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2개의 치과용 범용 접착제의 사전중합이 지르코니아에 대한 레진시멘트의 전단결합강도에 미치는 영향

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Effect of pre-curing of two universal adhesives on the shear bond strength of resin cement to zirconia

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본 연구에서는 범용 상아질 접착제를 지르코니아 프라이머로 사용 시 사전 광중합이 지르코니아와 레진 시멘트간의 결합강도 및 내구성에 미치는 영향을 알아보고자 하였다. 지르코니아 시편연마 후 프라이머의 종류와 사전 광중합 여부에 따라 여섯 군으로 나누어 표면을 샌드블라스팅하고 범용 접착제로는 All-Bond Universal 및 Single Bond Universal Adhesive를, 비교를 위한 일반 지르코니아 프라이머로는 Z-Prime Plus를 도포한 후 광중합 또는 미중합 하였다. 이중중합형 레진 시멘트인 Duo-Link를 올려 37℃에서 24시간 물에 보관 후 절반의 시편에 대하여 5,000회 열순환을 추가하고 (n = 12), 열순환 전후의 전단결합강도 측정 및 파절면 관찰을 시행하였다. 측정된 전단결합강도 값을 이원분산분석과 Tukey 사후검정을 이용하여 비교분석한 결과, 열순환 전의 결합강도는 범용 접착제의 광중합군이 미중합군에 비하여 높은 반면, Z-Prime Plus는 광중합에 영향을 받지 않았다. 열순환 후 범용 접착제의 광중합군과 미중합군 모두에서 결합강도가 감소되었으나 Z-Prime Plus에서는 감소가 없었으며 범용 접착제에 비하여 더 높은 값을 나타내었다. 파절양상의 경우 모든 군에서 접착면에서의 실패가 가장 많았으며, 미중합군의 경우 열순환 후 접착성 파절의 발생빈도가 증가하였다. 범용 상아질 접착제를 지르코니아 프라이머로 사용한 경우 사전 광중합이 지르코니아와 레진 시멘트간의 초기결합강도는 유의하게 증가시켰으나, 접착의 내구성 측면에서는 효과가 없었다.

색인단어 : 범용 상아질 접착제, 사전 광중합, 전단결합강도, 지르코니아, 프라이머

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Introduction

Zirconia-based ceramic has been increasingly used in dentistry due to its good biocompatibility, superior mechanical properties, and adequate esthetics (1). Despite having the excellent properties, its glass-free, polycrystalline structure makes the traditional hydrofluoric acid etching followed by silane application ineffective in surface conditioning. Especially when luting a non-retentive zirconia restoration, optimizing adhesive cementation is critical for a successful outcome. Among the several alternative surface treatments suggested for improved resin bonding to zirconia (2-4), the application of functional monomers with a chemical affinity to metal oxides, such as carboxylic and phosphoric acid esters was proposed as a simple and noninvasive procedure (5).

To create a chemical coupling between zirconia and resin cement, an organophosphate monomer, 10-meth acryloyloxydecyl dihydrogen phosphate (MDP) has been contained in various luting resins and primers. Although some study supports the use of MDP-containing primers as a substitute for airborne-particle abrasion (6), a combination of micromechanical interlocking and chemical bonding seems to be effective in achieving reliable and durable adhesion between zirconia and resin composite (7, 8)

MDP is also incorporated in multipurpose universal adhesives, which have been introduced for either direct or indirect use based on a simplified bonding strategy. Several recent studies have reported promising results with the use of universal adhesives on zirconia bonding (9-11). Notwithstanding the proven efficacy of universal adhesives as a priming agent for a zirconia restoration, instructions for clinical procedures are unclear without being specific about light-irradiation as well as application and drying methods. This ambiguity might contribute to the varying results from the different application protocols (12, 13).

For a stable structure with polymerized monomers in an adhesive applied to the substrate, proper light-curing of the adhesive is required prior to composite placement. An unpolymerized adhesive allows a flowable layer and pressure from the overlying composite may be enough to extrude the adhesive from the bonding interface, leaving little material needed for promoting adhesion (14). Moreover, compositional changes in the bonding interface caused by the uncured adhesive mixed with the resin cement may adversely affect the polymerization of the luting resin (15).

The purpose of this in vitro study was thus to evaluate the shear bond strengths of a resin cement to the air-abraded zirconia surface treated with the different surface conditioning protocols. The null hypothesis tested was that light-activation of priming agents prior to resin cement application would not affect early bond strengths and bonding durability between a resin cement and zirconia ceramic.

Materials and Methods

Two commercial universal adhesives (All-Bond Universal, ABU, Bisco Inc., Schaumburg, IL, USA; Single Bond Universal Adhesive, SBU, 3M ESPE, Seefeld, Germany) were selected in comparison with a conventional zirconia primer Z-Prime Plus (ZPP, Bisco Inc.) for this study. Their manufacturers, compositions, and application protocols are summarized in Table 1

Table 1. Zirconia priming agents and a dual-cured resin cement used in this study

Material (primer code)	Manufacturer (Lot No)	Composition	Application protocol
Z-Prime Plus (ZPP)	Bisco Inc., Schaumburg, IL, USA (1500003044)	Organophosphate monomer (MDP), carboxylic acid monomer (BPDM), Bis-GMA, HEMA, ethanol	1. Clean the internal surface of the restoration. 2. Apply 1-2 coats uniformly wetting the bondable surface. 3. Dry with an air syringe for 3-5 seconds.
All-Bond Universal (ABU)	Bisco Inc., Schaumburg, IL, USA (1500002118)	Organophosphate monomer (MDP), Bis-GMA, HEMA, ethanol, water, initiators	1. Prepare the surface of the restoration. 2. Apply 1 coat of All-Bond Universal and air-dry. 3. Light-cure for 10 seconds.
Single Bond Universal Adhesive (SBU)	3M ESPE, Seefeld, Germany (70201140426)	Organophosphate monomer (MDP), dimethacrylate resins (Bis-GMA, etc), HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane	1. Apply the adhesive to the cemented surface. 2. Allow it to react for 20 seconds. 3. Do not light-cure.
Duo-Link	Bisco Inc., Schaumburg, IL, USA (1500003824)	Base: Bis-GMA, TEGDMA, UDMA, glass fillers Catalyst: Bis-GMA, TEGDMA, glass fillers	1. Mix equal amounts of base and catalyst. 2. Seat the restoration and remove excess cement. 3. Light-cure for 40 seconds.

1. Preparation of zirconia ceramic specimens

The study design for shear bond strength testing is illustrated in Figure 1. A total of 36 zirconia blocks with dimensions of 10 x 15 x 2 mm were cut from pre-sintered blanks (Ceramill Zolid, Amann Girrbach AG, Koblach, Austria), wet-polished with 600- to 800-grit silicon carbide (SiC) papers, and sintered in an oven (Ceramill Therm, Amann Girrbach AG) at 1450℃ for 2 hours according to the manufacturer's instructions. The sintered blocks were embedded in round silicone rubber molds using poly(methyl methacrylate) resin, ensuring that one surface of the zirconia block remained uncovered for bonding. The exposed surface of all specimens was polished up to 1200-grit with wet SiC paper and cleaned in ultrasonic bath with 90% isopropyl alcohol for 3 minutes. Air abrasion was performed in a chairside air-abrasion device (MicroEtcher II, Danville Materials Inc., San Ramon, CA, USA) with 50 μm Al_2O_3 at 0.25 MPa for 15 seconds at a distance of 10 mm (8). The specimens were ultrasonically cleaned again in 90% isopropyl alcohol for 3 minutes, rinsed with deionized water, and finally air-dried.

2. Bonding procedures

The blocks were randomly divided into six groups, to which different priming agents were applied with or without photo-polymerization. For pre-cured groups (-LC), light-curing the priming agents was done after they had been applied to the zirconia surface in accordance with the respective manufacturer's instructions. In group ZPP-LC, a microbrush was used to apply two coats of

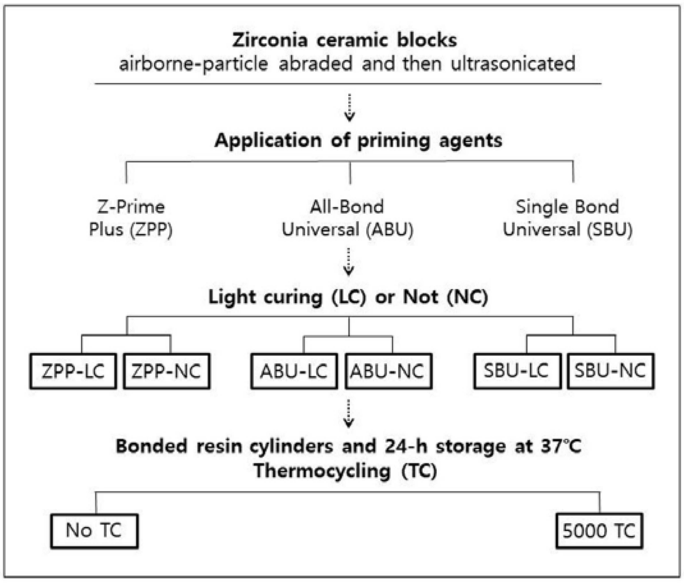

Figure 1. A study design for shear bond strength testing.

Z-Prime Plus, which was allowed to react for 20 seconds, followed by air-drying for 5 seconds and light-curing for 10 seconds using a LED light-curing unit (Bluephase 20i, Ivoclar Vivadent, Amherst, NY, USA; output intensity = 1000 mW/cm^2). For the specimens in groups ABU-LC and SBU-LC, one coat of All-Bond Universal and Single Bond Universal Adhesive, respectively was applied to the zirconia surface for 20 seconds with a microbrush, air-dried for 5 seconds, and then light-cured for 10 seconds as described above. For uncured groups (-NC), the specimens were treated in the same way as for those in the pre-cured groups with the omission of light-curing.

A freshly mixed paste of conventional Bis-GMA-based resin cement (Duo-Link, Bisco Inc.) was incrementally applied to the primed zirconia surface by packing the material into cylindrical-shaped silicone molds with an internal diameter of 2.0 mm and a height of 4.0 mm in two layers. Each cement layer was irradiated for 20 seconds and finally 40 seconds using a LED light-curing unit. In this manner, four bonded resin cylinders were made on one zirconia specimen and a total of 24 resin cylinders (i.e., six block specimens) were prepared for each group. Excess cement flash extruded beyond the bonding area was examined under the stereomicroscope (SZ4045, Olympus, Tokyo, Japan) and was carefully removed with a sharp knife. Prior to debonding, all bonded specimens were stored in water at 37℃ for 24 hours and half of them (n = 12) were additionally thermocycled 5,000 times between 5℃ and 55℃ water baths with a dwelling time of 30 seconds and an exchange time of 5 seconds between each bath.

3. Shear bond strength testing and failure mode analysis

The specimens were perpendicularly engaged at their bonded resin cylinder bases with a round-notched custom shear blade (a notch diameter: 2.5 mm) in a universal testing machine (Model 3343, Instron Inc., Canton, MA, USA). Each cylinder was stressed one by one by turning the specimen at a crosshead speed of 0.5 mm/minute until bonding failure occurred (16). Bond strengths (MPa) were calculated from the peak load of failure (N) divided by the bonded surface area. Following debonding, all specimens were examined under the stereomicroscope (SZ4045, Olympus) at a magnification of 25× to determine the failure mode: (A) adhesive failure at the zirconia-resin interface; (C) cohesive failure within resin cement; (M) mixed failure involving the combinations of these failure modes. Each type of failure mode was expressed as a percentage of the total number of specimens in that group.

4. Statistical analysis

As the normally distributed bond strength data (Kolmogorov-Smirnoff test) did not exhibited equal variances (Levene test), the bond strength values were log10 transformed to meet homogeneity of variance prior to analysis (17).

A two way analysis of variance (ANOVA) design was used to assess the effects of priming agent (pre-cured and uncured) and thermocycling (thermocycled and non-thermocycled), and the interaction of these two parameters on shear bond strength. Multiple comparisons were done using Tukey test and Student' s *t* test was also used to evaluate the effect of thermocycling on long-term stability of the adhesive interface for the same surface conditioning. Failure modes were analyzed using Fisher' s exact test. All statistical analyses were performed using SPSS 13.0 for Windows (SPSS Inc., Chicago, IL, USA) at a significance level of 0.05.

Results

Table 2 summarizes the shear bond strengths of resin cement to the zirconia surface treated with the different priming protocols prior to cementation. Two-way ANOVA showed statistically significant differences for the priming agent and thermocycling, and for the interaction between these factors (all $p < 0.001$).

Before thermocycling, the light-cured specimens of ABU and SBU showed higher bond strengths than the uncured ones of the materials ($p < 0.05$), whereas no significant difference was found between the pre-cured and uncured specimens of ZPP ($p > 0.05$). Group SBU-LC exhibited the highest bond strength when the comparisons were

Table 2. Table 2. The shear bond strengths of a resin cement to the primer-treated zirconia surface, mean (standard deviation) (n = 12)

Group	Bond strength (MPa)	
	No TC	5,000 TC
ZPP-LC	21.2 (5.1)^{AB,a}	18.3 (2.7)^{A,a}
ZPP-NC	17.4 (2.7)^{AC,a}	15.4 (2.4)^{AB,a}
ABU-LC	20.3 (2.9)^{AB,a}	13.2 (2.2)^{BC,b}
ABU-NC	15.4 (2.4)^{C,a}	11.4 (1.9)^{C,b}
SBU-LC	23.8 (3.5)^{B,a}	14.3 (2.1)^{B,b}
SBU-NC	18.4 (1.7)^{AC,a}	11.5 (1.7)^{C,b}

ZPP: Z-Prime Plus; ABU: All-Bond Universal; SBU: Single Bond Universal Adhesive; LC: Light-Curing; NC: No Light-Curing; No TC: No Thermocycling; 5000 TC: 5,000 Thermocycling

Within a column, values having similar upper-case letters did not exhibit statistical difference according to one-way ANOVA and Tukey's post hoc test ($p > 0.05$). Within a row, values having similar lower-case letters did not exhibit statistical difference according to Student's *t* test ($p > 0.05$).

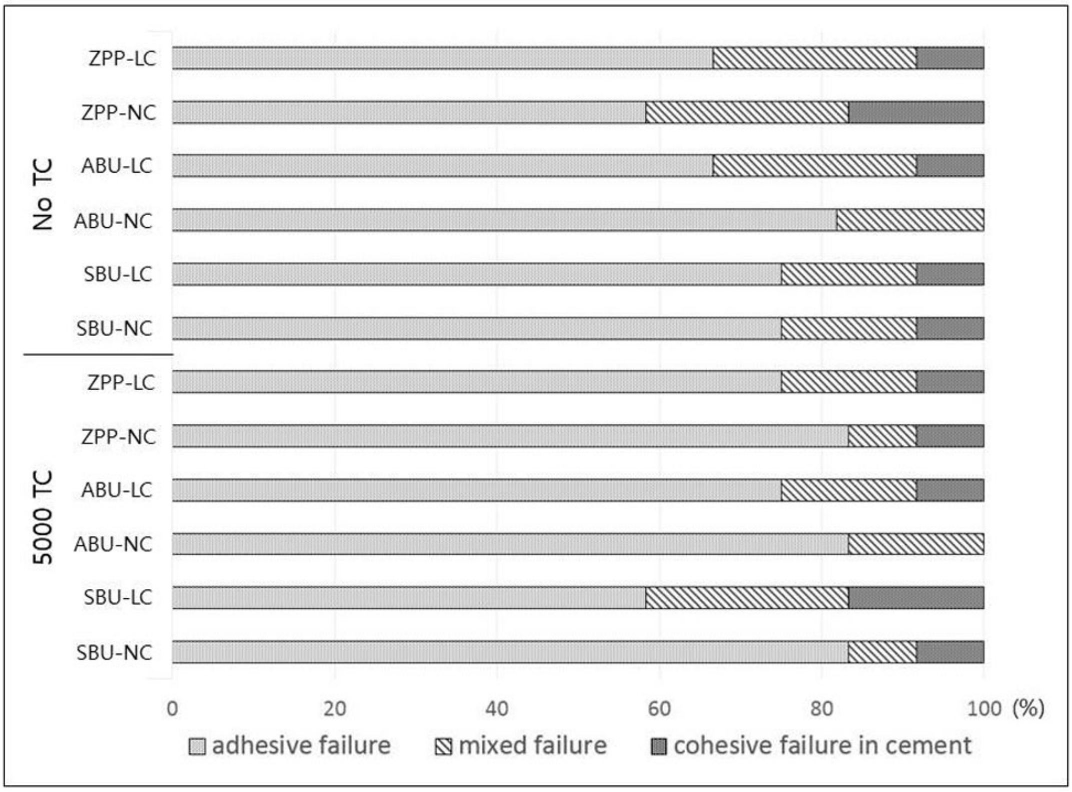

Figure 2. Distribution Of Failure Pattern (%) In The Test Groups.

made within the pre-cured groups, but the differences not being statistically significant ($p > 0.05$).

After aging by thermocycling, there were significant decreases in the bond strength for all specimens treated with the universal adhesives ABU and SBU ($p < 0.05$). ZPP, however, showed no significant difference after 5,000 thermal cycles ($p > 0.05$) and attained higher bond strength values for both the pre-cured and uncured specimens when compared to the two universal adhesives ($p < 0.05$). Regarding the effect of light-curing, no significant difference in the bond strength was found between the pre-cured and uncured specimens of ZPP and ABU ($p > 0.05$), while after aging the bond strength values of the uncured specimens were still inferior to those of the light-cured ones for SBU ($p < 0.05$).

Fisher's exact test indicated that there were no significant differences in failure modes among the groups regardless of light-curing and thermocycling ($p > 0.05$). Figure 2 shows the failure pattern distribution (%) as assigned using the light microscope. Although all modes of failures were observed in all test groups except ABU-NC, in which no cohesive failures were found, adhesive failures were predominant. Prevalence of adhesive failures appeared to increase with aging for the uncured groups, which was not shown in the pre-cured groups. Furthermore, after thermocycling, the uncured groups demonstrated a higher tendency of adhesive failures compared to the pre-cured groups.

Discussion

This *in vitro* study examined the effect of pre-curing the universal adhesives on the shear bond strengths of a luting resin to zirconia ceramic and compared it with that of the conventional zirconia primer. Since an adhesive functional monomer is not necessary in a resin cement if it is present in a primer for bonding to zirconia (18) and the present work focused on the bonding performance of MDP-containing priming agents, a composite-based resin cement (acidic monomers-free) Duo-Link was selected as the luting resin. The pre-cured two universal adhesives ABU and SBU yielded the superior initial bond strengths and the pre-cured SBU maintained the higher bond strength than the uncured one even after artificial aging. However, there were significant decreases in the bond strength after thermocycling, irrespective of light-curing or uncuring the two adhesives. Hence, the null hypothesis, which assumed pre-curing priming agents would not affect early bond strengths and bonding durability between a luting cement and zirconia ceramic, has to be partially rejected.

According to the respective manufacturer's instructions of the three priming agents, ABU should be light-cured for 10 seconds, SBU agitated for 20 seconds, and ZPP air-dried for 3-5 seconds before resin cement placement. Because of the lack of clarity in the clinical protocols, all the details about the application were standardized, thus enabling the light-curing to only influence the results. Considering that pre-curing improved 24-h bond strengths of ABU and SBU in this study, light-curing prior to cementation would be beneficial to zirconia bonding when using the universal adhesives as a priming agent. The inferior bonding performance of the uncured adhesives can be attributed to the collapse of the uncured layer and the resultant defective bonding interface caused by pressure during resin cement placement as described by Seabra et al. (14). It is commonly recommended that an adhesive applied on the internal surface of a restoration be kept unpolymerized before the restoration seated. As the thickness of a polymerized adhesive varies depending on both the adhesives type and the surface geometry of a restoration (19), a pooled adhesive may cause problems with restoration fit. However, from the clinical standpoint, co-curing of a luting resin and an underlying adhesive through opaque zirconia ceramic with limited light transmission leads to insufficient hardening of the adhesive joint, consequently contributing to early debonding failure of adhesive zirconia restorations (15, 20). Even though pre-curing failed to provide a durable bond between the two universal adhesives and zirconia, the tendency of adhesive failures to increase with thermocycling in the uncured adhesives may imply the formation of an unfavorable clinical bond without pre-curing.

Unlike the universal adhesives, light-curing the zirconia primer ZPP does not seem to significantly influence the early adhesion and long-term stability of resin-zirconia interface in the present study, which is in contrast to a previous study that demonstrated increased shear bond strengths with light-polymerized, two coats of ZPP (14). Although an uncured adhesive layer is seemingly unable to form a fully polymerized stable interphase due to its thinning-away between a luting resin and a restoration as well as light attenuation underneath zirconia ceramic as aforementioned (20, 21), this may be less apparent in ZPP, when considering the comparable bond strength results obtained in the cured and uncured ZPP. This result can be partly explained by a lower contact angle and better wettability of ZPP on zirconia surface than those of a universal adhesive (6).

As regard the long-term stability of the bond, the use of ZPP for conditioning zirconia can be advantageous rather than applying the universal adhesives, reflecting the differences in the chemical composition of the materials. Aside from the common ingredients such as MDP, HEMA, Bis-GMA, and ethanol in the three priming agents tested, water is incorporated in the universal adhesives for the ionization of acidic functional monomers. The addition of water renders the materials more susceptible to hydrolytic degradation especially under acidic aqueous conditions. Water mixed with acidic monomers becomes acidified and can hydrolyze the ester bonds of the unreacted methacrylate monomers and polymer networks, thereby compromise the integrity of resin-zirconia bond over time (22). The pH values for the two universal adhesives range from 2.4 ± 0.05 to 3.0 ± 0.05 (23). Meanwhile, ZPP has a carboxylate functional monomer BPDM in addition to MDP, which was reported to cooperate in the development of a chemical bond with metal oxide in the substrate (24). The synergistic coupling of these two monomers might result in the stable bonding in the current study. The increased hydrophobicity created by incorporating Bis-GMA in ZPP was claimed to offer a high priming efficacy to allow uniform spreading and improved adaptation of a resin cement (25). However, the presence of the extra resin does not appear to contribute to the bonding durability obtained with ZPP, because the other two universal adhesives are basically Bis-GMA-based.

SBU contains a silane primer to expand its use to silica-based ceramics. Although not significant, SBU exhibited the highest bond strength (23.8 ± 3.5 MPa) after 24-h storage, which was significantly decreased after additional thermocycling. The superiority in the initial bond strength is thought to be due to that the silane promoted wettability of the zirconia surface and thus enhanced spreading of the resin cement into the primed surface (26). However, while the increased wettability of the bonding surface could be a positive factor for the initial adhesion to zirconia, hydrophilic constituents from the silane film formed on the zirconia surface would be eventually detrimental to a long-lasting bonding with resin (27). Therefore, one of the critical steps during the application of any water-based adhesive, including universal adhesives, is evaporating the undesirable ingredients such as water, solvent and other reaction products from the bonding surface (28, 29). Meanwhile, Vitrebond copolymer in SBU was found to play a role in the improved initial bond strength and bonding durability of a resin cement to zirconia ceramic owing to its moisture-stabilizing effect (30-32). However, according to the reduction in the bond strength after 5,000 thermal cycles in this study, the inclusion of Vitrebond copolymer is not likely to result in any additional improvement in bond stability.

The degree of hydrophobicity and hydrophilicity of a primer-treated zirconia surface can be determined using contact angle measurements (33). Since the bond durability and interfacial characteristics of universal adhesives rely on the photo-irradiation conditions such as light intensity and irradiation time (34) and the bonding performance is related to the surface hydrophobicity of primer-coated zirconia (35), the surface characteristics of zirconia before and after light-curing priming agents obviously needs further investigation.

Adhesion is susceptible to mechanical, chemical, and thermal degradation in the oral environment (36). Even though bond strengths attained by the universal adhesives significantly decreased after thermocycling, they were still a bit higher than the suggested minimum value (10-13 MPa) for clinically acceptable bonding (37). However, considering the service life of zirconia restorations and the harsh conditions of the oral cavity (38), 5,000 thermal cycles used in the present study does not seem sufficient to simulate clinical conditions. Although a standardized, reliable thermal cycling protocol has not been developed yet, long-term water aging is required to evaluate the stability of the resin-zirconia interface formed by these new materials.

Conclusions

Within the limitations of this *in vitro* study, it can be concluded that when universal adhesives are used for surface treatment of zirconia ceramic, they should be light-cured prior to application of a resin cement, which may lead to significant improvement in early bond strengths. A conventional zirconia primer, Z-Prime Plus seems to provide a durable bond between a resin cement and zirconia. When adhesively cementing zirconia restorations, universal adhesives can be advantageously used for the both substrates tooth and zirconia without a need for a separate primer. However, despite the benefits of the simplified clinical technique, they may not show good performance in terms of bond stability compared to a conventional zirconia primer.

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Original Article

Effect of pre-curing of two universal adhesives on the shear bond strength of resin cement to zirconia

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This study aimed to investigate whether pre-curing the universal adhesives affect the resin bonding to zirconia ceramic. Two commercial universal adhesives (All-Bond Universal, ABU; Single Bond Universal Adhesive, SBU) were tested in comparison with a conventional zirconia primer (Z-Prime Plus, ZPP). Air-abraded zirconia specimens were divided into six groups, to which different priming agents were applied with or without photo-polymerization. After resin cylinders were built on the primed zirconia surface using a resin cement (Duo-Link), all bonded specimens were stored in water at 37℃ for 24 hours and half of them (n = 12) were additionally thermocycled 5,000 times. A shear bond strength test was performed at a crosshead speed of 0.5 mm/minute and failure modes were assessed using an optical microscope at 25× magnification. Before thermocycling, the light-cured specimens of ABU and SBU showed higher bond strengths than the uncured ones of the materials ($p<0.05$), whereas no significant difference was found between the pre-cured and uncured specimens of ZPP ($p > 0.05$). After thermal aging, there were significant decreases in the bond strength for all specimens treated with the universal adhesives ($p<0.05$). However, ZPP produced no significant difference after aging ($p > 0.05$) and attained higher bond strength values for both the pre-cured and uncured specimens when compared to the two universal adhesives ($p<0.05$). Adhesive failures were predominant in all test groups and thermocycling resulted in the higher incidences of adhesive failures for the uncured groups, which was not shown in the pre-cured groups.

Key Words : Pre-curing, Primer, Shear Bond Strength, Universal Adhesives, Zirconia