Original Article

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Effect of curing mode on micro tensile bond strength of resin composite cements

Phurinut Prateepamornkul[1], Piyaphong Panpisut[2], **Pong Pongprueksa**[3]

[1] *Master of Science Program in Operative Dentistry, Faculty of Dentistry, Mahidol University, Bangkok, Thailand.*
[2] *Division of Restorative Dentistry, Faculty of Dentistry, Thammasat University, Pathum Thani, Thailand.*
[3] *Department of Operative Dentistry and Endodontics, Faculty of Dentistry, Mahidol University, Bangkok, Thailand.*

Objectives: To investigate the micro-tensile bond strength (μTBS) to dentin of resin composite cements in different curing modes.

Materials and Methods: The μTBS of three resin composite cements—Panavia V5 (PV), Panavia SA Universal (SA), and RelyX Universal (RXU)— bonded to dentin were tested in two curing modes. PV was evaluated in auto polymerization (AA) and co-curing (AL) modes, while SA and RXU were tested in AL and separate light-curing (LL) modes. Failure modes were analyzed by light microscopy and SEM. Data were analyzed using one-way ANOVA and Tukey's post hoc test ($p<0.05$).

Results: PV had the highest bond strength, with no significant difference between AA and AL modes. In contrast, SA and RXU showed significantly lower μTBS in AL than LL. High rates of pretesting failures and adhesive failure modes were observed in the co-curing groups of SA and RXU. SEM analysis revealed voids and signs of water infiltration at the adhesive-dentin interface.

Conclusions: Co-curing adhesive-assisted resin composite cements (SA, RXU) significantly reduced bond strength. Primer-assisted resin composite cement (PV) maintained high bond strength across both curing modes.

Keywords: dentin-bonding agents, resin cements, tensile strength

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Introduction

Resin composite cements are categorized based on their application protocols: single-step self-adhesive resin composite cement and multi-step primer/adhesive-assisted resin composite cement [1]. Single-step self-adhesive resin composite cement offers the advantages of simplified application and reduced technique sensitivity. However, primer/adhesive-assisted resin composite cement has superior bond strength [1]. In response to clinical needs, manufacturers have introduced universal resin composite cement [1]. These cements are designed to function in both self-adhesive and adhesive modes when used in conjunction with recommended adhesives.

Multi-step adhesive-assisted resin composite cement yields the highest bond strength when the adhesive layer is separated and polymerized prior to resin composite cement cooperation [1]. Co-curing the adhesive layer with the resin composite cement can lead to water infiltration at the resin-dentin interface and compromise the bond strength [2, 3]. Despite the clear benefits of separate light-curing, some clinicians may hesitate to adopt this technique due to concerns about incomplete restoration seating. Additionally, some manufacturers recommend the co-curing mode

Corresponding author: Pong Pongprueksa
Department of Operative Dentistry and Endodontics, Faculty of Dentistry, Mahidol University,
6 Yothi Road, Ratchathewi, Bangkok 10400, Thailand
Tel: + 66 2200-7825 Email: pong.pon@mahidol.ac.th
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for their universal resin composite cement, further complicating the clinical decision-making process [1].

A novel type of primer-assisted resin composite cement utilizing the "Touch-Cure" system that incorporates chemical initiators in its paste, along with an additional accelerator in its primer [4]. By applying the accelerator to the tooth surface, polymerization upon contact with the cement occurs [5]. This "Touch-Cure" approach potentially leads to a higher degree of conversion at the interface compared to conventional resin composite cement [5]. Nonetheless, existing data regarding the recommended co-curing mode of adhesive-assisted resin composite cement remain limited. Therefore, the objective of this study is to investigate the micro-tensile bond strength to dentin of resin composite cement in different curing modes. This investigation could benefit the clinician in guiding how to achieve the optimal bond effectiveness of resin composite cement. The null hypotheses are that there is no significant difference in bond strength (1) among the resin composite cement systems and (2) between the curing modes of resin composite cement.

Materials and Methods

Forty-eight non-restored and non-carious human third molars were collected after approval by the Commission for Medical Ethics Committee (COA.No.MU-DT/PY-IRB 2024/DT115). A flat mid-coronal dentin surface was exposed using a semiautomated cutting machine (Accutom-100; Struers, Ballerup, Denmark) with a diamond saw (300 µm, M1D10; Struers). A standardized smear layer was created using P600-grit abrasive paper (CarbiMet; Buehler, IL, USA) [6].

The teeth were randomly divided into 3 groups according to three resin composite cement systems: Panavia V5 (PV; Kuraray Noritake Dental, Okayama, Japan) with Pavavia V5 tooth primer (Kuraray Noritake Dental), Panavia SA Universal (SA; Kuraray Noritake Dental) with CLEARFIL Universal Bond Quick (Kuraray Noritake Dental), and RelyX Universal (RXU; 3M, which was renamed to Solventum, St. Paul, MN, USA) with Scotchbond Universal Plus (Solventum). Then the specimens were further divided into two subgroups according to the curing mode as summarized in Table 2, resulting in a total of 6 experimental groups with a sample size of 8 in each group. 4 mm-thick resin composite blocks were pre-polymerized (Filtek Z350 XT, Shade A2, Solventum) and luted onto the dentin surfaces with resin composite cement under a constant seating force of 1 kg for 1 minute.

In the auto-curing mode (AA), specimens were solely auto-cure under load in a darkroom at 37°C for 30 minutes. In the co-curing mode (AL), the primer/adhesive was co-cured simultaneously with the resin composite cement via light polymerization through luted resin composite blocks at two opposite surfaces (buccal and lingual/palatal) and on top for 20 seconds each. The primer was not light-cured, but polymerized chemically through interaction with the resin composite cement—a process referred to as co-curing in this experiment. For the separate light-curing mode (LL), the adhesive was light-cured separately for 10 seconds and followed by light-curing of the resin composite cement through luted resin composite blocks at two opposite surfaces and on top for 20 seconds each. Light polymerization was conducted with a polywave LED light-curing unit (Bluephase N; Ivoclar, Schaan, Liechtenstein) at approximately 1,000 mW/cm². The light intensity has been regularly calibrated with a radiometer (Bluephase meter II, Ivoclar) throughout the experiment. Curing modes for PV were AA and AL. While the curing modes for SA and RXU were AL and LL. After polymerization, all specimens were stored in 100% humidity at 37°C for 24 hours, then transferred into pre-warmed artificial saliva at 37°C for an additional 6 days.

Table 1 The composition of material in each experimental group.

Group	Materials	Composition
PV	Panavia V5 Tooth Primer (Kuraray Noritake Dental) Lot: 920160	10-MDP, HEMA, Hydrophilic aliphatic dimethacrylate, accelerators, water
	Panavia V5 (Kuraray Noritake Dental) Lot: 8S0083	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, CQ, initiators-accelerators, silanated Ba-glass filler, silanated AlFSiO4 glass filler, colloidal SiO2, silanated Al2O3 filler, pigments
SA	CLEARFIL Universal Bond Quick (Kuraray Noritake Dental) Lot: 7V0465	10-MDP, Bis-GMA, HEMA, hydrophilic amide monomers, colloidal SiO2, MPTMS, NaF, CQ, ethanol, water
	Panavia SA Universal (Kuraray Noritake Dental) Lot: A50092	BisGMA, TEGDMA, HEMA, 10-MDP, hydrophobic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate, silane coupling agent, silanated Ba-glass filler, silanated colloidal SiO2, Al2O3 filler, Surface treated NaF, CQ, peroxide, catalysts, accelerators, pigments.
RXU	Scotchbond Universal Plus (Solventum) Lot: 10755325	Dibrominated aromatic dimethacrylate, 10- MDP, HEMA, VP-copolymer, MPTMS, APTES, CQ, DEGDMA, EDMAB, Cu (II) acetate monohydrate, ethanol, water
	RelyX Universal (Solventum) Lot: 10612250	TEGDMA, DUDMA, mixture of mono-, di-, tri- glycerol dimethacrylate esters with phosphoric acid, HEMA, MMA, TAHP, VCPAL, BHT, EDMAB, TPP, Cu (II) acetate monohydrate, MPTMS-silanated silica, MPTMS-and PTMS-silanated glass, YbF3

APTES: Aminopropyl triethoxysilane, BHT: Butylated hydroxytolouene, Bis-GMA: Bisphenol glycidyl dimethacrylate. CQ: Camphorquinone, DUDMA: Diurethane dimethacrylate, DEGDMA: diethylene glycol dimethacrylate. EDMAB: Ethyl-4 dimethylenamino benzoate, HEMA: 2- Hydroxyethyl methacrylate, MDP: 10-Methacryloyloxydecyl dihydrogen phosphate, MPTMS: Methacryloxypropyl trimethoxysilane, PTMS: Phenyl trimethoxysilane, TAHP: Tetr-amyl hyroperoxide, TPP: Triphenyl phosphite, TEGDMA: Triethyleneglycol dimethacrylate, VCPAL: L-ascorbic acid 6-hexadecanoate. VP-copolymer: Methacrylate-modified polyalkenoic acid copolymer

Table 2 The summary of different curing modes employed

Group	Primer or Adhesive	Resin Composite Cement	Curing Mode
PV AA	Auto-cure	Auto-cure	AA
PV AL	Auto-cure	Light-curing through luted resin composite	AL
SA AL	No separate light-curing	Light-curing through luted resin composite	AL
SA LL	Light-cure	Light-curing through luted resin composite	LL
RXU AL	No separate light-curing	Light-curing through luted resin composite	AL
RXU LL	Light cure	Light-curing through luted resin composite	LL

The bonded specimens were sectioned perpendicular to the bonded interface to obtain 1 x 1 mm micro-tensile sticks, following the non-trimming technique [6]. Sticks that fractured during sectioning were considered as pretest failures and referred to as zero MPa in statistical analysis. The sticks were attached to a Ciucchi's jig[6], then mounted to a universal testing machine (Instron 5566, Instron Ltd., Bucks, England). A preload force of 1.0 N was applied, and the specimens were loaded in tension at a crosshead speed of 1.0 mm/min until failure [6]. The bond strength was calculated in MPa.

Every specimen was examined under a Light microscope (DP22, Olympus Co., Shinjuku-Ku, Japan) to assess the failure modes. Representative specimens from each group were examined under a SEM (JSM-6610LV, JEOL Ltd., Tokyo, Japan). The failure modes were classified as follows[6]: (A) Cohesive failure at composite is a failure exclusively within the composite side; (B) Cohesive failure at dentin is a failure exclusively within the detin side; (C) Adhesive failure is a failure at the interface between the adhesive and dentin; (D) Mixed failure involves both interfacial de-bonding and cohesive fracture within dentin, adhesive, resin composite cement, or resin composite.

All data were analyzed with Shapiro-Wilk and Levene's tests to check normality and homogeneity of variance, respectively. Then μTBS data were analyzed using One-way ANOVA and Tukey's post-hoc test. The level of significance was set at α=0.05. The analysis was performed using a statistical software system (SPSS 27.0; IBM Corp., Armonk, NY, USA).

Results

The μTBS data are shown in Table 3. Statistically significant differences were observed among the tested groups ($p < 0.05$). Regarding curing condition, primer assisted resin composite cement (PV) exhibited the highest μTBS values, with no significant difference between AA and AL modes. Adhesive assisted resin composite cement (SA and RXU) demonstrated significantly lower μTBS in co-curing mode compared to pre-cured adhesive. High rates of pretesting failures were recorded in co-curing groups.

Table 3 The mean μTBS, standard deviation (SD), number of pre-testing failures per total number of specimens (ptf/n), and predominant failure mode of each experimental group.

Experimental group	Mean μTBS (MPa)	SD	ptf/n	Predominant failure
PV AA	38.7 a	8.6	0/36	Cohesive at Composite
PV AL	40.1 a	11.8	0/35	Cohesive at Composite
SA AL	2.0 d	2.4	15/34	Adhesive
SA LL	18.2 c	7.4	0/32	Adhesive
RXU AL	6.4 d	5.4	8/35	Adhesive
RXU LL	31.5 b	6.7	0/33	Adhesive

Statistically non-significantly different groups (Tukey multiple comparisons, $p > 0.05$) are labeled with the same lowercase letter.

Failure Analysis

Failure mode distribution varied notably among resin composite cements and curing modes (Figure 1). Representative SEM photomicrographs of failure interfaces of 6 experimental groups are shown in Figure 2. Adhesive failures predominated in SA and RXU in both curing mode groups. In contrast, PV demonstrated a higher proportion and consistent occurrence of cohesive failures within the composite or dentin.

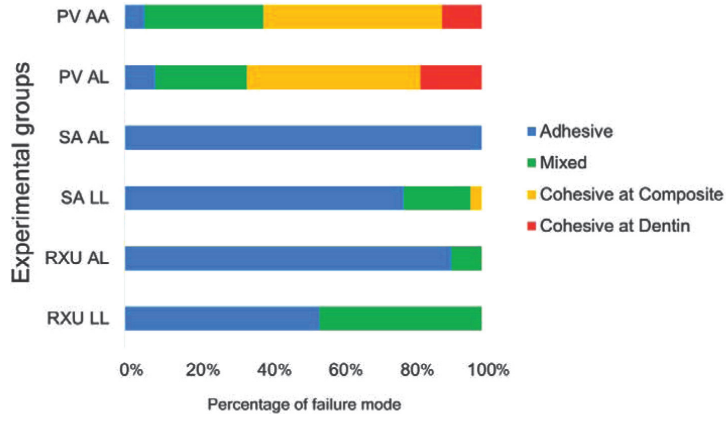


Figure 1 Results of the light-microscopy failure analysis for the different experimental groups.

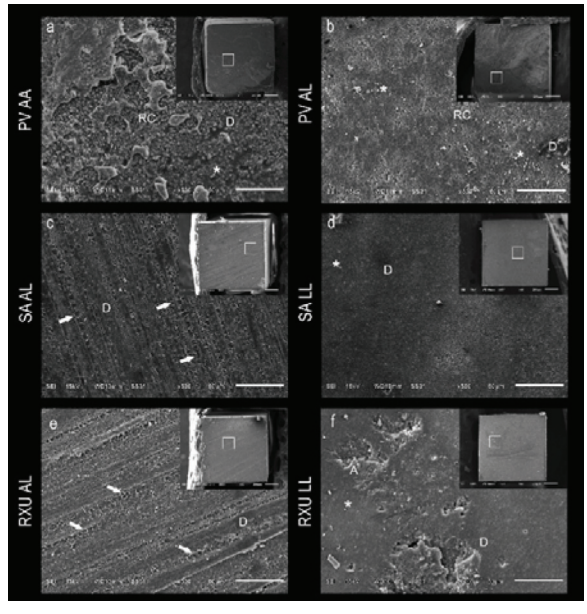


Figure 2 SEM photomicrographs of failure interfaces (dentin side) at magnification: 80× (top right) and 500× (center). Mixed failure of PV AA (a) and PV AL (b) were chosen rather than cohesive pattern to show resin tags and resin composite cement covering dentin surface, indicating optimal infiltration of the resin. Adhesive failure interface of adhesive-assisted resin composite cement in co-curing mode SA AL (c) and RXU AL (e) revealed a void pattern due to water remnant infiltration from dentinal tubules. SA LL (d) and RXU LL (f) showed dentin covered with adhesive. Some dentin tubules are open or slightly filled with adhesive. A-adhesive, Arrows – voids, Asterisk-resin tags, RC-resin composite cement, D-dentin.

Discussion

This study evaluated μTBS of resin composite cements under different curing modes, emphasizing the manufacturers' recommended co-curing protocols. Our results showed that there were statistically significant differences in μTBS between the composite cement systems and curing modes. Thus, both hypotheses were rejected.

The μTBS results of this study demonstrated that primer-assisted resin composite cements (PV) exhibited significantly higher μTBS compared to adhesive-assisted resin composite cements (SA and RXU) in all curing modes. The primary difference between the primer and adhesive lies in their composition, specifically high molecular weight hydrophobic monomers, such as Bis-GMA or DEGDMA, that can increase the viscosity [7]. It can compromise adequate solvent and water evaporation during adhesive application, resulting in trapped residual water, which ultimately causes nano leakage and reduced bond strength [7, 8]. In contrast, primers without any hydrophobic component have much lower viscosity, as they maintain homogeneity and consistent solvent evaporation, contributing to more durable bonding outcomes [7].

Adhesive-assisted resin composite cements (SA and RXU) had a significantly high proportion of adhesive failures as well as pretesting failures. This indicates weak interfaces due to an underdeveloped resin infiltration and incomplete adhesive polymerization. Uncured adhesives form thin, incomplete films and allow water penetration, impairing the adhesive-cement interface confirmed by SEM microphotographs. The failure interfaces showed voids and blister-like structures, due to infiltration of remaining water in dentinal tubules into uncured adhesives through osmosis [2, 3].

Several previous studies have reported that pre-curing universal adhesives results in significantly higher dentin bond strength compared to co-curing mode when used with dual-cure resin composite cements [2, 3, 9, 10]. In those studies, the adhesives were not specifically designed for co-curing, which explains their reduced performance under that protocol [2, 3, 9, 10]. In contrast, both SA and RXU are marketed as compatible with co-curing. Despite this, our findings showed a similar trend to earlier studies as bond strength was significantly reduced when the adhesive was not pre-cured.

In contrast, primer-assisted resin composite cement (PV) showed no significant differences in μTBS regardless of curing mode, showing a much higher proportion of cohesive failures. This suggests a stronger resin–dentin bond that exceeds the internal strength of the resin composite. The consistent performance across auto-curing and co-curing modes via light polymerization likely results from their “Touch-cure” accelerating primers [5]. Particularly, Panavia V5 tooth primer incorporates transition metal accelerator (vanadyl acetylacetonate) [5, 11]. These metals cycle between oxidation states (V(IV)/V(V)), enabling electron transfer reactions [12]. This creates a self-sustaining catalytic cycle, reducing reliance on external initiators and minimizing oxygen inhibition by converting oxygen-derived species into active initiators [12]. As a result, rapid chemical polymerization occurs at the primer-dentin interface, also known as ‘Touch-cure’ polymerization, helping to form stable hybrid layers and reducing susceptibility to moisture without reliance on light polymerization [5, 11, 13].

Composite blocks were used instead of ceramic due to their superior compatibility and bond strength with resin composite cements [14]. Additionally, the resin composite blocks were pre-polymerized prior to cementation, which helped minimize the effect of polymerization shrinkage stress

on the measured bond strength. This study has certain limitation as the number of resin composite cement systems evaluated may not be sufficient to represent primer/adhesive-assisted resin composite cement. Future studies could expand to a broader resin composite cement system. Additionally, further investigation into the degree of conversion and polymerization kinetics, specifically at the resin–dentin interface, would provide deeper insight into how different curing modes influence the interfacial quality of these systems.

Co-curing of adhesives was once proposed as a way to potentially reduce polymerization stress at the interface as when the adhesive and overlying cement cure together, the two would shrink as a single unit and minimize stress on the tooth interface [15]. Additionally, some manufacturers have recommended the co-curing mode for their universal resin composite cements [1]. Our findings indicate that co-curing adhesive-assisted cements results in weaker bond strength. Separate curing of the adhesive is recommended and should always be performed, rather than selectively as suggested by manufacturers. Primer-assisted cements with 'Touch-cure" system provide higher bond strength regardless of curing mode, a simpler clinical procedure and more reliable clinical outcomes.

Conclusion

By comparing the bond strength of resin composite cement with the manufacturer's recommended co-curing mode, adhesive-assisted cements have compromised bond strength due to water infiltration through the adhesive-dentin interface. It is recommended to separate the light curing for the adhesive. Primer-assisted resin composite cement containing "Touch-cure" primers maintained high bond strength regardless of curing mode.

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