



Effect of dentin pretreatment and curing mode on the microtensile bond strength of self-adhesive resin cements

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PURPOSE. The aim was to evaluate the effect of curing mode and different dentin surface pretreatment on microtensile bond strength (μ TBS) of self-adhesive resin cements. **MATERIALS AND METHODS.** Thirty-six extracted human permanent molars were sectioned horizontally exposing flat dentin surface. The teeth were divided into 12 groups (3 teeth/group) according to the dentin surface pretreatment methods (control, 18% EDTA, 10% Polyacrylic acid) and curing mode (self-curing vs. light-curing) of cement. After pretreatment, composite resin blocks were cemented with the following: (a) G-CEM LinkAce; (b) RelyX U200, followed by either self-curing or light-curing. After storage, the teeth were sectioned and μ TBS test was performed using a microtensile testing machine. The data was statistically analyzed using one-way ANOVA, Student T-test and Scheffe's post-hoc test at $P < .05$ level. **RESULTS.** For G-CEM LinkAce cement groups, polyacrylic acid pretreatment showed the highest μ TBS in the self-cured group. In the light-cured group, no significant improvements were observed according to the dentin surface pretreatment. There were no significant differences between curing modes. Both dentin surface pretreatment methods helped to increase the μ TBS of RelyX U200 resin cement significantly and degree of pretreatment effect was similar. No significant differences were found regarding curing modes except control groups. In the comparisons of two self-adhesive resin cements, all groups within the same pretreatment and curing mode were significantly different excluding self-cured control groups. **CONCLUSION.** Selecting RelyX U200 used in this study and application of dentin surface pretreatment with EDTA and polyacrylic acid might be recommended to enhance the bond strength of cement to dentin. [*J Adv Prosthodont 2015;7:317-22*]

KEY WORDS: Self-adhesive resin cements; Curing mode; Dentin surface pretreatment; Bond strength

INTRODUCTION

The increasing patients' and dentists' interest in tooth-colored restorations has led to the invention of many esthetic restoration materials and cements. Resin cements are cru-

cial materials for the longevity and durability of esthetic indirect restorations. Compared to conventional luting cements, resin cements provide improved retention, less microleakage, minimal solubility, and acceptable biocompatibility.¹ On the other hand, these resin cements require dentin surface pretreatment to modify or remove the smear layer, and acquire multiple steps to prime the collagen fibers and infiltrate the adhesive resin monomers to form a uniform hybrid layer.¹

Self-adhesive resin cements were introduced approximately 10 years ago to overcome the limitations of complex multistep applications, susceptibility to moisture and possible postoperative sensitivity of conventional resin cements as well as to simplify the bonding process.^{2,3} These cements combine an adhesive and a cement in a single application, eliminating the need for an additional pretreatment of the dentin surface.⁴

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Acidic monomers in self-adhesive resin cements demineralize and infiltrate the tooth substrate, providing micro-mechanical retention.⁴ Simultaneously, the reaction between the phosphoric acid monomers of the cements and hydroxyapatite of the tooth substrate can offer chemical retention.^{1,5}

On the other hand, many studies have reported the poor adhesion to dentin and the low bond strength as drawbacks of self-adhesive resin cements.^{1,2,6} Although they reduced the technique and postoperative sensitivity, the limited etching potential and superficial interaction with the dentin surface provided a lower bond strength than conventional resin cements.⁴ The smear layer-covered dentin surface impedes the proper infiltration of cement into dentin, and cement itself is too viscous to penetrate into the demineralized collagen fiber network.⁴

With the aim of enhancing the interaction between resin cement and dentin, many attempts have been made to remove or alter the smear layer of the dentin surface before applying the self-adhesive resin cements.^{1,3,4,6-8} The dentin surface can be treated either mechanically or chemically. The most common mechanical cleaning technique is using a rotary instrument with pumice or sandblasting with aluminum oxide particles.^{6,9-12} The chemical cleaning techniques include many chemical agents, such as chlorhexidine digluconate, sodium hypochlorite, hydrogen peroxides, polyacrylic acids (PA), and ethylenediaminetetraacetic acid (EDTA).^{3,4,6,8,13}

Most resin cements are dual-cured containing both self-cured and light-cured components and can initiate a polymerization reaction without light exposure.¹⁴ The cements were developed as dual-polymerization to compensate for the light attenuation either by the distance from the light source or by the thickness and opacity of the restoration.¹⁴⁻¹⁶ Nevertheless, several studies have demonstrated that self-curing alone is not as effective as light activation in a dual-cured resin cements when evaluating the degree of conversion, cement hardness, rate of polymerization, solubility, and bond strength.¹⁴⁻¹⁷ In other words, light-curing mode have a higher bond strength than self-curing mode when dual-cured resin cements are used.

G-CEM LinkAce™ (GC Corp., Tokyo, Japan) was introduced recently. It gives a reasonably high bond strength when self-cured that is comparable to light-cured cements.

This study examined the effects of different dentin surface pretreatments (EDTA vs. PA) and curing modes (self-curing vs. light-curing) on the microtensile bond strength (μ TBS) of self-adhesive resin cements.

MATERIALS AND METHODS

Thirty-six caries-free extracted human permanent molars were used in this study. This study was approved by the Institutional Review Board of Pusan National University Dental Hospital (IRB, PNUDH-2014-004). The teeth were cleaned and stored in distilled water at room temperature until used. The root portions of the teeth were embedded to the cervical level in self-cured acrylic resin (Tokuso

Curefast, Tokuyama, Tokyo, Japan) using a prepared square plastic mold. After removal from the plastic mold, the teeth were sectioned horizontally at the mid-coronal level to obtain a flat, sound dentin surface using a diamond saw (Accutom-50, Struers, Rødovre, Denmark) with constant water cooling. To create a uniform smear layer, the sectioned dentin surface was hand-polished with 600-grit silicon carbide abrasive paper for 60 seconds under running water and rinsed for 30 seconds with distilled water prior to cementation.

Composite resin blocks were made by increment layering of a 2-mm-thick light-cured composite resin (Filtek Z-250; 3M ESPE, St. Paul, MN, USA) into a cylindrical plastic mold (9 mm in diameter, 4 mm in thickness). After layering, the composite resin blocks were polymerized for 40 seconds using a LED light curing unit (BluePhase G2, Ivoclar Vivadent Inc., Amherst, NY, USA).

The teeth were divided randomly into 12 groups (3 teeth/group) according to the dentin surface pretreatment methods [control, 18% EDTA (Vision-PREP EDTA Gel, Metabiomed, Chungbuk, Korea), 10% PA (Dentin Conditioner, GC Corp., Tokyo, Japan)] and curing mode (self-curing vs. light-curing) of the cement. Two different self-adhesive resin cements, G-CEM LinkAce™ and RelyX™ U200 (3M ESPE, St. Paul, MN, USA), were used, and Table 1 lists their compositions and application procedures.

According to the dentin surface pretreatment, two experimental groups (n=10) were prepared: (a) 18% EDTA scrubbed with cotton ball for 60 seconds and rinsed with distilled water for 30 seconds; and (b) 10% PA scrubbed for 20 seconds and rinsed with distilled water for 30 seconds. Dentin surface without pretreatment were used as control. All treated surfaces were dried without desiccation.

After pretreatment, the composite resin blocks were cemented onto the pretreated dentin surface with the following: (a) G-CEM LinkAce™ and (b) RelyX™ U200, followed by either self-curing for 30 minutes or light-curing for 40 seconds at each side. The cemented teeth were stored in distilled water at room temperature for 24 hours.

The cemented teeth were sectioned vertically to produce a 1-mm-thick and 10-mm-long stick using a diamond saw with copious amount of flowing water. Out of 3 cemented teeth per each group, specimens which were appropriate for microtensile test were selected randomly, so that each group contained 10 specimens. Each stick was glued to the jig of the microtensile testing machine (Bisco, Schaumburg, IL, USA) with cyanoacrylic cement (Zapit, Dental Ventures of America, Corona, CA, USA) and was stressed to failure in tension at a crosshead speed of 1.0 mm/min. The maximum load at failure was recorded in MPa.

SPSS 15.0 software (SPSS Inc., Chicago, IL, USA) was used for the statistical analysis. A one-way analysis of variance (ANOVA) test was applied to test the significance of the differences between different dentin surface pretreatments, and a Student t-test was used to compare the differences within two curing modes (self-curing vs. light-curing)

Table 1. Compositions and application procedures of materials used in this study

Material	Composition	Application procedure
Vision-PREP EDTA Gel (Metabiomed, Chungbuk, Korea)	18% EDTA	<ul style="list-style-type: none"> • Scrub with cotton ball for 60 sec. • Rinse with distilled water for 30 sec. • Air Dry
Dentin Conditioner (GC Corp., Tokyo, Japan)	10% Polyacrylic Acid	<ul style="list-style-type: none"> • Scrub for 20 sec. • Rinse with distilled water for 30 sec. • Air Dry
Filtek Z-250 (3M ESPE, St. Paul, MN, USA)	Bis-GMA, UDMA, Bis-EMA, zirconia, silica	<ul style="list-style-type: none"> • 2 mm increment layering into cylindrical mold to 4 mm height • Light-cure for 20 sec. for each increment
G-CEM LinkAce™ (GC Corp., Tokyo, Japan)	Paste A: Fluoro-alumino-silicate glass, UDMA, dimethacrylate, silicon dioxide, initiator, inhibitor Paste B: Silicon dioxide, UDMA, dimethacrylate, initiator, inhibitor	<ul style="list-style-type: none"> • Apply cement through auto-mixing tip • Either self-cure for 30 min. or light-cure for 40 sec. at each side
RelyX™ U200 (3M ESPE, St. Paul, MN, USA)	Base paste: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizer, rheological additives Catalyst paste: Methacrylate monomers, alkaline fillers, silanated fillers, stabilizer, initiator components, pigments, rheological additives	<ul style="list-style-type: none"> • Apply cement through auto-mixing tip • Either self-cure for 30 min. or light-cure for 40 sec. at each side

EDTA: ethylenediaminetetraacetic acid, Bis-GMA: bisphenol A glycidyl methacrylate, UDMA: urethane dimethacrylate, Bis-EMA: ethoxylated bisphenol A dimethacrylate

Table 2. Effect of dentin pretreatment and curing mode on μ TBS (mean \pm SD in MPa) of G-CEM LinkAce™ U200

	Control	EDTA	PA
SC	10.16 \pm 2.15 ^a	12.06 \pm 3.15 ^{ab}	14.79 \pm 4.55 ^b
LC	11.79 \pm 2.06 ^a	13.22 \pm 2.90 ^a	13.72 \pm 1.89 ^a
t-test	$P > .05$	$P > .05$	$P > .05$

Different superscript letters in the row indicate statistically significant difference ($P < .05$).

μ TBS: microtensile bond strength, SD: standard deviation, EDTA: ethylenediaminetetraacetic acid, PA: polyacrylic acid, SC: self-curing, LC: light-curing

Table 3. Effect of dentin pretreatment and curing mode on μ TBS (mean \pm SD in MPa) of RelyX™ U200

	Control	EDTA	PA
SC	10.78 \pm 1.80 ^a	22.19 \pm 2.80 ^b	24.50 \pm 3.92 ^b
LC	18.03 \pm 2.94 ^a	22.84 \pm 4.11 ^b	23.35 \pm 3.14 ^b
t-test	$P < .05$	$P > .05$	$P > .05$

Different superscript letters in the row indicate statistically significant difference ($P < .05$).

μ TBS: microtensile bond strength, SD: standard deviation, EDTA: ethylenediaminetetraacetic acid, PA: polyacrylic acid, SC: self-curing, LC: light-curing

and to compare the differences between two self-adhesive resin cements. A Scheffe's test was used for a post hoc multiple comparison. All statistical tests were considered significant at $P < .05$.

RESULTS

Table 2 and 3 list the μ TBS of each cements.

In the self-cured group of the G-CEM LinkAce™ cement, the PA group showed the highest μ TBS followed in order by the EDTA and control group. The PA group showed significantly higher bond strength than the control

group ($P < .05$), but no significant differences were noted between the PA group and EDTA group ($P > .05$). In the light-cured group, no differences were observed, irrespective of the dentin surface pretreatment. Within the same surface pretreatment group, the bond strength of both the self-cured and light-cured groups was similar regardless of the dentin surface pretreatment ($P < .05$).

In the RelyX™ U200 cement group, the control group showed significantly lower bond strength than the EDTA and PA group, regardless of the curing mode ($P < .05$). The EDTA and PA group showed higher bond strength than the control group, but no significant differences were

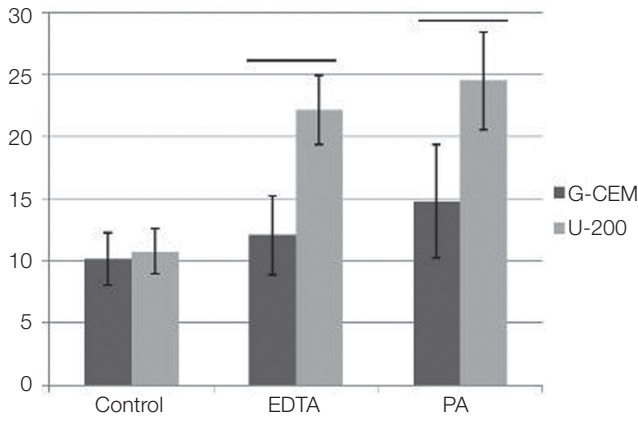


Fig. 1. Graph showing differences between two self-adhesive resin cements on μ TBS (MPa) when self-cured. The under bar indicates that there were significant differences ($P < .05$). Abbreviations; EDTA: ethylenediaminetetraacetic acid, PA: polyacrylic acid, G-CEM: G-CEM LinkAce™, U-200: RelyX™ U200.

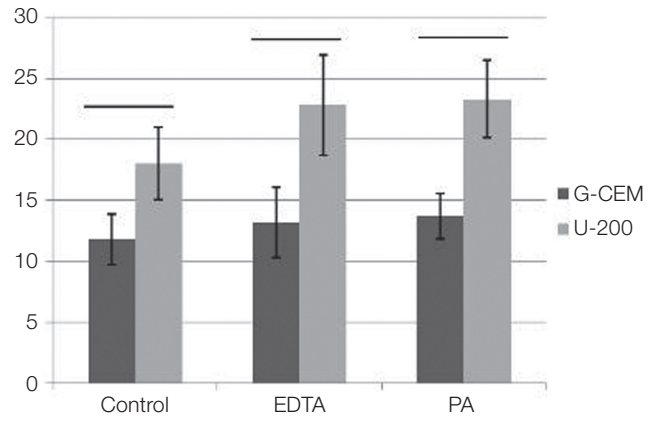


Fig. 2. Graph showing differences between two self-adhesive resin cements on μ TBS (MPa) when light-cured. The under bar indicates that there were significant differences ($P < .05$). Abbreviations; EDTA: ethylenediaminetetraacetic acid, PA: polyacrylic acid, G-CEM: G-CEM LinkAce™, U-200: RelyX™ U200.

observed between the EDTA and PA groups ($P > .05$). A comparison of the control groups according to the curing mode revealed the light-cured control group to have significantly higher bond strength than the self-cured one. On the other hand, comparing dentin surface pretreatment groups, the EDTA and PA groups showed the similar responses to the curing mode ($P > .05$).

Fig. 1 and Fig. 2 present the comparisons between the two self-adhesive resin cements. In the self-cured groups (Fig. 1), the RelyX™ U200 groups showed higher bond strength than the G-CEM LinkAce™ groups except for the control group ($P < .05$). In the light-cured groups (Fig. 2), all groups cemented with RelyX™ U200 showed higher bond strength than the G-CEM LinkAce™ groups ($P < .05$).

DISCUSSION

Self-adhesive resin cements are more user-friendly and less technique-sensitive. On the other hand, these cements do not fulfill the ultimate goal of the cementation of indirect restorations in that they revealed a low μ TBS. This is due to the formation of a smear layer on the dentin surface that was incorporated into the hybrid layer and impaired the deep infiltration of adhesive resin cements.

PA is a mild acid that is normally used as a cavity cleaning agent when the tooth cavity is restored with glass ionomer containing materials. This mild acid removes the smear layer partially but leaves smear plugs in the tubules.³ In addition, it leaves free calcium and phosphate ions on the dentin surface to promote a better chemical reaction with some of the restoration materials.³

On the other hand, EDTA is a mild calcium-chelating

agent at neutral pH.⁷ Many studies have shown that EDTA removes the hydroxyapatite of dental hard tissue selectively without destroying the collagen matrix structure.^{7,18,19} Cehreli *et al.* reported that when the dentin surface is pretreated with 17% EDTA for 60 seconds, approximately 30% of the smear plugs remained with the partially removed smear layer and no morphological change was observed.^{7,19}

In the present study, when PA was applied as a dentin surface pretreatment, both cements used in this study showed higher μ TBS than the control groups except for the light-cured G-CEM LinkAce™. On the other hand, no significant differences were observed according to the curing mode. When the dentin surfaces were pretreated with EDTA, the absolute μ TBS values showed an increasing tendency compared to the control groups, but only the RelyX™ U200 cement groups had significantly higher bond strength. These results suggest that although the removal mechanism of the smear layer is different, pretreatment of the dentin surface is an important factor that can influence the bond strength.

Dual-cured resin cements were developed to improve the polymerization efficiency. Because these cements are used under indirect restorations, light is applied only at the periphery of the restorations and it is difficult to allow the light pass through the restorations. The light penetration depths vary according to the types and thickness of an indirect restoration. Consequently, the self-curing initiator was added to compensate for this defect. Many studies, however, have shown that the dual-cured resin cements displayed higher bond strength when accompanied by light-curing.¹⁴⁻¹⁷

According to the manufacturer of the G-CEM LinkAce™,

they used a new innovative chemical initiator system offering the highest polymerization in self-curing mode. They claimed that it polymerizes within 4 minutes in self-curing mode and provides the maximum bond strength after 20 minutes.²⁰ They also emphasized that there is little difference in bond strength between the two curing modes (self and light-curing mode).

In the present study, the G-CEM LinkAce™ cement groups showed a similar μ TBS between the self-cured and light-cured groups and no significant differences were noted. These results support the manufacturer's claim that self-curing is sufficient to polymerize the G-CEM LinkAce™ cement compared to the light-curing mode. Under the same conditions, however, the μ TBS of the G-CEM LinkAce™ resin cement was inferior to that of RelyX™ U200 resin cement in all groups except for the self-cured control group.

For the RelyX™ U200 resin cement, the light-cured group showed higher bond strength than the self-cured group without a pretreatment. After the dentin surface pretreatment, no significant differences were observed between the self-cured and light-cured groups. This suggests that a pretreatment of the dentin surface can offset the effects of light-activation of the RelyX™ U200 resin cement.

Without considering the curing modes of the cements, RelyX™ U200 showed more dramatic results than the G-CEM LinkAce™ resin cement according to the dentin surface pretreatments. The difference in viscosity between these cements might be one explanation. RelyX™ U200 contains more filler particles (72%) than G-CEM LinkAce™ (60-70%) and is more viscous than G-CEM LinkAce™ during manipulation.^{20,21} The level of penetration decreases with increasing viscosity of the cement. With the aid of the dentin surface pretreatments, the high viscosity resin cement could penetrate deep into the dentinal tubules, which finally led to a dramatic increase in bond strength. For a strong bond strength, it is important not only to remove the smear layer, but also to improve the penetration potential of the cement.

These results indicate that pretreating the dentin surface with mild etchants, such as 10% PA and 18% EDTA to eliminate the smear layer and contaminants appears to be a desirable procedure for improving the μ TBS of the self-adhesive resin cements. On the other hand, the degree of the effects can vary according to the curing modes, types and compositions of resin cements.

CONCLUSION

In the G-CEM LinkAce cement groups, the effect of a dentin surface pretreatment only appeared in the self-cured groups, not in the light-cured groups. Overall, the effect of the PA pretreatment on the bond strength was significant. No significant differences were observed between the curing modes.

For the RelyX U200 cement groups, the EDTA and PA pretreatment showed effective results in both the self-cured and light-cured groups. Only the groups with no dentin

surface pretreatment (control groups) showed significant differences according to the curing modes.

In conclusion, selecting RelyX U200 used in this study and application of dentin surface pretreatment with EDTA and PA might be recommended to enhance the bond strength of cement to dentin.

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