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The Effect of Surface Preparation and Luting Agent on Bond Strength to a Zirconium-based Ceramic

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Clinical Relevance

Although clinical situations may exist where the adhesive luting of high-density milled ceramics for crown or fixed partial dentures are not required, certain clinical situations, such as the chairside repair of existing ceramic restorations, poor retention or inadequate abutment tooth size, will require a durable adhesive layer between the resin and ceramic. This study investigates the efficacy of modern "self-adhesive" luting cements on a zirconia-based ceramic following various surface preparations

SUMMARY

Objectives: To investigate the bond strength of modern "self-adhesive" resin cements to a zirconium-based dental ceramic following different surface preparations and storage conditions.

Methods: The surface of zirconium-based ceramic discs (12 x 2 mm) were either left untreated, prepared using alumina grit-blasting or tribochemical treatment. Resin composite cylinders were bonded to ceramic specimens using Panavia-F, RelyX Unicem or Maxcem resin cements. The shear bond strength of specimens (n=10) was tested "dry," following 24-hour water immersion or a thermocycling regime.

Results: For each surface preparation, a significant reduction in bond strength following 24-hour water immersion and thermocycling compared to "dry" storage conditions was identified for both Panavia-F and Maxcem. However, Unicem specimens exhibited statistically similar SBS values for tribochemically-treated specimens stored dry following 24-hour water immersion or thermocycling (11.7 ± 1.3, 14.1 ± 6.3 and 11.7 ± 4.9 MPa, respectively) (p>0.05). No significant differences in bond strength were identified for Panavia-F or Unicem specimens for any surface preparation following the thermocycling regime (p>0.05). In contrast, for each surface preparation following thermocycling (p<0.001), Maxcem exhibited a significant decrease in SBS

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compared with Panavia-F and Unicem specimens.

Conclusions: The pre-treatment of a zirconium-based ceramic surface with grit-blasting and tribochemical treatment improves the bond strength of resin cements. Following "wet" storage conditions, Panavia-F and Unicem demonstrated superior bond strength compared with Maxcem. Differences in ceramic surface preparation and the chemistry of resin cements will affect the nature of the bonding mechanism and durability of the adhesive layer.

INTRODUCTION

The properties of a luting agent and the preparation technique for ceramic intaglio surfaces prior to cement application play a major role in the clinical success of many indirect ceramic restorations. Luting agents should provide an adequate seal between the tooth and restoration to prevent the ingress of bacteria at the margins, while increasing fracture resistance of the restored tooth and restoration.[1] Many types of luting agents exist for the retention of fixed ceramic prostheses: contemporary zinc phosphate cements, glass-ionomers, resin-modified glass-ionomers and resin-based cements. Various investigations have reported that resin-based luting agents improve the retention and efficacy of indirect ceramic restorations,[2-3] particularly the provision of increased fracture resistance and an acceptable marginal seal of the ceramic restoration when compared to contemporary cements.[4]

To aid the adhesion of conventional silica-based ceramics, the glass phase at the surface of the ceramic can be readily etched using relatively low concentrations of hydrofluoric acid (HF). Subsequently, penetration of the resin cement into pores in the etched surface of the ceramic provides micromechanical retention, which may enhance the chemical bond strength of the silane-treated ceramic surface. However, high density alumina and zirconia-reinforced ceramics cannot be readily etched by HF using clinically acceptable acid concentrations, particularly for the chairside repair of existing restorations, since the high crystalline content provides resistance to acid attack. Furthermore, high-density, non-silica based ceramics, such as yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) materials, lack the silane binding groups needed for chemical adhesion of the resin to the ceramic surface. Tribochemical treatments can be used to provide a silicatized layer at the ceramic surface. Systems, such as Co-Jet and Rocatec (3M ESPE Dental Products, Seefeld, Germany), are used for chairside and laboratory ceramic preparation, respectively. These methods utilize mechanical energy (grit-blasting under pressure) to transfer silica-coated alumina particles to the surface of the ceramic substrate. Furthermore, the subsequent application of silane as a difunctional coupler to the silicatized surface is capable of achieving chemical retention between polymers and inorganic substances, which significantly improves bond strength.[5-7]

Some recently developed resin-based cements claim to be "self-adhesive," in that conventional surface pre-treatments are not required (such as one-step dentin bonding agents), with the ease of handling significantly improved over contemporary materials. The use of encapsulated, compared to separate base/catalyst, systems may also improve the effectiveness of the cement by reducing operator-induced variability and technique sensitivity.[8] It is well-known that surface pre-treatments and silane application to ceramic restorations can provide an increase in bond strength.[5-7] However, the adhesive nature to highly dense machinable ceramics, such as Y-TZP materials, has not been fully elucidated, and controversy still exists regarding applicable surface preparations, bonding methods and the type of luting agents on the success of adhesion to high-strength Y-TZP materials.

Consequently, this study investigated the bond strength of various modern resin cement systems to a Y-TZP dental ceramic following different surface preparations.

METHODS AND MATERIALS

Zirconium-based Y-TZP ceramic discs (Lava, 3M ESPE Dental Products, Seefeld, Germany) (12 mm in diameter, 2 mm thick) were provided fully sintered by the manufacturer. Disc specimens were cleaned for five minutes in an ultrasonic bath containing distilled water, air-dried, then divided into three test groups for each surface pre-treatment (Table 1). Subsequent to

Table 1: *Surface Pre-treatment Methods of the Zirconia-based Ceramic*

Group	Description	Manufacturer
1	**Grit-blast** Alumina particles Average particle size, 110 µm Nozzle pressure, 5 bars, Distance/time, 10 mm, 10 seconds	Korox, Bego, Bremen, Germany
2	**Co-Jet** Tribochemical coating and silanization Silica-coated alumina particles Average particle size, 30 µm Nozzle pressure, 2.8 bars, 2 mm, 15 seconds Silane, 3-MPS in ethanol, 5 minutes (ESPE-Sil)	3M ESPE Dental Products, Seefeld, Germany
3	**Control** No surface pre-treatment	

MPS, 3-methacryloxyprophyltrimethoxy silane the pre-treatment regimes, the ceramic surfaces were returned to the ultrasonic bath with clean distilled water to remove any wear debris. For each surface preparation group, the specimens were randomly divided into three additional groups for dry, 24-hour water immersion and thermocycling (TC) storage conditions (n=10). Resin-based composite (Tetric, shade A3; batch G01659, Ivoclar-Vivadent, Schaan, Liechtenstein) cylinders were prepared in an open-ended, white Delrin mold cavity (4 mm diameter, 4 mm thickness) with a light-curing unit (LCU) (Optilux 501, Kerr, UK) with an intensity of 940 ± 22 mW/cm^2, for a total polymerization time of 40 seconds from each side. Opposing mold cavity surfaces were covered with cellulose acetate to obtain a standardized cylinder height and to reduce oxygen inhibition of the outer layers of the resin composite material during polymerization.

The type, chemical composition and dispensers of the resin cements used in the current study are indicated in Table 2. Each cement was applied to the surface of the prepared group of ceramic specimens according to the manufacturers' instructions. For Panavia F 2.0 (PAN), equal amounts of ED primer (liquids A and B) were mixed and brushed onto the ceramic surface, left undisturbed for 30 seconds and gently dried. Following primer application, equal amounts (by equal revolutions of the syringe handles using the reference lines provided on the dispenser tubes) of base and catalyst pastes were dispensed and thoroughly mixed for 20 seconds, then applied to the primed ceramic surface. For RelyX Unicem (RXU), the encapsulated resin cement was activated, then immediately triturated using a CapMix high-speed mixer (3M ESPE) for 10 seconds at an oscillation speed of approximately 70 Hz, and the mixed cement was then applied to the ceramic surface. For Maxcem (MAX), the base and catalyst pastes were mixed through a dual-barrel syringe and applied directly to the ceramic surface.

Each ceramic-resin cement-composite cylinder sandwich was fabricated within a plastic alignment mold to allow for concentric placement of the resin-based composite cylinder and set under a load of 100 g for 30 seconds to ensure a consistent seating pressure. The excess resin cement was removed with a brush and left undisturbed for five minutes at 37°C ± 1°C to allow for chemical polymerization. While seating the composite cylinders for PAN specimens, Oxyguard 2.0 was applied to the exposed margins to minimize oxygen inhibition. Subsequently, each resin cement was light cured from the upper surface of the 2 mm ceramic disc for 40 seconds with the LCU.

Prior to the bond strength tests, for each resin cement and ceramic surface preparation (n=10), the

Table 2: *Type, Chemical Composition and Dispensers of the Modern Resin Cements Used in the Current Investigation*

Product/Batch/Code	**Type**	**Composition/Dispenser**	**Manufacturer**
Panavia F 2.0 batch: 41155 **code: PAN**	Dual cure ED Primer II A&B	2-HEMA, MDP, water accelerator, sodium benzene sulphinate	Kuraray Dental (Okayama, Japan)
	Adhesive	Resin: MDP, BPEDMA, hydrophobic-DMA, benzoyl peroxide, sodium aromatic sulfinate, N,N-diethanol p-toluidine, chemical and photoinitiators Filler: barium, silica, sodium fluoride filler (78 wt.%) **2 paste/hand-mixed**	
RelyX Unicem batch: 221806 **code: RXU**	*Dual cure* "self-adhesive" one-step	Resin: methacrylated phosphoric ester, multifunctional DMAs, chemical and photoinitiators Filler: alkaline filler, fumed silica (72 wt.%) **Capsule/mechanically mixed**	3M ESPE Dental Products (Seefeld, Germany)
Maxcem batch: 05-1347 **code: MAX**	*Dual cure* "self-adhesive" one-step	Resin: multifunctional DMAs, GPDM, proprietary Redox initiators and photoinitiators Filler: barium, fluoroaluminosilicate, fumed silica (66 wt.%) **2 paste/"auto-mix" dual barrel syringe**	Kerr, Sybron Dental Specialities (Orange, CA, USA)

HEMA, Hydroxyethylmethacrylate; MDP, 10-methacryloyloxy-decyl-dihydrogenphosphate; BPEDMA, bisphenol-A polyethoxy dimethacrylate; DMA, dimethacrylate; GPDM, glycerol dimethacrylate dihydrogen phosphate.

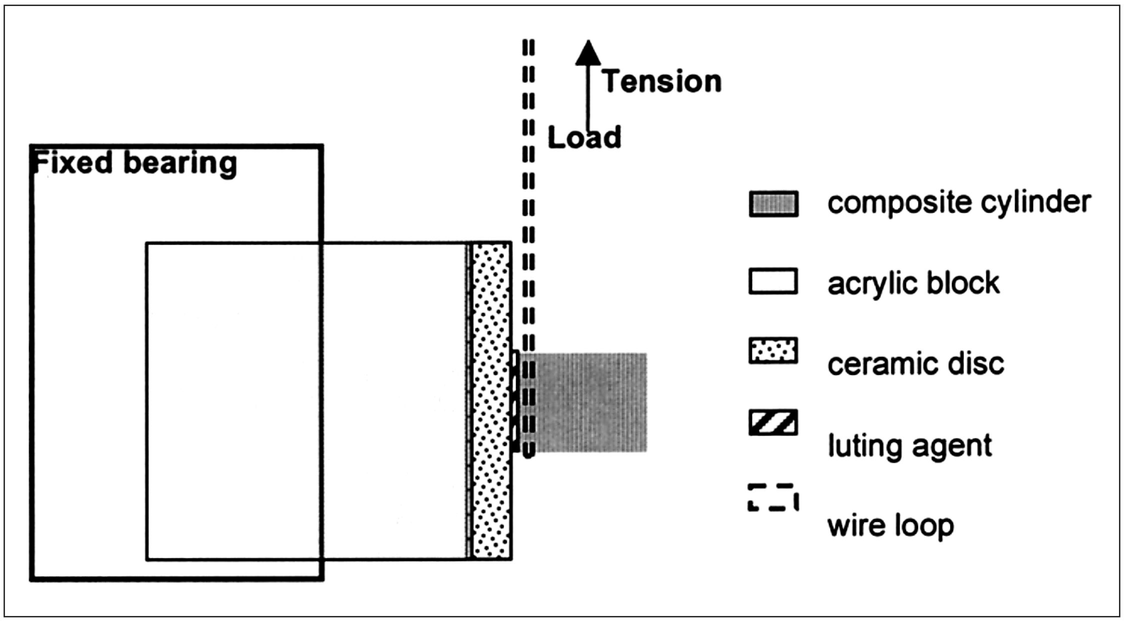

Figure 1. *Schematic drawing of the shear bond strength test apparatus illustrating a prefabricated composite cylinder attached with the specific luting agent to a ceramic disc with various surface preparations. A wire loop was positioned close to the edge of the composite cylinder and loaded in tension until failure of the specimen.*

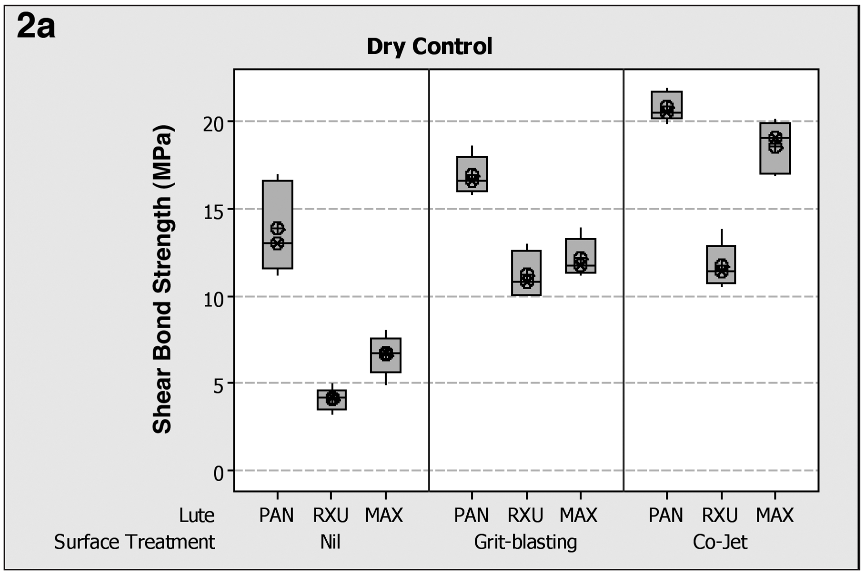

Figure 2: *Box and whisker plots of the shear bond strength of each resin cement with different surface pre-treatment. (a) Dry storage conditions. The boxes represent 50% of the bond strength values, the whiskers represent the highest and lowest strengths and the crosshair and line within the box indicates the mean and median, respectively.*

specimens were either stored a) in a dry light-proof container for 24 hours at 37°C ± 1°C, b) immersed in distilled water at 37°C ± 1°C for 24 hours or c) subject to a thermocycle (TC) regime. The TC regime consisted of 1000 cycles, where one cycle required 70 seconds to transfer the specimens between two water baths maintained at 4°C ± 1°C and 65°C ± 1°C, with a five second dwell time in each water bath.

The shear bond strength of the specimens was measured with a universal testing machine (Instron Model 5544, Instron Ltd, Buckinghamshire, England) at a crosshead speed of 0.5 mm/minute. During the testing procedure, the 0.4 mm stainless steel wire loop was positioned close to the edge of the resin composite cylinder adjacent to the ceramic disc specimen to ensure that the initial application of force took place parallel to the bonded surface (Figure 1). After bond failure, the diameter of each resin-based composite cylinder was measured with a micrometer (Moore and Wright, Sheffield, England) and the cross-sectional area calculated. The shear load at failure was recorded in N and converted to MPa as a function of the specimen area under test loading.

Specimens that failed prior to bond strength testing were recorded as 0 MPa and were included in the statistical analysis. In all cases, the Kolmogorov-Smirnov test was applied to test for a normal distribution. A non-parametric Kruskal-Wallis statistical test was employed to test data sets that did not exhibit normality or equal variances. Subsequently, a Mann-Whitney post-hoc test was performed to test paired group comparisons with a significance level of $p=0.05$.

RESULTS

The mean shear bond strengths (SBSs) are displayed in Table 3 and Figure 2, and the number of specimens that failed prior to bond strength testing is displayed in Table 4. The Kruskal-Wallis tests revealed that each resin cement, stored dry exhibited a significantly decreased SBS for the control group (no surface preparation) compared with either surface preparation type ($p<0.01$). For PAN and MAX, specimens treated with the Co-Jet regime exhibited significantly higher SBSs (20.8 ± 0.8 and 18.6 ± 1.5 MPa) than grit-blasting (16.9 ± 1.1 and 12.2 ± 1.1 MPa, respectively) (p<0.05). Whereas, for RXU, no significant difference was identified between specimen surfaces treated with either Co-Jet or grit-blasting (11.7 ± 1.3 and 11.2 ± 1.3 MPa, respectively). The Mann-Whitney paired group comparisons of test group medians revealed a significant increase in SBS of PAN compared with RXU or MAX specimens regardless of surface preparation type (p<0.05). The SBS of MAX was significantly greater than RXU for no surface treatment (6.6 ± 1.2 and 4.02 ± 0.63 MPa, respectively) and Co-Jet (18.6 ± 1.5 and 11.7 ± 1.3 MPa, respectively).

Generally, for each surface preparation, a significant reduction in SBS following 24-hour water immersion and TC compared with dry storage conditions was identified for both PAN and MAX. However, specimens of RXU exhibited statistically similar SBS values for Co-Jet surface preparations stored dry, following 24-hour water immersion or TC (11.7 ± 1.3, 14.1 ± 6.3 and 11.7 ± 4.9 MPa, respectively) (p>0.05; Figure 2). No significant differences in SBS were identified for PAN or RXU specimens for any surface preparation following the TC regime (p>0.05). In contrast, MAX exhibited a significant decrease in SBS compared with PAN and RXU specimens for either the control group, grit-blast or Co-Jet surface preparations following TC (p<0.001).

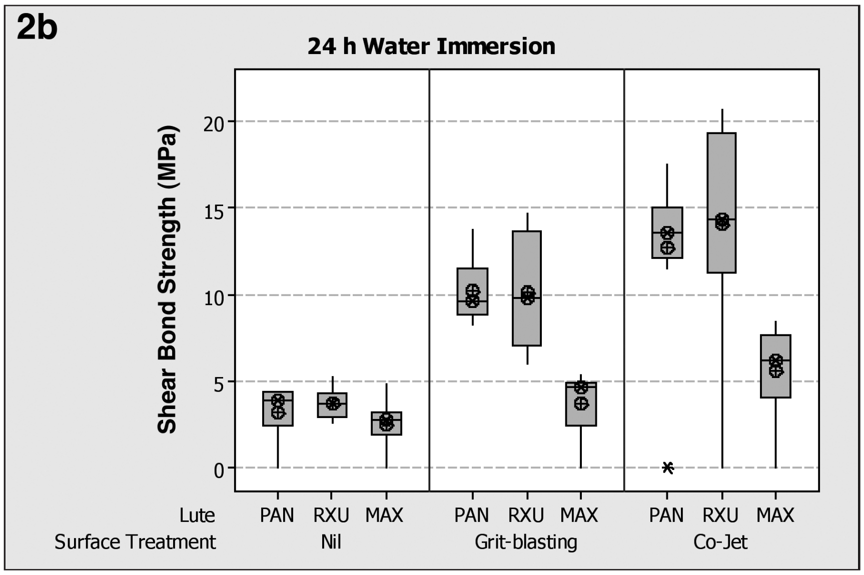

Figure 2b: *24 hour water immersion. The boxes represent 50% of the bond strength values, the whiskers represent the highest and lowest strengths and the crosshair and line within the box indicates the mean and median, respectively. Outlying values are indicated by an asterix (*).*

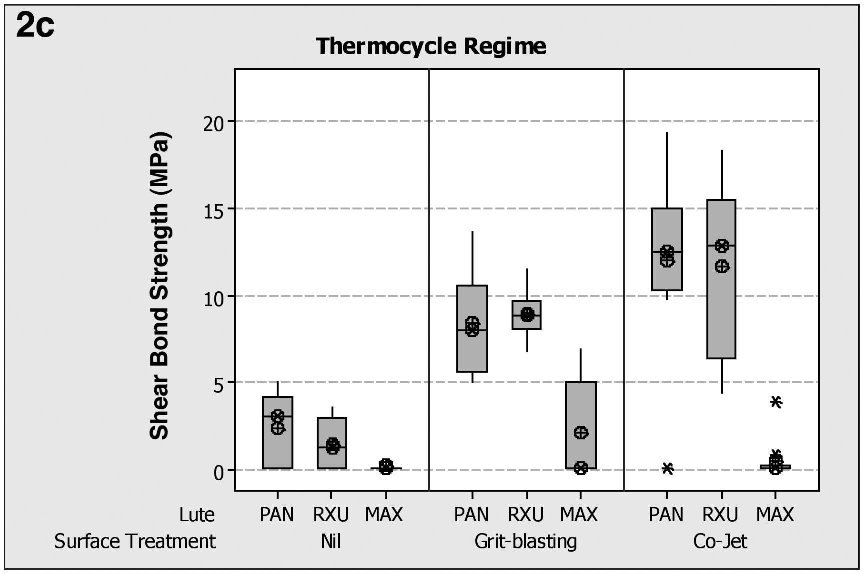

Figure 2c: *Thermocycling storage conditions. The boxes represent 50% of the bond strength values, the whiskers represent the highest and lowest strengths and the crosshair and line within the box indicates the mean and median, respectively. Outlying values are indicated by an asterix (*).*

DISCUSSION

Following the use of zirconium-based ceramic systems for orthopaedic prostheses, the interest in using such materials for the production of crowns and bridges for prosthodontic applications has increased. In particular, yttrium-stabilized zirconia materials offer high fracture strength and toughness due to inherent transformation toughening properties, which may significantly extend strength reliability and the longevity of specimens subject to specific pre-cyclic loading.[9] Counteracting forces (by cyclic loading) at the surface of Y-TZP ceramics may induce a volume increasing phase transformation to generate sufficient compressive stresses to resist crack propagation. However, previous studies have suggested that sharp-contact damage (by grit-blasting) can outweigh any strengthening effect from phase transformations of Y-TZP at critical numbers of cycles and load.[10-11]

Although clinical situations may exist where adhesive luting of a Y-TZP-based crown or fixed partial denture is not required,[12] adhesive luting provides the clinician with a "belt and suspenders" approach. Furthermore, certain clinical situations, such as the chairside repair of existing ceramic restorations, poor retention and inadequate abutment tooth size,[13] will require a

Table 3: *The mean shear bond strength (and standard deviation) for each resin cement following various surface preparations and storage conditions. Shear bond strengths exhibiting the same superscript letter within rows indicate no significant difference between surface preparations for the same resin cement type. Shear bond strengths exhibiting the same subscript number within columns indicate no significant difference between the different resin cements for similar surface preparations* ($p > 0.05$).

Storage Condition	Dry			24 Hour Water Immersion			Thermocycling		
Surface Preparation	**None**	**Grit Blast**	**Co-Jet**	**None**	**Grit Blast**	**Co-Jet**	**None**	**Grit Blast**	**Co-Jet**
PAN	13.9 (2.5)$_1^{C}$	16.9 (1.1)$_1^{B}$	20.8 (0.8)$_1^{A}$	3.2 (1.7)$_1^{E}$	10.2 (1.8)$_1^{D}$	12.7 (4.9)$_1^{C,D}$	2.4 (2.1)$_1^{E}$	8.4 (2.8)$_1^{D}$	12.0 (5.1)$_1^{D}$
RXU	4.0 (0.6)$_3^{C}$	11.2 (1.2)$_2^{A}$	11.7 (1.3)$_3^{A}$	3.7 (0.8)$_1^{C}$	10.2 (3.4)$_1^{A}$	14.1 (6.3)$_1^{A}$	1.4 (1.6)$_{1,2}^{D}$	8.9 (1.1)$_1^{B}$	11.7 (4.9)$_1^{A,B}$
MAX	6.6 (1.1)$_1^{C,D}$	12.2 (1.1)$_2^{B}$	18.6 (1.5)$_2^{A}$	2.5 (1.5)$_1^{F}$	3.7 (2.0)$_2^{E,F}$	5.6 (2.6)$_2^{D,E}$	0.2 (0.6)$_2^{F}$	2.1 (2.8)$_2^{F}$	0.5 (1.2)$_2^{F}$

Table 4. *The number of specimen failures prior to bond strength testing following 24-hour water immersion and thermocycling. For "dry" storage conditions, no specimens for each material and surface preparation failed prior to testing.*

Storage Condition	24 Hour Water Immersion			Thermocycling		
Surface Preparation	**None**	**Grit Blast**	**Co-Jet**	**None**	**Grit Blast**	**Co-Jet**
PAN	2	0	1	4	0	1
RXU	0	0	0	5	0	0
MAX	2	2	1	8	6	8

durable adhesive layer between the resin and ceramic. Highly dense ceramics, such as Y-TZP types, will require alternative bonding techniques from the conventional HF acid-etching. Previous reports suggest that etching does not result in an increased bond-strength, since the crystalline phase of the Y-TZP ceramic cannot be etched with clinically acceptable acid concentrations, resulting in decreased micromechanical retention.[5-7] In agreement, for no ceramic surface preparation, the current investigation revealed a significantly reduced bond strength for each resin cement, compared with grit-blasting and Co-Jet, following each immersion type (Figure 2). The roughened surface achieved with grit-blasting and Co-Jet may provide some degree of mechanical interlocking, while forming a larger surface area for the bond. In addition to the creation of a topographic pattern using the Co-Jet regime, chemical bonds are created between the silicatized ceramic surface, the ESPE-Sil silane agent and resin components of the luting agents. These mechanisms may explain the significant increase in bond strength for specimens pre-treated with the Co-Jet regime when compared with grit-blasting alone. It should be noted that the current investigation included pre-test failures, since analyzing bond strength data sets, which are based only upon surviving specimens, may bias the test towards an over-estimation of the bond strength.[14] Including pre-test failures in the current study reduced the normality and equal variance of data distributions and, as a consequence, non-parametric statistical analyses (Kruskal-Wallace and Mann-Whitney) were performed.

Wolfart and others[15] studied the durability of resin bond strength to zirconia with various surface pretreatments and suggested that, for Panavia-F, the bond was not stable following 150 days of storage or thermocycling. In this study, a significant decrease in bond strength was observed for PAN following 24-hour no surface treatment immersion and TC for ceramic specimens with either no or grit-blast surface preparation, which is in accordance with the findings of Wolfart and others,[15] where it may be assumed that the MDP monomer was not able to create a stable bond to a machined or abraded ceramic surface. However, following application of the Co-Jet regime in the current investigation, bond strength was significantly improved following 24-hour no surface treatment immersion and TC compared with grit-blasting alone. This may imply that a more durable bond to zirconia-based ceramics could be achieved using a silane application of the Co-Jet system, as well as the improved bonding potential of adhesive resin cements. In addition to silane contributing to bond strength through chemical linkages, it has been previously proposed that silanes may increase substrate surface energy and, therefore, improve surface wettability, leading to an increased bond strength.[16-17]

Numerous studies have reported the increased bond strength of PAN to metal oxides and have attributed the improvement to the phosphate ester group of the MDP-containing resin cement that directly bonds to metal oxides.[6-7,14] The data presented here, which indicates an increase in bond strength for Lava specimens prepared with PAN compared with RXU and MAX and tested dry, confirms the assumption that MDP-containing resin cements will also chemically bond to zirconia-based ceramics. Although RXU and MAX do not contain

MDP specifically, all three resin cements contain an adhesive phosphate monomer, which may explain the significant increase in bond strength of specimens tested following application of the Co-Jet regime compared with the absence of ceramic surface preparation (Table 2).

It has been frequently reported that silanized interfaces become unstable when in contact with moisture, which may explain why such studies almost always confirm lower strength values after storage in water and/or following thermocycling regimes.[5-7,15,18] In agreement, the bond strength values following water immersion and thermocycling recorded in the current study were generally lower than for dry conditions (Figure 2). However, the bond strength values of RXU samples were not significantly decreased following 24-hour water immersion or TC compared with dry conditions for Co-Jet surface preparations (Table 2). Similarly, Lüthy and others[18] also reported a non-significant decrease in the bond strength of RXU to a densely sintered zirconia ceramic following similar TC conditions. According to the manufacturer,[19] RXU consists of multifunctional phosphoric acid methacrylates and alkaline fillers. Apparently, as the material sets, water is produced as a by-product, initial acidity decreases and acidic functional groups are further neutralized with alkaline fillers.[19] This may be an important factor in the maintenance of a stable bond, where an acidic system (such as those used in a self-etch adhesive) may require neutralization following setting to avoid excess hydrolysis of the cement and a consequential decrease in bond strength following water immersion or even more severe TC storage conditions. Furthermore, the manufacturers claim that a secondary acid-base reaction of the fillers and phosphoric acid methacrylates accompanying neutralization result in a hydrophilic to hydrophobic property switch of the cured cement, which may also improve bond durability.

Interestingly, the bond strength of MAX following 24-hour water immersion and TC conditions for grit-blasted and Co-Jet prepared surfaces was inferior to both PAN and RXU (Figure 2). The bond strengths of MAX are not widely reported, although a recent study highlighted the lowest interfacial strengths and the highest amount of premature failures for MAX bonded to dentin or enamel,[12] which is in agreement with the data obtained in the current investigation (Tables 3 and 4). The GPDM monomer contained within MAX is purportedly responsible in part for its self-etching and adhesive properties.[20] Although the ability of GPDM-type phosphate molecules is known to etch enamel and dentin,[21] no information is given on the initial pH of the cement, which may be critical in understanding the chemical nature of adhesion to ceramic surfaces. The formation of siloxane bonds between the silanol (Si-O-CH_3) and hydroxyl (-OH) groups of the ceramic surface are initiated and accelerated by acid catalysis.[22] Consequently, the acid component within MAX may not provide the opportunity to enhance the formation of siloxane bonds, resulting in decreased bond strengths compared with PAN and RXU, as observed in the current investigation.

CONCLUSIONS

Within the limitations of this study, it can be concluded that pre-treatment of a Y-TZP ceramic surface with grit-blasting and/or tribochemical treatment improves the shear bond strength of resin cements. Differences in ceramic surface preparation and the chemistry of resin cements will affect the nature of the bonding mechanism and durability of the adhesive layer at high-density ceramic surfaces.

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