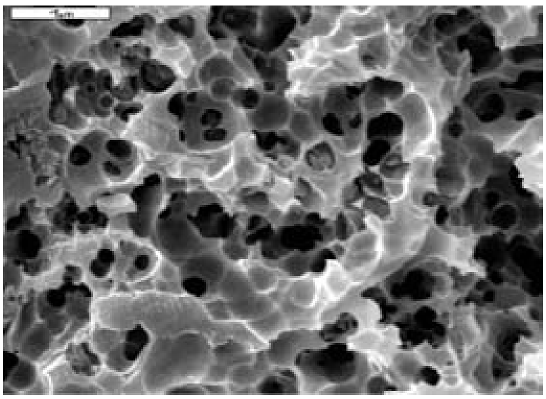

Fig. 1. IPS-Empress II sample etching with HFA at 5000X (SEM) showing the numerous cracks, edges, cavities and microporosities.

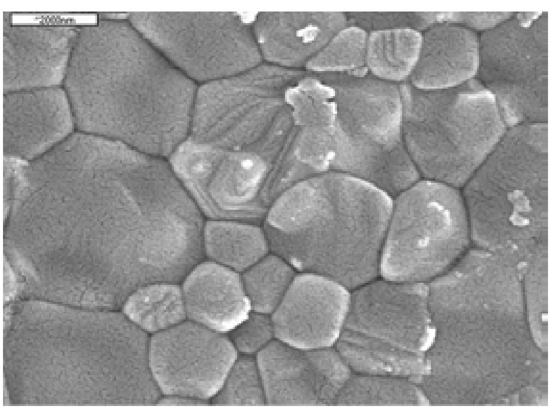

Fig. 2. AllCeram sample untreated at 10000X (SEM), there is no glassy matrix between the polygonal aluminum oxide crystals.

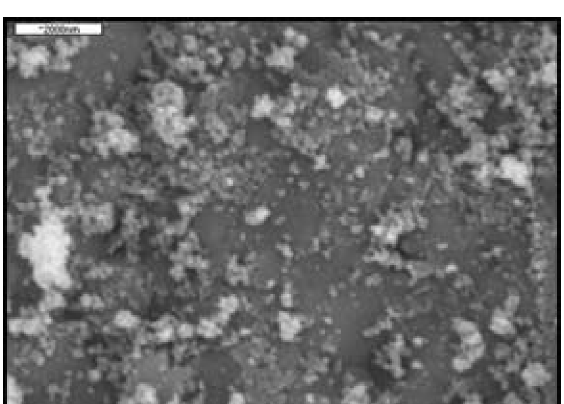

Fig. 3. Once silica coated a roughened surface appears with notable silica deposition at 10000X (SEM).

Discussion

Conventional hydrofluoric acid etching of silicate ceramic surfaces attacks the silica, creating a sufficiently microretentive surface for interlocking with the cement. The acid selectively eliminates the glassy matrix, exposing the leucite crystals. This elimination of ceramic material, in spite of the appearance in Figure 1, is minimal, and does not affect the fit of the restoration (12); it visibly increases the contact area on passing from a practically smooth surface to a three-dimensional one, and notably improves the bond strength between the two materials.

Since oxide ceramics contain very little or even no silica, there is no sense in acid etching (1). Hence, physical treatment of the internal ceramic surface is necessary, with the aim of transforming this surface from a relatively smooth to a rough or micro-retentive texture, thus increasing the ceramic-cement contact area and allowing incorporation of the resin into the microporosities in the ceramic before curing, so creating a micromechanical bond after polymerization of the cement. This mechanical treatment can be made by sandblasting or by silica coating, both of which favor the subsequent chemical bond with the cement (6). In addition to physical treatment of oxide ceramics, a chemical treatment is also recommended. On this key point two tendencies exist at the present time.

On one hand, some authors defend the use of a phosphate monomer adhesive (10-metacriloiloxidecil dihydrogen phosphate) (MDP), since this possesses a chemical affinity for metallic oxides.

On the other hand, diverse investigations (13), have also obtained good results with silanization of the ceramic before applying the adhesive and/or resin cement.

The detractors of silane for oxide ceramics explain that since there is practically no silica in the composition (present in In-Ceram Alumina and absent in Procera AllCeram) said silane would not react with the ceramic (it bonds with the active crystalline portions through oxygen bridges and atomic Si-O-Si bonds). However, those authors who do apply silane state that this increases the wettability of the porcelain by reducing the surface tension, facilitating a better coverage of the adhesive and cement (3,6).

If silica coating were carried out instead of sandblasting, with the consequent silica deposition, the application of silane would be justified independently of the ceramic composition and would explain the increase in bonding strength of the silica coating process against conventional sandblasting and silanization of the ceramic.

From our results we observe that Group 1 EHVA (IPS-Empress II and Variolink II cement) (the control group) achieved the highest bond strengths in the test. This was as expected, since the resin cement to silicate ceramic bond is widely endorsed by studies in vitro and clinicians

(14). We should also highlight that this group suffered a cohesive failure of the ceramic, and we can therefore suppose that the bond strength was in fact higher than the vertical loads applied to the ceramic. Results that coincide with those of diverse authors (7,14).

None of the ceramic oxide groups achieved similar values to those obtained with this bond, except group 7 ACVA, composed of silica-coated Procera AllCeram and a Variolink II cement cylinder, where a value approaching that of group 1 was found. In any case, there were no statistically significant differences between group 7 and group 6 (sandblasting without silane application + Panavia F), thus endorsing this last combination as the most valid for treating this porcelain when not silica-coating the ceramic. It seems that the combination of conventional sandblasting and the application of Clearfil SE Bond and Clearfil Porcelain Activator prior to Panavia F propitiates some very reliable, durable adhesion values (with tests with thermocycling) comparable to those obtained with silica coating (3,15). However, if silane is added to this combination, and normal Panavia adhesive (Primer A+B) is used, the ceramic-cement bonding mechanism is visibly hindered. Thus, Group 5 (sandblasting Procera AllCeram + silane + Panavia F) obtained the poorest results in the whole study, which seems to advise against its use, especially with regard to restorations retained by adhesion only, without friction, such as laminated porcelain veneers for example.

Group 11 with a combination of sandblasting and silica coating (sandblasting + ultrasound bathing in alcohol at 96% to clean the surface + silica coating) showed no improvement in bond strength.

With regard to the cements studied, statistically significant differences exist between the results of Variolink II and Panavia F. However, the differences disappear if the adhesive for Panavia F is modified, changing Primer A+B for Clearfil SE Bond + Porcelain Activator, we therefore consider this to be the combination of choice when using Panavia F to cement Procera AllCeram restorations.

With regard to oxide ceramic surface treatment, the silica coated groups (Groups 7 and 8) presented statistically significant improvement with respect to conventional sandblasting (Groups 4 and 5), which would seems to imply that silica coating improves adhesion. It is also true however, that no statistically significant differences were found between Group 6 and Group 7, probably because although the micromechanical bond is inferior, the chemical bond between ceramic and the Panavia F cement is notably improved by this combination.

When consulting other similar studies we observe a great variability between results in the shear tests, depending on the method used. Hence, we do not compare MPa figures obtained by different authors, but rather we prefer to explore tendencies in the behavior of the materials studied. Several authors (1,6,16,17) demonstrate that silica coating always improves adhesion by creating a homogeneous microretentive surface, containing a degree of silica, Figure 3, providing both a mechanical and chemical retention which would explain this increase in the oxide-ceramic to resin-cement bond strength. In all these studies, the Rocatec (particles of 110 µm) or the CoJet System (30 µm) were used for silica coating the oxide ceramics (6,17,18). However, as Hummel and Kern (2) explain, although the resulting bond strength after silica-coating is initially acceptable, after 150 days of storage and thermocycling the bond strength diminished considerably. Although studies also exist in vitro in which, after 180 days of storage and subsequent thermocycling, the combination of silica coating and resin cement is recommended as a valid and effective treatment (19).

When evaluating cements, the references consulted (1,2) reflect that of all the cements tested for Procera AllCeram, the cements of choice are Panavia F and Variolink II.

Regarding the macroscopic and optical microscopy examination, two clearly distinct behaviors were observed. As mentioned above, the IPS Empress II silicate ceramic combined with Variolink II suffered a cohesive fracture of the porcelain in 100% of cases, because the bond was stronger than the ceramic itself. The silicate ceramic to resin cement bond is stronger than the bond between cement and tooth (9). The tooth-cement interface should be considered as the weakest point of connection in porcelain restorations cemented to teeth, and to be biologically more important than the crown-cement interface (9,16). It would be ideal to achieve this same reliability for the oxide ceramics, however adhesive failures were observed in all the oxide ceramics samples of this study. Thus, we corroborate the adhesive failure of the oxide ceramic samples found by Blatz et al. in their test with Procera AllCeram (12). These data can be explained by the fact that oxide ceramics have a high flexion resistance (between 400 and 640 MPa)(3) and the bond strength values obtained with these were lower than for the silicate ceramic.

Using electronic microscopy the treated and untreated ceramic samples were compared. Thus, in the IPS-Empress II sample, it was seen that the hydrofluoric acid etching at 9.5% for two minutes converts a smooth, polished surface to one with a highly retentive morphology by the selective etching of the vitreous matrix. This is achieved by the dissolving of the glassy matrix which leaves behind a roughened crystallite surface, although excessive loss of the glassy matrix weakens the retentive capacity.

Examination of the In-Ceram Alumina ceramic reveals the basic structure of aluminum oxide crystals within a lanthanum oxide matrix, that is modified by the sandblasting showing a greater roughness (2) although not comparable with silicate ceramic etching.

Procera AllCeram without surface treatment is not at all retentive as it lacks a vitreous phase and because the crystals are so well organized that no gaps remain, see Figure 2. Once the ceramic is silica coated with CoJet Sand, the resulting surface presents a roughened texture formed by surface abrasion and the deposition of silica-coated particles (6). This creates the microretentive texture, facilitating the micromechanical bond. The chemical bond is established by the interaction of the silane with the silica present on the ceramic surface, as demonstrated by the spectrometric analysis. This physical-chemical bond is responsible for the increase in the adhesion values with respect to the sandblasted samples (20).

Conclusion

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

1. All the ceramics studied, after receiving the corresponding surface treatment, undergo a surface modification, increasing the contact area and therefore creating a more retentive morphology, and providing a better mechanical interdigitation with the cement.
2. Ceramic IPS-Empress II etched with hydrofluoric acid and cemented with Variolink II composite cement achieved the best results in the test, this combination being the current gold standard for bonding silicate ceramics.
3. The best bond strength values for the Procera AllCeram oxide ceramics were obtained with silica coating and the application of Variolink II, or with the combination of conventional sandblasting and the application of Clearfil SE Bond + Porcelain Activator and Panavia F cement.
4. The worst results in this study came from the combination of conventional sandblasting and the application of Panavia F.
5. The Variolink II and Panavia F cements, both dual polymerizing, showed better bond strength values than the autopolymerizing cements (Rely X and Multilink). With respect to the autopolymerizing cements used for Procera AllCeram ceramic, Rely X Unicem obtained better results than Multilink.
6. The In Ceram Alumina oxide ceramics prepared with conventional sandblasting obtained similar results with both the Variolink II and the Panavia F cements, therefore both can be recommended for cementing this ceramic. For full coverage restorations we consider the bond strength values obtained in our study to be sufficient.

Finally, as this is an in vitro study, we would like emphasize that these recommendations should be take with caution, and that the results cannot be extrapolated directly to the clinical environment.

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Acknowledgments:
The authors wish to acknowledge the invaluable help of Dr. Amparo Martínez González in the study design and final correction of the original text.