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Effect of Surface Treatments and Aging in Water on Bond Strength to Zirconia

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Clinical Relevance

Air abrasion and pretreatment with a metal primer seem to be an appropriate method for improving the bond strength of RelyX Unicem resin composite cement to hot isostatic pressed yttrium-oxide partially stabilized zirconia.

SUMMARY

The current study evaluated the effects of various pretreatments and aging in water on the bond strength to hot isostatic pressed yttrium-oxide partially stabilized zirconia. Sixty zirconia ceramic specimens (Denzir) were randomly divided into three groups of 20. One group of specimens (n=10) was then bonded to each other using a resin composite cement (RelyX Unicem), the second group (n=10) was bonded with RelyX Unicem and a metal primer (Metal Primer II), while the third group (n=10) was bonded with RelyX Unicem and a ceramic primer (Ceramic Primer). The specimens were then subjected to shear force before and after sandblasting and before and after aging in water for 180 days. Before aging, no significant differences (p>0.05) were seen within the different groups, either before or after sandblasting. Sandblasting and pretreatment with the metal (p<0.01) or ceramic (p<0.001) primers significantly improved the bond strength compared to that of the non-treated specimens. After aging, the bond strength of the sandblasted specimens with metal primer was significantly higher than that of the sandblasted specimens with ceramic primer (p<0.01) and the specimens with no primer (p<0.001). For the specimens that were sandblasted and pretreated with the metal primer, the bond strength was not significantly affected after aging (p>0.05), whereas, for those specimens with the ceramic primer (p<0.001) and no primer (p<0.05), the bond strength significantly decreased. Thus, air abrasion and pretreatment with a metal

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primer seems to be an appropriate method for improving bond strength.

INTRODUCTION

Good esthetics, strength and chemical stability are desirable characteristics in dental replacement materials, and sintered ceramics are materials said to fully possess these qualities.[1] One high-performance ceramic of special interest, because of its superior fracture toughness and chemical durability compared with other ceramics, is yttrium-oxide partially stabilized zirconia (Y-TZP).[2-3] Since Y-TZP has excellent biocompatibility and mechanical properties, it has been suggested that it has advantages over copings of alumina.[4] The research and development of zirconia as a biomaterial started 30 years ago and was first described in 1969 by Helmer and Driskrell as a material used in hip-joint replacements.[3] Ensuing research focused on zirconia-yttria ceramic characterized by fine-grained microstructures, known as yttria stabilized tetragonal zirconia polycrystals (Y-TZP). This material has a unique mechanism that inhibits crack propagation due to the transformation from a tetragonal structure to a monoclinic state that occurs at the crack tip.[3]

Dental restorations using Y-TZP are performed in two ways: 1) by milling enlarged restorations out of homogeneous ceramic green-body blanks of zirconia that are then sintered and shrunk to the desired final dimensions or 2) milling the restorations directly to the final dimensions out of highly dense sintered prefabricated Y-TZP blanks, so-called hot isostatic pressed (HIPed) zirconia blanks.[5-9] In dentistry, Y-TZP is used for posts, implant abutments, the cores of single crowns and fixed partial dentures (FPDs).[10]

The adhesion to enamel and dentin and the sealing of the marginal gap between the restoration and the tooth are dependent on the luting agent's ability to bond to the surface of the ceramic and, thus, it is crucial for the long-term durability of the restoration.[11] Consequently, the bond strength of various cements to Y-TZP, including zinc phosphate, glass-ionomer and resin composite, have been evaluated using various experimental setups and surface pretreatments.[11-16] Microtensile, shear or tensile testing methods with and without simulated aging and/or thermocycling have been used,[10,12,14-18] and conflicting results are reported regarding the effect on bond strength after aging in water and thermocycling.[10,16,18] Most studies, however, focus on determining the bond strength of various cementing agents to Y-TZP homogeneous green zirconia ceramic,[10,13,19-23] while only three articles were found that address the bonding properties of HIPed Y-TZP.[11-12,16] Since HIPed Y-TZP is used successfully in the fabrication of dental all-ceramic crowns and FPDs,[7-9] the bonding property of this type of zirconia ceramic is of interest. This study, therefore, evaluated the bonding property of a recently introduced dual-cured, self-adhesive resin-luting cement to HIPed Y-TZP and the effect of various pretreatments on bond strength and the effect of aging in water. The hypotheses considered were: 1) sandblasting and the use of various primers improve the bond strength to HIPed Y-TZP and 2) there would be no effect on the bond strength to HIPed Y-TZP from storage in water.

METHODS AND MATERIALS

The materials used, manufacturers and batch numbers are listed in Table 1. Sixty specimens made from hot isostatic pressed (HIPed) yttrium-oxide partially stabilized zirconia (Y-TZP) (Denzir) were delivered from the manufacturer. The specimens measured 14 x 14 x 3 mm and their shape is shown in Figure 1. Using the Decim CAD/CAM system (Cad.esthetics AB), the surfaces intended for bonding were plane-ground to each other. The area of the plane-ground surfaces was 14 mm x 14 mm.

Before bonding, all specimens were ultrasonically (Bransonic B221, Bransonic Ultrasonic Co, Danbury, CT, USA) cleaned for 30 minutes in distilled water, then wiped with 95 vol-% ethanol. The 60 specimens were then randomly divided into three groups of 20, one group of 10 pairs (n=10) was bonded with a resin composite cement (RelyX Unicem), the second group of 10 pairs (n=10) was bonded with RelyX Unicem and a metal primer (Metal Primer II), while the third group of 10 pairs (n=10) was bonded with RelyX Unicem and a ceramic primer (Ceramic Primer). In accordance with the manufacturers' instructions, the primers were dis-

Table 1: . *Summary of the Materials Used*

Material	Type of Material	Manufacturer	Batch #
Denzir	Hot isostatic pressed (HIPed) yttrium-oxide partially stabilized (Y-TZP) zirconia	Cad.esthetics AB, Skellefteå, Sweden	
RelyX Unicem	Dual-polymerizing resin luting agent	3M ESPE, St Paul, MN, USA	195276
Metal Primer II	Thiophosphoric methacrylate (MEPS)	GC Corporation, Tokyo, Japan	0506081
Ceramic Primer	Ethanol, methyl methacrylate	GC Dental Products Corp, Aichi, Japan	0504121
Ultradent Porcelain Etch	9.5% buffered hydrofluoric acid	Ultradent Products Inc, South Jordan, UT, USA	31TK

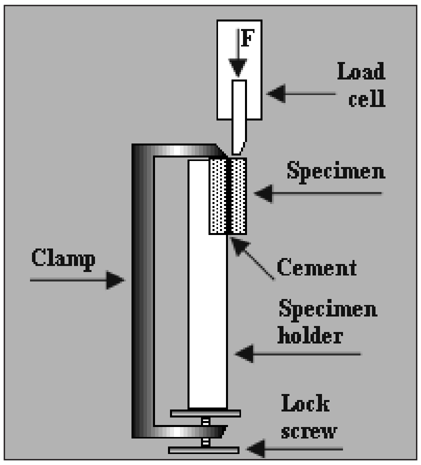

Figure 1. *Cross section of a bonded HIPed Y-TZP specimen in the specimen holder and subjected to a vertical load by means of a hardened, chisel-shaped steel blade.*

pensed, mixed and applied immediately with a microbrush. The specimens bonded with RelyX Unicem and the ceramic primer were, in accordance with the manufacturer's instructions, pretreated by etching the surfaces using 9.5% hydrofluoric acid (Ultra-dent Porcelain Etch) for 60 seconds, then rinsed in water and gently air dried.

The resin composite cement was mixed for 15 seconds using an amalgam vibrator (Duomat, Degussa, Germany), then used to bond the plane ground surfaces of the specimens to each other at room temperature. A constant load of 75 N was applied on the specimens and the excess cement was removed with an explorer. The resin composite cement was then polymerized on each side for 20 seconds using a halogen lamp (Norlite, Germany). After the bonding procedures, the specimens were stored in distilled water for 24 hours at 37°C prior to the bonding test.

Next, the bonded specimens were fixed in a holder (Figure 1). Using a universal testing machine (Alwetron TCT 5/10, Lorentzon and Wettre, Spånga, Sweden), the specimens were subsequently subjected to a vertical load by means of a hardened, chisel-shaped steel blade (0.7 mm thick and 6.5 mm wide). The load was applied perpendicular to the outer part of the specimens (Figure 1) at a crosshead speed of 0.5 mm/minute until failure occurred. The loads required to fracture the specimens were automatically recorded when the force was 1% below the highest level recorded during the test. To examine the mode of fracture, the debonded area was viewed under a light microscope (Wild M5A, Heerbrugg Ltd, Heerbrugg, Switzerland) at 25x magnification. The mode of fracture was classified as cohesive if failure occurred within the cement and adhesive if failure occurred at the junction of the cement and ceramic.

The plane-ground surfaces of the specimens were then sandblasted with 110 µm Al_2O_3 for 60 seconds at a standoff distance of 2 mm with 3 bars pressure. To ensure that there was no damage to the specimens or that no residual cement remained, the specimens were examined using a light microscope (Wild M5A) at 25x magnification. The bonding and loading procedures and evaluation of the mode of fracture were then repeated as described above.

In addition, after fracture, the plane-ground surfaces of the specimens were again sandblasted with 110 µm Al_2O_3 for 60 seconds at a standoff distance of 2 mm with three bars pressure and examined using the light microscope as described above. Thereafter, bonding was carried out as described above and the specimens were stored in the dark in distilled water for 180 days at 37°C. The water was changed monthly. After 180 days in water, the specimens were subjected to loading and the mode of fracture was examined as described above.

Statistical Analysis

The values were statistically analyzed using the Kruskal-Wallis test supplemented with Bonferroni at a significance level of $p<0.05$.

RESULTS

Data, as medians, and 1st and 3rd quartiles of the bond strength (MPa) of the nine series of specimens are presented in Figure 2. The results of the statistical analysis are summarized in Table 2.

Before aging in water, no significant ($p>0.05$) differences were seen within the various groups either before or after sandblasting. Sandblasting and pretreatment with metal ($p<0.01$) or ceramic ($p<0.001$) primers significantly improved bond strength compared with no pretreatment.

After aging, the bond strength of the sandblasted specimens bonded with metal primer was significantly higher than that of the sandblasted specimens bonded with ceramic primer ($p<0.01$) and specimens with no primer ($p<0.001$). No significant difference ($p>0.05$) was observed between the specimens bonded with no primer and those bonded with ceramic primer.

Aging significantly reduced bond strength to the sandblasted specimens pretreated with ceramic primer ($p<0.001$) and those pretreated with no primer ($p<0.05$). For those specimens sandblasted and pretreated with metal primer, the bond strength was not significantly ($p>0.05$) affected after aging in water.

All the specimens fractured within the cemented joint during the load-to-fracture test. Visual inspection, using the light microscope after fracture within the joint, showed that some cement remained on the surfaces, but most of the areas were free of cement.

Table 2: *Summary of Statistical Analysis of Bond Strength to HIPed Y-TZP Ceramic Specimens*

Specimen	R	RS	RSA	RC	RSC	RSCA	RM	RSM	RSMA
R									
RS	ns								
RSA	ns	*							
RC	ns	ns	**						
RSC	***	ns	***	ns					
RSCA	ns	ns	ns	ns	***				
RM	ns	ns	**	ns	ns	ns			
RSM	**	ns	***	ns	ns	**	ns		
RSMA	**	ns	***	ns	ns	**	ns		

Results of the Kruskal-Wallis test supplemented with Bonferroni.
ns denotes no statistically significant difference $p>0.05$.
*** denotes statistically significant difference* $p<0.01$.
**** denotes statistically significant difference* $p<0.001$. *Ten specimens in each group.*

R; RelyX Unicem with no primer, RC; Relyx Unicem with Ceramic Primer, RM; Relyx Unicem with Metal Primer, RS; RelyX Unicem with no primer but sandblasted, RSC; Relyx Unicem with Ceramic Primer and sandblasted, RSM; Relyx Unicem with Metal Primer and sandblasted, RSA; RelyX Unicem with no primer but sandblasted and aged for 180 days in water, RSCA; Relyx Unicem with Ceramic Primer, sandblasted, and aged for 180 days in water, RSMA; Relyx Unicem with Metal Primer, sandblasted, and aged for 180 days in water.

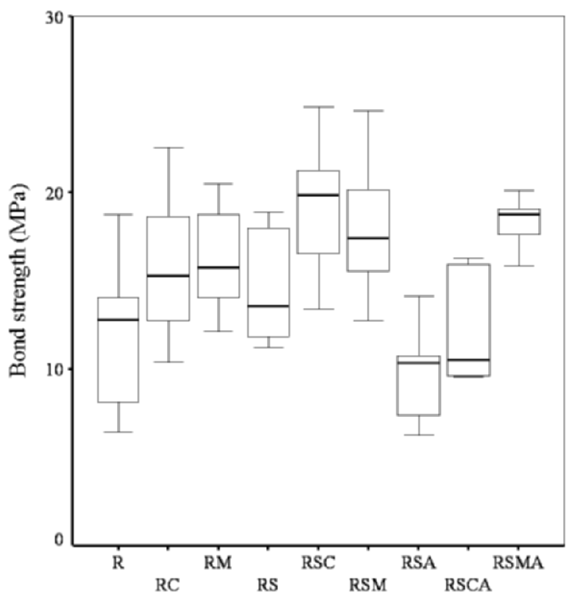

Figure 2. *Box-plot diagram comprising the load at fracture (MPa) of nine different types of pretreatments of the specimens. Ten specimens in each test group. Data are presented as medians and 1st and 3rd quartiles. A horizontal line within the box presents the median.*
R; RelyX Unicem with no primer, RC; Relyx Unicem with Ceramic Primer, RM; Relyx Unicem with Metal Primer, RS; RelyX Unicem with no primer but sandblasted, RSC; Relyx Unicem with Ceramic Primer and sandblasted, RSM; Relyx Unicem with Metal Primer and sandblasted, RSA; RelyX Unicem with no primer but sandblasted and aged for 180 days in water, RSCA; Relyx Unicem with Ceramic Primer, sandblasted, and aged for 180 days in water, RSMA; Relyx Unicem with Metal Primer, sandblasted, and aged for 180 days in water.

This implies a combination of adhesive and cohesive failure at the ceramic-cement interface.

DISCUSSION

The results of this study show that there are significant differences in bond strength between the sandblasted and pretreated specimens and the non-treated specimens. Aging in water significantly affected the bond strength of those specimens pretreated with ceramic primer and those not treated with primer. Therefore, the hypothesis that sandblasting and various primers improve bond strength was sustained; whereas, the hypothesis that storing the specimens in water would have no effect on bond strength was partly rejected.

There are several different bonding systems currently available on the market with varying numbers of preconditioning steps, such as etching, silanization and bonding agents. In the current study, the metal and ceramic primers and the resin composite that was used were obtained from different manufacturers. However, each manufacturer's instructions stated that the primers should promote bonding to acrylic resins and composites without limiting which resin composite could be used in combination with those primers. It was, therefore, of interest to the authors of the current study to study the effect of these primers on the bond strength to zirconia in combination with a recently-introduced dual-cured resin cement. In the current study, the same pairs of specimens were recemented after debonding. To ensure that no residual cement remained or that the pairs of specimens sustained no damage, they were examined under a light microscope. Since sandblasting should have removed the primer and resin composite from the surfaces, the texture of the specimens should have been equivalent to unused sandblasted specimens. Thus, it seems unlikely that repeated bonding would have notably influenced the outcome.

The shear bond test was selected, since it is said to be the most commonly used testing method[24] and to make it easier to compare the results obtained against previous studies of HIPed Y-TZP.[12,16] It has been stated that using the shear bond test may lead to undesired stress pattern distribution, resulting in cohesive failures and incorrect interpretation of the data, and that the microtensile bond strength test can reduce these problems.[25] However, most of the failures in the current study were ranked as "adhesive." Based on these findings and a statement in the previous study,[25] the weakest link in the current study seems to be bonding between the composite and ceramic surface.

The results in the current study showed that bond strength was influenced by different pretreatment methods. Sandblasting with Al_2O_3 prior to bonding resulted in the highest bond strength when used in combination with a metal or ceramic primer before storage in water. After storage in water, bond strengths significantly decreased in the group that had been sandblasted and pretreated with ceramic primer and in the group that had been sandblasted but with no primer, but it did not significantly affect the group that had been sandblasted and pretreated with metal primer. The fact that sandblasting and various primers influence the bond strength of dental ceramics has also been demonstrated in other studies.[10,12,26] However, consideration should be given to the possibility that sandblasting could affect the ceramic surfaces by creating microcracks and/or phase transformation, which, in turn, could influence the mechanical properties of the material.[9] There are studies stating that resin bonding agents strengthen the ceramic materials by repairing microcracks caused by sandblasting.[27-28] In the current study, there was no statistically significant improvement in bond strength within the various test groups before and after sandblasting. Therefore, since various results are reported regarding the effect of pretreatments on ceramics, more studies are needed to elucidate the effects of surface treatments on bond strength and on the mechanical properties of zirconia ceramics.

Piwowarczyk and others conducted tests on the bond strengths of RelyX Unicem to human dentin.[18] After 150 days of storage in water, their results showed a mean bond strength of 3.3 MPa. One interesting finding in their study[18] was that the mean bond strength increased to 6.2 MPa after storage in water for 150 days and 37,500 thermal cycles. In another study,[16] it was reported that the shear bond strengths of RelyX Unicem or Panavia F resin composite cements to HIPed Y-TZP were not significantly affected by storage in water for one week, thermocycling after 24 hours aging in water or air abrasion. Water storage and thermocycling are commonly used as aging parameters for bonding durability and generally reduce bond strength.[10,18] In a study of specimens made of green-body zirconia blanks and bonded after various pretreatments with two types of resin cements, Variolink II and Panavia F, Wolfart and others reported that all but the sandblasted specimens that were bonded with Panavia F showed very low bond strength or debonded spontaneously after storage in water for 150 days.[10] In other words, the bond was not stable over time. It was suggested that this resulted from a degeneration of the resin.[10] Similar observations were reported by Özcan and others after 6,000 thermal cycles[29] and, in a study by Blatz and others, it was shown that artificial aging in water and thermal cycling significantly reduced bond strengths.[14] In a pilot study preceding the current study, the bond strength of five specimens that were sandblasted and pretreated with a ceramic primer (Ceramic Primer) significantly decreased after 180 days in water and two of the specimens debonded spontaneously.

Another interesting finding in the current study was that bond strength was improved in sandblasted specimens that were pretreated with metal primer and bond strength was not significantly affected after storing in water. According to the manufacturer's information, the metal primer contained a special monomer, thiophosphoric methacrylate (MEPS), which was meant to promote bonding to all types of metal. In a study of bond strength to unalloyed titanium, it was shown that a primer containing MEPS was effective, and it was speculated that MEPS reacted with the thin oxide film on the titanium surface, which increased bond strength and durability.[30] The explanation for the improved bond strength to HIPed zirconia obtained in the current study for the sandblasted specimens pretreated with metal primer remains, however, unclear and further studies are needed to reveal the mechanism(s) of this improvement.

In the current study, hydrofluoric acid etching was included in the pretreatment of specimens with ceramic primer, despite Y-TZP being a non-silica-based ceramic material that should not be affected by etching with acid,[20] because the manufacturer's instructions included hydrofluoric acid etching during pretreatment. Although the authors of the current study did not expect the etching to have any effect on the Y-TZP specimens, they were, nevertheless, etched in order to follow the instructions of the manufacturer of the ceramic primer.

The authors of the current study could only locate three articles that addressed bond strength to HIPed Y-TZP.[11-12,16] In these earlier studies, the bond strength to HIPed Y-TZP of the adhesive resin cements Panavia 21 and RelyX Unicem was determined.[11-12,16] In one of the studies,[11] a bond strength of 12 MPa was achieved. In the other studies, which addressed the bond strength to HIPed Y-TZP, the reported values ranged between 7-11 MPa[12] and 16-21 MPa.[16] That is, the values reported in the former study[11] differed somewhat from those obtained in the current study, while those in other studies[12,16] were in closer agreement with the current study. A direct comparison between the studies is, however, difficult, because the experimental setups were different. In the former study,[11] bond strength was estimated using a push-out test; whereas, the shearing test was used in the latter studies.[12,16] Thus, the test methods in the studies were somewhat different, which may be one explanation for the differences in values obtained. In addition, in the current study, the size of the specimens was larger than in the other studies.[11,16] This could have influenced the results, because a large area often results in it being more difficult to achieve an even distribution of the cement and light polymerization, both of which can arise in clinical situations. Assessment of the clinical significance of the values achieved for bond strengths *in vitro*, however, is difficult. It has been suggested that 10-13 MPa should be the minimum for clinical bonding;[31-32] whereas, in another study,[33] it was stated that bond strengths to dentin and enamel should be higher than 20 MPa. However, if the bond strength between the core material and the cement exceeds the bond strength to dentin, the possibility that the bond to the core should be sufficiently strong for clinical use cannot be excluded. In this context, it should be noted that the values reported are based on *in vitro* studies.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions can be drawn:

1. Before aging, the bond strength of RelyX Unicem to HIPed Y-TZP specimens was significantly improved by sandblasting and pretreatment using a metal ($p>0.01$) or ceramic primer ($p>0.001$) when compared to specimens bonded without any pretreatment.
2. After aging, the bond strength of the sandblasted specimens bonded with ceramic primer ($p<0.001$) or without any primer ($p<0.05$) was significantly inferior when compared to the sandblasted specimens before aging. Aging did not significantly affect ($p>0.05$) the bond strength of the sandblasted specimens with metal primer.

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