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Ceramic Primer Heat-Treatment Effect on Resin Cement/Y-TZP Bond Strength

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Clinical Relevance Statement

The adhesion of yttrium-stabilized tetragonal zirconia polycrystal ceramics to resin cements is unstable. Even if heat treatment of the metal/zirconia primer improves early bond strength, it is not effective for bond improvement under aging and therefore should not be recommended.

SUMMARY

The purpose of this study was to evaluate the effect of different heat-treatment strategies for a ceramic primer on the shear bond strength of a 10-methacryloyloxydecyl-dihydrogen-phosphate (MDP)-based resin cement to a yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic. Specimens measuring 4.5 × 3.5 × 4.5 mm^3 were produced from Y-TZP presintered cubes and embedded in polymethyl methacrylate (PMMA). Following finishing, the specimens were cleaned using an ultrasound device and distilled water and randomly divided into 10 experimental groups (n=14) according to the heat treatment of the ceramic primer and aging condition. The strategies used for the experimental groups were: GC (control), without primer; G20, primer application at ambient temperature (20°C); G45, primer application + heat treatment at 45°C; G79, primer application + heat treatment at 79°C; and G100, primer application + heat treatment at 100°C. The specimens from the aging groups were submitted to thermal cycling (6000 cycles, 5°C/55°C, 30 seconds per bath) after 24 hours. A cylinder of MDP-based resin cement (2.4 mm in diameter) was con-

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structed on the ceramic surface of the specimens of each experimental group and stored for 24 hours at 37°C. The specimens were submitted to a shear bond strength test (n=14). Thermal gravimetric analysis was performed on the ceramic primer. The data obtained were statistically analyzed by two-way analysis of variance and the Tukey test (α=0.05). The experimental group G79 without aging (7.23 ± 2.87 MPa) presented a significantly higher mean than the other experimental groups without aging (GC: 2.81 ± 1.5 MPa; G20: 3.38 ± 2.21 MPa; G100: 3.96 ± 1.57 MPa), showing no difference from G45 only (G45: 6 ± 3.63 MPa). All specimens of the aging groups debonded during thermocycling and were considered to present zero bond strength for the statistical analyses. In conclusion, heat treatment of the metal/zirconia primer improved bond strength under the initial condition but did not promote stable bonding under the aging condition.

INTRODUCTION

Reliable adhesion of resin cements to yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP)-based ceramics would improve marginal adaptation, prevent microleakage, and increase retention in situations where it does not exist.[1] However, adhesion between Y-TZP ceramics and resin cements is still a challenge because this ceramic substrate is highly crystalline and dense and consequently is characterized as a nonetchable material.[1,2]

Studies have shown that modifications on the Y-TZP ceramic surface using airborne particle-abrasion approaches, such as the tribochemical silica coating method followed by silanization[1,3] or sandblasted with aluminum oxide particles followed by application of ceramic primers with a chemical functional group increase the bond strength with 10-methacryloyloxydecyl-dihydrogen-phosphate (MDP)-based resin cement. The metal/zirconia primer contains an adhesive phosphate monomer that consists of a bifunctional molecule with 1) a polymerizable organic chain that reacts with the restorative material, 2) an acidic group that reacts with oxides present on the Y-TZP surface, and 3) a spacer group that affects the hydrophilicity, flexibility, and wettability of this molecule.[4,5]

Studies suggest that heat treatment of silanes can improve both their resistance to hydrolysis and the bond strength of glass ceramics to resin cements.[6–8] This treatment provides energy to the system, promoting the evaporation of the solvent and by-products formed in the chemical reactions of this layer. Furthermore, heat could enhance the polymerization of the molecule's organic portion.[9] Due to the similar molecular structure (bifunctional molecules), these mechanisms could also occur in the ceramic primer; consequently, heat could optimize the resin bond strength to Y-TZP ceramics.

Thus, the purpose of this study was to evaluate the effect of different heat-treatment strategies for a ceramic primer on the shear bond strength (SBS) of a MDP-based resin cement to a Y-TZP ceramic. The hypothesis was that the heat treatment of the ceramic primer would improve the resin bond strength.

MATERIALS AND METHODS

Y-TZP Specimen Production

The brand name, manufacturer, and chemical composition of the materials used in this study are listed in Table 1.

Sintered, block-shaped Y-TZP ceramics (Vita Zanhfabrik, Bad Säckingen, Germany) measuring $4.5 \times 3.5 \times 4.5$ mm^3 were embedded in polymethyl methacrylate (PMMA) (Classico, São Paulo, Brazil), maintaining one side of the block free for resin-cement application. They were wet ground finished with 200- to 1500-grit sandpapers (Politriz PSK-2V, ERIOS Equipamentos Técnicos e Científicos Ltda, São Paulo, Brazil) and then ultrasonically cleaned in distilled water for 10 minutes (Vitasonic II, Vita Zanhfabrik).

Subsequently, the blocks were randomly divided into 10 groups according to the surface conditioning method applied and the aging condition (n=14). The experimental groups and treatment strategies are schematically presented in Table 2.

Surface Conditioning Methods

All the testing groups, with the exception of GCd and GCa (control groups, no surface treatment), received an application of metal/zirconia primer (Ivoclar Vivadent Ltda, São Paulo, Brazil) using a microbrush rubbed upon the zirconia surface for 20 seconds. The primer was left for 180 seconds, in accordance with the manufacturer's recommendations, at room temperature and 50% relative humidity, to allow the chemical reactions of the primer and Y-TZP surface to occur. Then, the excess primer was removed by air spray free from oil contamination.

A heat treatment in a ceramic oven (EDG 1800, EDG Equipamentos, São Paulo, Brazil) was per-

Table 1: *Materials Used in the Study*

Brand	Type and Material	Composition	Manufacturer
InCeram 2000 YZ	Yttria stabilized tetragonal zirconia	Zircon oxides, yttrium oxides	Vita Zanhfabrik, Germany
Metal/zirconia primer	Ceramic primer	Tertiary butyl alcohol, methyl isobutyl ketone, phosphonic acid acrylate, benzoylperoxide	Ivoclar Vivadent, Schaan, Leichtenstein
Panavia F (paste A)	Resin cement	10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, initiators	Kuraray Medical Inc, Kurashiki, Okayama, Japan
Panavia F (paste B)	Resin cement	Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler catalysts, accelerators, pigments	Kuraray Medical Inc, Kurashiki, Okayama, Japan

formed for 60 seconds in the G45, G79, and G100 testing groups, in accordance with the temperature previously described for each group (Table 2).

Bonding Procedure

A dual-cure resin cement (Panavia F 2.0, Kuraray Medical Inc, Osaka, Japan) was mixed and applied in a standardized cylindrical plastic mold (2.4 mm diameter, height ~2 mm) and placed upon the zirconia surface of the specimens. The cement was mixed in accordance with the manufacturer's instructions, inserted into the plastic mold using a plastic hand instrument, and light activated (Dentsply SmartLite PS, Dentsply International, York, PA, USA) from above for 40 seconds. Removal of the plastic mold was performed gently by cutting the side with a no. 15 scalpel blade. The specimens from the heat-treated groups (Table 2) were stored for 24 hours at room temperature to guarantee the polymerization of the resin cement and then submitted to the SBS test. The specimens from the aging groups (Table 2) were submitted to the thermal cycling (6,000 cycles, 5°C/55°C, 30 seconds per bath) after 24 hours.

SBS Test and Failure Analysis

The specimens were mounted in the jig of a universal testing machine (EMIC, São José dos Pinhais, Brazil), and a knife-edge shearing rod running at a crosshead speed of 1.0 mm/min was used to load the adhesive interface until failure occurred. The maximum force to produce failure was recorded (N) using a corresponding software. Then the bond strength (MPa) was calculated (force in N / adhered area in mm^2).

Following the shear bond test, the fractured specimens were analyzed under an optical microscope (Mitutoyo FM, Mitutoyo, São Paulo, Brazil) at

Table 2: *Testing Groups (n=14)*

Groups	Heat-treatment Strategies of the Metal/Zirconia Primer	Condition
GCd	None (Control)	Dry[a]
G20d	Room temperature 20°C	
G45d	Thermal treatment at 45°C	
G79d	Thermal treatment at 79°C	
G100d	Thermal treatment at 100°C	
GCa	None (control)	Aging[b]
G20a	Room temperature 20°C	
G45a	Thermal treatment at 45°C	
G79a	Thermal treatment at 79°C	
G100a	Thermal treatment at 100°C	

[a] *The shear bond test was performed 24 hours after cement application.*
[b] *The shear test was performed after the aging regime.*

Table 3: *Results for the SBS by the Tukey Test*

Condition	Groups (n=14)	Shear Bond Strength (MPa)	Homogeneous Grouping[a]
Dry	GCd	2.81 ± 1.5	C
	G20d	3.38 ± 2.21	C
	G45d	6.00 ± 3.63	AB
	G79d	7.23 ± 2.87	A
	G100d	3.96 ± 1.57	BC
Aging	GCa	0.0	D
	G20a	0.0	D
	G45a	0.0	D
	G79a	0.0	D
	G100a	0.0	D

[a] *Different letters in same column show statistical differences with a significance level of 5%.*

Table 4: *Percentage of Fracture Types for the Experimental Groups (Dry Condition)*

Groups	Type of Failure			
	A	B	C	D
GC	100	0	0	0
G20	100	0	0	0
G45	71.44	0	0	28.57
G79	71.44	0	14.28	14.28
G100	78.57	0	0	21.42

20× magnification and by scanning electron microscopy (JEOL JSM-7401F, JEOL, Tokyo, Japan) using the secondary electron (SE) mode (70×) to inspect the failure type. The failure types were classified according to the following criteria: (A) along the ceramic/cement adhesive interphase; (B) cohesive in the ceramic; (C) cohesive in the resin cement; and (D) mixed (A+C).

Bond strength data were analyzed by two-way analysis of variance (ANOVA) and the Tukey test (α=0.05).

Thermal Analyses

A metal/zirconia primer solution was positioned in a platinum sample holder, its mass considered to be 15 mg. Thermal gravimetric and differential scanning calorimetry analyses (TG and DSC, respectively) were performed, which initiated at room temperature and increased to 200°C with a heat rate of 10°C/min at 50 mL/min under nitrogen flow.

Thermal analysis is used as a complementary technique in the identification of weight variation with heat treatment (TG) and to measure the enthalpy of the system and the heat capacity of reactions (DSC) when the temperature is increased.

RESULTS

All the specimens in the aged groups debonded during thermocycling and were considered to present zero bond strength for the statistical analyses. All the failures of these prematurely debonded specimens presented a type (A) adhesive failure of the Y-TZP ceramic and cement resin.

Two-way ANOVA revealed that the SBS values were significantly affected by surface treatment (p=0.000) and the interaction with aging (p=0.000). The results of the Tukey multiple comparison test revealed that G79d (7.23 ± 2.87 MPa) presented higher values compared with negative (GCd: 2.81 ± 1.5 MPa) and positive (G20d: 3.38 ± 2.21 MPa) control groups and G100d (3.96 ± 1.57 MPa). G45d (6 ± 3.63 MPa) presented statistically similar results compared with G79d (Table 3).

The fracture analysis of the specimens revealed different fracture patterns: adhesive failure along the interfacial region between the luting agent and the Y-TZP ceramic surface, mixed failure (cohesive fracture of the luting agent combined with adhesive failure), and cohesive in the luting agent (Table 4). The typical mixed fracture pattern is presented in Figure 1.

The TG and DSC analyses (Figure 2) showed mass loss accompanied by an endothermic reaction with a peak at 57°C and a reverse exothermic reaction at 79°C that peaked at 142°C, followed by a slightly downfall, then remained constant from 162°C.

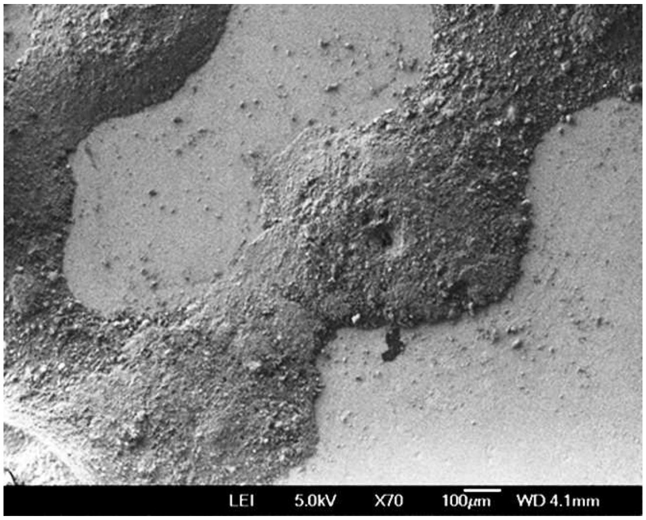

Figure 1. *Representative SEM micrograph of a mixed fracture pattern specimen.*

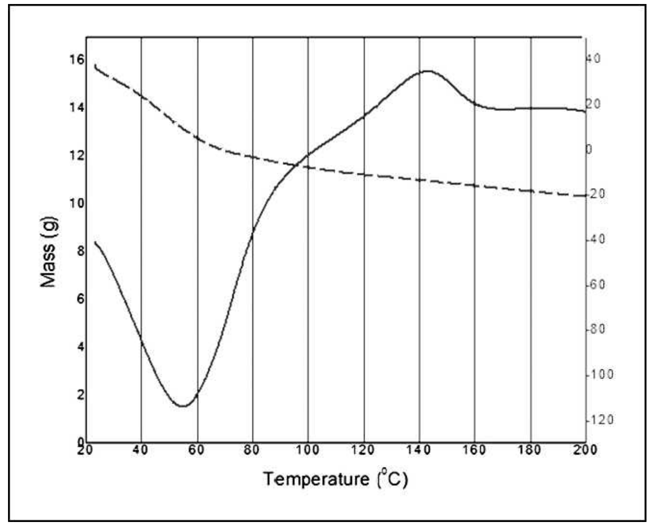

Figure 2. *Thermal behavior of the metal/zirconia primer due to temperature increase: TG (dashed line) and DSC (continuous line).*

DISCUSSION

It is known that the cohesive resistance of Y-TZP ceramics surpasses the adhesive resistance between them and resin cements, decreasing the chances of ceramic failure during the test. The choice of the SBS test was made considering this fact and to simplify comparison of the results with other studies reported in the literature that used the same methodology.[1,5,10,11,12]

Many studies regarding adhesion to Y-TZP ceramic have been conducted due to the fact that the bond between this material and resin cement is not stable.[1] Its nonetchable characteristic prevents the formation of an adhesive interface capable of resisting hydrolysis, leading to degradation and bonding failure.[13]

The heat treatment for bonding agents indicated for etchable ceramics improves the bond strength resistance with resin cements.[1,7] This positive effect could have been caused by the increase in siloxane links in the bonding agent and the solvent evaporation present in the mixture.[7,8] Solvent molecules stored in the adhesive interface could influence the degradation process, accelerating hydrolysis.[14]

The current study applied heat treatment to the metal/zirconia primer used for dentistry and indicated the positive influence of this treatment on the SBS in the dry condition. The primer was subjected to this process up to 200°C, as demonstrated in the TG analysis (Figure 2). It displayed continuous mass loss due to the increase in temperature, suggesting a continuous chemical reaction. The objective of the DSC analysis in this study was to detect the initial temperature of the thermal processes responsible for mass loss and its qualitative characterization, endothermic or exothermic.[15] The modifications that occurred in the material are linked to the chemical processes and transformations caused by the absorption and release of heat, which are clearly present in Figure 2. In the first phase, the primer absorbs heat and results in the loss of volatile substances (solvent and water produced during the primer reaction with the ceramic surface) up to 60°C. Beyond this temperature, mass loss decreased and an inversion of the curve began at 80°C, indicating the onset of exothermic reactions in the primer, with a peak at 140°C caused by the polymerization and posterior chemical degradation of the material, starting at 162°C. Polymerization began at 80°C, as verified by the DSC, negatively influencing the bond strength results for the G100d group.

One important fact is that the chemical composition of the primer used in this study is 70% alcohol (solvent) and its ebullition point is close to 80°C. Within this temperature range, alcohol molecules and by-products resulting from the hydrolysis of phosphonic acid stored in the polymeric network are probably eliminated, according to the results obtained for bond strength, which were greater for the G79d group.

Other studies suggest that silane copolymerization could retard the diffusion of water molecules, preventing the impact of hydrolysis on the adhesive interface.[16] Nevertheless, even with its positive influence on the results, the heat treatment promoted values that were too low to consider it an efficient alternative for resin bonding improvement for non-etchable Y-TZP ceramics. Although this consideration is related only to the metal/zirconia primer, the aging groups confirm that heat treatment alone was insufficient to promote stable bonding between resin cements and Y-TZP ceramics when only chemical adhesion is required. A consideration must be made regarding the heat treatment time, which was standardized to the time recommended by the manufacturer when the primer is applied under room temperature. However, extending the heat-treatment time and also changing the temperature of ceramic surface could improve the bond strength.

In agreement with the current results, previous research showed that the use of primer solution before resin cement application improved initial bond strength to zirconia ceramics[17]; however, no chemical adhesion was observed between Panavia (Kuraray Medical) and a flat Y-TZP ceramic when this interface was submitted to aging.[18,19]

Several techniques to improve this bond resistance have been reported in the literature, and the use of advanced technologies and chemical modification of the ceramic surface have demonstrated some success at improving the bond strength. The use of film deposition,[20,21,22] selective infiltration etching as a surface pretreatment, nanostructural alumina coating of the Y-TZP surface, or the addition of glass ceramics to the internal region of the zirconia-based ceramics in order to improve bond resistance and permit the use of conventional silane treatment have all been reported.[23,24,25]

Other studies have been trying to find alternative adhesion approaches that are functional in the working environment. Further studies regarding bonding agents have also been conducted.[17,23] The efficiency of this primer is associated with the increased roughness of the surface, consequently promoting better micromechanical retention, improving surface free energy, and increasing the contact area between the ceramic and the bonding agent.[19] That there are so many studies attempting to improve the bond resistance of this adhesive interface is due to the fact that no consensus exists regarding the best treatment to be applied at the Y-TZP adhesive surface. A surface treatment that permits an adhesive cementation of restorations made of Y-TZP frameworks would maximize their clinical application and contribute to preventing marginal infiltration and discoloration.[1,5,10,17,18,21,26]

Heat treatment of the ceramic primer associated with air abrasion using alumina particles may produce relevant results, though sandblasting methods seem to promote crack formation, compromising the material's performance in fatigue tests.[5,27] Clinical studies are necessary to validate this surface treatment as a valid alternative.

Therefore, new studies are required to further investigate the importance of copolymerization on bond resistance following interfacial aging, using other ceramic primers and heating ceramic primers associated with different surface treatments. Heat treatment of ceramic primers should be studied separately to find an optimal temperature capable of promoting the optimal results for each bonding agent.

CONCLUSION

Within the limitations of this study, it can be concluded that heat treatment of the metal/zirconia primer improved the early bond strength but did not promote stable bonding under the aging condition. The aging condition is fundamental for assessing the real capacity for bond improvement of any pretreatment of the Y-TZP surface.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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