

Effect of Various Surface Conditioning Methods on the Adhesion of Dual-cure Resin Cement with MDP Functional Monomer to Zirconia after Thermal Aging

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This study evaluated the effect of chairside and laboratory types of surface conditioning methods on the adhesion of dual-cure resin cement with MDP functional monomer to zirconia ceramic after thermocycling. Disk-shaped (diameter: 10 mm, thickness: 2 mm) Y-TZP ceramics (Lava™, 3M ESPE) were used (N=40) and finished with wet 1200-grit silicon carbide abrasive paper. Specimens were randomly divided into four experimental groups according to the following surface conditioning methods (n=10 per group): Group 1—Chairside airborne particle abrasion with 50- μm Al_2O_3 + Alloy Primer (Kuraray); Group 2—Airborne particle abrasion with 50- μm Al_2O_3 + Cesead II Opaque Primer (Kuraray); Group 3—Airborne particle abrasion with 50- μm Al_2O_3 + Silano-Pen + silane coupling agent (Bredent); Group 4—Laboratory tribochemical silica coating (110- μm Al_2O_3 + 110- μm SiO_x) (Rocatec) + silane coupling agent (ESPE-Sil). Adhesive cement, Panavia F 2.0 (Kuraray), was bonded incrementally to the ceramic surfaces using polyethylene molds (diameter: 3.6 mm, height: 5 mm). All specimens were thermocycled (5 and 55°C, 6,000 cycles) and subjected to shear bond strength test (1 mm/min). Data were statistically analyzed (one-way ANOVA, $\alpha=0.05$), whereby no significant differences were found among the four groups (8.43 ± 1.3 , 8.98 ± 3.6 , 12.02 ± 6.7 , and 8.23 ± 3.8 MPa) ($p=0.1357$). Therefore, the performance of chairside conditioning methods used for zirconia was on par with the laboratory alternative tested.

Keywords: Surface conditioning, Panavia F 2.0, Zirconia

INTRODUCTION

The increasing demand for metal-free fixed partial dentures (FPD) has fueled the development of all-ceramic materials with optimized mechanical properties. One of the most commonly used all-ceramic core materials is yttrium tetragonal zirconia polycrystal (Y-TZP) (henceforth known as zirconia). The high fracture strength and fracture toughness of zirconia stem from the superior physical properties of partially stabilized zirconia. As for the long-term stability of ceramics, it is closely related to subcritical crack propagation and stress corrosion caused by water in the saliva reacting with glass structure, leading to decomposition of the glass structure¹. However, zirconia cores are glass-free by virtue of their polycrystalline microstructure, and hence display excellent long-term stability².

Presently, zirconia is the only ceramic material that has the potential to substitute the metal used in the porcelain-fused-to-metal technology. Unfortunately, the only problem related to their performance is the inferior adhesion of resin cements to such ceramics³⁻⁵. The zirconia ceramic is composed of a glass-free, polycrystalline microstructure⁶, characterizing it as acid-resistant or non-etchable material⁷. In fact, the reliable adhesion of resin cements to zirconia improves marginal adaptation, prevents microleakage, and increases retention in situations where mechanical retention does not exist⁸.

While clinical evidence is lacking for an optimal method for conditioning reinforced ceramics, existing *in vitro* studies did not reveal reliable results using resin-based cements in combination with zirconia⁹. For this reason, there have been efforts by some manufacturers and researchers to modify the surface properties of zirconia by using various methods of airborne particle abrasion followed by silanization⁹⁻¹³. One suggested method is tribochemical silica coating⁹⁻¹¹. This method is usually applied in dental laboratories using Al_2O_3 particles of 110 μm particle size and coated with silica. The blasting pressure results in silica-coated alumina particles being embedded on the ceramic surface, rendering the silica-modified surface chemically more reactive to the resin through silane coupling agents.

The silane molecules react with water to form silanol groups (-Si-OH) from the corresponding methoxy groups (-Si-O-CH₃). The silanol groups then react further to form a siloxane (-Si-O-Si-O-) network with the silica surface. The most commonly applied silane in dental laboratories and chairside applications is 3-trimethoxysilylpropyl methacrylate (MPS). Any resin composite material that contains methacrylate groups in their composition could therefore be used in conjunction with MPS silanes¹⁴.

However, tribochemical silica coating in dental laboratories runs the risk of contamination when restorations are delivered from the laboratory to the clinic¹⁵. Besides, laboratory airborne particle abrasion techniques have also been recently criticized for

possible subcritical crack growth within zirconia¹⁶⁾. Against this backdrop of unfavorable feedback against laboratory techniques, there arises a greater need for chairside versions.

One alternative to laboratory type of airborne particle abrasion is the application of sand particles using a chairside air abrasion device. This procedure increases the surface area and surface energy for adhesion of resin cements and promotes micro-mechanical retention. Further, it also decreases surface tension, thereby enabling optimal wetting of silanes or adhesive media¹⁷⁾. On the note about improving bonding, the use of metal primers — following chairside airborne particle abrasion — has recently been suggested to act as adhesion promoters¹⁸⁾. Metal primers are easy to apply and do not need complicated and expensive apparatus. Most metal primers are in liquid form, consisting of a monomer to polymerize in the solution. Initially, such metal primers were indicated for the repair of fractured metal-ceramic FPDs with metal exposure. However, subsequently, they were also indicated for conditioning reinforced ceramics including zirconia. Results with metal primers are not always favorable, chiefly because they reach a high bond strength under dry circumstances, but controversial reports emerge concerning their hydrolytic stability^{13,18-20)}.

It has been reported that the chairside version of Silicoater technology (Silicoater™ MD, Heraeus-Kulzer), applied by a handheld device (Silano-Pen or PyrosilPen) and using a flame treatment approach, could deliver reliable adhesion^{21,22)}. In this system, the flame has a reactive zone where tetraethoxy silane is decomposed to silicium-organic fragments ($\text{SiO}_x\text{-C}$ fragments). These fragments cover the substrate surface with an adhesively bound layer (*via* van der Waals forces)²²⁾, whereby this extremely thin layer ($\sim 0.1 \mu\text{m}$) has glass-like properties and can be silanized with MPS silane. Similar to metal primers, this method was initially advised for metal conditioning but has also recently been indicated for ceramics¹³⁾.

One of the most frequently used resin cements in dentistry is Panavia F 2.0. It consists of a bifunctional monomer, 10-methacryloyloxydecyldihydrogen-phosphate (MDP). In the use of dental resin cements, clinicians are constantly faced with the dilemma of whether to follow the cement manufacturers' instructions during the cementing of zirconia FPDs, or to modify the instructions by following the instructions of some other manufacturers that suggest separate surface conditioning protocols. A recent study demonstrated that a modified application mode of Panavia F 2.0 delivered improved adhesion of this cement to zirconia; otherwise, practically no adhesion was achieved when only

the manufacturer's instructions were followed which did not suggest any conditioning method²³⁾.

To make estimations for the long-term survival of cemented zirconia FPDs, information is needed about the hydrolytic stability of adhesion promoters used in conjunction with surface conditioning methods¹²⁾. The objective of this study, therefore, was to evaluate the effects of various surface conditioning methods on the bond strength of dual-cure composite resin cement, which contained MDP as a functional monomer, to zirconia after thermocycling. Since heat application on metals and ceramics increases surface hydroxyl groups^{21,22)}, the tested hypothesis was that the conditioning method based on heat treatment would deliver higher results than those of metal primers with reactive methacrylate groups.

MATERIALS AND METHODS

Disk-shaped (diameter: 15 mm, thickness: 2 mm) zirconia ceramics (Lava™, 3M ESPE, Seefeld, Germany) were embedded in polyethylene molds using polymethyl methacrylate (PMMA) (Condular AG, Wager, Switzerland), with one side of the disk exposed for cement bonding. They were ground finished with 1200-grit silicone carbide abrasive under water cooling (Struers RotoPol 11, Struers A/S, Rodovre, Denmark), and then ultrasonically cleaned in distilled water for three minutes.

Subsequently, the disks were randomly divided into four groups according to the surface conditioning method to be applied ($N=40$, $n=10$ per group): Group 1—Chairside airborne particle abrasion with $50\text{-}\mu\text{m}$ Al_2O_3 + Alloy Primer; Group 2—Airborne particle abrasion with $50\text{-}\mu\text{m}$ Al_2O_3 + Cesead II Opaque Primer; Group 3—Airborne particle abrasion with $50\text{-}\mu\text{m}$ Al_2O_3 + Silano-Pen + silane coupling agent; Group 4—Laboratory tribochemical silica coating ($110\text{-}\mu\text{m}$ Al_2O_3 + $110\text{-}\mu\text{m}$ SiO_x) + silane coupling agent (ESPE-Sil).

Table 1 shows the brand names, compositions, batch numbers, and manufacturers of the silane coupling agents, metal primers, and resin cement used in this study. Table 2 summarizes the experimental groups, surface conditioning methods, and procedures according to each manufacturer's instructions.

Surface conditioning methods

The following surface conditioning methods were employed per experimental group:

Group 1 — Chairside airborne particle abrasion with $50\text{-}\mu\text{m}$ alumina particles (Korox, Bego, Bremen, Germany) using an intraoral air abrasion device (Dento-Prep™, Ronvig, Denmark). The nozzle was held perpendicular to the surface from a distance of approximately 10 mm for 15 seconds/ cm^2 at a pressure of 2.3 bar. The substrate surface was rinsed

Table 1 Experimental groups, surface conditioning methods and procedures according to each manufacturer's instructions

Brand name	Composition	Batch numbers	Manufacturer
ESPE®-Sil	3-methacryloxypropyltrimethoxysilane(MPS) in ethanol	68411	3M ESPE, Seefeld, Germany
Alloy Primer	6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol, -dithione tautomer (VTD) and 10-methacryloxydecyl dihydrogen phosphate (MDP) in acetone	00494A	Kuraray Medical, Inc., Osaka, Japan
Cesead II Opaque Primer	10-methacryloxydecyl dihydrogen phosphate (MDP) and initiator in solvent	00106B	Kuraray Medical, Inc., Osaka, Japan
Haftvermittler	3-methacryloxypropyltrimethoxysilane (MPS), Ethanol	11	Bredent, Senden, Germany
Panavia F 2.0	10-methacryloxydecyl dihydrogen-phosphate (MDP) Paste A: BPEDMA/MDP/DMA Paste B: Al-Ba-B-Si glass/silica containing composite	41144	Kuraray Medical, Inc., Osaka, Japan

Table 2 Brand names, compositions, batch numbers, and manufacturers of the silane coupling agents, metal primers, and resin cement

Group	Surface conditioning	Silane coupling agent/metal primers ³	Resin cement
Gr1	Chairside	Alloy Primer	Panavia F 2.0
Gr2	air-abrasion	Cesead II Opaque Primer	Panavia F 2.0
Gr3	Chairside air-abrasion +heat treatment	Haftvermittler	Panavia F 2.0
Gr4	Laboratory tribochemical silica coating	ESPE®-Sil	Panavia F 2.0

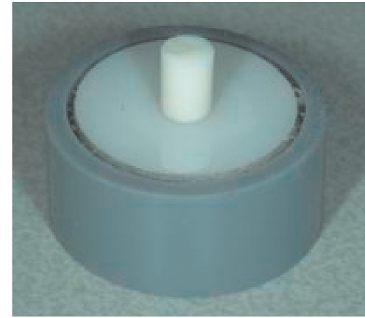


Fig. 1 The Y-TZP disk specimen embedded in PMMA mold with the cementation surface exposed.

for 20 seconds and air-dried for five seconds. Alloy Primer was applied to the surface with a disposable brush.

Group 2 — Chairside airborne particle abrasion with 50- μm alumina particles (Korox, Bego, Bremen, Germany) using an intraoral air abrasion device (Dento-Prep™, Ronvig, Denmark). The nozzle was held perpendicular to the surface from a distance of approximately 10 mm for 15 seconds/cm² at a pressure of 2.3 bar. The substrate surface was rinsed for 20 seconds and air-dried for five seconds. Cesead II Opaque Primer was applied to the surface with a disposable brush and left for 30 seconds for its reaction to be completed.

Group 3 — Chairside airborne particle abrasion with 50- μm alumina particles (Korox, Bego, Bremen, Germany) using an intraoral air abrasion device (Dento-Prep™, Ronvig, Denmark). The nozzle was held perpendicular to the surface from a distance of approximately 10 mm for 15 seconds/cm² at a pressure of 2.3 bar. The substrate surface was rinsed for 20 seconds and air-dried for five seconds. Heat treatment was achieved by applying the flame of Silano-Pen (Bredent, Senden, Germany) for 5 seconds/cm² at the surface. The surface was then

left to cool down at room temperature. Following which, the corresponding silane (Haftvermittler, Bredent) was applied to the surface with a disposable brush and left for three minutes for its reaction to be completed.

Group 4 — Laboratory silica coating (Rocatec, 3M ESPE, Seefeld, Germany) first with 110- μm alumina particles (Rocatec Pre, 3M ESPE, Seefeld, Germany), followed by 110- μm alumina particles coated with silica (Rocatec Plus). The nozzle was held perpendicular to the surface from a distance of approximately 10 mm for 13 seconds/cm² at a pressure of 2.8 bar. MPS silane (ESPE-Sil, 3M ESPE) was applied to the surface with a disposable brush and left for five minutes for its reaction to be completed.

Bonding procedure

A dual-cure resin cement (Panavia F 2.0, Kuraray Medical Inc., Osaka, Japan) was mixed and applied in a standardized way by the same operator on the specimens embedded in translucent polyethylene molds (inner diameter: 3.6 mm, height: 5 mm). The cement was mixed according to manufacturer's instructions, packed into the mold incrementally

using a hand instrument, and light-polymerized for 40 seconds from each direction (Demetron LC, SDS Kerr, Orange, CA, USA; light intensity: 800 mW/cm²). The polyethylene molds were then gently removed (Fig. 1). A polyethyleneglycol/glycerin gel to prevent oxygen inhibition (Oxyguard II, Kuraray Medical Inc., Osaka, Japan) was applied around the margins of the cement-ceramic interface, and the specimens were further light-polymerized for another 40 seconds. After three minutes, the glycerin gel was rinsed off thoroughly.

All specimens were kept at 37°C for 24 hours and then subjected to thermocycling for 6,000 cycles between 5 and 55°C in deionized grade 3 water (Willytech, Gräfelfing, Germany). The dwelling time at each temperature was 30 seconds, and the transfer time from one bath to the other was two seconds.

Shear bond test

The specimens were mounted in the jig of a universal testing machine (Zwick ROELL Z2.5 MA 18-1-3/7 Ulm, Germany), and load was applied to the adhesive interface until failure occurred (crosshead speed: 1.0 mm/min). The maximum force to produce failure was recorded (MPa) using a corresponding software.

Statistical analysis

Statistical analysis was performed using SAS System for Windows, release 8.02/2001 (Cary, NC, USA). Bond strength data were analyzed by one-way analysis of variance (ANOVA). P values less than 0.05 were considered to be statistically significant in all tests.

RESULTS

No significant differences were found among the four groups (Groups 1 to 4: 8.43 ± 1.3 , 8.98 ± 3.6 , 12.02 ± 6.7 , and 8.23 ± 3.8 MPa respectively) ($p =$

0.1357) (Fig. 2).

DISCUSSION

It has been previously demonstrated that the bond strength of resin-based materials to acid-resistant ceramics, especially zirconia, is neither durable nor stable³⁻⁵. Such an inferior bonding performance thus poses a formidable challenge to the clinicians, especially when bonding non-retentive zirconia FPDs. Against this background, this study set out to evaluate the influence of various surface conditioning methods on improving bond strength. According to the results of the present study for both laboratory and chairside surface conditioning methods, mean shear bond strengths to the zirconia material tested were not significantly different.

Some studies have evaluated ceramics with different microstructures, and reported that high-strength ceramics are compact materials that are difficult to grit-blast²⁴. On this note, one would expect that air abrasion with bigger particle size would produce higher surface roughness, and hence higher micromechanical retention. The laboratory airborne particle abrasion system used in this study, Rocatec, was based on application of 110- μ m alumina followed by 110- μ m silica-coated alumina particles. However, higher micromechanical retention was not achieved, since its shear bond strength result was not significantly different from the other experimental groups where surface roughening was achieved with 50- μ m alumina particles. One possible explanation could be the phenomenon of less wettability and contact angle²⁵ between the silane coupling agent and the deep grooves on the zirconia surface, which occurred after air abrasion with bigger particle size. This finding also indicated the importance of chemical adhesion as opposed to micromechanical retention.

In the present study, no significant differences were obtained between the laboratory and chairside conditioning methods. However, in a clinical setting, chairside air abrasion with small particle size should be considered due to possible material loss with bigger particles²⁶ — especially at the margins of restorations. Aggressive air abrasion could lead to ditching between the resin cement and zirconia substrate surface, when bigger grain-sized particles are used during airborne particle abrasion.

Metal primers are easy to apply, and saddled with other advantages of good price-performance and no need for proprietary apparatus. They are usually supplied as single-liquid primers composed of a polymerizable monomer in a suitable solvent. These products are often called primers despite the fact that they are also coupling agents. Typically, these systems are considered as simplified chairside

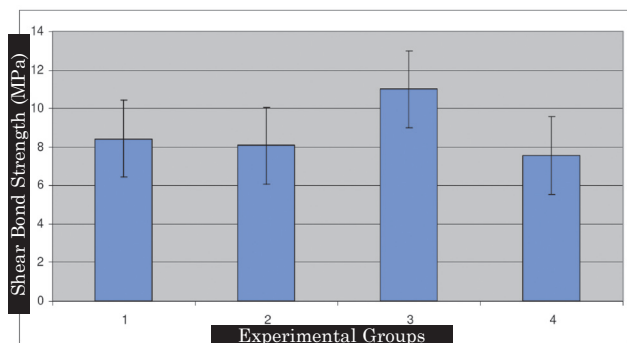


Fig. 2 Shear bond strengths (MPa) of the experimental groups. Vertical lines represent the standard deviations. See Table 2 for group names

applications. However, a careful study of the manufacturer's instructions would reveal that prior to metal primer application, Al_2O_3 airborne particle abrasion is also required. Therefore, bond strength is not derived purely from the metal primer, but that the need for airborne particle abrasion remains. Although these primers deliver high bond strength results at dry conditions, a reduction of 30–90% after thermocycling has been noted¹³.

In the present study, Cesead II Opaque Primer and Alloy Primer were used as metal primers, whereby both contained MDP monomer. In a previous study¹⁹, significant differences were observed between Cesead II Opaque Primer and Alloy Primer in both dry and thermocycled conditions, with the latter leading to significant decrease after thermocycling when they were applied on titanium. This was attributed to differences between the two materials in terms of monomer composition, initiator, and solvent. Since MDP monomer has a polymerizable methacryloyl group and a dihydrogen phosphate functional group, it is very important for phosphate monomers to have a hydrophobic spacing component such as the decyl group of the MDP. In the present study, the reaction between the metal primers tested and the hydroxyl groups on zirconia seemed to be different from those of the alloys.

Of late, there have been some developments on zirconate coupling agents¹³. Zirconate coupling agents might slightly bond with the hydroxyl groups on the zirconia surface. Shear bond strength has been reported to improve before thermal cycling, but significant decrease was observed after thermal cycling depending on the silane concentration. It was thus evident that more studies need to be conducted in order to develop silane coupling agents with better chemical affinity to zirconium oxides, and which at the same time exhibit stability hydrolytically.

The phosphate ester monomer of MDP, on the other hand, was reported to bond directly to metal oxides such as chromium, nickel, aluminum, tin, titanium, and zirconium oxides²⁷. Conversely, with zirconia ceramics, Valandro *et al.*⁵ found — by means of a microtensile test setup — inferior Panavia-zirconia adhesion results, with dramatic decrease after 12,000 times of thermocycling and/or 300 days of long-term water storage. Reactions might have formed between the hydroxyl groups in MDP monomer and the hydroxyl groups on zirconia ceramic surface, but these chemical reactions did not maintain their strength after thermal cycling. In the present study, the aging effect on resin cement adhesion was investigated with 6,000 times of thermal cycling in water. However, to evaluate the effect of long-term aging on the hydrolytic stability of the material combinations tested in this study, the reaction mechanism of metal primers on zirconia

requires further surface analysis.

It was expected that heat application would increase the surface hydroxyl groups, hence rendering the zirconia surface more reactive for silane coupling agent. However, this was not so as no significant differences were observed between Group 3 and the other groups. Therefore, the hypothesis was rejected. One possible explanation could be the uncontrolled firing process with Silano-Pen — a hand-held instrument, such that the hydroxyl groups were destroyed at room temperature. Other possible reasons could be simply due to a mismatch between different brands of materials, namely the phosphate monomer-containing resin cement was not adequate for the silane coupling agents used in this study. Therefore, further research is needed with other commercial silanes and metal primers with and without surface conditioning.

Presently, satisfactory bond strength values of resin cement to high-strength ceramics are yet to be determined for clinically successful performance. Nonetheless, the bond values obtained for the ceramic tested in this study could be considered sufficient with all the herein-evaluated surface conditioning methods. As for the bonding of ceramics to tooth substance, it is based on the adhesion of luting cement to the ceramic substrate together with the adhesion of luting cement to enamel and/or dentin. It has recently been reported that the marginal gaps with zirconia ceramic crowns (Procera) varied between 7 and 529 μm ²⁸, indicating the important role of adhesive cementation.

In this study, shear test was employed. Although it is often used in dental research, shear bond strength test has been previously shown to create uneven stress distribution at the adhesive interface²⁹. In this connection, results of this study should also be verified with microtensile strength test.

CONCLUSIONS

In the present study, chairside conditioning methods were based on airborne particle abrasion with 50- μm Al_2O_3 followed by a metal primer or heat application followed by silane treatment. The laboratory method employed was airborne particle abrasion with 110- μm silica-coated alumina followed by silanization. After 6,000 cycles of thermal cycling, no significant differences in the adhesion of resin cement to zirconia were found upon comparing the chairside methods against the laboratory method.

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