Comparative bond strength and adhesive layer thickness of self-adhesive resin cements to zirconia depending on light-curing of universal bonding

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Recently, as various self-adhesive resin cements have emerged, adhesion to zirconia materials has been carried out without additional adhesive bonding. However, there are still doubts about the adhesive strength when only self-adhesive resin cement is used alone, and there is a claim that a better adhesive strength can be achieved by using universal adhesive bonding. The purpose of this study is to compare the adhesive strength and adhesive layer thickness to zirconia depending on light curing when adding universal adhesive bonding to self-adhesive resin cement.

Ninety-eight zirconia specimen block (10mm x 10mm x 10mm) was prepared and embedded in acrylic resin blocks. The four different surface-conditioning agents Single Bond Universal (SBU), All-Bond Universal (ABU), Prime&Bond Universal (PBU), Z-prime plus(ZPP) applied to zirconia surfaces. And then, the groups were divided according to the presence of pre-curing. All specimen surfaces were bonded with RelyX U-200 resin cement cylinders and subjected to thermocycling for 10,000 cycles between 5°C and 55°C. The shear bond strengths, fracture type and bonding layer thickness were evaluated. Statistical analyses were performed by analysis of variance and oneway ANOVA, at a significance level of 95%.

All agents used showed an increase in bond strength compared to the control group. Prime&Bond Universal showed significantly lower values than the other three bonding

agents with pre or co-curing ($p<0.05$). There was no significant difference between pre and co-curing group. The mean bonding thickness using SBU, ABU, PBU with co-curing was 10.34μm, 9.28μm, and 9.14μm respectively, and with pre-curing, the thicknesses were 10.52μm, 9.42μm, and 10.02μm respectively. All three agents showed a slight increase in thickness with pre-curing. Among the three universal adhesive agents, adhesive failures (70% to 100%) were dominant with co-curing, but in the group using ABU and PBU, the ratio of mixed failure increased with pre-curing.

It can be concluded that universal bonding agents and self-adhesive resin cement could replace the conventional zirconia primer and make the clinical situation simpler and less time consuming.

Keyword: Universal adhesive, self-adhesive resin cement, zirconia restoration, shear-bond strength, bonding layer thickness

Comparative bond strength and adhesive layer thickness of self-adhesive resin cements to zirconia depending on light-curing of universal bonding

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I. Introduction

As expectations for aesthetics increase, interest in non-metallic restorations is increasing, and this demand extends beyond the anterior to the posterior.[1] Recently developed zirconia ceramics are widely used in the field of dental restorations, such as fixed prostheses, posts, and implant abutments because of their high mechanical strength. Advances in CAD / CAM technology have further increased the use of all-ceramic restorations using zirconia ceramics.

Zirconia has a very stable chemical structure with excellent biocompatibility, as well as a hard and dense structure with high wear resistance. This surface stability requires a different adhesion method from the existing all-ceramic restorations using hydrofluoric acid or silane treatment.[2-5]

A bonding agent using a functional monomer for zirconia adhesion has been introduced that induces a reaction between methacrylic acid and phosphoric acid.[5-7] This new type of bonding agent contains a functional monomer, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) and simplifies the conditioning process of the tooth and restoration surfaces. This MDP-containing bonding agent can be used for both total-etch and self-etch, hence the name 'universal' adhesive.[8] In addition, it can adhere not only to the tooth, but also to metal, ceramic, and zirconia materials.[9]

Moreover, adhesive resin cements have been developed that can increase the chemical bonding force and simplify the adhesive process. They contain functional monomers such as 10-MDP and 4-META and adhere to metal-, alumina-, and zirconia-based ceramics.[9] The phosphate-based functional monomers of self-adhesive resin cements shorten the procedure time, reduce the possibility of contamination, and can form stable bonds with metal and ceramic restorations. More specifically, monomers such as 10-MDP directly bond with zirconia through chemical bonds.[10, 11]

As various self-adhesive resin cements have emerged, adhesion to zirconia materials has been carried out without additional adhesive bonding. However, there are still doubts about the adhesive strength when a self-adhesive resin cement is used alone; in addition, it is claimed that a better adhesive strength can be achieved using universal adhesive bonding.

When bonding is applied to the inner surface of the crown before the cement for zirconia crown adhesion, the manufacturer recommends applying cement after light-curing of the adhesive (pre-curing). However, some claim that the fit of the crown is compromised because a thick adhesive layer is formed during light-curing.[12] Therefore, it is recommended to use cement without light-curing (co-curing after cementation) after adhesive application or to replace the adhesive with zirconia primer. However, when co-curing, deterioration of bond strength is suspected as it is against the manufacturers' recommendation, and when using the zirconia primer, the challenge is the need to purchase and use additional materials.

Studies of the differences in zirconia adhesive strength and adhesive layer thickness depending on light-curing when using universal adhesive before self-adhesive resin cement use have not yet been conducted.

The purpose of this study is to compare the adhesive strength and adhesive layer thickness to zirconia depending on light-curing when adding universal adhesive bonding to self-adhesive resin cement. The null hypothesis was that there is significant difference in bond strength, and there is no increase in bonding layer thickness when pre-curing universal adhesive.

II. Materials and methods

Preparation of zirconia specimens

A partially sintered zirconia block (Cercon base, DeguDent, Hanau, Germany) was sectioned to obtain 98 specimen blocks, which were then sintered according to the manufacturer's instructions. The fully sintered specimens (10 mm x 10 mm x 10 mm) were embedded in acrylic resin blocks. The zirconia surface was polished with up to 600-grit silicon carbide paper under water cooling; thereafter, the samples were subjected to ultrasonic cleaning in isopropyl alcohol for 3 minutes (Fig. 1).

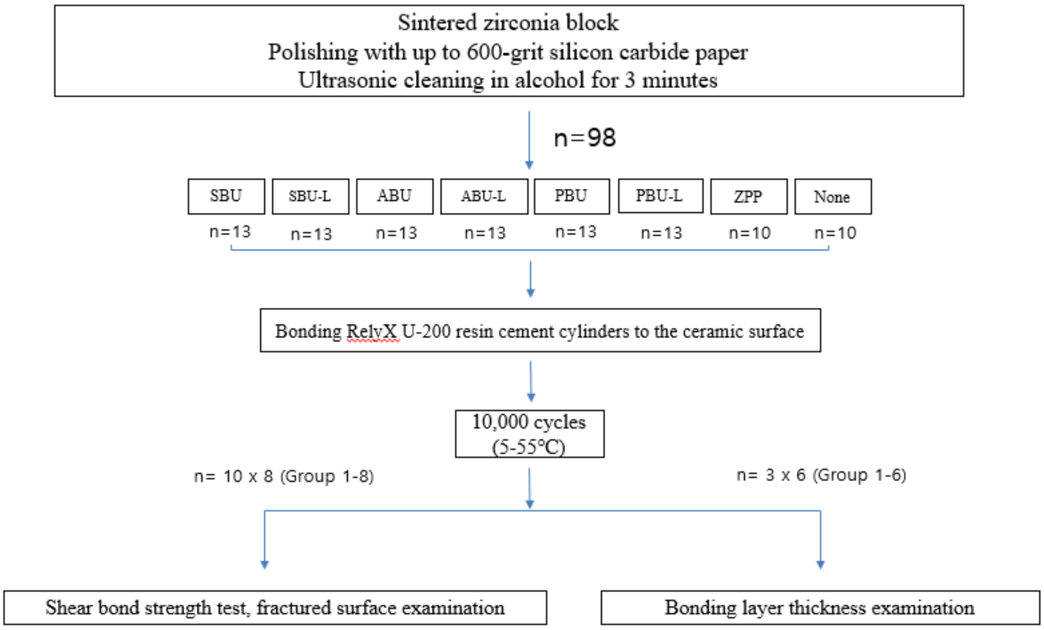

Figure 1. Experimental design on whether light curing was done before or after resin cement application on yttria-tetragonal zirconia polycrystal (Y-TZP) specimens in this study. SBU: Co-curing of Single Bond Universal, SBU-L: Pre-curing of Single Bond Universal, ABU: Co-curing

of All-Bond Universal, ABU-L: Pre-curing of All-Bond Universal, PBU: Co-curing of Prime&Bond Universal, PBU-L: Pre-curing of Prime&Bond Universla, ZPP: Z-prime plus, Conrol: None

Experimental groups and adhesive applications

The specimens were randomly divided into eight groups of 10 specimens each according to the applied surface-conditioning agent.

Group SBU: Co-curing of Single Bond Universal

Group SBU-L: Pre-curing of Single Bond Universal

Group ABU: Co-curing of All-Bond Universal

Group ABU-L: Pre-curing of All-Bond Universal

Group PBU: Co-curing of Prime&Bond Universal

Group PBU-L: Pre-curing of Prime&Bond Universal

Group ZPP: Z-prime plus

Group control: None

The four different surface-conditioning agents used in this study are summarized in Table 1.

Table 1. Compositions of the materials used in this study

Material	Abbreviation	Manufacturer	Composition	Lot number
Single Bond Universal	SBU	3M ESPE, St Paul, MN, USA	MDP, bis-GMA, HEMA, DMA, methacrylate functional copolymer, filler, ethanol, water, initiators, silane	1400306119

All-Bond Universal	ABU	Bisco Inc, Schaumburg, IL, USA	MDP, bis-GMA, HEMA, ethanol, water, initiators	1900004577
Prime&Bond Universal	PBU	Dentsply Sirona, Milford DE, USA	Bi-and multifunctional acrylatel, PENTA, phosphoric acid modified acrylate resin, initiator, stabilizer, isopropanol, water	LT00897089
Z-prime plus	ZPP	Bisco Inc., Schumburg, IL, USA	BPDM, HEMA, ethanol, MDP	1800006387
RelyX U-200		3M ESPE, St Paul, MN, USA	Base: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, initiators, stabilizers Catalyst: Methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers	4325505

HEMA: 2-hydroxyethyl methacrylate; MDP: methacryloyloxydecyl dihydrogen phosphate; bis-GMA: bisphenol glycidyl methacrylate; TEGDMA: Triethylene glycol dimethacrylate; PENTA: dipentaery thritol penta acrylate monophosphate.

Preparation of composite cylinders

Composite cylinders (2 mm in diameter and 10 mm in height) were fabricated. Four different MDP-containing agents were applied to the polished zirconia specimens strictly in accordance with the respective manufacturer's instructions. Resin cement (RelyX U200, 3M ESPE) was applied to the composite cylinder, which was then placed on the zirconia specimen. After excess resin cement was removed with a microbrush, glycerin gel was applied around the bonded interface. The resin cement was light-cured from four directions for 20 s per side with the LED curing unit (VALO light irradiator, Ultradent, South Jordan, UT). After 30 minutes at room temperature, the bonded specimens were stored in distilled water at 37 °C for 24 h.

Shear bond strength measurements

All specimens of each group were subjected to thermocycling for 10,000 cycles between 5 and 55 °C with a 25 s-dwell time before the bond-strength test. The shear bond-strength test was performed with a universal testing machine (LF Plus, Lloyd Instruments, Fareham, UK). The shear force was applied at a crosshead speed of 0.5 mm/minutes until failure occurred (Fig. 2). The shear bonding forces were recorded in newtons (N) and altered as megapascals (MPa) by dividing by the bonding surface area (mm^2).

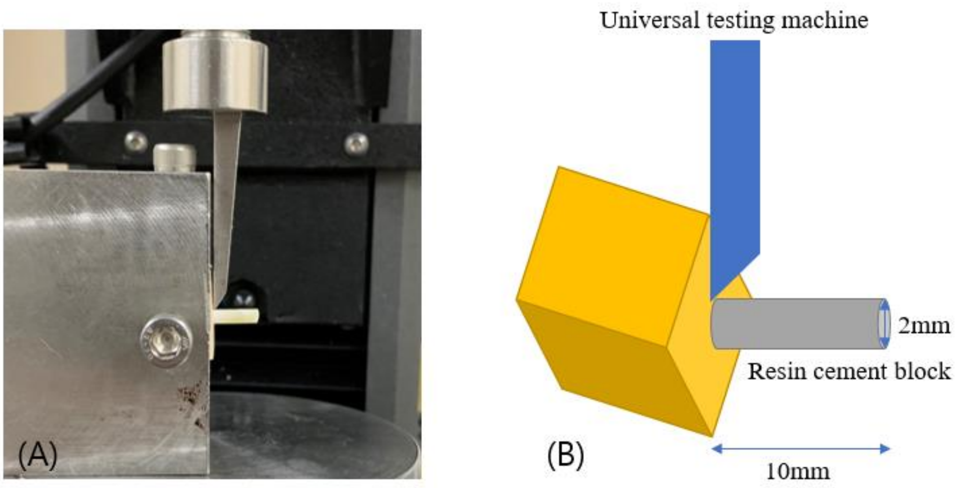

Figure 2. (A) Zirconia specimen fixed onto the universal testing machine; (B) Schematic diagram.

Bonding layer thickness by scanning electron microscopy analysis

The remaining 18 specimens were divided into six groups according to the use of SBU, ABU, and PBU. The thickness of the bonding layer after co-curing and pre-curing was measured using scanning electron microscopy (SEM).

Failure types analysis

The fractured surfaces of the specimens were examined with an optical microscope (OPMI pico, Carl Zeiss, Gottingen, Germany) at 40x magnification to determine the failure mode. The failure was classified into two types: (i) adhesive failure (occurring between the zirconia ceramic and the resin cement); and (ii) mixed failure (adhesive failure and cohesive fracture occurred simultaneously within the resin cement). In cases of mixed failure, the surface of the zirconia was partially covered by the remaining resin cement.

Statistical analysis

Bond-strength data were analyzed using a statistical software (SPSS, SPSS Inc, Chicago, IL, USA). One-way ANOVA was conducted, where a P-value less than .05 was considered statistically significant. After all statistical analyses were conducted, the Scheffe test was performed to assess the differences among the surface-conditioning agents.

III. Results

Shear bond strength measurements

The mean shear bond strength values and standard deviations are shown in Table 2 and Fig. 3. All agents used showed an increase in bond strength compared with the control group. With co-curing, group 1 (SBU) showed the highest value, and with pre-curing, group 4 (ABU-L) showed the highest value. Prime&Bond Universal showed significantly lower values than the other three bonding agents with pre- or co-curing ($p < 0.05$). Although there was no significant difference, the bond strength increased with pre-curing in all groups, except the group using SBU. SBU and ABU did not show any significant difference with the conventional MDP-containing zirconia primer (Z-prime plus), regardless of light-curing ($P < .05$).

Table 2. Mean shear bond strengths and standard deviations (SD) (n=10)

Group	Adhesive	Shear bond strength (MPa)	SD
1	SBU	52.58	16.25
2	SBU-L	47.66	4.78
3	ABU	43.74	18.1
4	ABU-L	48.76	21.43
5	PBU	21.18	9.86
6	PBU-L	23.8	12.95
7	ZPP	40.29	12.07
8	none(control)	12.1	4.31

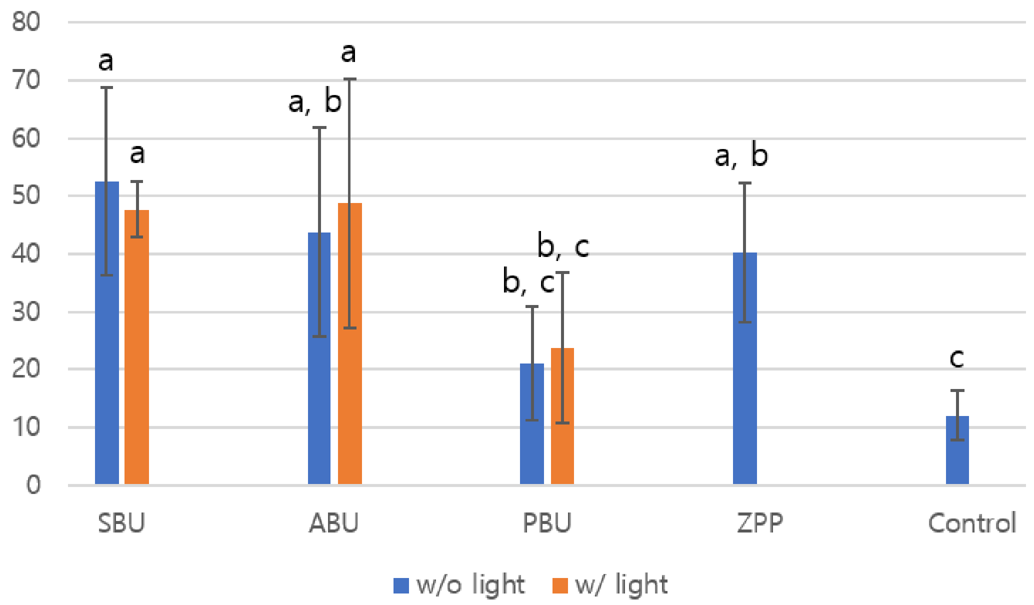

Figure 3. Mean shear bond strengths.

The error bars represent standard deviations (n=10). a,b,c: Different lowercase letters above the bars represent statistically significant difference ($p<0.05$)

Bonding layer thickness by scanning electron microscopy analysis

The average bonding layer thickness and SEM images of the bonding after pre- or co-curing are shown in Fig. 4 and Fig. 5. The mean bonding thicknesses using SBU, ABU, and PBU were 10.34, 9.28, and 9.14 μm, respectively, with co-curing, and 10.52, 9.42, and 10.02 μm, respectively, with pre-curing. All three agents showed a slight increase in thickness with pre-curing.

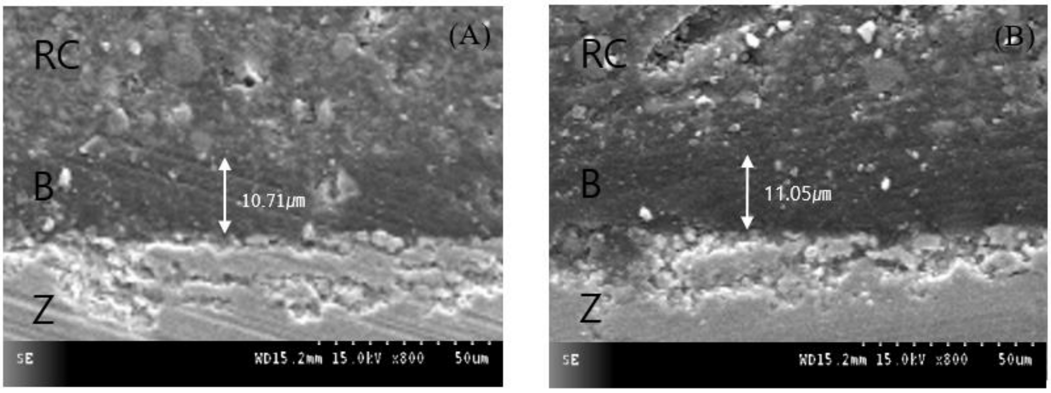

Figure 4. Representative and characteristic SEM micrographs showing Prime&Bond Universal bonding thickness (B) between Resin cement (RC) and Zirconia (Z) with pre or co-curing (800x magnification). (A) Bonding thickness with co-curing; (B) Bonding thickness with pre-curing.

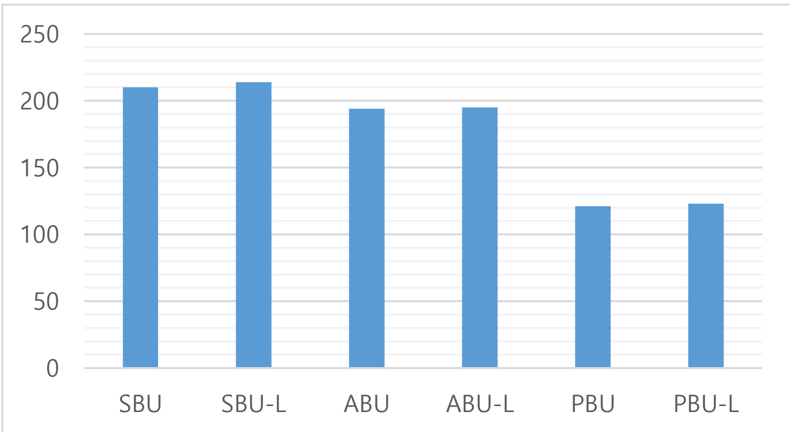

Figure 5. The Average bonding layer thickness with pre or co-curing using three MDP-containing bonding agents.

Failure types analysis

The distributions of failure modes after pre- and co-curing are shown in Fig. 6. All specimens in group 5 (PBU) showed adhesive failure after fracture. Among the three universal adhesive agents, adhesive failures (70% to 100%) were dominant with co-curing, but in the group using ABU and PBU, the ratio of mixed failure increased with pre-curing.

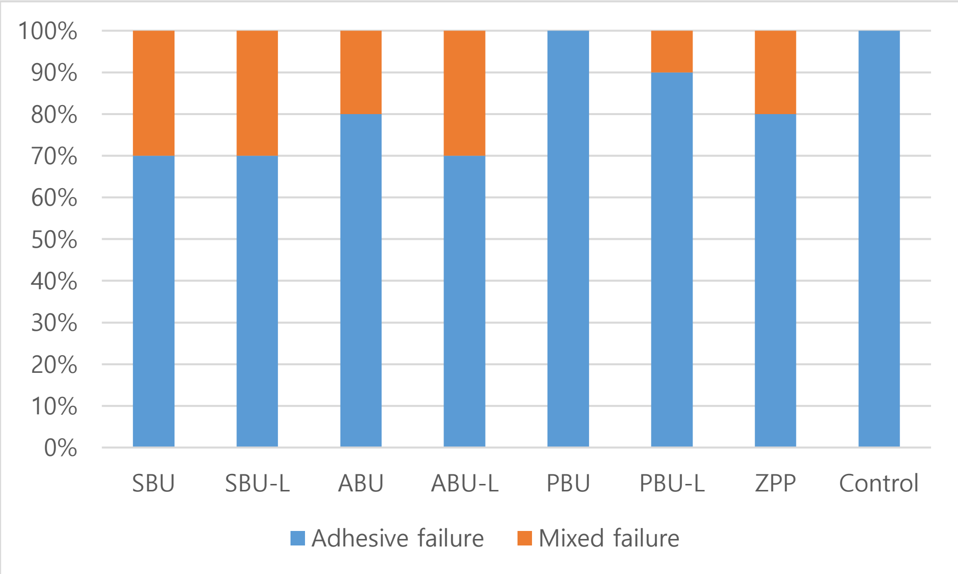

Figure 6. Percentage distribution of failure modes pre or co-curing

IV. Discussion

The aim of this study was to observe the change in shear bond strength and thickness depending on pre- or co-curing of universal bonding agents when used with a self-adhesive resin cement, which has recently been used to bond zirconia, and to compare with the conventional MDP-containing zirconia primer. Universal bonding agent and self-adhesive resin cement are often used together to reduce the treatment time and the possibility of contamination because of the simplicity of the procedure,[3, 13, 14] but studies on multi-purpose agents and MDP-containing resin cement are scarce. In this experiment, the null hypothesis was rejected, there was no significant difference in bond strength after pre-curing, and an increase in bonding layer thickness was observed. Shear bond[15-19] or microtensile bond[20-22] testing is often used to measure the bond strength between resin and zirconia. However, both methods have limitations in measuring true adhesion strength. Microtensile tests are useful for removing macro-size flaws during specimen preparation and can be used to apply a homogeneous force to the adhesive surface, thereby reproducing the ideal adhesion test.[23] Unfortunately, it is time-consuming to prepare a specimen for measurement using this method, and damage to the bonding interface may occur when cutting the specimen because of the high physical properties of zirconia. In addition, there are reports that a large number of premature failures are observed in specimens.[24] Conversely, shear bond-strength tests are limited by the fact that uneven force is applied to the adhesive interface, which may cause cohesive failure of the specimen;[25, 26] however, because of the high physical properties of zirconia, cohesive failure of zirconia is rarely observed.

In this study, in the shear bond tests, SBU, ABU, and ZPP showed significantly higher values than PBU and the control. These values were slightly higher than the findings by Lee, Kim [27] and Kim, Chae [28]; these studies differed from this study because they used a resin cement that does not contain MDP. The difference in shear bond strength may be a result of the RelyX U-200 used in this study, which contains MDP. The high bond strength may have been achieved by the phosphate group of the MDP interacting with the hydroxyl groups on the zirconia surface via van der Waals forces or hydrogen bonds.[6, 9] In this study, all groups except PBU showed significantly higher bond strength than that of the control group. In addition, the MDP components in the universal bonding agent have strong bonds with enamel, dentin, and metal and form chemical bonds with zirconia (Zr-O-P).[8, 29] The advantage of a universal adhesive is that it can attach to various substrates and can achieve suitable bonding strength without special treatment at the adhesive interface. Furthermore, because it is easy to use in the clinic because of the simplicity of the procedure, it is considered a suitable alternative to conventional primers.

PBU is a recently developed universal bonding agent that showed a significantly lower bonding strength in this study. Unlike SBU and ABU, PBU contains not only MDP but also the phosphoric acid modified acrylate resin PENTA (dipentaerythritol pentacrylate phosphate), and this functional monomer may have been affected during thermocycling. In this study, all specimens were thermocycled for 10,000 cycles, which is generally accepted to represent 1 year of clinical use.[30] Bond strength tests were not conducted before thermocycling, and studies on adhesion of PBU and zirconia are very rare; therefore, further investigation is needed.

When pre-cured, although not significant, ABU and PBU showed an increase in bond strength, and in the case of SBU, adhesive strength decreased. The manufacturers' instructions recommend pre-curing for ABU and PBU. When a pre-cured adhesive forms a uniform hybrid layer, it acts as an elastic barrier for the cavity-absorbing part of the polymerization shrinkage of the resin cement, thereby improving the marginal fit, reducing the microleakage of the restoration,[31, 32] and increasing the bond strength.[33, 34] Conversely, if the pre-curing process of the adhesive is skipped and co-curing is performed with the resin cement, the adhesive layer is depressed by the pressure of resin cement above the unpolymerized adhesive, resulting in low bonding strength.[35] The adhesive located under the composite resin may cause incomplete polymerization as a result of insufficient light transmission because of thickness of the restoration, thus decreasing the bonding strength.[36, 37] Conversely, the SBU showed better performance with co-curing. The SBU manufacturer's instruction does not recommend light-curing the adhesive prior to indirect cementation. Considering the decrease in adhesive strength and increase in thickness when pre-curing SBU, it is recommended to not pre-cure SBU as instructed by the manufacturer.

In this study, the mean film thicknesses of SBU, ABU, and PBU showed a slight increase, from 10.34, 9.28, and 9.14 μm to 10.52, 9.42, and 10.02 μm, respectively, when pre-cured. Although the above mean film thickness is so thin that it does not interfere with the fit of the indirect restoration,[38] it may interfere with the fit of the indirect restoration in a particular area because the film thicknesses are not uniform. Previous studies reported that the thickness of the bonding layer was not uniform and had a varying thicknesses.[39, 40] Because of the variability of bonding layer thickness, it is difficult to measure objective thickness in the SEM photograph.

Fracture mode was observed after the shear bond strength test. In the experiment, all PBU specimens showed adhesive failure with co-curing, demonstrating the lowest bond strength. An increase in mixed failure was seen only in the pre-cured ABU group. No cohesive failure was observed within the resin cement and zirconia specimens, indicating that the interface between the resin cement and the zirconia was the weakest part.

In this study, the effect of pre-curing on shear bond strength was dependent on the adhesive agent. Pre-curing the adhesive increased the adhesive layer thickness for all the bonding agents tested, but if used properly in the clinical situation, the setting of the indirect restoration may not be significantly affected. In fact, in this experimental design, light-curing was performed directly on zirconia and resin cement, but in a clinical situation, light-curing is performed on the outer surface of zirconia. Because of this, it may be difficult for curing light to penetrate the zirconia restoration, and sufficient polymerization may not be achieved. Further investigation is necessary to determine the influence of the pre-curing and adhesive thickness on indirect restorations.

V. Conclusions

Within the limitations of this study, the universal adhesives showed a similar degree of bonding strength to the conventional zirconia primer, and these multi-purpose bonding agents and self-adhesive resin could make the clinical situation simpler and less time-consuming. Considering the benefits and risks of bonding strength and bonding layer thickness, it is recommended that ABU and PBU be pre-cured and SBU not pre-cured.

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국문 초록

자가접착 레진 시멘트 사용 시 Universal adhesive 의 light-curing 여부에 따른 지르코니아에 대한 접착력 및 접착층의 두께 비교

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최근, 다양한 자가접착 레진시멘트가 등장함에 따라, 추가의 접착 과정 없이 지르코니아 재료에 대한 접착이 시행되었다. 그러나, 자가접착 레진시멘트 단독으로 사용하는 경우의 접착 강도에 대해서는 여전히 의문이 있으며, universal adhesive 를 사용함으로써 더 우수한 접착이 이루어 질 수 있다는 주장이 있다. 이 연구는 자가접착 레진시멘트에 universal adhesive 를 지르코니아 접착에 함께 사용할 때, 광중합에 따라 shear bond strength 와 bonding layer thickness 를 비교하고자 하였다.

96 개의 지르코니아 시편 (10mm x 10mm x 10mm)을 준비하여 레진블록에 고정시켰다. 지르코니아 표면에 4 가지의 bonding agent, SBU (Single Bond Universal), ABU (All-Bond Universal), PBU (Prime & Bond Universal), Z-prime plus (ZPP)를 적용하고 pre-curing 유무에 따라 그룹을 나누었다. 모든 시편 표면을 RelyX U-200 레진 시멘트와 접착하고 5 ℃와 55 ℃ 사이에서 10,000 사이클 동안 thermocycling 시행하였다. Shear bond strength, Fracture type, bonding layer

thickness 에 대해 평가하였다. 통계 분석은 95 %의 유의구간을 기준으로 ANOVA 로 통계적으로 분석하였다.

사용된 모든 agent는 대조군과 비교하여 bond strength의 증가를 보여 주었다. Prime & Bond Universal 은 pre 혹은 co-curing 된 다른 세 가지 bonding agent 보다 유의차 있게 낮은 값을 나타냈습니다 ($p <0.05$). pre 혹은 co-curing 그룹 사이에는 유의한 차이가 없었다. Co-curing 한 SBU, ABU, PBU 의 평균 bonding layer thickness 는 각각 10.34μm, 9.28μm 및 9.14μm 이고, co-curing 에서는 두께가 각각 10.52μm, 9.42μm 및 10.02μm 였다. 세 가지 agent 모두 pre-curing 시에 두께가 약간 증가한 것으로 나타났다. 3 가지 universal adhesive 중에서, Adhesive failure (70 % ~100 %)가 co-curing 시에 지배적 이었지만, ABU 및 PBU 를 사용하는 그룹에서, mixed failure 의 비율은 pre-curing 시에 증가 하였다.

Universal adhesive 및 자가 접착 레진 시멘트는 기존의 지르코니아 프라이머를 대체할 수 있으며 임상 상황을 보다 단순하고 술식 시간의 단축을 가져올 수 있다는 결론을 낼 수 있다.

핵심 되는 말: Universal adhesive, 자가 접착 레진 시멘트, 지르코니아 수복물, shear bond strength, 접착층 두께