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Evaluation of the Microshear Bond Strength of MDP-containing and Non–MDP-containing Self-adhesive Resin Cement on Zirconia Restoration

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Clinical Relevance

To enhance the adhesion between zirconia restorations and self-adhesive resin cements (SARC), clinicians need to consider the use of zirconia primer. If using MDP-containing SARC, then zirconia primer is not necessary. If using non–MDP-containing SARC, then zirconia primer is recommended.

SUMMARY

Objectives: The purpose of this study was to measure the microshear bond strength (μSBS) of four different self-adhesive resin cements with/without 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-containing primer to zirconium ceramics and to evaluate the effect of zirconia primers on these self-adhesive resin cements (SARCs).

Methods and Materials: Zirconia blocks (20 × 20 × 8 mm^3) were prepared and divided into eight groups (n=20). They were sandblasted (50 μm Al_2O_3) and treated as follows: no primer or primer (Z-Primer Plus). Four self-adhesive resin cements (MDP-containing: Permacem 2.0 [PC], Clearfil SA luting [CS]; non–MDP-containing: Rely-X U200 [RU], Maxcem Elite [ME]) were bonded to the zirconia surface. After thermocycling, a μSBS test was performed. The failure mode was analyzed using light microscopy. Statistical analysis of μSBS was performed using one-way analysis of variance and two-sample *t*-test with post hoc Tukey test. The loss rate was evaluated using the Fisher's exact test and χ^2 test with *post hoc* Tukey test ($p<0.05$).

Results: Within the no primer groups, the PC and CS groups showed higher bond strength than the RU and ME groups. Comparing the μSBS of the no primer and primer groups in the same SARCs, the RU/P group was higher

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than the RU group, and the ME/P group was higher than the ME group. No significant difference was observed between the PC and PC/P groups and between the CS and CS/P groups.

Conclusions: Non–MDP-containing SARC showed the increased bonding value with MDP-containing primer to zirconia ceramics. The bond strength of MDP-containing SARCs was not affected significantly by the use of zirconia primer.

INTRODUCTION

In recent years, zirconia-based ceramics have been widely used in dentistry, including inlays, crowns, and fixed dental prostheses (FDPs), particularly with the development of dental computer-assisted design/computer-assisted manufacturing (CAD/CAM) systems. Yttria-stabilized tetragonal zirconium polycrystal (Y-TZP) ceramics demonstrate superior mechanical properties, such as increased hardness and fracture toughness, in addition to its esthetic properties and biocompatibility.[1]

Compared to silica-based ceramics, Y-TZP ceramics show difficulty in forming reliable and durable bonds to the resin cements. The highly-crystalline structures of zirconia ceramics provide an acid-resistance, and the silica- and glass-free structure of zirconia cannot form a siloxane network with silane coupling agents.[2] Therefore, conventional bonding protocols (hydrofluoric acid etching and silanization) are not effective between Y-TZP and resin cements.[2] To resolve this bonding problem of zirconia restorations, alternative surface treatment methods have been tried using mechanical and chemical approaches. These approaches consist of surface grinding, airborne-particle abrasion, laser application, selective infiltration etching, and priming with acidic adhesive monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and 4-methacryloyloxyethy trimellitate anhydride (4-META).[2,3] It has been reported that micromechanical bonding using airborne-particle abrasion followed by chemical bonding using an MDP monomer is effective.[4,5]

MDP is a functional adhesive monomer used in dentistry. It has been suggested that phosphate groups of MDP monomers chemically react with the hydroxyl groups of the zirconia ceramics. Consequently, the MDP monomer increases the bond strength for a longer period.[6,7] It has been reported that the application of MDP-containing bonding and luting systems provide higher bonding values to zirconia than other systems.[8-11] There are several ways to apply MDP monomers for zirconia bonding. The first method is to use MDP-containing adhesives.[9,12] Commercially available "universal" or "self-etch" adhesives, which contain MDP monomers, have been launched. The second method is to use zirconia primer, which contains MDP as an ingredient.[8,9] The third method is the use of MDP-containing resin cements.[8,10,11] When these MDP-containing products are used in combination, such as resin cement after adhesive application or resin cement after primer application, reports on the overlapped effect of MDP monomers are rare.

In recent years, self-adhesive resin cements (SARCs) have been preferred clinically for their ease of use.[13] Whereas the conventional resin cement, used in combination with an adhesive, requires a multistep procedure and makes the cementation technique sensitive and time consuming, SARCs need a simplified cementation procedure such that acid etching and adhesive application can be skipped on the tooth surface.[13,14] SARCs are defined as cements based on filled polymers designed to adhere to tooth structure without the use of etchant or adhesive. Instead of etchant, the cement's composition contains acid-functionalized monomers, such as acrylate monomers with either carboxylic acid groups (4-META, pyromellitic dianhydride glycerol dimethacrylate [PMGDM]) or phosphoric acid groups (Phenyl-P, 10-MDP) for demineralization of the tooth structure.[15]

However, there is limited information regarding the bonding results between the self-adhesive resin cements and zirconia ceramics. There are various types of commercially available self-adhesive resin cements, and some of these cements contain the MDP component itself.[15] The manufacturer may instruct to use only MDP-containing self-adhesive resin cement with zirconia restorations without the priming procedure, however little is known about the result of applying both MDP-containing primer and MDP-containing cement in the bonding procedure of zirconia restorations. Therefore, it is necessary to determine whether the additional use of the MDP-containing primer affects the bond strength between self-adhesive resin cements and zirconia ceramics.

The purpose of this study was (1) to measure the microshear bond strength (μSBS) of four different self-adhesive resin cements with/without MDP-containing primer to zirconium ceramics and (2) to evaluate the effect of zirconia primers on these self-adhesive resin cements. The null hypotheses were

(1) there was no significant difference in the bond strength between MDP-containing and non–MDP-containing SARC, and (2) an additional primer application would not affect the bond strength of either MDP-containing or non–MDP-containing self-adhesive resin cement to zirconium ceramics.

METHODS AND MATERIALS

Specimen Preparation

Twenty partially sintered Y-TZP blocks (D max Omega Dark, DMAX Co., Daegu, Korea) were cut with diamond discs and then sintered in 1500°C for two hours. The final dimensions of the blocks were 20 mm × 20 mm × 8 mm. The experimental surfaces of each specimen were ground with 600-grit sandpaper for standardization of the surface roughness. Next, all the specimens were sandblasted with 50-μm Al_2O_3 particles (44-74 μm, blasting medium, Dentaurum, Ispringen, Germany) at a distance of 10 mm perpendicular to the Y-TZP surface for 10 seconds (Micro-etcher II A, Danville, Carlsbad, CA, USA). Air pressure was applied at 0.3 MPa, and the surface of each block was then rinsed with water for 30 seconds and air-dried for 30 seconds.

All specimens were randomly divided into eight groups (n=20) according to four self-adhesive resin cements with/without primer. The materials tested in this study are described in Table 1. Four commercially available self-adhesive resin cements were tested (MDP-containing: Permacem 2.0 [PC], Clearfil SA luting [CS]; non–MDP-containing: Rely-X U200 [RU], Maxcem Elite [ME]). For the pretreated groups with primer, one commercially available MDP-containing primer (Z-Prime Plus, Bisco, Schaumburg, IL, USA) was used. One even coat of Z-Prime Plus was applied with a disposable microbrush. After 30 seconds, the solvent was evaporated with an air syringe for five seconds. Next, cylindrical translucent molds (Tygon tubing, E3603, ACF00001, Saint-Gobain Performance Plastics, Akron, OH, USA) were prepared. The each mold had an internal diameter of 0.8 mm and height of 2 mm. The cylindrical molds were filled with freshly auto-mixed self-adhesive resin cements using an endodontic condenser (S-Kondensor, NiTi 40-80 SS, Obtura Spartan, Algonquin, IL, USA) resulting in eight randomly bonded microshear test specimens for each Y-TZP block. After positioning, each mold was light cured from the top surface by a LED curing unit (LEDEX WL-090, Dentalmate Technology, New Taipei City, Taiwan) at 1200 mW/cm^2 for 20 seconds. All of the specimens were left to incubate for 24 hours at room temperature (23±1°C) for further polymerization.

Subsequently, the specimens were thermocycled for 5000 cycles between 5 °C and 55 °C with a dwell time of 30 seconds at each temperature. Then, the cylindrical molds were removed with a #25 blade (Feather surgical blade, Feather Safety Razor, Osaka, Japan).

μSBS Test and Surface Analysis

Measurement of μSBSs was performed as follows: each Y-TZP block was attached to the testing device with cyanoacrylate adhesive (LOCTITE, Henkel, Dublin, Ireland). The μSBS test was performed using a universal testing machine (EZ test, Shimadzu Co., Kyoto, Japan). The shear load was applied by a thin metal wire (wire-loop method) positioned as close as possible to the adhesive interface with a cross-head speed of 1.0 mm/min until bond failure of the specimen occurred (Figure 1). Next, the μSBS was calculated and presented in MPa (N/m^2): Newtons per bonded circular resin cement area.

The failed bond surface was analyzed using a light-microscope at 40× magnification (OPMO pico, Carl Zeiss, Oberkochen, Germany). The failure mode was classified as "adhesive" at the interface of the cement–zirconia, "cohesive" in cement, or "mixed." The loss rate was calculated as the percentage of the number of pre-test failure specimens divided by the total number of specimens in each experimental group.

Statistical Analysis

Statistical analysis was performed using SAS (version 9.3, SAS Inc, Cary, NC, USA). Normal distribution was confirmed using the Shapiro-Wilk test and Kolmogorov-Smirnov test. In each of the primer and no primer groups, one-way analysis of variance was performed to compare the μSBSs between the four self-adhesive resin cements, followed by a *post hoc* Tukey test. In each of the self-adhesive resin cement conditions, independent two-sample *t*-tests were performed to compare the μSBSs between the primer and no primer groups.

The differences between the loss rate of the specimens were evaluated using Fisher's exact test and χ^2 test with a *post hoc* Tukey test; $p<0.05$ was considered statistically significant for all tests.

RESULTS

The mean and standard deviation of the μSBS of all experimental groups are presented in Table 2.

Table 1: *Self-Adhesive Resin Cements and Primer*

Material	Composition	LOT No.	Manufacturer
Permacem 2.0	Base paste: Bis-phenol A diglycidylmethacrylate(Bis-GMA), Triethyleneglycol dimethacrylate (TEGDMA), Ethoxylated bisphenol A dimethacrylate (EBPADMA), UDMA, 2-Hydroxyethyl methacrylate (HEMA), Silanated glass filler, Camphorquinone, Amine, Pigments, Butylated hydroxytoluene (BHT), NaF Catalyst paste: 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Triethyleneglycol dimethacrylate (TEGDMA), Ethoxylated bisphenol A dimethacrylate (EBPADMA), Urethane dimethacrylate (UDMA), Silanated glass filler, Butylated hydroxytoluene (BHT), Benzoyl peroxide	755814	DMG
Clearfil SA Luting	Paste A: 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Bis-phenol A diglycidylmethacrylate (Bis-GMA), Triethyleneglycol dimethacrylate (TEGDMA), Hydrophobic aromatic dimethacrylate, dl-Camphorquinone, Benzoyl peroxide, Initiator, Silanated barium glass filler, Silanated colloidal silica Paste B: Bis-phenol A diglycidylmethacrylate (Bis-GMA), Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Accelerators, Pigments, Surface treated sodium fluoride, Silanated barium glass filler, Silanated colloidal silica	3G0089	Kuraray
RelyX U200	Base paste: Methacrylate monomers containing phosphoric acid groups, Methacrylate monomers, Silanated fillers, Initiator components, Stabilizers, Rheological additives Catalyst paste : Methacrylate monomers, Alkaline (basic) fillers, Silanated fillers, Initiator components, Stabilizers, Pigments, Rheological additives	629949	3M ESPE
Maxcem Elite	Glycerol phosphate dimethacrylate (GPDM), Methacrylate ester monomers, Proprietary self-curing redox activator, Camphorquinone, Fluoroaluminosilicate glass fillers, Barium glass filler, Silica, Activators, Stabilizers	5989391	Kerr
Z-Prime Plus	10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Bisphenyl dimethacrylate (BPDMA), 2-Hydroxyethyl methacrylate (HEMA), Aromatic substituted carboxylic acid, Ethyl alcohol	1600002233	Bisco

Within the no primer groups, the highest bond strength was obtained in the PC and CS groups, whereas the lowest bond strength was observed in the ME group. Within the primed groups with Z-Prime Plus, the PC/P group showed higher bond strength than the CS/P and ME/P groups. There were no significant differences in bond strengths between the PC/P and RU/P groups. In addition, no significant differences were observed among the CS, RU, and ME groups. Comparing the μSBS of the no primer and primer groups in the same SARCs, there was no significant difference between the PC and PC/P groups and between the CS and CS/P groups. The bond strength of the RU/P group was higher than the RU group, and the bond strength of the ME/P group was higher than the ME group with a highly significant difference ($p<0.0001$).

The loss of the specimens is listed in Table 3. Within the no primer groups, the loss rate of the ME group was 90%, which was significantly higher than the other groups. Within the primer groups, the PC/P group showed the lowest loss rate, 0%. Comparing the loss rate within the same SARCs, the ME/P group was lower than the ME group, whereas no significant difference was observed in the other SARCs groups.

The result of failure mode classification using a light-microscope (40×) is shown in Figure 2. In all experimental groups except the ME and ME/P groups, mixed failure mode was observed with a

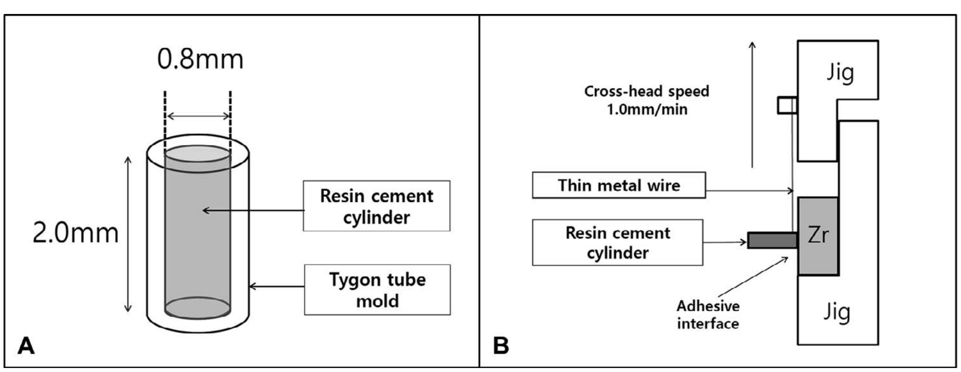

Figure 1. *A, Dimension of Tygon tube mold and resin cement cylinder. B, Microshear bond strength test.*

Table 2: *Microshear Bond Strength (MPa) to Zirconia (Means ± SD, N=20)*[a]

	PC	CS	RU	ME
No primer	20.596 ± 9.591 aA	17.968 ± 12.498 abA	12.612 ± 9.431 bA	1.039 ± 3.209 cA
Primer	21.248 ± 8.971 aA	12.761 ± 10.346 bA	19.75 ± 9.538 abB	12.805 ± 10.983 bcB

[a] *Different lowercase letters represent statistically significant differences within the same line ($p<0.05$; horizontal comparisons). Different capital letters represent statistically significant differences within the same row ($p<0.05$; vertical comparisons).*

higher proportion than adhesive or cohesive failure modes. In the ME/P group, adhesive failure was observed as often as mixed failure.

DISCUSSION

This study compared the bond strengths of four different self-adhesive resin cements to zirconia ceramics and examined the effect of application of the primer to MDP-containing or non–MDP-containing self-adhesive resin cement.

MDP is a functional monomer that chemically bonds with the zirconia surface and increases bond strength. It has been suggested that the phosphate groups of MDP react with the hydroxyl groups of zirconia ceramics.[6,7] Blatz and others reported that the bond strengths of self-adhesive resin cements to zirconium oxide ceramics were increased by airborne particle abrasion, and resin cements containing adhesive functional monomers (MDP) present better bond strengths than other compositions.[4] In addition, Koizumi and others reported that adhesive MDP monomers provide better bond strength to zirconia. In our study, we obtained similar results. Without primer application, MDP-containing SARCs, Permacem 2.0 (PC) and Clearfil SA luting (CS), showed higher bond strength than non–MDP-containing SARCs, Rely X U200 (RU) and Maxcem Elite (ME). However, there were no significant differences in bond strengths between the CS and RU groups. In the ME group, the bond strength demonstrated a significantly lower value compared with the other groups. Thus, the first null hypothesis was rejected.

While thermocycling, loss of the resin cylinder in some specimens occurred, and thus the bond strengths were not measurable. *In vitro* studies revealed that artificial aging, including thermocycling, reduced the bond strengths.[16] Loss of the specimens is due to a lack of bond strength capable of withstanding the stresses exerted by thermocycling. Thus, the μSBS of a lost specimen after thermocycling was calculated as 0 MPa. In particular, the loss rate was 90% in the ME group, and this value was statistically significant. In our study, low μSBS was observed in the ME group. This result corresponded with findings obtained in previous studies that the mechanical strength of Maxcem Elite to zirconia was lower than that of the other self-adhesive resin cements.[17,18] Zorzin and others reported this result with the pH neutralization behavior of the resin cements. The pH of Maxcem Elite remained low (pH 3.9) until 24 hours after the beginning of polymerization and demonstrated the lowest pH value of all tested materials. It was observed that the pH neutralization ability was insufficient.[17] Ferracane and others reported that excessive hydrophilicity due to a low pH value can cause water absorption and swelling, which may lower mechanical strength and dimensional stability.[15]

Within the limitations of optical light microscopy observations, a mixed failure mode was mainly observed. In shear bond tests, stress distributions are nonhomogeneous on the adhesive zone, so the failure often starts in one brittle point of substrates and not at the adhesive interface.[11] This is considered one reason for the observation of some cohesive failure patterns. There is also a possibility of misinterpretation due to a limitation of this study. In the ME/P group, an adhesive failure mode was observed as often as a mixed failure mode. This adhesive failure was a result of debonding at the interface of the resin cement and the zirconia surface.

Table 3: *Loss of Specimen (Loss Rate, %)*[a]

	PC	CS	RU	ME
No primer	2(10.00)aA	4(20.00)aA	4(20.00)aA	18(90.00)bA
Primer	0(0.00)aA	5(25.00)bA	1(5.00)abA	6(30.00)bcB

[a] *Different lowercase letters represent statistically significant differences within the same line ($p<0.05$; horizontal comparisons). Different capital letters represent statistically significant differences within the same row ($p<0.05$; vertical comparisons).*

With primer (Z-Prime Plus) application, Permacem 2.0 (PC/P) showed higher bond strength than Clearfil SA luting (CS/P) and Maxcem Elite (ME/P), and there was no significant difference in bond strength between the PC/P and RU/P groups and among the RU/P, CS/P, and ME/P groups. These results showed that bond strengths in MDP-containing primer application were not dependent on whether SARCs contained or did not contain 10-MDP in their composition. The loss rate of specimens also presented a similar tendency.

When comparing the effect of primer application, MDP-containing SARCs, Permacem 2.0 and Clearfil SA luting, presented no significantly different bonding value with/without primer; however, non–MDP-containing SARCs, Rely X U200 and Maxcem Elite, showed increased bond strengths with primer application. Therefore, the second null hypothesis was also rejected.

In this study, 10-MDP–containing primer had a positive effect on bonding values between zirconia and non–MDP-containing SARCs. This feature was particularly evident in the ME and ME/P groups. In Maxcem Elite, the μSBS was increased, and the loss rate was lowered with primer application.

Based on previous studies, the effect of an MDP-containing primer is controversial. Stefani and others reported that an MDP-containing alloy primer did not enhance the bond strength of an MDP-containing SARC (Clearfil SA luting) to the zirconia ceramics.[19] However, other studies reported that the use of an MDP-containing primer increased bond strength between Clearfil SA luting and zirconia.[9,20]

In our experiment, the use of an MDP-containing primer was effective only for the non–MDP-containing SARCs, RU and ME. The use of MDP-containing primer did not increase the bond strength in the MDP-containing SARCs, PC and CS. This result showed that the increased MDP concentration in both the primer and SARC did not lead to an additional enhancement in bond strength.

There are a few studies reporting optimal MDP concentrations for maximum bond strength to zirconia ceramics. Nagaoka and others reported that higher concentrations of 10-MDP in primers yielded higher shear bond strengths with a concentration dependency and suggested that a minimum 1-ppb MDP was needed to bond to zirconia.[21] However, it should be considered that the experiment was performed with 0.1 ppb∼1wt% 10-MDP of a low and narrow concentration range. However, Llerena-Icochea and others reported that there was

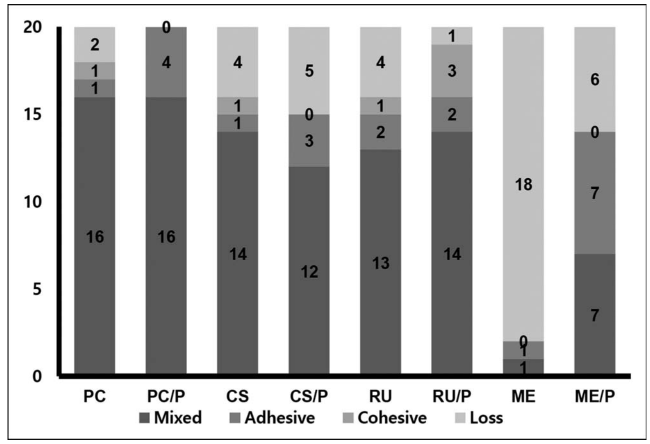

Figure 2. *Results of failure mode classification.*

no linear correlation between bond strength and concentration of 10-MDP in experimental adhesives with a 10-MDP concentration range of 0% to 15% (0%, 3%, 6%, 9%, 12%, and 15%), whereas the highest bonding value was observed in commercially available adhesives (Signum Zirconia Bond, Heraeus Kulzer, Hanau, Germany).[12] Because the concentration ranges of MDP evaluated in these two studies were very different from each other, it is difficult to determine whether bond strength exhibited a linear correlation with the concentration of MDP.

Commercial 10-MDP–containing primers have more than 1wt% 10-MDP, whereas 10-MDP–containing resin cements also contain higher concentrations of 10-MDP than primers.[21] In the case of Permacem 2.0, the manufacturer reported that it contains 2% 10-MDP in the mixed state of the base paste and catalyst paste. According to Llerena-Icochea and others, the commercially available MDP-containing adhesives showed high bonding values and indicated bonding to Y-TZP.[12] However, above a certain level, it could be considered that MDP no longer increases the bond strength.

Therefore, further studies are needed to evaluate the mutual effect of MDP between resin cements and primers and to detect the optimal level of MDP for maximum bonding ability.

CONCLUSION

Non–MDP-containing SARCs showed increased bonding values with the addition of MDP-containing primer to zirconia ceramics. The bond strength of MDP-containing SARCs was not affected significantly by the use of zirconia primer.

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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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