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# Effects of Multipurpose, Universal Adhesives on Resin Bonding to Zirconia Ceramic

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### Clinical Relevance

Single Bond Universal and All-Bond Universal significantly improved the bond strength of resin cement to zirconia ceramic compared with Alloy Primer.


## SUMMARY

**This study evaluated the effects of single-bottle, multipurpose, universal adhesives on the bond strength of resin cement to zirconia ceramic. Polished zirconia ceramic (Cercon base) discs were randomly divided into four groups (n=40) according to the applied surface-conditioning agent: Single Bond 2, Single Bond Universal, All-Bond Universal, and Alloy Primer. Cured composite cylinders (Ø 0.8 mm × 1 mm) were cemented to the conditioned zirconia specimens with resin cement (RelyX ARC). The bonded specimens were subjected to a microshear bond-strength test after 24 hours of water storage and after 10,000 cycles of thermocycling. The surface-conditioning agent significantly influenced the bond strength ($p<0.05$). Single Bond Universal showed the highest initial bond strength (37.7 ± 5.1 MPa), followed by All-Bond Universal (31.3 ± 5.6 MPa), Alloy Primer (26.9 ± 5.1 MPa), and Single Bond 2 (8.5 ± 4.6 MPa). Artificial aging significantly reduced the bond strengths of all the test groups ($p<0.05$). After 10,000 cycles of thermocycling, All-Bond Universal showed the highest bond-strength value (26.9 ± 6.4 MPa). Regardless of artificial aging, Single Bond Universal and All-Bond Universal showed significantly higher bond strengths than Alloy Primer, a conventional metal primer.**


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## INTRODUCTION

As patient demand for esthetic restorations has increased, zirconia ceramics have been frequently used as frameworks for metal-free restorations.[1] The development of computer-aided design and manufacturing technology has contributed to the popularity of zirconia ceramics as substitutes for dental metal alloys, which are generally processed by the lost wax technique. Zirconia restorations can be cemented with conventional cements because of their superior mechanical properties. However, a wide variety of clinical applications, such as partial coverage coronal restorations, Maryland-type resin-bonded fixed partial dentures, and intracoronal restorations, require a long-term durable bond to zirconia ceramics.

Numerous studies have proposed various methods for modifying the zirconia surface to improve resin bonding, including plasma spraying,[2] glass micro-pearls,[3] selective infiltration etching,[4] and a vapor phase deposition technique.[5] However, these methods require further investigation for clinical application. Although there is no consensus on the most suitable surface treatment method for zirconia, the combination of airborne-particle abrasion with $Al_2O_3$ particles for micromechanical interlocking and conditioning with a primer containing phosphate monomer for chemical bonding has been recommended.[6–10]

Commercially available surface-conditioning agents for zirconia generally contain functional monomers that are derived from the reaction of methacrylic acid with phosphoric or carboxylic acid.[9,11-13] One agent, 10-methacryloyloxydecyl dihydrogen phosphate (MDP; Figure 1), has been shown to provide chemical bonds between methacrylate-based materials and zirconia ceramics.[7,9,11,14] MDP was first introduced by Kuraray Medical Inc (Okayama, Japan) and has been included in the resin cements of Panavia, Alloy Primer, Clearfil Ceramic Primer, and Clearfil SE Bond. Recently, other manufacturers have introduced new MDP-containing adhesives to the dental market. These single-bottle adhesives are called "universal" adhesives because they can be used in etch-and-rinse or self-etch modes on the tooth substrates.[15,16] In addition, the manufacturers have suggested that these adhesives promote the bonding of methacrylate-based materials to various indirect restorative substrates, such as zirconia and dental non–precious metal alloys, with no need for an additional primer. However, little information is available about how these universal adhesives affect resin bonding to zirconia.

The purpose of this study was to evaluate the effects of single-bottle, multipurpose, universal adhesives on the bond strength of resin cement to zirconia ceramic compared with a conventional MDP-containing primer. The null hypothesis tested was that there would be no differences in bond strength or durability from a zirconia ceramic with different surface-conditioning agents. The bonded specimens were subjected to the microshear bond-strength test after 24 hours of water storage and after 10,000 cycles of thermocycling.

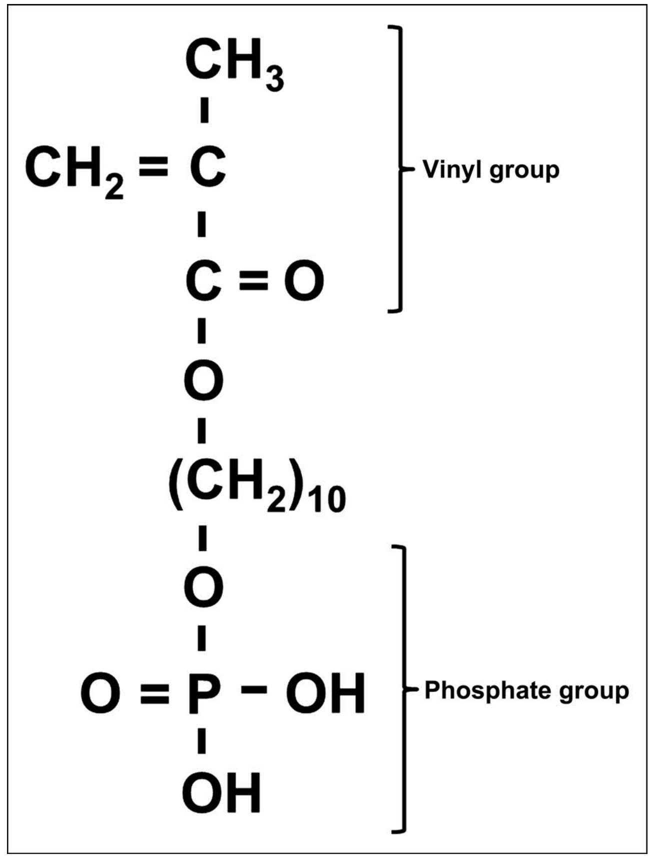


Figure 1. *Amphiphilic structure of the MDP monomer.*

## METHODS AND MATERIALS

A partially sintered zirconia block (Cercon base, DeguDent, Hanau, Germany) was sectioned to produce 160 square specimens, which were then sintered according to the manufacturer's instructions. The fully sintered specimens (10 mm × 10 mm × 3 mm) were embedded in acrylic resin blocks. The zirconia surface was sequentially polished with up to 600-grit silicon carbide paper using an automatic polishing machine (Rotopol-V, Struers, Ballerup, Denmark) under water cooling and then underwent ultrasonic cleaning in isopropyl alcohol for 3 minutes. The specimens were randomly divided into four groups of 40 specimens each according to the applied surface-conditioning agent: Single Bond 2, Single

Table 1: *Surface Conditioning Agents and Resin Cement Used in This Study and Their Application Procedures*

| Product (Batch No.) | Composition | Manufacturer | Application Procedure |
|---|---|---|---|
| Single Bond 2 (N412273) | bis-GMA, HEMA, DMA, methacrylate functional copolymer, filler, ethanol, water, photoinitiator | 3M ESPE, St Paul, MN, USA | 1. Apply the adhesive |
| | | | 2. Allow it to react for 20 s |
| | | | 3. Gently air dry for 5 s |
| Single Bond Universal (502225) | MDP, bis-GMA HEMA, DMA, methacrylate functional copolymer, filler, ethanol, water, initiators, silane | 3M ESPE, St Paul, MN, USA | 1. Apply the adhesive |
| | | | 2. Allow it to react for 20 s |
| | | | 3. Gently air dry for 5 s |
| All-Bond Universal (1200013674) | MDP, bis-GMA, HEMA, ethanol, water, initiators | Bisco Inc, Schaumburg, IL, USA | 1. Apply the adhesive |
| | | | 2. Air dry |
| | | | 3. Light cure for 10 s |
| Alloy Primer (00436A) | VBATDT, MDP, acetone | Kuraray Medical Inc, Okayama, Japan | 1. Apply the primer |
| | | | 2. Leave it for drying |
| RelyX ARC resin cement (N441122) | bis-GMA, TEG-DMA, zirconia/silica filler, DMA, amine, photoinitiator, BP, pigment | 3M ESPE, St Paul, MN, USA | 1. Dispense the cement onto a mixing pad and mix for 10 s |
| | | | 2. Apply a thin layer of the cement to the bonding surface |

*Abbreviations: bis-GMA, bisphenol A diglycidyl ethermethacrylate; BP, benzoyl peroxide; DMA, dimethacrylate; HEMA, hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEG-DMA, triethylene glycol dimethacrylate; VBATDT, 6-(4-vinylbenzyl-n-propyl amino)-1,3,5-triazine-2,4-dithione.*

Bond Universal, All-Bond Universal, and Alloy Primer. The four different surface-conditioning agents used in this study are summarized in Table 1.

Polyethylene tubes (Tygon R-3603 tubing, Saint-Gobain Co, Courbevoie, France) were used to fabricate composite cylinders (0.8 mm in diameter and 1 mm in height). The tube was filled with composite resin (Filtek Z-250, 3M ESPE, St Paul, MN, USA) and light-polymerized from four directions for 20 seconds per side with a light-emitting diode (LED) curing unit (Elipar FreeLight 2, 3M ESPE). The light intensity of 800 $mW/cm^2$ was frequently monitored with a radiometer (Demetron 100, Demetron Research Co, Danbury, CT, USA). After light-polymerization, the composite cylinder was removed from the tube.

Three different MDP-containing agents were applied to the polished zirconia specimens strictly in accordance with the respective manufacturers' instructions as summarized in Table 1. Single Bond 2 was used as a negative control for the MDP-containing agents and applied according to the instructions of Single Bond Universal. Resin cement (RelyX ARC, 3M ESPE) was mixed and applied onto the composite cylinder, which was then placed on the zirconia specimen under a fixed load of 0.4 N. After excess resin cement was removed with a microbrush, glycerin gel was applied around the bonded interface. The resin cement was light-polymerized from four directions for 20 seconds per side with the LED curing unit. After 30 minutes at room temperature, the bonded specimens were stored in distilled water at 37°C for 24 hours. Next, 20 specimens of each group were immediately subjected to the bond-strength test. The remaining 20 specimens of each group were subjected to thermocycling for 10,000 cycles between 5°C and 55°C with a 25-second dwell time before the bond-strength test.

The microshear bond-strength test was performed with a universal testing machine (LF Plus, Lloyd Instruments, Fareham, UK). The experimental setup for the test is schematically shown in Figure 2. A stainless steel orthodontic wire (0.2 mm in diameter) was used to apply a shear force to the bonded interface. The wire, which was attached to the load cell, was looped around the composite cylinder as close as possible to the bonded interface. The shear force was applied at a crosshead speed of 0.5 mm/min until failure occurred.

The fractured interfaces of the specimens were examined with a stereomicroscope (SZ4045, Olympus Optical Co Ltd, Tokyo, Japan) at 40× magnification to determine the failure mode. The failure mode was classified as "adhesive failure" when it occurred between the zirconia ceramic and the resin cement and was classified as "mixed failure" when the adhesive failure and cohesive fracture occurred simultaneously within the resin cement. In cases of mixed failure, the surface of the zirconia was partly covered by the remaining resin cement.

Bond-strength data were analyzed using statistical software (SPSS 18.0, SPSS Inc, Chicago, IL, USA). One-way analysis of variance, followed by the

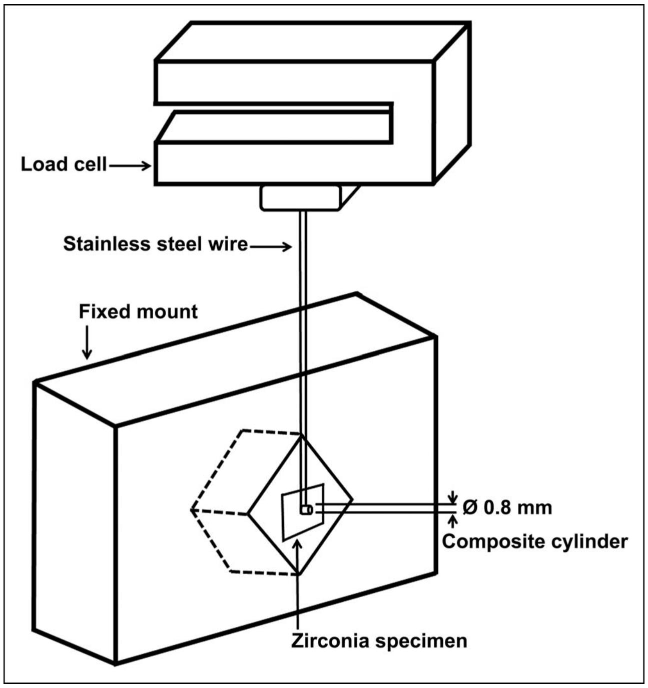


Figure 2. *Schematic drawing of the microshear bond-strength test.*

Tukey honestly significant difference test for *post hoc* pairwise comparisons, was performed to assess the differences among the surface-conditioning agents. For each agent, the effect of thermocycling on bond strength was investigated using a two-sample *t*-test. The analyses were performed at a significance level of $\alpha$=0.05.

## RESULTS

The mean bond-strength values and standard deviations are summarized in Table 2. Single Bond Universal, All-Bond Universal, and Alloy Primer significantly improved the bond strength of resin cement to zirconia compared with Single Bond 2 ($p<0.05$). The universal adhesives (Single Bond Universal and All-Bond Universal) showed significantly higher bond strengths than the conventional MDP-containing primer (Alloy Primer) regardless of the storage condition ($p<0.05$).

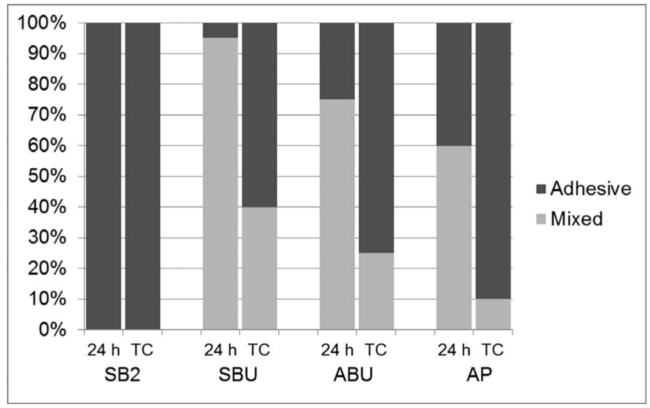


Figure 3. *Percentage distribution of failure modes after 24 hours or 10,000 cycles of thermocycling. Abbreviations: SB2, Single Bond 2; SBU, Single Bond Universal; ABU, All-Bond Universal; AP, Alloy Primer.*

Before thermocycling, Single Bond Universal showed the highest bond-strength value ($p<0.05$). The bond strengths for all of the conditioning agents were significantly reduced after thermocycling ($p<0.05$). All-Bond Universal showed a significantly higher bond strength than Single Bond Universal after thermocycling ($p<0.05$).

The distribution of failure modes after the microshear bond-strength test is presented in Figure 3. Regardless of the storage condition, all of the specimens for Single Bond 2 were classified as adhesive failure after fracture. With the three MDP-containing agents, mixed failures (60% to 95%) predominated before thermocycling, but adhesive failures (60% to 90%) occurred more frequently than mixed failures (10% to 40%) after 10,000 cycles of thermocycling.

## DISCUSSION

This study examined the effects of multipurpose, universal adhesives on the bond strength of resin cement to zirconia ceramic and compared them to

Table 2: *Means (Standard Deviations) of Microshear Bond Strength (in MPa) of Resin Cement to Zirconia Ceramic with Different Surface Conditioning Agents (n=20)*

| Product | Feature of Agent | 24 h[a] | 10,000 Cycles of Thermocycling[a] |
|---|---|---|---|
| Single Bond 2 | Conventional single-bottle adhesive | 8.5 (4.6) $^{D}_{a}$ | 0.3 (0.1) $^{D}_{b}$ |
| Single Bond Universal | MDP-containing single-bottle adhesive | 37.7 (5.1) $^{A}_{a}$ | 20.7 (6.4) $^{B}_{b}$ |
| All-Bond Universal | MDP-containing single-bottle adhesive | 31.3 (5.6) $^{B}_{a}$ | 26.9 (6.4) $^{A}_{b}$ |
| Alloy Primer | Conventional MDP-containing primer | 26.9 (5.1) $^{C}_{a}$ | 10.7 (4.2) $^{C}_{b}$ |

*Abbreviation: MDP, 10-methacryloyloxydecyl dihydrogen phosphate.*
[a] *Different superscript uppercase letters indicate significant differences between data within the same column ($p<0.05$). Different subscript lowercase letters indicate significant differences between data within the same row ($p<0.05$).*

that of a conventional MDP-containing primer. Resin cements containing phosphate monomers have been shown to improve resin bonding to zirconia ceramic without any additional surface treatment.[6,8,17,18] Because the focus of the present study was on evaluating the performance of the MDP-containing adhesive agents, RelyX ARC, which does not contain any functional phosphate monomer, was selected as a luting cement and allowed for the differentiation of the effects of the surface-conditioning agents. The universal adhesives, namely Single Bond Universal and All-Bond Universal, produced higher initial bond strengths and maintained higher bond strengths after aging than did Alloy Primer.

The microshear bond-strength test was selected for this study. Various bond-strength tests, including shear,[2,3,8,9,13,14,19-21] tensile,[6,10] microshear,[18] and microtensile methods,[4,5,22] have been used to evaluate the effects of surface treatments on resin-zirconia bonding. Microtensile bond-strength tests allow for a more homogeneous distribution of stress and for the evaluation of the bond strength of a small region of interest within a substrate.[23] However, it is extremely difficult to section bonded zirconia specimens into microbeams without damaging the bonded interface because of the superior mechanical properties of zirconia. In addition, a high incidence of premature failure in the specimens has been reported with the microtensile bond-strength test, which would decrease the discriminative power of the test.[24]

Shear bond-strength tests have been widely used in the studies on resin-zirconia bonding.[2,3,8,9,13,14,19-21] However, the shear bond-strength test has been criticized for nonhomogeneous stress distribution at the bonded interface, inducing cohesive failures within the substrates and misinterpretations of the results.[25,26] However, these concerns were reduced because cohesive failures within zirconia have been rarely reported.[3,21] In the present study, the microshear bond-strength test allowed for the differentiation of the effects of the surface-conditioning agents with relatively small standard deviations. The microshear bond-strength test maximizes shear stresses at the bonded interface and gives precise results due to the reduced bonding area.[27] The test protocol for the microshear bond-strength test is simpler than that of the microtensile bond-strength test.

In the present study, the failure-mode distribution after the microshear bond-strength test was in line with the bond-strength data. All of the specimens for Single Bond 2, which showed the lowest mean bond strength, presented adhesive failures regardless of the storage condition. The specimens for the MDP-containing agents presented primarily mixed failures before thermocycling, whereas adhesive failures increased after thermocycling. The differences in the failure-mode distribution can be explained by the reduced bond strengths after thermocycling. Cohesive failures within the composite cylinders or the zirconia specimens were not observed, which implies that the interface between the resin cement and zirconia was the weakest link in the structure.

MDP chemically bonds to non–precious metals and tooth substrates.[28-30] MDP has an amphiphilic structure, with the vinyl group as the hydrophobic moiety and the phosphate group as the hydrophilic moiety (Figure 1). The vinyl group can copolymerize with the resin monomer of the resin-based materials applied later. MDP has also been shown to be effective in improving the resin bonding to zirconia ceramics.[6,8,10,11,13,14] It has been assumed that the hydroxyl groups of the phosphate moiety in MDP interact with the hydroxyl groups on the zirconia surface through Van der Waals forces or hydrogen bonds.[11] In the present study, the universal adhesives containing MDP showed significantly higher bond strengths than Single Bond 2. According to the manufacturer, Single Bond Universal differs from Single Bond 2 primarily in the addition of MDP and silane. There was a need to differentiate between the effects of conventional adhesive formulation and MDP on bond strength. For this, Single Bond 2 served as the negative control for the universal adhesives. Although Single Bond 2 showed the lowest bond strength among the groups, it seems that Single Bond 2 slightly improves the initial bond strength of RelyX ARC. In our preliminary test the bond strength of RelyX ARC to polished zirconia was almost zero without any primer or adhesive. The conventional adhesive formulation would allow the resin cement to flow more easily and adapt to the zirconia surface. However, the bond strength for Single Bond 2 was significantly lower than those of the universal adhesives. The higher bond strengths with universal adhesives can be explained by the addition of MDP to conventional adhesive formulations.

Single Bond Universal showed the highest initial bond strength. In contrast to All-Bond Universal, Single Bond Universal contains a silane in addition to MDP. The silane cannot contribute to the chemical bond to zirconia because zirconia lacks silica. However, the silane could increase the wettability of the zirconia surface and as a result improve the initial bond strength.[11,31] However, in spite of the high initial bond strength, Single Bond Universal showed a significantly lower bond strength than All-Bond Universal after thermocycling. The silane could increase the hydrophilicity of Single Bond Universal, thereby predisposing the adhesive layer to hydrolytic degradation.[32] This assumption needs further investigation.

Single Bond Universal and All-Bond Universal showed significantly higher bond strengths than Alloy Primer, a conventional MDP-containing primer. Although Alloy Primer was originally designed to enhance the bond between resin-based materials and dental metal alloys, it has provided a superior bond to zirconia compared with other primers containing phosphate monomer.[10,13,17] In contrast to Alloy Primer, the universal adhesives have resin adhesive components, which could allow the resin cement to flow more easily and strengthen the interfacial layer through copolymerizing with the resin cement. Based on the result of the present study, separate primers for conditioning the zirconia surface, such as Alloy Primer, can be substituted with these universal adhesives. The universal adhesives have also shown comparable performance on the tooth substrates compared with conventional adhesives.[15,16] The clinical procedure of cementing zirconia restorations could be simpler and more efficient with the single-bottle universal adhesives.

In the present study, the polished zirconia specimens were used without airborne-particle abrasion in order to focus on the role of the surface-conditioning agent. Airborne-particle abrasion is a crucial factor for a durable bond to zirconia.[6,7,10,13,14] Airborne-particle abrasion increases the surface roughness and area, thereby improving micromechanical retention. However, airborne-particle abrasion has a limited effect in improving the bond strength of resin cement to zirconia.[6,9,10] Another consideration is that airborne-particle abrasion with $Al_2O_3$ particles can induce phase transition and produce microcracks within the zirconia surface, which influence the mechanical properties of zirconia.[20,33,34] Özcan and others[34] reported that air abrasion with 50 μm $Al_2O_3$ particles at 2.8 bar pressure decreased the biaxial flexural strength of the zirconia. Large particles at a high blasting pressure increased surface roughness but did not result in a higher bond strength of resin cement to zirconia.[9,20,35] The initial bonding to zirconia seems to be mainly promoted by chemical bonds. However, previous long-term studies have shown that the chemical bonds are not water resistant,[6,7,10,13,14] which is in accordance with the present results. Therefore, airborne-particle abrasion, which has a surface activation and cleaning effect, is required to promote chemical bonds and increase bond durability.[10] Airborne-particle abrasion with silica-coated $Al_2O_3$ particles at reduced pressure has been recommended for producing a durable bond to zirconia with minimal influence on the mechanical properties of the material.[33,34,36] Further investigations that include various airborne-particle abrasion protocols should be conducted to achieve a durable bond to zirconia with the universal adhesives.

## CONCLUSION

Within the limitations of the present study, the new universal adhesives showed better performance in terms of the bond strength of resin cement to zirconia ceramic compared with a conventional MDP-containing primer. Although artificial aging significantly reduced the bond strengths for all of the conditioning agents, the bond strengths of Single Bond Universal and All-Bond Universal remained higher than that of Alloy Primer. All-bond Universal presented the highest bond strength after thermocycling. The universal adhesives containing the MDP functional monomer could make the clinical procedure of cementing zirconia restorations simpler and more efficient.

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#### Conflict of Interest

The authors 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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