

Shear Bond Strength of Resin Cements to Human Dentin under Self-Curing Mode

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Abstract

To measure the shear bond strength (SBS) of a dual-cured resin cement to human dentin under self-curing mode, and compare it with the SBS of two self-cured cements. The SBS of two self-cured resin cements (Super-Bond C&B, Sun Medical Co. Ltd, Kyoto, Japan (SB); C&B Cement, Bisco Inc., Schaumburg, IL, USA (CB)) and one dual-cured resin cement (NX3 Nexus, Kerr Corporation, Orange, CA, USA (NX)) were measured according to ISO 11405. All resin cements were used according to the manufacturers' instructions to bond to the dentin of premolar teeth using a stainless-steel cylinder mold and allowed to self-cure. Shear bond strengths were measured after 1h and 24h storage in a humidified lightproof box at 37°C. Super-Bond C&B exhibited the highest bond strength at both 1h and 24h (19.2 ± 2.8 MPa, 34.0 ± 5.1 MPa respectively) and the lowest was C&B Cement (5.2 ± 1.1 MPa, 10.5 ± 2.8 MPa respectively) ($p \leq 0.05$). The bond strength at 24h in all groups were significantly higher than at 1h. Resin cements with different chemical formulations yield significantly different bond strengths to human dentin. The self-cured acrylic resin cement showed the highest bond strength and should therefore be preferred in clinical situations where the photo-curing light cannot transmit through the restoration. Time after bonding increased the bonding performance of all resin cements.

Keywords: Resin cement, Self-curing mode, Shear bond strength

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Introduction

The excessive loss of the tooth structure, e.g., large carious lesions, root canal treatment and severe attrition, weakens the tooth and renders it more susceptible to fracture.^{1,2} In these cases, a direct restoration may be inadequate to support the remaining tooth structure. Cusp-coverage indirect restorations are more appropriate treatments to increase the fracture resistance of the tooth.^{3,4}

There are many factors that affect the success of an indirect restoration, such as the height of the tooth, the type of the preparation⁵ and the luting cement.⁶ To lute indirect restorations, methyl methacrylate-based luting agents or resin cements are increasingly used due to their additional advantages of higher compressive strength, flexural strength and bond strength to tooth structure, compared to conventional cements. Furthermore, resin cements have less microleakage and excellent color match.⁷ According to their polymerization mechanism, they may be classified into light-cured, self-cured and dual-cured.⁸

If the restoration is a ceramic or indirect resin composite through which light can pass, an appropriate resin cement can be light-cured or dual-cured.⁹ A self-cured resin cement is appropriate for luting metal restorations because they are opaque. However, many manufacturers claim that dual-cured resin cements can be used for luting metal restorations.

Several studies indicated that the rate and degree of conversion (C=C to C-C, or the extent of the curing reaction) of dual-cured resin cement without light activation was less when compared to cement exposed to light.¹⁰⁻¹³ The degree of conversion may affect the surface hardness of resin cements.¹⁴ Many studies also showed that surface hardness was less when dual-cured resin cement was polymerized by a self-curing mode only^{8,15-19} and this would affect bond strength and the success of the treatment if lute metal restoration with dual-cured resin cement.

There are no studies on bond strength to the tooth of dual-cured resin cements in a self-curing mode. The objectives of this study were to measure the bond strength of a dual-cured resin cement to dentin under self-curing mode and compare it with self-cured resin cements. The null hypotheses were that (1) there is no difference in shear bond strength between the types of resin cement; (2) there is no difference between 1h and 24h shear bond strength of the same resin cements.

Materials and Methods

Preparation of the specimens

The study received approval from the Human Research Ethics Committee of the Faculty of Dentistry, Chulalongkorn University (No.43/2015). Ninety non-carious human premolars extracted for orthodontic treatment were frozen at - 4°C and used within 6 months after extraction.

The apices of the teeth were cut off 10 mm below the cemento-enamel junction and the crown cut off at the dentine-enamel junction using a low-speed diamond saw under water cooling (Isomet 1000; Buehler, Lake Bluff, IL). The resulting roots were embedded in epoxy resin in polyvinylchloride rings (diameter 22 mm, height 20 mm) with their occlusal surfaces exposed. The surfaces were polished with wet 600-grit silicon carbide paper (SiC) on a polisher (NANO 2000; PACE Technology, AZ) to create a standardized smear layer. The specimens were randomly divided into three groups for each of the three resin cements (n=30), and each group further divided into two sub-groups (n=15) for 1h and 24h shear bond strength testing. All groups comprised the same number of upper and lower teeth.

Bonding procedure

Super-bond C&B (Sun Medical Co., Ltd, Kyoto, Japan), C&B Cement combined with All-Bond 3 (Bisco Inc., Schaumburg, IL, USA), and NX3 Nexus combined

with Optibond Solo Plus (Kerr Corporation, Orange, CA, USA) were used (Table 1). A stainless steel cylindrical mold, inner diameter 3.5 mm, height 3 mm, was localized onto the dentin surface by a shear bond strength rig (Fig 1; SDI Ltd, Bayswater, Victoria, Australia). The respective resin cement was placed into the mold, and the mold

covered with a 5x5 mm lightproof glossy paper. A load of 9.8 N was placed on the paper for 10 min. All resin cements were allowed to cure in self-curing mode according to the respective manufacturer's instructions (Table 2). Thereafter, the specimens were store in a humidified lightproof box at 37°C for 1h or 24h.

Table 1 Materials, components, and manufacturers

Resin cement (Classification)	Manufacturer	Components
Super-Bond C&B (Self-cured)	Sun Medical Co. Ltd (Kyoto, Japan)	Catalyst V: TBB, Hydrocarbon Monomer: MMA, 4-META Polymer: PMMA, pigment Green activator: Citric acid, FeCl ₃
C&B Cement (Self-cured)	Bisco Inc. (Schaumburg, IL, USA)	Base: Bis-GMA, Ethoxylated Bis-GMA, Triethyleneglycol Dimethacrylate Catalyst: Bis-GMA, Triethyleneglycol Dimethacrylate ALL-BOND 3: Part A: Ethanol, NTG-GMA Salt Part B: Bis-GMA Uni-Etch: 32% Phosphoric acid
NX3 Nexus (Dual-cured)	Kerr Corporation (Orange, CA, USA)	Uncured methacrylate ester monomers, nonhazardous inert mineral fillers, nonhazardous activators and stabilizers, radiopaque agent OptiBond Solo Plus: Ethyl alcohol, Alkyl dimethacrylate resins, Barium aluminoborosilicate glass, Fumed silica (silicon dioxide), Sodium hexafluorosilicate Kerr gel etchant: 37.5% Phosphoric Acid, Water, fumed silica, dye colorant

*TBB: tri-butylborane. MMA: methyl methacrylate. PMMA: polymethyl methacrylate. 4-META: 4-methacryloyloxyethyl trimellitate anhydride. FeCl₃: Ferric Chloride. Bis-GMA: bisphenol A glycidyl methacrylate. NTG-GMA: N-*o*-toluene glycine and glycidyl methacrylate*

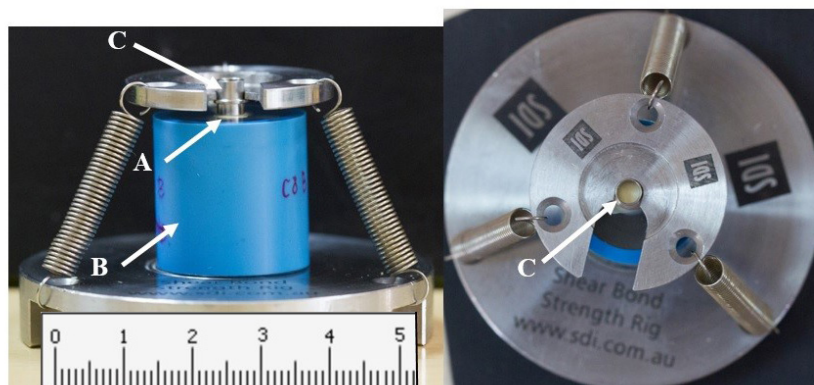


Figure 1 Stainless steel mold seated on dentin with shear bond strength rig. A: prepared dentin, B: polyvinylchloride rings, C: stainless steel mold

Table 2 Resin cements and application procedure

Resin cements	Etching	Dental adhesive/ primer	Application procedure
Super-Bond C&B	Green Activator	None	<ul style="list-style-type: none"> - Apply the Green Activator using a sponge pledget on dentin for 10 s. - Rinse thoroughly with water for 15 s and air dry for 10 s. - Dispense 4 drops of monomer and 1 drop of catalyst V into the chilled dispensing dish and stir lightly with brush. - Add 1 standard spoon of polymer powder and stir lightly for 10 s. - Apply the cement to dentin immediately after mixing.
C&B Cement	Uni-Etch	ALL-BOND 3	<ul style="list-style-type: none"> - Etch dentin for 15 s and rinse thoroughly with water for 15 s. - Remove excess water using a gauze for 10 s. - Dispense an equal number of drops of ALL-BOND 3 Parts A and B (1:1) into a mixing well and mix with a brush for 5 s. - Apply adhesive onto the dentin with a light agitating motion for 5 s and gently air dry starting from 5 cm for 5 s. - Light cure for 10 s. - Mix equal amounts of C&B base and catalyst into a uniform paste (10-15 s). - Apply the cement to dentin with dispenser gun.
NX3 Nexus	Kerr gel etchant	OptiBond Solo Plus	<ul style="list-style-type: none"> - Etch dentin for 15 s and rinse with water for 15 s. - Remove excess water using a gauze for 10 s. - Apply OptiBond Solo Plus for 15 s using light brushing motion. - Air thin for 3 s. - Light cure for 10 s. - Dispense NX3 dual-cure cement onto dentin.

Shear bond strength testing

Shear bond strength was determined according to ISO/TS 11405:2003 using a universal testing machine (EZ-S; Shimadzu, Japan) at a cross head speed 1 mm/min. Load at failure (N) was recorded and bond strength calculated (MPa). The de-bonded dentin surfaces were inspected visually under a stereomicroscope (ML 9300; MEIJI, Japan) at x40 magnification to determine failure mode, and classified into one of four categories:

- Complete adhesive failure at the resin-dentin interface (Adh)
- Cohesive failure in the resin cement (CohC)
- Cohesive failure in dentin (CohD)
- Adhesive failure and cohesive failure in the resin cement and/or Dentin (Mixed)

SEM examination

Two specimens were randomly selected from each group, cut in a sagittal section in bucco-lingual direction to obtain a 2-mm thick dentin disk. Specimens were surfaced with wet SiC papers of increasingly finer grit (#600, #800, #1000 and #1200), polished with soft clothes with diamond pastes of decreasing grit sizes (6, 3, 1 and 0.25 μm) and ultrasonically cleaned in deionized water. Specimens were superficially demineralized with 32 % phosphoric acid for 10 s, immersed in a 1 % sodium hypochlorite solution for 60 s, dehydrated for 20 min in increasing concentrations of ethanol (25 %, 50 %, 95 % and 100 %), immersed in hexamethyldisilazane (HMDS) for 10 min and sputter coated with gold (JFC-1200, JEOL,

Japan). Gold coated specimens were observed using SEM (JSM-5410 LV, JEOL, Japan) at 10-25 kV (x3000 and x6000 magnification).

Statistical analysis

Statistical analysis was performed using SPSS 22 software for Windows (SPSS Inc., Chicago, IL, USA) at a significance level of 5 % ($p < 0.05$). Descriptive statistics were applied to the data. A comparison of shear bond strength between different resin cements at the same time was done using 2-way ANOVA. An independent *t*-test was used to compare between groups of the same resin cement.

Results

Shear bond strengths

The results of the shear bond strength measurements are summarized in Fig 2. At 1h, no significant difference was seen between the shear bond strengths of SB and NX group (19.2 ± 2.8 MPa, 17.5 ± 3.4 MPa). CB group showed significantly the lowest bond strength (5.2 ± 1.1 MPa). At 24h, the shear bond strength of all groups was significantly higher than at 1h (SB = (34.0 ± 5.2) MPa, NX = (28.8 ± 4.7) MPa, CB = (10.5 ± 2.8) MPa). The bond strength of the three cements was significantly different ($p \leq 0.05$) at 24h.

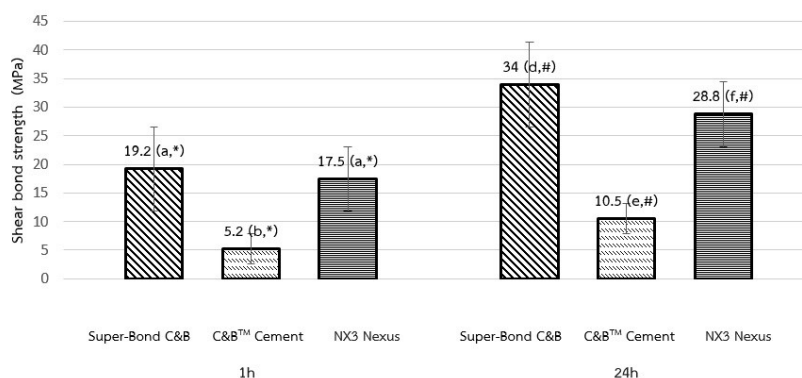


Figure 2 Shear bond strength of three resin cements at 1h and 24h.

Lowercase letters indicate comparisons between material at the same time intervals. Symbol indicate comparisons within materials at different time. Mean values with the same superscript letters or symbol were not statistically different ($P \leq 0.05$).

The failure mode distribution (%) of the three resin cements indicated by stereomicroscopy is shown in Fig 3. Nearly all (67- 87 %) failures in the SB and NX groups were mixed at both 1h and 24h. CB group specimens

had the highest percentages of adhesive failure at both evaluation times. The failure modes at 1h and 24h among all groups were not different.

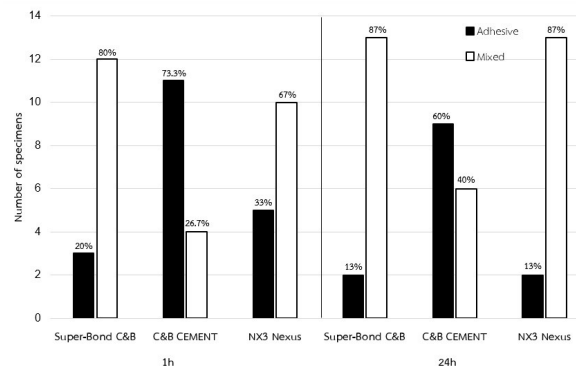


Figure 3 Failure mode of three resin cement at 1h and 24h.

SEM observation

Figure 4 show examples of the resin cement-dentin interface bonded with the three cements. A well-defined hybrid layer with a thickness approximately 2.8-3 μm in the SB group and 3.8-4 μm in the NX group

was observed. In the CB group, a gap between resin cement and dentin was found. A hybrid layer could not be observed clearly but resin tags were still evident at both times.

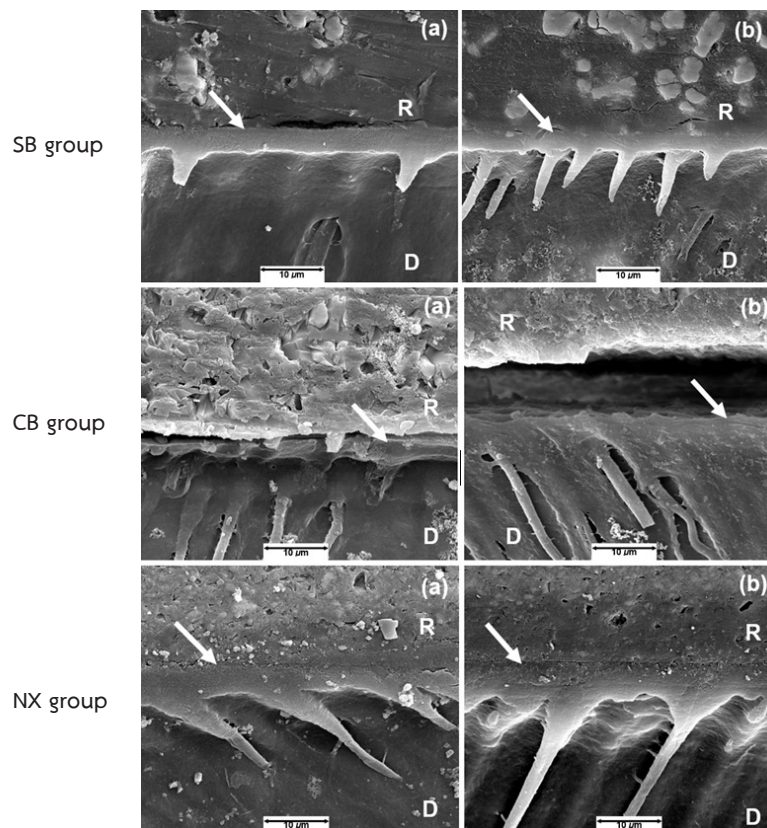


Figure 4 Scanning electron micrographs of the cement-dentin interfaces. (a): 1h, (b): 24h, R: resin cement, D: dentin, Arrow: hybrid layers.

Discussion

The stresses at the interface of restoration and tooth structure are complex, but can be identified mainly as either tensile or shear. When chewing, the forces of displacement tend to be closer to shear²⁰, so a shear bond strength test was used according to ISO TR 11405.²¹ The SB group exhibited the best SBS results, while the lowest bond strength was found for the CB group at both 1h and 24h. Based on the results, the first null hypothesis, that there is no difference between materials regarding the shear bond strength, was rejected. The shear bond strength of the 24h group was significantly higher than the 1h group for all cements ($p \leq 0.05$), so the second null hypothesis was also rejected.

The relatively high bond strength values of the SB group at 1h may be attributed to the use of 10 % citric acid combined with 3 % ferric chloride solution to condition the dentin, while other two resin cements used 32 % and 37.5 % phosphoric acid (CB and NX group respectively). A previous study reported that a 4-META/MMA resin cement (known commercially as Super-Bond, Sun Medical Co. Ltd Kyoto, Japan), when initiated with oxidized tri-n-butylborane (TBBO), provided excellent bond strength to dentin when the dentin surface was pre-treated with citric acid solution containing ferric chloride.²² Ferric ions diffuse into collagen in dentin during acid conditioning and help to maintain spaces between the collagen fiber networks, even after air-drying, so that monomers can penetrate and form better hybridized dentin after polymerization.²³ Furthermore, ferric chloride accelerates free radical polymerization that is initiated by TBBO.²⁴ The predominant failure mode in this group was mixed (80 %), which may imply that the bond strength at the resin-dentin interface exceeds the cohesive strength of the cement itself in some areas. A previous study also reported the same correlation between failure mode and bond strength as in our study.²⁵ Moreover, SEM evaluation showed the well-defined and uniform 3- μ m thick hybrid layer that may provide an adequate

support for the good bond strength of this cement.

Even though NX is a dual-cured resin cement, the 1h bond strength was not significantly different from that of the SB group. This indicates the effect of light-curing to adhesive, applied to the dentin before the resin cement, in stabilizing the hybrid and adhesive layer. By immediately sealing the dentin and thus preventing water uptake from the tubules by osmosis²⁶, the favorable immediate bond strength can be obtained even though this resin cement is polymerized by a self-cured mode. Lührs *et al.* found that favorable bond strengths of resin cements were achieved when the adhesive was light-cured even resin cement was polymerized in self-cured mode, except for a group of RelyX Ultimate (3M ESPE, St. Paul, MN, USA) with Scotchbond Universal (3M ESPE, St. Paul, MN, USA)²⁷. In our study, most of the NX specimens failed in mixed mode (67 %), indicating the high bond strength between resin cement and dentin that exceeds the cohesive strength of the cement itself. SEM evaluation also found a hybrid layer approximately 3.8 μ m thick.

In the CB group at 1h, there was a significantly lower shear bond strength when compared to SB and NX groups. This resin cement is used combined with All-Bond 3 adhesive system, following the manufacturer's instructions for an indirect restoration, that only a hydrophilic mixture of part A and B was used without subsequently applying a hydrophobic All-Bond 3 Resin. Previous studies showed that water can be taken up from dentin if only a hydrophilic primer is applied, polymerization of resin cement and the bond strength will be impaired.^{28,29} Hydrophobic resin should be applied after a primer in order to seal the dentin and enhance the bond strength. The failure mode of the CB group supports the result of shear bond strength, in that most of the failure mode is adhesive (73.3 %), indicating that the weakest point is at the interface between resin cement and dentin. This agrees with a previous study which reported that adhesive failures were consistently

found in association with lower bond strengths.³¹ SEM analysis also confirms the result, as there is a gap between resin cement and dentin such that the hybrid layer could not be clearly observed.

When comparing the shear bond strength between the 1h group and 24h group, that at 24h was significantly greater for all resin cements. This greater bond strength could be ascribed to greater degree of conversion as shown in a previous study.³⁰

At 24h, SB group presented the highest bond strength, followed by NX and CB. In dark conditions, self-cure resin cement has a higher degree of conversion than dual-cured resin cement³¹, so the SB group provided a favorable bond strength. In contrast, the CB group, which is also a self-cure resin cement, provided the lowest bond strength. This might due to not applying hydrophobic resin following the hydrophilic primer before luting with the cement, thus water uptake from dentin may have impaired the polymerization process and bond strength. In general, for dual-cure resin cement, adhesive/resin cement combinations presented a lower degree of conversion after auto-curing than when they were light-cured.^{12,32} Also in a previous study, self-cure resin cement had a higher surface hardness than dual-cured resin cement polymerized by the self-cure reaction only.¹⁴ This low degree of conversion would impair the bond strength of NX group.^{33,34}

For all cements, the failure mode at 24h was the same as at 1h. For SB and NX, 87 % of specimens showed mixed failure mode, while 60 % of CB specimens showed an adhesive failure mode. The failure mode of CB specimens was nearly all adhesive at both times, which may because of the low bond strength. A previous meta-analytical study showed a strong correlation between bond strength and fracture mode, in that the higher bond strength, the higher incidence of cohesive failure.³⁵ In our study, there was no cohesive failure, which might be because the bond strength was not higher than the strength of the cement or the dentin.

The result from SEM evaluation at 24h revealed

a hybrid layer in SB and NX group specimens of approximately 2.8 and 4 μm thick, respectively. There was no significant different in hybrid layer thickness compare to 1h group. In the CB group, a gap between resin cement and dentin was still observed and a hybrid layer could not be clearly seen. According to the low bond strength of this group, a gap could be formed while preparing specimen for SEM evaluation. Bond strength may depend on hybrid layer quality rather than thickness.³⁶

A shear bond strength test is to quantify adhesion. Clinically, there are many criteria which can be used to assess the effectiveness of a resin cement, such as the retention form of the preparation, marginal integrity and clinical microleakage. Choosing an appropriate resin cement for an indirect restoration is one important key to success. In this study, the self-cured acrylic resin cement showed the highest bond strength and could therefore be preferred in clinical situations where light transmission cannot penetrate through the restoration. Applying hydrophobic resin before luting is necessary when using a self-cured dimethacrylate cement. The patient should be advised to avoid using the indirect restoration after luting with resin cement for at least 24 hours.

Conclusion

Resin cements with different chemical formulations yield significantly different bond strengths to human dentin. Under only self-curing mode, self-cured acrylic resin cement created the highest bond strength. The shear bond strength of all resin cements were significantly greater after 24h compared to 1h.

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