

ความแข็งแรงยึดติดระหว่างเซลฟ์แอดฮีซีฟเรซินซีเมนต์ และเนื้อฟันส่วนตัวฟันโดยใช้สารยึดติดเซลฟ์เอตซ์แบบขั้นตอนเดียว

Shear Bond Strength between Self-Adhesive Resin Cement and Coronal Dentin using One-Step Self-Etch Bonding

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ชม. ทันตสาร 2558; 36(2) : 81-88

CM Dent J 2015; 36(2) : 81-88

บทคัดย่อ

วัตถุประสงค์เพื่อศึกษาความแข็งแรงยึดติดระหว่างเซลฟ์แอดฮีซีฟเรซินซีเมนต์กับเนื้อฟันส่วนตัวฟันที่เตรียมผิวฟันด้วยสารยึดติดเซลฟ์เอตซ์แบบขั้นตอนเดียว ใช้ฟันกรามน้อยจำนวน 50 ซี่ กรอดัดรากฟันออกที่บริเวณรอยต่อเคลือบฟันและเคลือบรากฟัน กรอดัดตัวฟันในแนวแก้ม-ลิ้น ได้ชิ้นงานฟันส่วนใกล้กลางและชิ้นงานฟันส่วนไกลกลางจำนวน 100 ชิ้น ดัดตัวฟันด้านใกล้แก้มในแนวใกล้กลาง-ไกลกลางห่างจากรอยต่อเคลือบฟันและเนื้อฟัน 1 มิลลิเมตร ใช้เทปกาวด้านเดียวที่มีเส้นผ่านศูนย์กลาง 1 มิลลิเมตรติดบริเวณด้านใกล้แก้มที่ใช้ทดสอบ ยึดติดแท่งเรซินคอมโพสิตบนเนื้อฟันด้วยเรซินซีเมนต์ตามคำแนะนำของบริษัท โดยแบ่งชิ้นงานออกเป็น 5 กลุ่มๆ ละ 20 ชิ้นโดยวิธีสุ่ม ดังนี้

Abstract

The aim of this study was to evaluate shear bond strength between self-adhesive resin cement and coronal dentin using one-step self-etch bonding. Fifty human premolar teeth were cut at the cemento-enamel junction. The crowns were divided into mesial and distal parts by splitting them longitudinally in a bucco-lingual direction. The 100 split crowns were cut longitudinally on the buccal side in a mesio-distal direction 1mm into dentin from the dentino-enamel junction. The buccal sides of the sectioned crowns were used as the substrate for testing. A one-sided adhesive tape

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กลุ่มที่ 1 กลุ่มควบคุม ยึดชิ้นงานด้วยพานาเวียเอฟ 2.0 กลุ่มที่ 2 ยึดชิ้นงานด้วยรีไลเอกซ์ ยูร้อย กลุ่มที่ 3 เตรียมผิวฟันด้วยสารยึดติดแอดเปออร์ อีซีวัน แล้วยึดชิ้นงานด้วยรีไลเอกซ์ ยูร้อย กลุ่มที่ 4 ยึดชิ้นงานด้วยแมกเซม อีลิท กลุ่มที่ 5 เตรียมผิวฟันด้วยสารยึดติด ออฟดิบอนด์ ออลอินวัน แล้วยึดชิ้นงานด้วยแมกเซม อีลิท นำชิ้นงานทั้งหมดไปแช่ในอ่างน้ำกลั่นอุณหภูมิ 37 องศาเซลเซียส เป็นเวลา 24 ชั่วโมง ก่อนนำไปทดสอบความแข็งแรงยึดเหนี่ยวด้วยเครื่องทดสอบอินตรอน 5566 ที่ความเร็วหัวกด 0.5 มิลลิเมตรต่อนาที นำข้อมูลที่ได้มาทำการวิเคราะห์ทางสถิติเปรียบเทียบความแข็งแรงยึดเหนี่ยวโดยใช้การวิเคราะห์ความแปรปรวนแบบทางเดียว และทดสอบเชิงซ้อนด้วยวิธีทูกี้ พบว่าค่าความแข็งแรงยึดเหนี่ยวเฉลี่ยของกลุ่มควบคุมและกลุ่มที่ใช้สารยึดติดแอดเปออร์ อีซีวัน แล้วยึดชิ้นงานด้วยรีไลเอกซ์ ยูร้อย ไม่แตกต่างกันอย่างมีนัยสำคัญทางสถิติ ($p > 0.05$) แต่มากกว่ากลุ่มอื่นอย่างมีนัยสำคัญทางสถิติ ($p < 0.05$) ภายใต้อัจฉริยภาพของการทดลองนี้พบว่าค่าความแข็งแรงยึดเหนี่ยวกลุ่มที่ทำการเตรียมผิวฟันด้วยสารยึดติดชนิดเซลฟ์ เอตซ์แบบขั้นตอนเดียวแล้วยึดชิ้นงานด้วยเซลฟ์ แอด-ฮีซีฟเรซินซีเมนต์ มีค่าสูงกว่ากลุ่มที่ไม่ได้เตรียมผิวฟันก่อนการยึดชิ้นงานด้วยเซลฟ์แอดฮีซีฟเรซินซีเมนต์

คำสำคัญ: เซลฟ์แอดฮีซีฟ เซลฟ์เอตซ์ เรซินซีเมนต์

with a 1 mm-diameter hole was fixed on the dentin surface. The composite resin rods were randomly divided into five groups, each group containing 20 specimens: a control group (Panavia F2.0), a Rely X U100 group, an Adper Easy One with Rely X U 100, a Maxcem Elite group and an Optibond All-In-One with Maxcem Elite group. The rods were bonded to selected dentin surfaces using matching cementing agents and bonding systems from the same manufacturers. The bonded specimens were immersed in 37°C distilled water for 24 hours and then, subjected to a shear bond test using a universal testing machine (Instron 5566) at a cross-head speed of 0.5 mm/min. One-way ANOVA was used to determine statistical differences ($p < 0.05$) in shear bond strength. Tukey's test revealed no significant difference between the control cement and the Rely X U100 with Adper Easy One but showed significantly higher shear bond strength than the other tested cements or combination of cement and bonding agent. One-step self-etch bonding exhibited increased shear bond strength between self-adhesive resin cement and coronal dentin.

Keywords: Self-adhesive, Self-etch, Resin cement

Introduction

The clinical success of an indirect restorative procedure depends in part on the cementation technique used to create a link between the restoration and the tooth⁽¹⁻³⁾. Currently, resin-based adhesive luting materials are widely used for the fixation of inlays, onlays, crowns, posts and veneers⁽⁴⁾. The bonding of resin-based cements traditionally requires the additional use of a total-etch adhesive system, whereby phosphoric acid is used to completely dissolve the smear layer and create a zone of partial-

ly demineralized dentin, to which the primers and resins are then applied to achieve micromechanical bonding⁽⁵⁻⁸⁾. Alternatively, a self-etch adhesive system utilizes acidic resin monomers to simultaneously demineralize and infiltrate the smear layer-covered dentin⁽⁹⁻¹¹⁾.

Recently-developed self-adhesive resin cements do not require pretreatment of the dentin. Because these cements do not use an adhesive system, they drastically reduce the number of application steps, shortening clinical treatment time and decreasing

technique sensitivity, since they minimize errors throughout the treatment procedure^(9, 12-14). They are based on a new monomer, filler and initiation technology. The manufacturers report that the organic matrix consists of newly-developed multifunctional phosphoric acid methacrylates. The phosphoric acid methacrylates can react with the basic fillers in the luting cement and hydroxyapatite of the hard tooth tissue^(9, 14-16).

Many studies have evaluated the bond strength of self-adhesive resin cements to dentin using microtensile or shear testing methods⁽¹⁶⁻²²⁾. However, the self-adhesive resin cements nearly always show lower bond strength than resin cements that use adhesive bonding systems, such as Panavia F and Variolink II^(19, 22-25). Some studies^(9, 16, 19, 26, 27) have been conducted to identify the best way to improve this bond strength. When tested with pre-etching of dentin by phosphoric acid, self-adhesive resin cements have been shown to have lower bond strength than when tested without pre-treatment⁽¹⁶⁾. This has been attributed to the self-adhesive resin cement's inability to infiltrate the collagen depleted by the etching step^(16, 29). However, bonding of self-adhesive resin cements to coronal dentin presents a challenge to clinical use because it is difficult to predict the long term success of restorations retained with such cements. Therefore, the present study focused on investigating the effect of pretreating dentin with one-step self-etch bonding. The aim of this study was to compare shear bond strength between self-adhesive resin cement and coronal dentin used with or without one-step self-etch bonding. The null hypothesis of the study was that self-adhesive resin cement with or without dentin adhesive agent has no significant influence on the shear bond strength between the dentin and composite resin restoration.

Materials and Methods

Self-adhesive resin cements were used as the adherence substrate materials with one-step self-etching bonding agents. The control resin cement was Panavia F2.0. The composition and applications of all materials used are listed in Table 1.

Tooth preparation

Fifty non-carious human premolar teeth were stored in 0.5% chloramine solution and used within six months after extraction. Each tooth was cleaned with pumice and a prophylaxis cup at low speed for 10 seconds. The teeth were cut at the cemento-enamel junction using a diamond disc under water lubrication (Fig. 1a). The crowns were divided into mesial and distal parts by splitting them longitudinally in a bucco-lingual direction (Fig. 1b). The 100 split crowns were cut longitudinally on the buccal side in a mesio-distal direction 1mm into dentin from the dentino-enamel junction. All measurements were made at least 2 mm coronal to the cemento-enamel junction. The split crown was cut 1 mm from DEJ on the buccal aspect (Fig. 1c). The buccal sides of the sectioned crowns were used as the substrate for testing (Fig. 1d). The prepared crowns were mounted in stainless steel rings with self-cure acrylic resin (Tempron, GC Corporation, Tokyo, Japan) (Fig. 1e). The adherend surface of the tooth was flat-ground with 600-grit alumina abrasive, cleaned ultrasonically in distilled water and air dried. The specimens were randomly divided in to five groups, each group containing 20 specimens. The tooth surfaces were kept moist throughout the specimen preparation procedure.

Composite resin rod preparation

A microhybrid composite resin (Filtek Z250, 3M ESPE, Seefeld, Germany) was shaped into 2mm-thick, 2 mm-diameter cylindrical rods by the split mold technique, and light polymerized for two

Table 1 List of materials used in this study.

Group/Material	Composition	Application
1.Panavia F2.0 (Kuraray, Osaka, Japan)	Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments, others, sodium fluoride	Mix ED primer (A&B), apply to tooth surface for 60 sec., gently air blow 5 sec., mix cement (paste A&B), lute resin rod using light pressure, light cure (20 sec) for each side, apply oxyguard for at least 3 min.
2.Rely X U100 (3M ESPE, Seefeld, Germany)	Glass powder, methacrylated phosphoric acid esters, triethylene glycol dimethacrylate (TEGDMA), silane-treated silica, sodium persulfate, sodium p-toluene sulfinate, calcium hydroxide.	Mix cement, lute resin rod using light pressure, light cure (20 sec) for each side.
3.Adper Easy One+Rely X U 100 (3M ESPE, Seefeld, Germany)	2-hydroxyethyl methacrylate (HEMA), bisphenol A glycol dimethacrylate (Bis-GMA), methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, methacrylate functionalized polyalkenoic acid, finely dispersed bonded silica filler, ethanol, water, camphorquinone, stabilizers. Rely X U100 see above.	Apply primer (20sec), gently air dry (20sec), light cure (10sec). Cement: Rely X U100 see above.
4.Maxcem Elite (Kerr, Orange, CA, USA)	Glycerol phosphate dimethacrylate (GPDM), co-monomer (mono-, di-, and tri-functional methacrylate monomers), proprietary self-curing redox activator, photo-initiator camphoroquinone, stabilizer, fluoro-aluminosilicate glass, fused silica, barium glass, ytterbium fluoride	Mix cement, lute resin rod using light pressure, light cure (20 sec) for each side.
5.Optibond All-In-One+Maxcem Elite (Kerr, Orange, CA, USA)	Glycerol phosphate dimethacrylate monomer (GPDM), mono-, di- and multi-methacrylate co-monomers, water, acetone and ethanol, camphorquinone, filler, sodium hexafluorosilicate, ytterbium fluoride. Maxcem Elite see above.	Apply two consecutive coats of adhesive primer (20sec), gently air dry (20sec), light cure (10sec). Cement: Maxcem Elite see above.
Composite resin Filtek Z250 (3M ESPE, Seefeld, Germany)	Bisphenol A glycol dimethacrylate (Bis-GMA), ethoxylated bisphenol A glycol dimethacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA), urethane dimethacrylate (UDMA), nanosilica filler	-

minutes with an LED light unit operating at 1000 mW/cm² (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein). The bonded surface of each composite resin rod was airborne-particle abraded using 50 µm alumina oxide for 10 seconds at a distance of 10 mm, cleaned with ultrasonic cleanser in distilled water and air dried for 30 seconds.

Specimen preparation for shear bond test

The bonding areas on the flat dentin specimens in the rings were located 2mm coronal to the cemen-

to-enamel junction. A one-sided adhesive tape with a 1 mm-diameter hole was fixed on the dentin surface. The composite resin rods were randomly divided into five groups: a control group (Panavia F2.0), a Rely X U100 group, an Adper Easy One with Rely X U 100, a Maxcem Elite group and an Optibond All-In-One with Maxcem Elite group. The rods were bonded to selected dentin surfaces using matching cementing agents and bonding systems from the same manufacturers. (Table I, Fig. 1f). After one day, the bonded specimens were immersed in 37°C distilled water for

24 hours. The specimens were subjected to a shear bond test using a universal testing machine (Instron 5566, Norwood, MA, USA) at a crosshead speed of 0.5 mm/min.

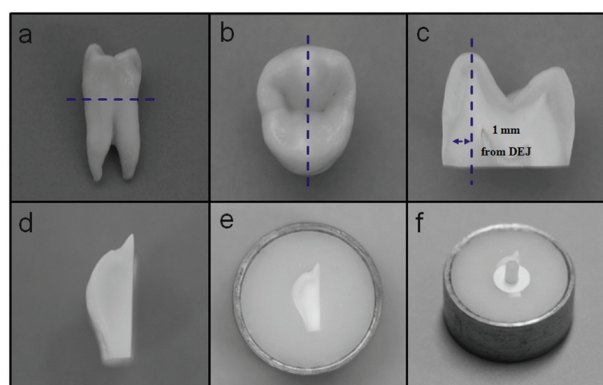


Figure 1 Schematic illustration of specimen preparation for testing: (a) the tooth was cut at the CEJ; (b) the crown was divided into mesial and distal parts; (c) the split crown was cut 1 mm from DEJ on the buccal aspect; (d) the prepared tooth; (e) the prepared crown was mounted in a stainless steel ring; (f) the bonded specimen.

After the shear bond test, all specimens, all de-bonded interfaces between composite rods and coronal dentin were observed using a light microscope (Meiji Techno Co. Ltd., Tokyo, Japan) at x40 magnification. Failure modes were classified into the following categories: type I – cohesive failure in the resin cement; type II – cohesive failure in the dentin; type III – adhesive failure; type IV – mixed failure: cohesive failure in resin cement and dentin; and type V – mixed failure: cohesive failure in the resin cement and in the bonding region.

Statistical analysis

The mean and standard deviation of the shear bond strength values for each load at failure were calculated, and the results were subjected to one-way analysis of variance (ANOVA) followed by

the Tukey multiple comparisons test at the 5% confidence level. The calculations were performed using statistical software (SPSS ver 17.0, SPSS Inc. Chicago, IL, USA).

Results

The means and standard deviations of shear bond strength values are presented in Table II. The highest mean shear bond strength on coronal dentin was for Rely X U 100 with Adper Easy One and also demonstrated a significant difference compared to the mean shear bond strength of the cements without self-etch bonding. The Panavia F2.0 group and the an Adper Easy One with Rely X U 100 group did not show a statistically significant difference in shear bond strength but they each showed significantly higher shear bond strength than did the other groups. The Optibond All-In-One with Maxcem Elite group exhibited higher mean shear bond strength than did the Maxcem Elite group without Optibond All-In-One.

The failure pattern distribution is demonstrated in Table III. A predominance of adhesive failures was demonstrated in the Rely X U 100 group and the Maxcem Elite group. A remarkably higher incidence of type III failure was noted for both the Adper Easy One with Rely X U 100 group and the Optibond All-In-One with Maxcem Elite group when dentin was treated with a one-step self-etch bonding agent.

Table 2 Means (Standard deviations) in Megapascals (MPa)

Group	Mean (Standard deviation)
1. Panavia F2.0	29.08(5.32) ^c
2. Rely X U100	12.72(4.65) ^a
3. Adper Easy One + Rely X U100	29.77(7.92) ^c
4. Maxcem Elite	8.92(3.81) ^a
5. Optibond All-In-One + Maxcem Elite	22.37(5.68) ^b

*different letters mean statistically significant differences

Table 3 Failure modes for each group.

Type Group	I	II	III	IV	V
1	-	-	4	-	16
2	2	-	14	-	4
3	1	-	5	-	14
4	3	-	15	-	2
5	1	-	6	-	13

Discussion

The shear bond strength between self-adhesive resin cement and coronal dentin was higher with one-step self-etch bonding than without. The adhesion mechanisms of Rely X U100 and Maxcem Elite are claimed by the manufacturers to rely on both micromechanical retention and chemical interaction between the phosphate monomer acidic group and hydroxyapatite in each cement,⁽¹⁶⁾ and methacrylate phosphate esters in Rely X U100, and glycerol dimethacrylate dihydrogen phosphate monomer (GPDM) in Maxcem Elite.

The phosphate monomer acidic groups chelate the calcium ions of hydroxyapatite, promoting chemical adhesion. Therefore, an ionic bond can also be formed between these cements and the tooth's hydroxyapatite, a bond that positively influences the chemical bond. The chemical bond might account for the superior performance of self-adhesive cements. However, in this study, there was no significant difference in shear bond strength between the two groups of self-adhesive resin cement. This lack of difference was probably due to the presence of the same functional phosphate monomer in each cement^(12,28).

The finding that one step-self-etch bonding pre-treatment of dentin significantly enhances the shear bond strength might be the result of micromechanical retention produced by the hybrid layer and resin tags in the dentin (Fig. 2). Bonding to dentin with one-step self-etch adhesive, such as Adper Easy One and Optibond All-In-One, involves dissolving

the inorganic smear layer and demineralization of the intertubular dentin. Simultaneously, the adhesive penetrates the demineralized dentin to form the hybrid layer and flows into the dentin tubule to create resin tags. Adper Easy One self-etch adhesive includes a phosphoric ester, which under aqueous conditions etches the surface of the dentin to allow for the micromechanical retention of a self-adhesive resin cement. Moreover, the phosphoric ester and the Viterbond copolymer in Adper Easy One form a chemical bond to the hydroxyapatite by forming a complex with the calcium ions, but Optibond All-In-One can form chemical interaction with GPDM only.

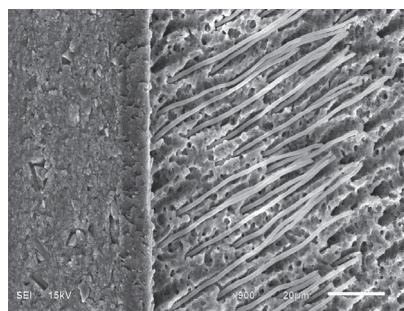


Figure 2 Scanning electron microscopy image of the dentin surface of a specimen bonded with Adper Easy One + Rely X U100

Based on the results obtained in this study, the null hypothesis that self-adhesive resin cement with or without dentin adhesive agent has no significant influence on the shear bond strength between the dentin and composite resin restoration was rejected.

Conclusions

Within the limits of this study, it was concluded that one-step self-etch adhesive pre-treatment significantly increased shear bond strength of self-adhesive resin cement. Adper Easy One with Rely X U 100 showed similar shear bond strength to Panavia F2.0.

Acknowledgments

This study was carried out with partial financial support from the Faculty of Dentistry, Chiang Mai University. The authors would like to thank Dr. M. Kevin O Carroll, Professor Emeritus of the University of Mississippi, School of Dentistry, USA and Faculty Consultant at Chiang Mai University, Faculty of Dentistry, Thailand, for his editorial assistance in the preparation of the manuscript.

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