Research Articles

Effects of Provisional Cements on Shear Bond Strength of Resin Cements to Dentin

Pattamon Sethsoontree[1], Pattaranat Banthitkhunanon[1] and Sitthichai Wanachantararak[2]

[1]Department of Prosthodontics, Faculty of Dentistry, Chiang Mai University, Chiang Mai, Thailand.

[2]Department of Oral Biology and Oral Diagnosis Sciences, Faculty of Dentistry, Chiang Mai University, Chiang Mai, Thailand.

Abstract

This study evaluated the effects of provisional cements on the shear bond strength (SBS) of permanent resin cements to dentin. The buccal cusps of extracted human mandibular first premolars (n=144) were sectioned to expose dentin at 3 mm from the buccal cusp tip. The specimens were first equally divided into four groups according to the provisional cements used: control (no cementation), zinc oxide eugenol, zinc oxide non-eugenol and calcium hydroxide. The provisional cement was mixed and applied on the dentin surface with an acrylic rod placed over with 10 N constant load until the cement was set. The test specimens were stored in distilled water at 37°C for one week. The acrylic rods were removed and provisional cement remnants were cleaned with spoon excavator and pumice-water slurry. The specimens were then divided equally into three subgroups for testing permanent resin cements: self-adhesive, self-etch and total-etch. Permanent resin cement was used to cement a composite resin stick onto dentin surface. After 24 hours, all specimens were processed to test the shear bond strength with a universal testing machine. Data were analyzed with two-way ANOVA and Tukey's test. There was no interaction between provisional and permanent resin cement groups. The bond strength obtained when using calcium hydroxide and zinc oxide eugenol provisional cement were similar to no provisional cement contamination. Zinc oxide non-eugenol provisionalization had a significantly lower shear bond strength than the others. The shear bond strength of the total-etch cement group was the highest while that of the self-adhesive group was the lowest. In conclusion, the shear bond strength of three permanent resin cement is not affected when using zinc oxide eugenol and calcium hydroxide as provisional cements, but is reduced when using zinc oxide non-eugenol cement.

Keywords: Provisional cement, Resin cement, Shear bond strength (SBS)

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Correspondence to:

Pattaranat Banthitkhunanon, Department of Prosthodontics, Faculty of Dentistry, Chiang Mai University, Chiang Mai, 50200, Thailand

Tel: 0869209595 E-mail: pat.dentcm@gmail.com

Introduction

During the processes of fabrication of the permanent fixed restoration, the provisional restorations with provisional cementation are required to maintain chewing function, pulpal protection and esthetic.[1] The provisional cement should be firm enough to retain the provisional restoration in place, while it should be easy to be removed and has no effect on permanent cementation.[2] Available in the market are calcium hydroxide liners such as Dycal® (DENTSPLY Caulk, Milford, DE, USA), zinc oxide cements such as Temp-Bond™ (Kerr, Orange, CA, USA) and zinc oxide non-eugenol cements such as Temp-Bond™ NE (Kerr, Orange, CA, USA).[2-4]

The reduction in the bond strength of permanent cementation has been found after the tooth surface is contaminated with provisional cements.[5-7] Eugenol (4-allyl 2-methoxy phenol) in provisional materials has been blamed to interfere with the polymerization process of resin materials, because of its antioxidizing property.[8] It might interfere with the retention of permanent resin cement.[1,9] As a result, the zinc oxide non-eugenol has been manufactured to serve this purpose.

Calcium hydroxide liners such as Dycal® has also been used as provisional cement with the advantage of higher mechanical and adhesive properties over zinc oxide cement.[10] However, in the moist condition, it releases hydroxyl ions that creates alkaline environment which initially irritates dental pulp, but later stimulates reparative dentin formation.[11] Its alkaline pH could also neutralize a mild acidic resin primer of the self-etch system and a mild acidity of the self-adhesive cement.[6,12]

The bonding ability of permanent cementation determines the success of fixed restoration.[13] Resin cements have been widely used for permanent cementation due to their high bond strength, low solubility and acceptable esthetic.[14] They have been classified into three systems based on adhesive mechanisms; total-etch, self-etch and self-adhesive. Total-etch system (such as Variolink® N, Ivoclar Vivadent, Schaan, Liechtenstein) treats dentin surface with 35-37 % phosphoric acid to create microporosities before applying primer and bonding agent to form hybrid layer and resin tag for micromechanical interlock.[15] Self-etch system (such as PANAVIA™ F2.0, Kurarey, Osaka, Japan) prepares tooth surface with acidic functional monomers, for example, 10-MDP (10-methacryloyloxydecyl dihydrogenphosphate) to enhance chemical bond to calcium ions in hydroxyapatite on tooth surfaces before applying bonding agent.[16,17] Self-adhesive system (such as Rely X™ U200, 3M ESPE, Seefeld, Germany) has been recently developed by combined all acidic, primer, and adhesive agents into a single mixture of cement for the simplest use with less chance of postoperative hypersensitivity.[18-20]

There are controversial results from the literatures, whether eugenol, or residual of provisional cement disturb the etching quality, impair infiltration of adhesive into dentin or inhibit the polymerization of resin cement causing the reduction in the bond strength.[1,3,21] The aim of this study was to verify the effect of three provisional cements (zinc oxide eugenol, zinc oxide non-eugenol and calcium hydroxide) and the control with no provisional cement contamination on the shear bond strength of three resin cements (total-etch, self-etch and self-adhesive) to dentin.

Materials and Methods

This study has been approved by The Human Experimentation Committee of the Faculty of Dentistry, Chiang Mai University, Thailand. (Certificate of ethical clearance No. 1/2019). Human non-carious permanent mandibular first premolar teeth (n=144) were extracted as part of the orthodontic treatment. The samples were cleaned and stored in 0.1 % thymol solution. The root was cut off at the level of 3 mm apically to the cemento-enamel junction. Dentin under the buccal cusp was exposed by sectioned off with slow-speed diamond saw sectioning machine (Isomet® 1000 precision saw, Buehler, U.S.A) under water coolant, at the level of 3 mm from the buccal cusp tip (Fig. 1). The tooth specimen was positioned and held with dental stone type IV in polyvinylchloride (PVC) tube with the exposed cut tooth surface upward.

Dentin surface of all specimens was polished with 600-grit silicon carbide paper with water for 10 seconds to create standard smear layer. The prepared dentin surfaces were evaluated under a stereomicroscope system and digital camera (SZX7 & SZ2-ILST led illuminator stand & E-330, Olympus, Tokyo, Japan) at 40x magnification to verify complete enamel removal, no pulpal exposure or crack.

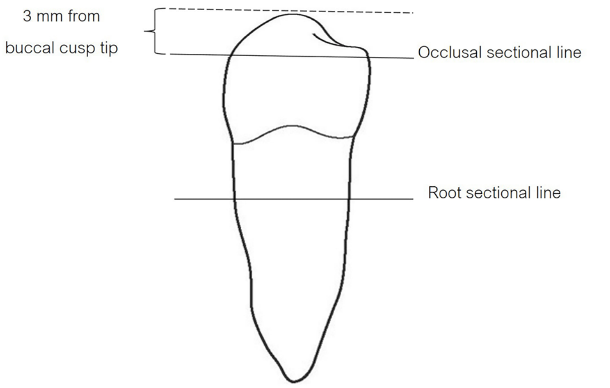

Figure 1 *Schematic diagram shown sectional lines for tooth specimen preparation.*

For testing the effect of provisional cements, the specimens were randomly divided equally into four main groups (n=36). One group served as a control while the other three groups were used for testing three provisional cements; calcium hydroxide liner (Dycal®), zinc oxide non-eugenol cement (Temp-Bond™ NE) and zinc oxide eugenol cement (Temp-Bond™). The provisional cement was applied onto the dentin surface before placing an acrylic rod (12 mm in diameter and 5 mm in height) over with 10 N constant load. The excess cement was gently removed using sharp tip explorer. After the cement had set, the specimen was kept in distilled water at 37°C.

After seven days, the acrylic rods were removed from the tooth surface. The cement remnants were removed using spoon excavator followed by polishing with slurry water of fine grain pumice using prophylaxis rubber cup with slow speed handpiece for 10 seconds and cleaned with water spray for 5 seconds. An adhesive tape (Paper Masking Tape No.720, Nitto Denko, Osaka, Japan) with 100 μm thickness and 3 mm hole was placed over the cut tooth surface for restricting the bonding area between permanent resin cement and dentin.

Resin rods (3 mm in diameter and 3 mm in height) were made from a light-cured composite resin (Filtek™ Z350 XT, 3M ESPE, Seefeld, Germany). Theirs bonding surface was prepared by air blasting with a 50 μm aluminum oxide under 35 PSI of pressure using airborne-particle abrasive unit (Basic Classic, Renfert GmbH, Hilzingen, Germany) and cleaned with ultrasonic cleaner machine (Easyclean, Renfert GmbH, Hilzingen, Germany).

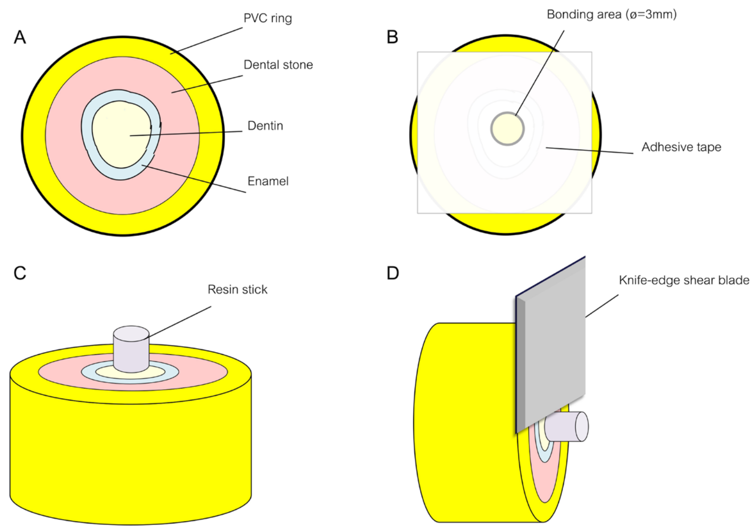

Figure 2 *(A) prepared tooth specimen was embedded in PVC ring (top view) (B) an adhesive tape with 3 mm hole was placed on prepared dentin surface for restricting the bonding area (top view) (C) resin rod was place on the tooth specimen with 10 N constant load (D) SBS test using knife-edge shear blade at resin-dentin interface.*

Each main group (n=36) was divided further into three subgroups (n= 12) for testing three permanent resin cements; total-etch, self-etch and self-adhesive. The tooth surface was prepared and three permanent resin cements were used according to the manufacturer's instruction. In brief, for self-adhesive cement (Rely X™ U200), the tooth surface was prepared by dropping distilled water on dentin with gentle air blow until the dentin surface was slightly shiny with moist, then resin base and catalyst were mixed. For self-etch cement (PANAVIA™ F2.0), self-etching primers (ED Primer II Liquid A and B) were mixed and applied on the moist bonding area with agitating technique for 15 seconds, waited for 15 seconds and air-dried until no movement of the liquid. Resin base and catalyst pastes were mixed for 20 seconds. For total-etch cement (Variolink® N), dentin surface was treated with 37 % phosphoric acid for 15 seconds, rinsed with water for 20 seconds, gently air blew until dentin surface appeared moist, applied Syntac primer and left it dried for 15 seconds, applied Syntac adhesive, dried after waiting for 10 seconds, applied Heliobond and blew to a thin layer, then mixed base and catalyst pastes of the resin cement.

The mixed resin cement was applied onto both prepared dentin surface and treated surface of resin rod. The resin rod was placed in the hole of adhesive tape onto dentin surface with 10 N constant load for controlling the cement thickness (according to ISO/TS 11405, 2015).[22] The curing light from a light-curing unit (Elipar™ LED Curing Light, 3M ESPE, Seefeld, Germany) with radiances of 1000-1200 mW/cm^2 was shone on the cement for 2 seconds then removed surrounding excess cement, and subsequently applied light for 20 seconds in four different directions of resin-dentin interface for complete polymerization.

The specimens were kept in distilled water at 37°C for one day before preparing for the SBS test in a universal testing machine (Instron® 5566, Instron Limited, Massachusetts, U.S.A) with 50 kg load cell. A knife-edge shear blade was positioned to compress at the cement interface of dentin and resin rod with a cross head speed of 0.5 mm/minute. The force was read out in newton (N) and was calculated to be the shear bond strength values in megapascals (MPa) by divided with the area of the bonding interface.

The failure mode of specimens was evaluated using a stereomicroscope at 50x magnification. The fracture surfaces were classified as adhesive failure (failure at dentin-resin interface), cohesive failure (within dentin, cement or resin rods) and mixed failure. Moreover, the six specimens of each group were randomly selected to be examined in more details under scanning electron microscope-SEM (JSM-5910LV, Jeol, Massachusetts, USA). The selected specimens were sectioned longitudinally. The cut surface was treated with 37 % phosphoric acid for 15 seconds to demineralize an inorganic part, followed by immersion in 5.25 % sodium hypochlorite for 20 minutes to remove an organic part and put in ultrasonic cleaner to remove all debris from the surface and examined under SEM.

Two-way ANOVA statistical analysis was used to investigate the interaction between provisional and permanent cements and Tukey's post hoc test for pair-wise comparisons (α = 0.05) to identify the difference within the same group.

Table 1 Shear bond strength (SBS) was presented in mean ± standard deviation values for both provisional cement and permanent cement groups.

Provisional cement (n=36)	Permanent cement (n=12)	SBS Mean±SD (MPa)	
None	Self -adhesive	6.41±1.06	** (vs Self-etch); * (vs Total-etch)
	Self-etch	7.94±1.23	
	Total-etch	8.71±1.14	
Calcium hydroxide	Self-adhesive	6.64±1.13	* (vs Total-etch)
	Self-etch	7.38±1.03	
	Total-etch	8.42±1.20	
Zinc oxide eugenol	Self-adhesive	6.59±1.28	** (vs Total-etch)
	Self-etch	7.47±0.78	
	Total-etch	8.16±1.59	
Zinc oxide-non eugenol	Self-adhesive	5.91±0.84	* (vs Total-etch)
	Self-etch	6.17±1.15	** (vs Total-etch)
	Total-etch	7.74±1.45	

**indicated the significant difference (p<0.001)*

***indicated the significant difference (p<0.05)*

Results

Two-way ANOVA statistical analysis suggested that there was no interaction between provisional and permanent cements ($p \geq 0.05$), but found a significant difference within the provisional cement main groups ($p<0.05$) and within the permanent cement main groups ($p<0.001$). The mean ± standard deviation of the shear bond strengths in MPa among control and the 3 provisional cement groups with the 3 permanent cement groups were presented in Table 1.

The multiple comparison using Tukey's test among subgroups of the cements suggested that zinc oxide non-eugenol group had a significant ($p<0.05$) lower shear bond strength (6.61±1.40 MPa) than that of the control (7.69±1.47 MPa), calcium hydroxide (7.48±1.32 MPa) and zinc oxide eugenol (7.41±1.39 MPa) (Fig. 3A) and there were significant differences among the three permanent resin cements, while the total- etch cement had the highest SBS (8.26±1.36 MPa) and the self-adhesive cement had the lowest SBS (6.39±1.09 MPa) (Fig. 3B).

For the self-etching cement, zinc oxide non-eugenol group had a significant lower bond strength (6.17±1.15 MPa) when compared to the control (7.94±1.23 MPa) ($p<0.001$) and the zinc oxide eugenol group (7.47±0.78 MPa) ($p<0.05$) while other groups were not different. There was no significant difference when comparing among provisional cement groups for total-etch and self-adhesive cements.

For all permanent cement groups, total-etch cement and self-etch cement had only the mixed type of failure mode (100 %), while the adhesive type of failure mode when using self-adhesive were 58.33 %, 66.67 %, 66.67 % and 83.33 % for calcium hydroxide group, zinc oxide eugenol group, zinc oxide non-eugenol group and control group, respectively. From SEM images, long resin tags with lateral branches, with an average length of 20-50 µm, were found in the total-etch group. Self-etch group showed absent or short resin tags with the average length about 5-10 µm, while there was no resin tag in none of the specimen in the self-adhesive group.

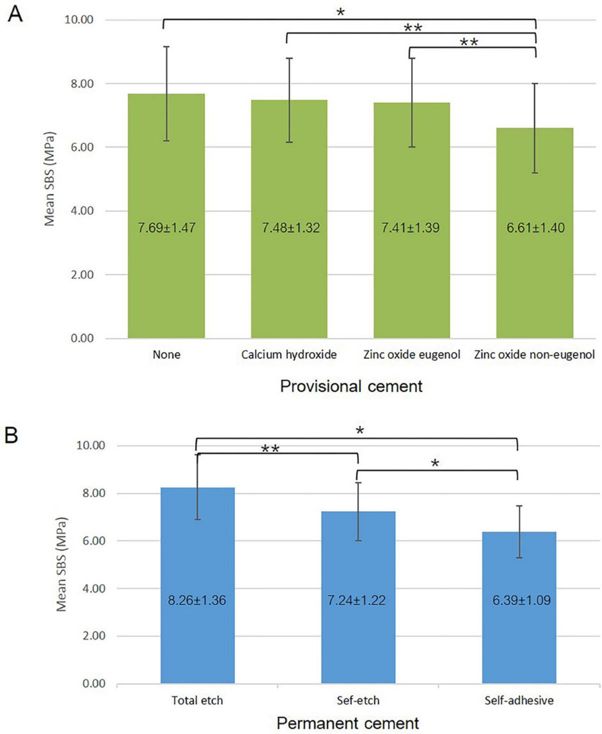

Figure 3 *Shear bond strength mean values ± standard deviation within cement groups (A) provisional cement groups (B) permanent cement groups. [*indicated the significant difference (p<0.001) **indicated the significant difference (p<0.05)]*

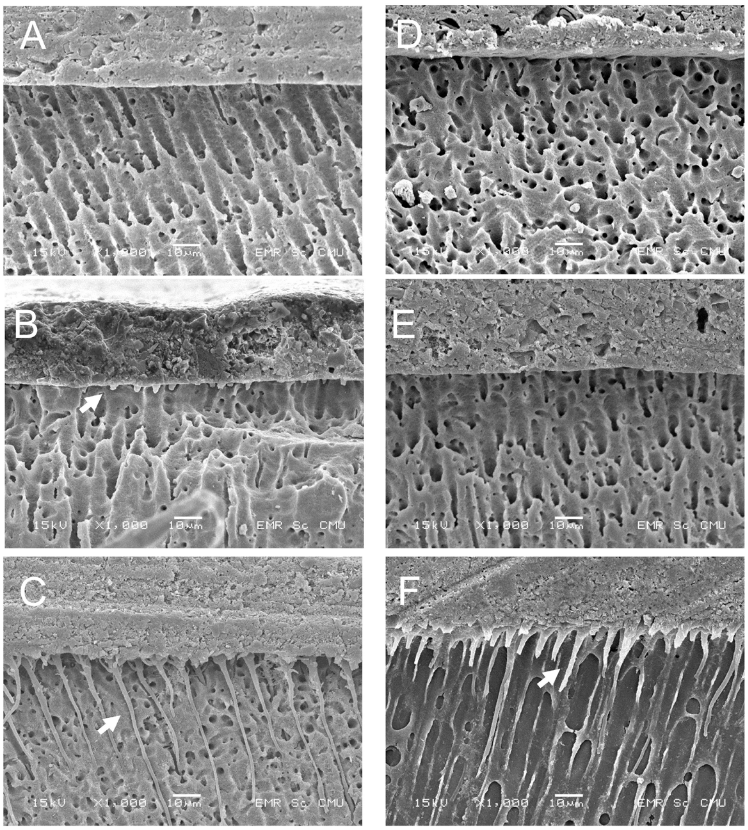

Figure 4 *SEM images of resin-dentin interface demonstrated resin tags (arrow) and dentinal tubule at the magnification of x1,000 (A-C) a sample in the control groups, (D-F) zinc oxide non-eugenol groups (A,D) samples were permanently cemented with self-adhesive cement, (B,E) self-etch cement, (C,F) total-etch cement. The SEM images of calcium hydroxide and zinc oxide eugenol showed similar results with these two groups.*

Discussion

Zinc oxide non-eugenol cement had a significant lower shear bond strength when compared to the control, calcium hydroxide cement and zinc oxide eugenol groups. This coincided with the observation by Altintas and colleagues[1] that zinc oxide non-eugenol had the lowest SBS compared to no provisional cement contamination and calcium hydroxide cement groups. Moreover, this supported the evidences that the use of eugenol-containing cement did not have an effect on bond strength of permanent resin cement,[23] but the use of zinc oxide non-eugenol did. In contrast to some studies[4,9] that showed the opposite result.

The bonding between resin cement and dentin could be affected by multiple factors. The duration of seven days for provisional restoration should be suitable for clinical practice because it is relevant to the duration for laboratory processes to make permanent fixed restoration. According to a systematic review by Ajaj and colleagues, eleven studies showed no significant adverse effect on the bond strength of permanent resin cementation when tooth surfaces were contaminated with eugenol-containing provisional materials for seven days,[24] while another study suggested that provisionalization with calcium hydroxide cement for a short-term as 7 or 30 days did not affect the bond strength of permanent adhesive resin to dentin.[25] The alkaline property of calcium hydroxide cement was the concern that it might neutralize the acidic capacities of adhesive systems or alter the organic matrix in the dentin.

On the contrary, other studies found a significantly reduced bond strength when provisionalization with eugenol-containing cement for one day.[24] Hume and colleagues[26] suggested that eugenol was released from hydrolysis of zinc oxide eugenol into dentin at the highest rate during the first day of placement and decreased thereafter. This supports the result of this study that the zinc oxide eugenol provisional cementation for 7 days has no effect on the bond strength of permanent resin cement.

The result of the bond strength of permanent resin cement, of this study, showed no significant difference among the control, calcium hydroxide and zinc oxide eugenol groups. This suggests that the surface of dentin of these two provisional cementation groups are not different from that of the control group. Moreover, the technique of using spoon excavator to remove cement remnants on the tooth surface followed by polishing with pumice-water slurry could provide acceptable bond strength and simple to use in the clinic.[24,27] However, the reduction in bond strength of zinc oxide non-eugenol has been unclear and could be caused by other factors.

Feitosa and colleagues[28] reported that the presence of Zn^{2+} in the form of zinc oxide might compromise the performance of MDP in forming MDP–Ca salts and reduce the bonding of MDP-based self-etch adhesives. The provisional cements used in this study contain different proportion of zinc oxide in the base. Under acidic condition, zinc oxide could dissociate from cement and bind to phosphoric functional monomer instead of calcium ion resulting in compromised bonding interface.

In moist condition, calcium hydroxide can be dissolved and release hydroxyl ions[29] which could disturb the acidic capacity of resin cement, deteriorate organic matrixes and reduce strength of dentin structures.[30, 31] The result of this study found the bond strength, when using calcium hydroxide as provisional cement, to be similar to that of the control which is in accordance with others studies.[5,32] It could be attributed to the high solubility in water of Dycal® (4.21 %) so this cement could be easily eliminated during the cleaning process.[33]

Similar to other studies,[32,34] total-etch cement has the highest bond strength, followed by self-etch and self-adhesive cements. The bond strength of self-etch cement was reduced significantly when using Temp-Bond™ NE as a provisional cement similar to the study by Carvalho and colleagues.[23] The total-etch system has the advantage of a higher bond strength than the others, but it also has disadvantage on several steps of application. Strong phosphoric acid used in total-etch system is most effective in removing smear layer, smear plug and other inorganic materials allowing bonding agents to penetrate deep into the dentinal tubule and form long resin tags[34-36] as shown in Figure 4C, 4F. Self-etching system simplifies clinical procedure and reduces technique sensitive of the total-etch system. Primer has mild acidic property (pH >2) which is less effective on removing smear layer and smear plug. Short or absent resin tags were found in the hybrid layers in SEM images (Fig. 4B, 4E) which is in accordance with other investigations.[37,38] Self-adhesive cement (Rely X^{TM} U200) has an advantage on clinical application, but it has low acidity (pH = 2.8)[39] resulting in the lowest potential to remove smear layer or cement debris and the lowest bond strength compared to other systems.[12,23,34] The SEM images (Fig. 4A, 4D) showed no resin tag penetration and the tubular orifices was covered with smear layers.

This study used extracted teeth which might have some limitations such as the lack of continuous outward flow of dentinal fluid, but a small area of the bonding area can be controlled to be comparable between specimens. Dentinal fluid in vital tooth could disturb hybrid layer formation and resin infiltration into the dentinal tubule to form resin tags. So, the long resin tags found in total-etch cement in this *in vitro* study could be shortened or absent when the dentinal fluid flow is present. Further studies should be conducted to evaluate the bond strength of permanent resin cement under simulated pulpal pressure or in vivo. Moreover, residual element particles of provisional cement remnants on the dentin surface should be investigated

Conclusion

Under the conditions used in this experiment, the shear bond strength of three permanent resin cements is not affected when using zinc oxide eugenol and calcium hydroxide as provisional cement, but is reduced when using zinc oxide non-eugenol cement. Among the three permanent cements, the total- etch adhesive cement yields the highest SBS, while the self-adhesive cement gives the lowest value.

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Graduate School, Chiang Mai University, Thailand

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