Clinical and laboratory evaluation of CAD/CAM All-ceramic crowns
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Citation for published version (APA):
Begazo, C. M. C. (2004). Clinical and laboratory evaluation of CAD/CAM All-ceramic crowns

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CHAPTER 4

Shear Bond Strength of Different Type of Luting Cements to an Aluminum Oxide Containing Glass Ceramic Core Material

Abstract

Purpose: The aim of this study was to find the optimal choice of luting cement to Synthoceram, an aluminum oxide reinforced glass ceramic material. The bond strength of five different commercially luting cements to the ceramic material was evaluated. The effect of surface treatments, etching, sandblasting, silanizing, and combinations of these treatments was also investigated.

Materials and Methods: Shear bond strength tests were performed using the ceramic material as substrate with each of the luting cements. Cement rods were prepared on pre-treated ceramic surfaces. The shear bond strength was determined 24 h after cementation. The effect of surface treatments: etching, sandblasting, and sandblasting followed by etching respectively, on the morphology of the material was investigated with SEM.

Results: The shear bond strength increases significantly from Ketac Cem, Rely X Luting, Fuji Plus, Panavia F to Xeno Cem. The surface treatments etching and/or sandblasting followed by silanization generally provides the highest bond strength values.

Conclusion: Based on the results of this study the use of resin composite based cements is preferred for cementation of an all-ceramic restoration with an aluminum oxide reinforced glass ceramic base. As surface treatment etching and/or sandblasting followed by silanization is recommended.
**Introduction**

All-ceramic restorations show excellent aesthetic qualities and a good biocompatibility. For these reasons an increase of the use of all-ceramic restorations by dentists is recognized, even for the stress bearing regions of the mouth. However, even with introduction of stronger modern ceramic systems, the dentist is still facing problems related to fracture of these restorations. Amongst others, strength of the ceramic materials is one of the parameters that determine the longevity of an all-ceramic restoration\(^1\). The preparation, the way of production of the restoration, as well as the composition of the ceramic material, and the cement are of influence on the durability of the restoration\(^2\).

In search of high strength ceramic materials 'new' production methods are used as casting, pressing, and milling of ceramic caps. Automatic production methods may exclude the variance in strength related to manual manipulation of ceramic materials. CAD/CAM technology is an example of a method for making dental ceramic restorations without manual interference. Cerec, and Procera are the most used CAD/CAM systems\(^3\). Also layered ceramic restorations can be fabricated by CAD/CAM\(^3,4\). Here a coping is made of an aluminum oxide reinforced glass ceramic to improve the overall strength of the crown. Furthermore, two layers of different veneering glass ceramics are applied for an aesthetic appearance. Synthoceram is a high content aluminum oxide reinforced glass ceramic, which can be used as coping in these latter CAD/CAM crowns.

Even with the application of automated ways of production the cement can be the 'Achilles heel' of an all-ceramic restoration\(^5\). For brittle materials, as ceramics are, the integrity and longevity of the tooth-cement-ceramic interface is of main importance for the risk of fracture of the restoration. The adhesive capacity and the cement stiffness are important material properties for the longevity of all-ceramic restorations. Conventional glass ionomer cements do show adhesive properties to metals and have a relative high stiffness\(^6\). For that reason glass-ionomer luting cements are common used to cement metal crowns. Also for some all-ceramic restorations glass-ionomer cements are advised as luting cement. However, resin composite cements are preferred as luting material for all-ceramic restorations due to their bonding ability to ceramics\(^7\). For the latter cements the ceramic surface has to be treated and/or conditioned, while for glass ionomer-based cements this treatment
or conditioning may not be required. For many cases hydrofluoric acid-etching followed by silane application resulted in the most durable bond with the highest bond strength\textsuperscript{8-16}.

The aim of this study was to find the optimal luting cement for a high content aluminum oxide reinforced glass ceramic, Synthoceram. Different luting cements and surface treatments were evaluated by means of their shear bond strength to the aluminum oxide reinforced glass ceramic material.

**Materials and Methods**

In this study shear bond strength of five different luting cements to ceramic discs made of Synthoceram was evaluated (See Table 1). Synthoceram consists of aluminum oxide (66%), siliciumoxide (20%) and other materials (14%). Hundred twenty-eight ceramic discs \((d=20\ \text{mm},\ h=3\ \text{mm})\) were fabricated according manufactures procedures. The ceramic discs were sanded in a mould with a constant load of 5 N on a rotating disk with silicon carbide paper of 320 grit (2 min), followed by silicon carbide paper of 600 grit (2 min) and finally polished with silicon carbide paper of 1200 grit (2 min) to obtain a standard surface roughness. The samples were polished up to 1200 grit to obtain a smooth surface. Bonding to a smooth surface and sandblasted or etched surface, respectively, reveals information about chemical bonding or micromechanical interlocking to the surface. The disks were cleaned with tap water and dried before further treatment. The ceramic discs were divided in eight groups that receive different surface treatments (see Figure 1).

Group 1 received no further surface treatment.
Group 2 was etched for 150 s with 9.5% hydrofluoric acid.
Group 3 was silanized by a mixture of the bonding agent in Clearfil liner bond 2V and Clearfil porcelain bond activator.
Group 4 was etched as group 2 and silanized as Group 3.
Group 5 was sandblasted for 20 s with aluminum oxide (50\mu m) at 200 kPa air pressure.
Group 6 was sandblasted as group 5 and etched as group 2.
Group 7 was sandblasted as group 5 and silanized as group 3.
Group 8 was sandblasted as group 5, etched as group 2 and silanized as group 3.
One of the five cements (see Table 1) was tested, giving sixteen samples per method. Before re-using the samples the sanding procedure was repeated and damaged samples were substituted.

On the treated ceramic surface a cylindrically shaped split-mold (d = 4.0 mm, h = 4.0 mm) was placed. The mold was filled with the luting cement and cured according the manufacturers instructions. The dual-cure cements were light cured with an Elipar II light curing device (3M-ESPE, Seefeld, G). After setting of the luting cements, the split-mold was carefully removed leaving an adhesively bonded cylinder of cement on the ceramic surface. Before testing the sample were stored 24 hours in 100% humidity at room temperature.

![Figure 1. Surface treatment procedures of the eight groups.](image)
Table 1. Materials used.

<table>
<thead>
<tr>
<th>Material</th>
<th>Type</th>
<th>Main Composition</th>
<th>Manufacturer</th>
<th>Batch</th>
</tr>
</thead>
<tbody>
<tr>
<td>Synthocera m</td>
<td>Aluminum oxide</td>
<td></td>
<td>Elephant Dental B.V., Hoorn, the Netherlands</td>
<td>A-873 &amp; A-1071</td>
</tr>
<tr>
<td>Clearfil liner bond 2v &amp;</td>
<td>Adhesive &amp;</td>
<td></td>
<td>Kuraray Medical, Tokyo, Japan</td>
<td>00055A</td>
</tr>
<tr>
<td>Clearfil porcelain bond</td>
<td>Silanizing Agent</td>
<td></td>
<td></td>
<td>41123</td>
</tr>
<tr>
<td>activator</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Panavia F</td>
<td>Dual-cure resin composite cement</td>
<td>Silanated barium glass and silica powder, bis-phenol A polyethoxy dimethacrylate, 10-methacryloyldecyl dihydrogen phosphate (MDP) and photochemical and chemical initiators.¹</td>
<td>Kuraray Medical, Tokyo, Japan</td>
<td>046AA</td>
</tr>
<tr>
<td>Xeno Cem</td>
<td>Dual-cure resin composite cement</td>
<td>Fluoraluminosilicate glass, UDMA, HEMA, polyacrylic acid and photochemical and chemical initiators.²</td>
<td>Dentsply/Sankin, Tokyo, Japan</td>
<td>334-001</td>
</tr>
<tr>
<td>Rely X Luting</td>
<td>Glass-lonomer cement</td>
<td>Fluoraluminosilicate glass, polyalkenoic acid and HEMA, chemical initiators.²</td>
<td>3M-ESPE, Seefeld, Germany</td>
<td>20000425</td>
</tr>
<tr>
<td>Fuji PLUS</td>
<td>Glass-lonomer cement</td>
<td>Maleic acid, HEMA, tartaric acid, water, chemical initiators.¹</td>
<td>GC, Tokyo, Japan 9910261</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Conventional</td>
<td>Fluoraluminosilicate glass, polycarboxylic acid, tartaric acid, water.²</td>
<td></td>
<td></td>
</tr>
<tr>
<td>KetacCem</td>
<td>Glass-lonomer cement</td>
<td></td>
<td>3M-ESPE, FW Seefeld, Germany</td>
<td>063589</td>
</tr>
</tbody>
</table>

¹ from ref [25] ² manufacturers date.

The specimens thus obtained were mounted in a universal testing machine (Instron Ltd. Wycombe, UK), and shear stress was applied at a crosshead speed of 0.2 mm min⁻¹. Shear load at failure was recorded and converted to strength. The mode of failure was evaluated by using light microscopy (10x magnification). The treated ceramic surfaces were examined by scanning electron microscopy (Phillips SEM XL 20, Eindhoven, NL).
Shear bond strength of different type of luting cements

SPSS 10.0 (SPSS inc., Chicago, USA) was used as to carry out the statistical analysis. One-way analysis of variance (ANOVA) and Tukey HSD tests was used to determine differences in shear bond strength values between the groups and cements. A p-value<0.05 was considered significant.

Results

The shear bond strength values are presented in Table 2 and graphically depicted in Figure 2. The observed shear bond strength ranged from 0 to ca. 21 MPa. For all groups with a shear bond strength higher than 19 MPa cohesive fractures in the ceramic was observed.

Resin composite cements (Panavia F and Xeno Cem) showed that all extra surface treatments did result in a significant increase of shear bond strength value. For both cements, sandblasting only resulted in significant lower shear bond strength values with respect to the other treatments. The highest shear bond strength of Panavia F was observed for the treatments etching, etching and silanizing, sandblasting and etching, sandblasting and silanizing, and sandblasting, etching and silanizing. With Xeno Cem the significant highest bond strength was found with treatments sandblasting and etching, sandblasting and silanizing, and sandblasting, etching and silanizing.

Resin modified glass ionomer cements (Rely X Luting and Fuji Plus): For the resin modified glass ionomer cements all extra surface treatments did result in similarly in an increase of the shear bond strength compared to no treatment (see Figure 2). For Rely X Luting the highest values were found with a surface treatments silanizing, and sandblasting followed by etching. For Fuji Plus the surface treatment of sandblasting or etching resulted lower shear bond strength values compared to the other treatments.
Table 2. The mean shear bond strength (MPa) (n=16) with the standard deviation in parentheses, of different luting cements depending of ceramic treatment.

<table>
<thead>
<tr>
<th>Luting Cement</th>
<th>Group 1</th>
<th>Group 2</th>
<th>Group 3</th>
<th>Group 4</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>NT</td>
<td>E</td>
<td>Si</td>
<td>E &amp; Si</td>
</tr>
<tr>
<td>Panavia F</td>
<td>3.9 (2.4)\textsuperscript{aB}</td>
<td>12.5 (5.6)\textsuperscript{bC}</td>
<td>9.7 (3.4)\textsuperscript{bA}</td>
<td>13.0 (4.7)\textsuperscript{bcAB}</td>
</tr>
<tr>
<td>Xeno Cem</td>
<td>4.7 (2.0)\textsuperscript{aA}</td>
<td>12.1 (3.3)\textsuperscript{bA}</td>
<td>11.3 (3.4)\textsuperscript{cA}</td>
<td>15.5 (2.8)\textsuperscript{cDA}</td>
</tr>
<tr>
<td>Rely X Luting</td>
<td>0.4 (0.5)\textsuperscript{aC}</td>
<td>4.8 (1.3)\textsuperscript{bC}</td>
<td>5.0 (1.9)\textsuperscript{bcB}</td>
<td>3.8 (1.8)\textsuperscript{bcC}</td>
</tr>
<tr>
<td>Fuji Plus</td>
<td>2.9 (1.2)\textsuperscript{aB}</td>
<td>8.4 (2.7)\textsuperscript{bB}</td>
<td>9.3 (1.5)\textsuperscript{bcA}</td>
<td>10.7 (2.3)\textsuperscript{bcB}</td>
</tr>
<tr>
<td>KetacCem</td>
<td>0.7 (0.5)\textsuperscript{aC}</td>
<td>3.0 (1.3)\textsuperscript{bC}</td>
<td>0.0 (0.1)\textsuperscript{cC}</td>
<td>0.3 (0.7)\textsuperscript{cD}</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Luting Cement</th>
<th>Group 5</th>
<th>Group 6</th>
<th>Group 7</th>
<th>Group 8</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sa</td>
<td>Sa &amp; E</td>
<td>Sa &amp; Si</td>
<td>Sa &amp; E &amp; Si</td>
</tr>
<tr>
<td>Panavia F</td>
<td>10.7 (6.1)\textsuperscript{bB}</td>
<td>11.7 (5.3)\textsuperscript{bcB}</td>
<td>14.4 (4.6)\textsuperscript{bc}</td>
<td>16.4 (5.2)\textsuperscript{c}</td>
</tr>
<tr>
<td>Xeno Cem</td>
<td>11.7 (2.3)\textsuperscript{bA}</td>
<td>19.4 (4.4)\textsuperscript{bA}</td>
<td>18.0 (4.1)\textsuperscript{de}</td>
<td>20.9 (4.2)\textsuperscript{e}</td>
</tr>
<tr>
<td>Rely X Luting</td>
<td>4.8 (1.7)\textsuperscript{bcC}</td>
<td>6.8 (2.0)\textsuperscript{bcC}</td>
<td>3.9 (1.5)\textsuperscript{b}</td>
<td>4.6 (2.4)\textsuperscript{b}</td>
</tr>
<tr>
<td>Fuji Plus</td>
<td>8.3 (2.0)\textsuperscript{bB}</td>
<td>11.5 (3.5)\textsuperscript{bcB}</td>
<td>9.5 (1.9)\textsuperscript{bc}</td>
<td>9.7 (3.9)\textsuperscript{bc}</td>
</tr>
<tr>
<td>KetacCem</td>
<td>2.6 (1.7)\textsuperscript{bcC}</td>
<td>4.1 (1.4)\textsuperscript{bcC}</td>
<td>0.0 (0.0)\textsuperscript{a}</td>
<td>0.0 (0.0)\textsuperscript{a}</td>
</tr>
</tbody>
</table>

Treatment: NT = No Treatment, E = Etched, Si = Silanized, Sa = Sandblasted.
No significant differences were observed for the surface treatment if the mean shear bond strength is quoted with the same small letter. No significant differences were observed for the cement if the mean shear bond strength is quoted with the same capital letter.

For the conventional glassionomer cement (Ketac Cem) any increase in surface roughness by means of sanding, sandblasting and/or etching of the ceramic surface improved the bond strength value. A silanized surface had a detrimental effect on the interface between the ceramic and cement resulting in complete debonding of the cement.

In general, the surface treatment of etching, sandblasting, sandblasting followed by etching in combination with silanizing (Groups 4, 7 and 8) resulted in significant higher shear bond strength values compared to the untreated surface (Group 1). The shear bond strength increases significantly from Ketac Cem, Rely X Luting, Fuji Plus, Panavia F to Xeno Cem, for the Groups 7 and 8. For Group 4 the same order was found, but there were no significant differences between Fuji Plus and Panavia F, and Panavia F and Xeno Cem.
SEM analysis showed that sanding with 1200 grit silicon carbide paper produced only slight surface scratches (Figure 3A). Hydrofluoric acid etching and sandblasting resulted in a surface with relief and undercuts (Figure 3B and 3C), which became more pronounced if the surface was sandblasted and etched (Figure 3D).

**Figure 2.** Shear bond strength (MPa) of different luting cements depending of ceramic treatment. NT = No Treatment, E = Etched, Si = Silanized, Sa = Sandblasted.
Figure 3. SEM Micrograph of the ceramic surface (magnification 1000x) A. Specimen sanded with 1200-grit SiC paper. B. Specimen sanded with 1200-grit SiC paper and etched for 150 s with 9.5% hydrofluoric acid. C. Specimen sanded with 1200-grit SiC paper and sandblasted during 20 s with aluminum oxide (50μm) at 200 kPa air pressure. D. Specimen sanded with 1200-grit SiC paper and sandblasted during 20 s with aluminum oxide (50μm) at 200 kPa air pressure and etched for 150 s with 9.5% hydrofluoric acid.

Discussion

This study was carried out to evaluate the shear bond strength of different luting cements to an aluminum oxide reinforced glass ceramic material. After polishing the surface of the ceramic surface, different treatments were applied and the effect on the shear bond strength was investigated. For the resin composite and resin modified glass ionomer cements surface treatments that involve roughening and silane application resulted in the highest shear bond strength values. These results are in agreement with previously published results, showing that an efficiently etched ceramic surface in combination with a silane treatment usually provides the highest bond strengths.\(^8\)\(^{16}\) It should be noted that silanization is crucial in the durability of the bond, since the bond strength of unsilanized surface seems to decrease in time.\(^{11}\)\(^{14}\) SEM analysis showed a similar topographical relief for acid etching and sandblasting, while sandblasting followed by etching created a greater topographical relief. These findings correspond with the observed bond strengths, showing that micromechanical interlocking plays an important role in bonding. Beside the micromechanical interlocking of the cement and the ceramic surface, chemical
bonding also contributes to the bond strength. Comparing an untreated surface with a silanized surface (Groups 1 and 3) shows an increase in bond strength of ca. 5 MPa. For the resin composites a significant increased bond strength for silanized surfaces were found for the sandblasted samples (5 vs 7), showing a combination of micromechanical interlocking and chemical bonding. For conventional glass ionomer cement, Ketac Cem, an increase in surface roughness resulted in improved bond strength, but silanization of the surface had a detrimental effect on the bond strength. Apparently, the chemical interaction of polyacrylic acid and the ceramic surface contributes to shear bond strength, which was cancelled out by the use of the silanization agent.

Shear bond strength of resin based cement to ceramic materials of maximal 21 MPa with a rather large standard deviation is in agreement with previous research9-11,13,14,16. The brittle character of the cements is often taken as the origin of the rather large standard deviations. The results of this study compared to other studies are probably due to differences in porcelain, acid concentration and etching time, sand blasting pressure and differences shear bond strength determination. Figure 2 and Table 2 showed that the shear bond strength increases in the order of Ketac Cem, Rely X Luting, Fuji Plus, Panavia F, Xeno Cem. For the interpretation of these values one has to take the internal strength of the cement used into account. Ultimately, cement with a bond strength that competes with the strength of the cement or one of the substrates to be bonded to, can be used. The tensile strength of glass iononomer cement is much lower than that of resin modified glass ionomer cements, which have a lower strength compared to resin composite cements17. This fact is reflected in the highest shear bond strength values of all cements tested. In general, the ranking of the bond strength results mounted up from glass ionomer cements to resin modified glass ionomer cements to resin based cements. This trend may be related to the intrinsic strength of the cement. The higher the resin contents the higher the strength.

Since, the all-ceramic restoration is cemented to dentine not only the cement-ceramic interface is important but also the dentine-cement interface can be an important factor that determine the longevity of the restoration. The shear bond strength of human dentine was found to be 13.4 Mpa18. The shear bond strength of dentine to Panavia F18, Fuji Plus19, and Ketac Cem20-22 were 7.7 MPa, 7.0 MPa and 1.6 – 12.9 MPa, respectively. Due to variation in experimental set-up or preparative procedures the reported shear bond strengths in the literature are hard to compare.
Nevertheless, the reported shear bond values are much lower than the shear bond strength values found for the ceramic-cement interface. Microtensile bond strength test of dentin and Cerec 2 inlays cemented with Panavia F showed a similar result; de-bonding occurred more often at the cement-dentin interface than at the cement-inlay interface. In short, based on the previous considerations the use of the two resin composite cements will give the most reliable bond to the ceramic material and fracture will most probably occur at the cement-dentin interface.

In principle the two resin cements Panavia F and Xeno Cem are the favorable cements for clinical use. But, for the clinical application of luting cements more variables have an influence on the final retention of the restoration. The handling of the lute during mixing, placement and setting is an important factor as well as the amount of steps of the bonding procedure. The increase in steps may affect the risk to fail too as the time to carry out the whole adhesive procedure will be enlarged. Moreover, the risk of blood and/or saliva contamination of the preparation may increase with enlarging the procedure time. To decrease operator-related mistakes it may be desirable to use or develop simple, fast systems.

Conclusions

Based on the results of this study the use of resin composite based cements is mandatory for cementation of an all-ceramic restoration with an aluminum oxide reinforced glass ceramic base. As surface treatment etching and/or sandblasting followed by silanization is recommended. The etching and sandblasting provides the micromechanical interlocking and the silanization is crucial for the durability of the bond. Furthermore the bonding procedure is clinically simple to carry out.

References


