

치과용 지르코니아 전처리제와 레진시멘트의 조합이 치과용 지르코니아의 전단결합강도에 미치는 영향

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〈Abstract〉

Effect of priming agent and resin cement combinations on shear bond strength of dental zirconia

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본 연구에서는 치과용 지르코니아 전처리제와 레진시멘트의 조합이 치과용 지르코니아의 전단결합강도에 미치는 영향을 평가하였다. 336개의 치과용 지르코니아 디스크 시편(직경15mm×두께1mm)을 실험에 사용한 지르코니아 전처리제(대조군, Monobond Plus, Z-PRIME Plus, Single Bond Universal)와 레진시멘트(Rely X ARC, RelyX Ultimate, RelyX U200), 열순환 처리 유무에 따라 24개 군으로 분류하였다. 연마된 시편을 각 실험군에 따라 전처리제를 도포한 후 레진시멘트를 적용하고, 24시간 동안 실온에서 자가 중합하였다. 실험군의 반은 24시간 동안 37°C 증류수에 저장한 후, 나머지 반은 5,000회 열순환처리 후 전단결합강도를 측정하였다. 실험 결과, 24시간 동안 증류수에 저장한 후 전단결합강도를 측정한 군은 전처리제 처리 후 전단결합강도의 유의한 증가를 보였으나, 전처리제와 레진시멘트 종류 간의 유의한 차이는 없었다. 5,000회 열순환처리 후 측정된 전단결합강도는 Single Bond Universal을 도포한 군이 Monobond Plus를 도포한 군보다 유의하게 높은 결합강도를 보였고, Z-PRIME Plus를 도포한 군과는 유의차가 없었다. RelyX Ultimate과 RelyX U200의 적용은 RelyX ARC보다 유의하게 높은 결합강도를 보였다. 열순환처리는 치과용 지르코니아와 레진시멘트의 결합강도를 유의하게 감소시켰다. 결론적으로, 아무 처리하지 않은 치과용 지르코니아 표면에 전처리제의 적용은 치과용 지르코니아와 레진시멘트의 결합강도를 개선시킬 수 있다. 또한 치과용 지르코니아에 대한 결합 강도를 강화하기 위해, 레진시멘트로 RelyX Ultimate과 RelyX U200을 선택하고 기능성 모노머가 있는 전처리제 도포가 추천된다.

Key words : Resin Cement, Shear Bond Strength, Dental Zirconia Primer

I . INTRODUCTION

The patient demand for esthetic restorations has increased recently, and yttria partially stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramic is used frequently as a metal-free restoration alternative (Tsuo Y et al., 2006; Magne P et al., 2010; Shin YJ et al., 2014). Owing to its high flexural strength, optical properties and biocompati-

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bility, dental zirconia has been used in a variety of clinical applications such as a core material for ceramic restorations, posts and implant abutments (Blatz MB et al., 2004; Tsuo Y et al., 2006; Wolfart M et al., 2007). Computer-aided design/computer-aided manufacturing (CAD/CAM) systems have contributed to the increased popularity of dental zirconia restorations in dentistry (Miragaya L et al., 2011; Maeda FA et al., 2014).

One of the major drawbacks regarding the use of dental zirconia, however, is the difficulty in establishing a durable bond between dental zirconia and resin cement because of its high crystalline content, acid-resistance and silica-free characteristics (Yi YA et al., 2014). Unlike conventional silica-based ceramics, the hydrofluoric acid (HF) etching and the application of a silane coupling agent cannot produce a reliable bond between dental zirconia and resin cement (Ozcan M et al., 2003; Miragaya L et al., 2011).

In an attempt to achieve a durable bond between dental zirconia and the resin cement, several pre-treatments of the dental zirconia surface have been suggested, such as airborne-particle abrasion with alumina, selective infiltration etching and tribochemical silica coatings (Wolfart M et al., 2007; Aboushelib MN et al., 2008; Yi YA et al., 2014). Any alteration of the dental zirconia surface can create micromechanical interlocking. But this is controversial, because such dental zirconia restorations may be prone to long-term degradation via hydrolytic and phase transformations (Ozcan M et al., 2003; da Silva EM et al., 2014).

An alternative approach to improve the bond strength to dental zirconia has been developed: a chemical interaction between the dental zirconia surface and resin cement, and the use of a primer containing a phosphate/phosphonate monomer (Tsuo Y et al., 2006; Magne P et al., 2010). In recent years, 10-methacryloyloxydecyl dihydrogen phosphate (MDP) has been reported to be a promising component for improving the bond strength of dental zirconia (Tsuo Y et al., 2006; Wolfart M et al., 2007;

Magne P et al., 2010; Maeda FA et al., 2014). Other phosphate/phosphonate monomers such as 4-methacryloxyethyl trimellitic anhydride (4-META), and thiophosphoric acid methacrylate (MEPS) provides an additional chemical bond to the dental zirconia surfaces (Magne P et al., 2010). These functional monomers have an affinity for metal oxides on the dental zirconia surface (Miragaya L et al., 2011). The phosphate monomers form chemical bonds with the dental zirconia surface and have resin terminal end groups that enable bonding to the resin cement (Yang B et al., 2010). Recently, many manufacturers have incorporated functional monomers into various bonding and luting products, such as primers, adhesives and resin cements. A range of commercial primers for bonding to dental zirconia have been introduced to the market.

Fixed partial dentures (FPDs) and dental zirconia crowns can be cemented conventionally, as recommended by the manufacturers. Many different cements are used to lute crowns, including zinc phosphate, glass ionomer, resin, compomer, and resin-modified glass-ionomer cements (Ernst CP et al., 2005). But resin cement is recommended because it improves the marginal adaptation, prevents microleakage, and increases the fracture strength and retention of restorations (Ozcan M et al., 2003). Self-adhesive resin cements eliminate the need for a pre-treatment of the tooth and restoration, and can be used in a single step (Radovic I et al., 2008). Dental zirconia also has an advantage when using a self-adhesive resin cement containing a functional monomer which forms chemical bonds with dental zirconia surfaces (Miragaya L et al., 2011). On the other hand, because self-adhesive resin cements are hydrophilic as a result of the acidic resin components, it is theoretically better to use a dual-cured, hydrophobic resin cement that does not contain acidic/hydrophilic monomers for the long-lasting strength of the cement (Byoung I. Suh, 2013).

This study examined the effects of combinations of

priming agents and resin cement on the shear bond strength of dental zirconia to provide more favorable clinical guidelines on dental zirconia bonding.

II. MATERIALS AND METHODS

1. Dental zirconia disk preparation

A total of 336 disk specimens (24 groups, n = 14) with a diameter of 15 mm and a thickness of 1 mm that were manufactured industrially from a densely sintered dental

zirconia ceramic (Zirmon, 94.7% ZrO₂ and 5.2% Y₂O₃, Kuwotech Co., Ltd., Gwangju, Korea) were used. Table 1 lists the materials used in this study.

The surface of each specimen was polished by hand-grinding on wet 600-grit silicon carbide abrasive paper and cleaned ultrasonically in ethanol for 10 minutes and air-dried.

2. Bonding procedure

The specimens were assigned randomly to 4 groups (n = 14) according to the following priming agents: no

Table 1. Materials Used in this Study

Material	Brand	Composition	Manufacturer
Dental zirconia	Zirmon	94.7% ZrO ₂ , 5.2% Y ₂ O ₃	Kuwotech Co., Ltd., Gwangju, Korea
Primer	Monobond Plus	Ethyl Alcohol, silane methacrylate, phosphoric acid methacrylate, sulphide methacrylate	Ivoclar Vivadent, Schaan, Liechtenstein
	Z-PRIME Plus	MDP, BPDM, methacrylates, ethanol	Bisco, Schaumburg, IL, USA
	Single Bond Universal	MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane	3M ESPE, St. Paul, MN, USA
Resin cement	RelyX ARC	BisGMA, TEGDMA, zirconia/silica filler, DMA, amine, photoinitiator, BP, pigment	3M ESPE, St. Paul, MN, USA
	RelyX Ultimate	Base: methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives Catalyst: methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives, fluorescence dye, dark cure activator for Scotchbond Universal adhesive	3M ESPE, St. Paul, MN, USA
	RelyX U200	Base: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives Catalyst: methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives	3M ESPE, St. Paul, MN, USA

*Abbreviations: MDP, 10-methacryloyloxydecyl dihydrogen phosphate; BPDM, biphenyl dimethacrylate; HEMA, hydroxyethyl methacrylate; BisGMA, bisphenol-A-diglycidylether dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; DMA, dimethacrylate; BP, benzoyl peroxide.

primer (control), Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein), Z-PRIME Plus (Bisco, Schaumburg, IL, USA), and Single Bond Universal Adhesive (3M ESPE, St. Paul, MN, USA). The dental zirconia disks of each group were divided further into 3 subgroups (n = 14), depending on the resin cements used: RelyX ARC (3M ESPE) and RelyX Ultimate (3M ESPE), which are conventional dual-cured resin cements; and RelyX U200 (3M ESPE), which is a self-adhesive resin cement.

The priming agents were applied to the dental zirconia surface with a microbrush according to the respective manufacturers' instruction (Table 2). No primer was applied to the specimens of the control group. Three types of resin cements (RelyX ARC, RelyX Ultimate and RelyX U200) were mixed according to the manufacturers' instruction, and packed into a no. 9 size gel-cap (internal diameter: 2.5 mm, height: 4.3 mm) using a hand instrument. The gel-cap was

positioned on the prepared dental zirconia surface and any excess was removed. For 24 hours at a room temperature, all the specimens were left to polymerize without light curing. Then half of the specimens were immersed in distilled water at 37 °C for 24 hours. The other half of the groups were subjected to thermocycling for 5,000 cycles between 5 °C and 55 °C in a water bath. The dwelling time in each bath was 30 seconds, and the transfer time from one bath to the other was 20 seconds.

3. Shear bond strength (SBS) testing

After storing the samples under the different storage conditions, the shear bond strength tests were performed at a crosshead speed of 0.5 mm/min in a shear bond tester (T-63010K, Bisco Inc., Schaumburg, IL, USA.). The maximum load (N) to produce failure was recorded in the

Table 2. Materials and Application instructions

Material	Brand	Application instructions
Primer	Monobond Plus	<ol style="list-style-type: none"> 1. Apply a thin coat with a brush. 2. Allow it to react for 60 seconds. 3. Disperse any remaining excess with a strong stream of air.
	Z-PRIME Plus	<ol style="list-style-type: none"> 1. Apply 1-2 coats, uniformly wetting the surface. 2. Dry with an air syringe for 3-5 seconds.
	Single Bond Universal	<ol style="list-style-type: none"> 1. Apply the adhesive 2. Allow it to react for 20 seconds. 3. Gently air dry for 5 seconds.
Resin cement	RelyX ARC	<ol style="list-style-type: none"> 1. Dispense the cement onto a mixing pad. 2. Mix for 10 seconds. 3. Apply a thin layer of the cement to the bonding surface. 4. Light cure for 40 seconds or allow to selfcure for 10 minutes.
	RelyX Ultimate	<ol style="list-style-type: none"> 1. Dispense the cement onto a mixing pad. 2. Mix for 20 seconds. 3. Light cure for 20 seconds.
	RelyX U200	<ol style="list-style-type: none"> 1. Dispense the cement onto a mixing pad. 2. Mix for 20 seconds. 3. Light cure for 20 seconds.

samples that had undergone water storage for 24 hours and thermocycling. The maximum load at failure (N) was converted into MPa.

4. Statistical Analysis

Statistical analysis was performed using statistical software (SPSS 18.0, SPSS Inc., Chicago, IL, USA). Two-way analysis of variance (ANOVA) and a Tukey's multiple comparison test were performed for comparison with SBS as the dependent variable, the priming agents and the resin cements as the independent factors. A student's t-test was used to compare the SBS of the specimens of water storage for 24 hours and thermocycling of 5,000 cycles. Statistical significance was set at $p < 0.05$ for all tests.

III. RESULTS

Table 3 lists the means and standard deviations of the SBS for the different combinations of priming agent and resin cement after water storage for 24 hours. Monobond Plus, Z-PRIME Plus and Single Bond Universal showed significantly higher bond strength of the resin cement to dental zirconia compared to the control group ($p < 0.05$). The control groups that did not treat with the primer showed the lowest SBS. Before thermocycling, no statistical differences in SBS were observed for the priming agents regardless of the resin cement ($p > 0.05$). Among the resin cements, there were no significant differences in SBS ($p > 0.05$).

Table 4 presents the SBS of the specimens after thermocycling for 5,000 cycles. The Single Bond Universal showed a significantly higher bond strength than Monobond Plus after thermocycling ($p < 0.05$). The SBS was similar in the groups treated with Z-PRIME Plus and Single Bond Universal ($p > 0.05$). Furthermore, the SBS obtained for Monobond Plus and Z-PRIME Plus were similar

($p > 0.05$). The groups of these priming agents resulted in a significantly higher SBS than the control group ($p < 0.05$). All of RelyX ARC samples without the primers were debonded during the thermocycling. For the priming agent groups, the SBS of RelyX Ultimate and RelyX U200 were similar ($p > 0.05$), but RelyX ARC was significantly different from RelyX Ultimate and RelyX U200 ($p < 0.05$).

Figure 1 shows the SBS after water storage for 24 hours and 5,000 thermocycling. For all groups of priming agents, the SBS decreased significantly after thermocycling ($p < 0.05$).

IV. DISCUSSION

This study examined the SBS of dental zirconia to different resin cements in combination with various priming agents. The results showed that the bond strength to dental zirconia was affected significantly by the application of a primer without any mechanical surface pre-treatment.

In this study, when the dental zirconia surface was primed with one of three priming agents, the SBS was improved significantly compared to the control group, regardless of the resin cements and thermocycling ($p < 0.05$). This result is in accordance with other studies showing that a high bond strength could be achieved on air-abraded surfaces using a primer containing a phosphate monomer (Wolfart M et al, 2007). The adhesive functional monomer (phosphoric acid methacrylate in Monobond Plus, MDP, BPDM in Z-PRIME Plus, and MDP in Single Bond Universal) can react with the oxide groups on the dental zirconia surface through Van der Waals forces or hydrogen bonds (Magne P et al., 2010; Miragaya L et al., 2011; Kim JH et al., 2014). These interfacial forces might have improved the wettability of the dental zirconia surface, thereby increasing the interlocking with resin cements (Yang B et al., 2010). Before thermocycling, the resin cements and priming agents showed no significant differences in SBS.

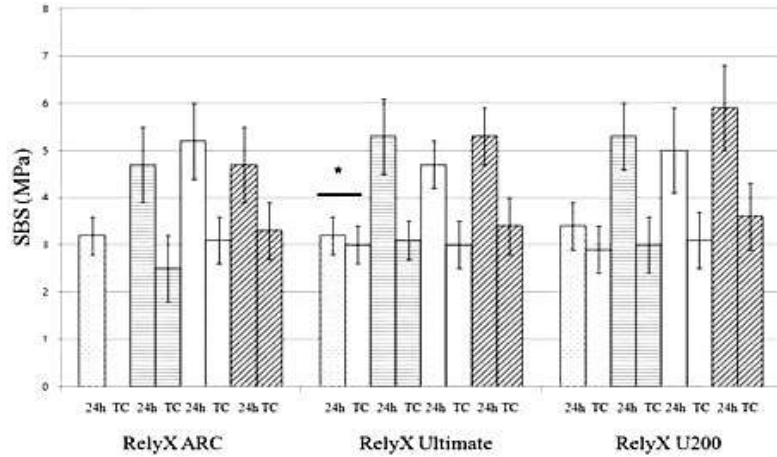


Figure 1. SBS after water storage for 24 hours (24h) and 5,000 thermocycling (TC). Vertical lines represent the standard deviations. The asterisk indicates the no significant difference ($p > 0,05$).

Table 3. Means and standard deviations of SBS (MPa) after water storage for 24 hours.

	No primer ¹	Monobond Plus ²	Z-PRIME Plus ²	Single Bond Universal ²
RelyX ARC ^A	3,24 ± 0,36	4,72 ± 0,84	5,23 ± 0,76	4,65 ± 0,85
RelyX Ultimate ^A	3,17 ± 0,44	5,27 ± 0,81	4,68 ± 0,52	5,26 ± 0,60
RelyX U200 ^A	3,35 ± 0,52	5,33 ± 0,71	4,98 ± 0,88	5,90 ± 0,90
<i>p</i> -value	$\alpha < 0,001,$	$\beta = 0,025,$	$\alpha \times \beta = 0,014$	

Statistically significant difference on resin cements is shown by superscript letters^{A,B} (columns) and on priming agents by superscript numbers^{1,2} (rows). Same letters or numbers are not significantly different ($p > 0,05$).

On *p*-value, the letters α and β denote priming agent and resin cement, respectively.

Table 4. Means and standard deviations of SBS (MPa) after 5,000 thermocycling.

	No primer ¹	Monobond Plus ²	Z-PRIME Plus ^{2,3}	Single Bond Universal ³
RelyX ARC ^A	-	2,53 ± 0,70	3,11 ± 0,51	3,30 ± 0,56
RelyX Ultimate ^B	3,01 ± 0,36	3,08 ± 0,45	3,03 ± 0,49	3,38 ± 0,57
RelyX U200 ^B	2,87 ± 0,55	3,00 ± 0,65	3,08 ± 0,64	3,58 ± 0,72
<i>p</i> -value	$\alpha < 0,001,$	$\beta < 0,001,$	$\alpha \times \beta < 0,001$	

Statistically significant difference on resin cements is shown by superscript letters^{A,B} (columns) and on priming agents by superscript numbers^{1,2} (rows). Same letters or numbers are not significantly different ($p > 0,05$).

On *p*-value, the letters α and β denote priming agent and resin cement, respectively.

Previous studies reported that some conventional and self-adhesive resin cements have low bond strength when applied to intact, untreated dental zirconia surface under light curing (Aboushelib MN et al., 2008; Yun JY et al., 2010; da Silva EM et al., 2014). In this study, the SBS of the untreated dental zirconia surfaces were relatively low and decreased over time, compared to the results of other studies using different surface treatments and resin cements. Dental zirconia is an opaque material, and even translucent dental zirconia ceramics have significant low light transmission (Turp V et al., 2011). Light transmission through dental zirconia is difficult when bonding the dental zirconia restorations to the teeth under clinical conditions. Therefore, light irradiations was excluded from the study. In addition, the polished dental zirconia specimens were used to standardize the dental zirconia surfaces. Airborne-particle abrasion was not considered because the main focus of the study was to evaluate the chemical bonding between the adherent surfaces. One explanation for the low bond strength to the untreated dental zirconia surfaces of this study might be the limited experimental conditions. According to Lüthy et al., the minimum acceptable bond strength required to provide adequate clinical performance and longevity was suggested to be approximately 10 to 13 MPa (Lüthy H et al., 2006; de Souza G et al., 2014). A Table 3 and 4, the bond strength between dental zirconia and the resin cements ranged from 2.53 to 5.90 MPa, which is considered low. Establishing a strong bond between the resin cements and the intact, untreated dental zirconia surface would be difficult without any mechanical pre-treatment and light cure.

Significant differences in the SBS were observed among the priming agents after thermocycling. Single Bond Universal, which shows significantly higher bond strength than Monobond Plus ($p < 0.05$), contains both MDP and silane. Although Monobond Plus had a lower SBS than Single Bond Universal after thermocycling ($p < 0.05$), the

significantly higher value compared to the control group can be explained by the presence of a silane material mixed with phosphoric acid methacrylate. According to the literature, MDP has great affinity to metal oxides such as zirconium dioxide, and creates a stable bond to airborne-particle abraded dental zirconia before and after thermocycling (Blatz MB et al., 2004; de Souza G et al., 2014). On the other hand, the dental zirconia ceramic is composed of a glass-free material, which means that the silane cannot contribute to the chemical bond to dental zirconia (Yi YA et al., 2014). Silane, however, can increase the wettability of the dental zirconia surface and improve the bond strength as a result (Tsuo Y et al., 2006). Silane monomers are less stable under acidic conditions (Matinlinna JP et al., 2004). These finding are in accordance with the present study in that Monobond Plus showed the largest decrease in SBS (-46.4% for RelyX ARC, -41.6% for RelyX Ultimate and -43.7% for RelyX U200) compared to the result before thermocycling. In contrast, Single Bond Universal contains resin adhesive components, such as dimethacrylate resins, which can improve the flowability of the resin cement and reinforce the interfacial layer by copolymerizing with the resin cement (Kim JH et al., 2014).

Z-PRIME Plus used in this study includes two types of acidic monomers, BPDm and MDP. BPDm has a synergistic effect with the phosphate monomer, improving the bond strength of dental zirconia and other various materials (Maeda FA et al., 2014; Yi YA et al., 2014). In this study, the groups treated with Z-PRIME Plus showed a similar SBS to the groups treated with Single Bond Universal.

The resin cements used in this study showed a significantly different SBS after thermocycling. Without the use of a primer, RelyX ARC lost its bond strength completely after thermocycling ($p < 0.05$). This conventional resin cement is not supposed to have a chemical interaction with the dental zirconia surface because RelyX ARC does not contain phosphate monomers, and was used as a control. This

result is supported by a previous study in that the self-adhesive resin cement had a higher bond strength to the air-abraded dental zirconia surface than RelyX ARC, a conventional resin cement (Miragaya L et al., 2011). On the other hand, the bonding ability of RelyX Ultimate was similar to that of RelyX U200 after thermocycling ($p > 0.05$). According to Blatz et al., an adhesive functional monomer might not be essential in the resin cement when using a primer containing a phosphate monomer (Blatz MB et al., 2004). RelyX Ultimate has an integrated dark cure activator for Single Bond Universal, which was designed for optimal performance when combined with Single Bond Universal. RelyX U200, however, yielded a higher bond strength to the dental zirconia surface than RelyX ARC. According to the manufacturer, RelyX U200 contains methacrylate monomers with phosphoric acid groups and silanated fillers in its chemical composition. This cement has the characteristics of self-etching phosphorylated methacrylates, which are designed to bond directly to both the enamel and dentin (Radovic I et al., 2008). On the other hand, an acidic component included in the self-adhesive resin cements makes it more hydrophilic (Ito S et al., 2005). The bond strength durability showed that the self-adhesive resin cements showed a significant decrease in bond strength after thermocycling (da Silva EM et al., 2014). In this study, for RelyX U200, there was a 43.7% decrease in the SBS treated with Monobond Plus, 38.2% for Z-PRIME Plus and 39.3% for Single Bond Universal (Figure 1). A larger decrease in the SBS was observed in RelyX U200 compared to RelyX Ultimate, a hydrophobic resin cement. This might be due the acid-based component of the self-adhesive resin cement, which is not enough to resist the hydrolytic effect of the water storage condition (Aboushelib MN et al., 2008). In addition, for all resin cements, the dental zirconia surface with the phosphate monomer was incapable of maintaining the bond stability over thermocycling. This behavior is supported by previous studies, and may be

explained by the poor hydrolytic stability of the phosphate monomer based primer (Yun JY et al., 2010; da Silva EM et al., 2014).

Although the three priming agents tested in this study contain phosphate monomers, the differences in SBS suggest that the additional monomers found in their composition can affect the bond strength (Maeda FA et al., 2014). Furthermore, variations in the chemical composition, wettability, viscosity, and mechanical properties of each resin cement may play an important role in the bonding capacity to dental zirconia ceramics (Derand T et al., 2005; de Souza G et al., 2014).

Thermocycling in a water bath is used frequently to simulate the intraoral conditions and evaluate the hydrolytic stability of the bond interface (da Silva EM et al., 2014). Gale and Darvell suggested that approximately 10,000 cycles represented a service year (Gale MS et al., 1999). In the present study, 5,000 cycles were adopted and might represent 6 months, which might be not a sufficient time to assess the long-term bond durability. After thermocycling, the SBS of the samples treated with the priming agents decreased significantly ($p < 0.05$). Therefore, it can be assumed that the phosphate monomer cannot create a stable bond to an untreated dental zirconia surface. Further studies with a longer artificial aging time will be needed to evaluate the long-term durability of the resin cements to dental zirconia restorations.

V. CONCLUSION

1. The application of priming agents onto the untreated dental zirconia surface can improve the bond strength between dental zirconia and resin cements.
2. The SBS of the samples that had undergone water storage for 24 hours was similar regardless of the priming agents and resin cements ($p > 0.05$).

- To enhance the bond strength to dental zirconia, the selection of RelyX Ultimate and RelyX U200 and the application of a primer containing a functional monomer is recommended.

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