

# ผลของการสลับไพรเมอร์และแอตชีฟเรซินต่อกำลังแรงยึด แบบเฉือนของแบรacketทางทันตกรรมจัดฟัน Effects of Alternation of Primers and Adhesive Resins on Shear Bond Strength of Orthodontic Brackets

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## บทคัดย่อ

การศึกษาเพื่อเปรียบเทียบกำลังแรงยึดแบบเฉือนของแบรacketทางทันตกรรมจัดฟัน โดยการใช้วัสดุยึดติดแบบแข็งตัวด้วยแสงสองชนิดที่ใช้ทั่วไป ชนิดที่หนึ่งคือทรานสบอนด์เอกซ์ที และชนิดที่สองคือเอนไลท์ โดยมีการสลับส่วนของไพรเมอร์และแอตชีฟเรซิน นำฟันกรามน้อยที่ถูกถอน จำนวน 120 ซี่มาแบ่งโดยการสุ่มเป็น 4 กลุ่มๆ ละ 30 ซี่ ทำความสะอาดส่วนของผิวเคลือบฟันด้วยฟลูออไรด์แล้วล้างและเป่าแห้ง ทำการยึดติดแบรacketกับผิวฟันโดยแบ่งออกเป็น 4 วิธี ดังนี้ วิธีที่ 1 ใช้ไพรเมอร์และแอตชีฟของวัสดุชนิดที่หนึ่ง วิธีที่ 2 ใช้ไพรเมอร์ของวัสดุชนิดที่หนึ่งและใช้แอตชีฟของวัสดุชนิดที่สอง วิธีที่ 3 ใช้ไพรเมอร์ของวัสดุ

## Abstract

This study evaluated the shear bond strength of metal orthodontic brackets bonded to enamel surfaces using two common brands of light-cured bonding systems (Transbond™ XT and Enlight®) and the effects of alternation of the primers and adhesive resins. One hundred and twenty extracted premolar teeth were randomly divided into four groups of 30. The teeth were pumiced, rinsed and air dried. Four bonding approaches were planned. The first and the fourth bonding approaches used the primers and the adhesive resins from each brand respectively.

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ชนิดที่สองและใช้แอตชีฟของวัสดุชนิดที่หนึ่ง และวิธีที่ 4 ใช้ไพรเมอร์และแอตชีฟของวัสดุชนิดที่สอง จากนั้นเก็บในน้ำกลั่นที่อุณหภูมิ 37 องศาเซลเซียสนาน 24 ชั่วโมง แล้วนำไปผ่านขบวนการเทอร์โมไซเคิลระหว่างอุณหภูมิ  $5\pm 2$  องศาเซลเซียสและ  $55\pm 2$  องศาเซลเซียสจำนวน 1,000 รอบ แล้วนำไปทดสอบค่ากำลังแรงยึดแบบเฉือนด้วยความเร็วหัวกด 0.5 มิลลิเมตรต่อวินาที ทำการตรวจระดับตัวแอตชีฟที่หลงเหลืออยู่บนผิวฟันโดยการใช้อัลตร้าซาวด์ 3 เท่า

ผลการทดสอบพบว่า ค่ากำลังแรงยึดแบบเฉือนเฉลี่ยของการใช้สารยึดติดชนิดที่หนึ่งสูงกว่าของสารยึดติดชนิดที่สองอย่างมีนัยสำคัญทางสถิติ การสลับส่วนของไพรเมอร์และแอตชีฟเรซินระหว่างวัสดุทั้งสองชนิดให้ค่าแรงยึดติดแบบเฉือนไม่ต่างกันโดยสัมพันธ์กับชนิดของแอตชีฟเรซิน ซึ่งสามารถนำมาใช้ทดแทนกันในทางคลินิกได้

**คำสำคัญ :** กำลังแรงยึด, ไพรเมอร์ทางทันตกรรมจัดฟัน, สารยึดติดทางทันตกรรมจัดฟัน

The second method used the first primer with the second adhesive resin, while the third method used the second primer and the first adhesive resin. All specimens were stored in distilled water for 24 hours at  $37^{\circ}\text{C}$ . The aging process was operated through thermocycling for 1,000 cycles between  $5\pm 2^{\circ}\text{C}$  and  $55\pm 2^{\circ}\text{C}$ . Shear bond strength was tested using an Instron<sup>®</sup> testing machine at a cross head speed of 0.5 mm/min. ARI scores were recorded under a light microscope with 3X magnification. Mean shear bond strength with the first brand was significantly higher than that with the second brand. Alternation of primers and adhesive resins produced no significant difference in bond strength related to the adhesive resin, which implies a possible clinical use.

**Keywords:** bond strength, orthodontic primers, orthodontic adhesive resins

## Introduction

Techniques in orthodontic bonding are applied from conventional restorative treatment. The conventional bonding of orthodontic brackets to enamel uses the total etching and bonding approach and provides good adhesion. The total etching two-step bonding procedure with light-cured activation is commonly applied in the Orthodontic Clinic, Faculty of Dentistry, Chiang Mai University. A bonding agent is used to provide an initial bond with the tooth surface. Then, an adhesive bulk with a bracket is added with a chemical bond being provided by the bonding agent.

Adhesive bond strength is governed by the presence of stress concentrations in the adhesive or at the interface, rather than by local forces of

attraction at the interface.<sup>(1)</sup> Moreover, the adhesive tends to have poorer mechanical properties than the substrates being bonded.<sup>(1)</sup> The surfaces and internal defects can play a major role in determining the bond strength of adhesive joints.<sup>(1)</sup> Adhesion at the bracket-cement interface is achieved by a mechanical undercut, into which the adhesive extends before polymerization. Micromechanical retention of resin composites to acid-etched enamel may not be due only to formation of resin tags but also to the formation of an interfacial resin-enamel interdiffusion zone within the lateral sites of the remaining enamel protuberance.<sup>(2)</sup>

For directing bonding, there are three main types of adhesive resins in orthodontics classified according to the method of polymerization

initiation.<sup>(2)</sup> They are self-cured, light-cured, and dual-cured systems. A self-cured orthodontic adhesive can be either a two-paste or a one-paste system. A two-paste self-cured system, for example, Phase II<sup>®</sup> (Reliance Orthodontic Products, Inc., Itaska, Illinois, USA), Concise<sup>™</sup> (3M Unitek, Monrovia, California, USA), requires the mixing and application of two liquids (bonding resins) to the enamel and the mixing and application of two pastes to the bracket base.<sup>(3)</sup> Mixing of two components leads to some defects such as surface porosity and air voids in the material. Consequently, no mix or one-paste self-cured systems have been introduced, for example, Unite (3M Unitek), Rely a Bond<sup>®</sup> (Reliance Orthodontic Products, Inc.), Right On<sup>®</sup> (TP Orthodontics, La Porte, Indiana, USA), and System 1+ (Ormco, Orange, California, USA), with which teeth and brackets are preloaded with the liquid component and paste before being applied.<sup>(4)</sup> Light-cured adhesives are good alternatives to two-paste systems.<sup>(2)</sup> They provide ease of use, extended working time in bracket placement, easier clean-up of flashes, faster cure of composite, and allow immediate archwire placement.<sup>(5)</sup> There are many commercially available light-cured adhesives, such as Transbond<sup>™</sup> XT (3M Unitek), Light Bond<sup>®</sup> (Reliance Orthodontic Products, Inc.), Enlight<sup>®</sup> (Ormco). The last one is commercially claimed to be a light-cured adhesive with a dark-cure mechanism. Polymerization is initiated by a reaction between the catalyst in the adhesive and the photons emitted by the light-curing source. The extent of polymerization depends on several factors: exposure time, photo-initiator concentration, light intensity from the curing unit at the peak absorbance wavelength of the photo-initiator, and the filler volume fraction.<sup>(2)</sup> However, bonding metal brackets with light-cured adhesives may be problematic.<sup>(4)</sup> Incorporation of

self-cured and light-cured modes of activation produces a dual-cured adhesive.<sup>(4)</sup> The purpose of a double mechanism in dual-cured resins is, primarily, to boost the polymerization and to achieve a high degree of conversion, especially in areas remote or hidden from the light source.<sup>(6)</sup> An example of a dual-cured adhesive is Phase II Dual Cure<sup>®</sup> (Reliance Orthodontic Products, Inc.).

In our Orthodontic Clinic, two common orthodontic bonding brands are used. They are the Transbond<sup>™</sup> XT bonding system (3M Unitek), which is a light-cured system, and the Enlight<sup>®</sup> bonding system (Ormco), which is commercially claimed as a dual-cured system. Sometimes, one component of each brand is out of stock, while the other remains. Even though we know that the materials should be used within the same brand, there remains the question of whether the orthodontic bonding primer and the adhesive resin can be switched between these two brands to achieve optimal bond strength. The null hypothesis was that “there is no difference in mean shear bond strength with alternation of primers and adhesive resins between two bonding systems”.

## Materials and Methods

One hundred and twenty human upper premolar teeth extracted for orthodontic reasons were used. All teeth had intact enamel on the buccal surfaces with an absence of caries, restorations, fluorosis, or other defects. The teeth were stored in 0.1% (weight/volume) thymol solution after extraction for one to eight months prior to the bonding process. The teeth were randomly categorized into four groups of 30. The teeth were prepared by sectioning with a carborundum disc about 3 mm apical to the cemento-enamel junction. The buccal surface of each tooth was polished with fluoride-free pumice slurry and a rubber cup for 15 seconds, and rinsed

with water for 15 seconds. The excess water was removed from the surface with oil-free compressed air. The lingual half of each sample was placed in a clay block as a holder during the bonding process. The long axis of the sample was laid as parallel as possible to the base of the block. Each buccal surface was etched with Gel Etch<sup>®</sup> (Ormco) using a microbrush for 15 seconds, washed with water

for 15 seconds, and air-dried to achieve a frosty appearance. A stainless steel bracket for upper premolar teeth (3M Unitek) was bonded on the enamel surface. Two brands of adhesive system were used; the compositions are shown in Table 1.<sup>(7-11)</sup> Four bonding approaches were used as shown in Table 2.

**Table 1** Compositions and materials used in this study.

**ตารางที่ 1** ส่วนประกอบของวัสดุที่ใช้ในการศึกษา

		<b>Compositions</b>	
<b>Etchant</b>	<b>Ormco<sup>®</sup> Etching Gel (Gel Etch<sup>®</sup>)</b>	37% phosphoric acid, Water, Fumed silica	
<b>Bonding system</b>		<b>Primer</b>	<b>Adhesive</b>
	<b>Transbond<sup>™</sup> XT Light cure Adhesive</b>	Bisphenol A diglycidyl ether dimethacrylate Triethylene glycol dimethacrylate 4-(dimethylamino)-benzeneethanol DL-camphoroquinone Hydroquinone	Bisphenol A diglycidyl ether dimethacrylate (BisGMA) Bisphenol A Bis(2-hydroxyethyl ether) dimethacrylate Silane treated quartz Silane treated silica Diphenyliodonium hexafluorophosphate
	<b>Enlight<sup>®</sup> Bonding system</b>	<b>Ortho Solo<sup>™</sup> :</b> HydroxyEthylMethAcrylate (HEMA) Ethyl Alcohol Disodium HexaFluorosilicate 4 Methoxyphenol (MEHQ) Inert fillers Pigments	<b>Enlight<sup>®</sup> :</b> Uncured methacrylate ester monomers Activators Inert mineral fillers Fumed silica Preservatives

**Table 2** The bonding approaches in this study.

**ตารางที่ 2** การออกแบบการยึดแบร็กเกตตามชนิดของไพรเมอร์และแอคซีซีฟเรซิน

<b>Group</b>	<b>Bonding approach</b>	
	<b>Primer</b>	<b>Adhesive resin</b>
I	Transbond <sup>™</sup> XT (3M Unitek)	Transbond <sup>™</sup> XT (3M Unitek)
II	Transbond <sup>™</sup> XT (3M Unitek)	Enlight <sup>®</sup> (Ormco)
III	Ortho Solo <sup>™</sup> (Ormco)	Transbond <sup>™</sup> XT (3M Unitek)
IV	Ortho Solo <sup>™</sup> (Ormco)	Enlight <sup>®</sup> (Ormco)

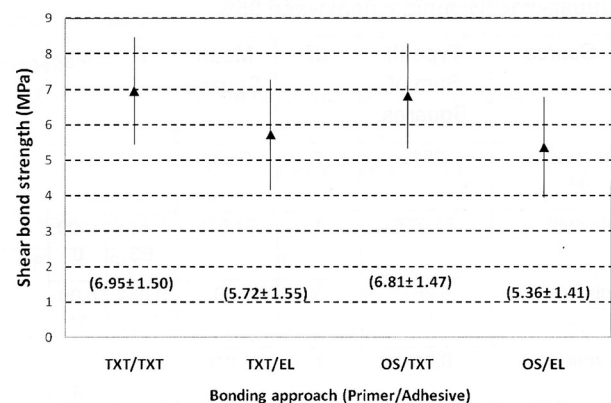
Primer was applied on the buccal surface with a microbrush, followed by placement of the bracket lined with the adhesive resin on the tooth. The long axis of the bracket was positioned parallel to the tooth surface, under firm pressure. Excessive adhesive resin was removed with an orthodontic sickle. The adhesive resin was cured with a DEMI™ LED curing unit (Kerr, Sybron Dental Specialties, Orange, California, USA) for 10 seconds on each proximal portion of the bracket. The light tip was placed less than 4 mm from the bracket base. A 2-inch section of 0.019 x 0.025-in rectangular stainless steel wire was attached to the bracket with an elastic module. The assembly was positioned on a 2-inch square plastic sheet with a central 20-mm diameter hole and secured with adhesive tape and then placed on the center of a 25-mm diameter PVC ring. The PVC ring was filled with self-curing acrylic resin to the level of the bottom of the specimen. When the resin was completely cured, the elastic module, the wire, and the plate were removed, leaving only the specimen, resin and PVC ring. All specimens were incubated in distilled water at 37°C for 24 hours. The specimens were processed in a thermocycling machine (Model TC 301, Medical and Environmental Equipment Research Laboratory, King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand) between 5°C and 55°C for 1,000 cycles. The immersion time for each bath was 20 seconds, and the transferring time was 10 seconds. After this aging process, all specimens underwent a shear test using an Instron® universal testing machine (Model number 5566, Instron Calibration Laboratory, Canton, Massachusetts, USA). A cross-head speed of 0.5 mm/min was used with a 1 kilo-Newton load cell in an occluso-gingival direction until bracket dislodgement. The force value was recorded in Newtons, and was divided by the bracket base area of 10.61 mm<sup>2</sup>. The force

per unit area was obtained as shear bond strength in Mega Pascals (MPa).

Meanwhile, the adhesive remnant index (ARI) was also recorded under a light microscope according to the method of Artun and Bergland.<sup>(12)</sup> Analysis of variance (ANOVA) and two post-hoc comparison tests (Tukey HSD test and t-test) were performed to evaluate and compare the mean shear bond strength values. Descriptive analysis was performed to evaluate the ARI scores.

## Results

Mean shear bond strength values (MPa) for the different bonding approaches are demonstrated in Figure 1.



**Figure 1** Mean shear bond strength and standard deviation values (MPa) according to bonding approach (TXT = Transbond™ XT, EL = Enlight®, OS = Ortho Solo™)

**รูปที่ 1** ค่าเฉลี่ย และส่วนเบี่ยงเบนมาตรฐานของกำลังแรงยึดติดแบบเหนียว (เมกะปาสคาล) ในแต่ละกลุ่ม

Two-way ANOVA at  $\alpha = 0.05$  was applied; there was no statistically significant interaction between primer factor and adhesive factor. Mean bond strength values between each primer were also not significantly different. However, a significant difference in mean bond strength values was found between adhesive types, as shown in Table 3.

**Table 3** Comparison of mean shear/peel bond strength with two-way ANOVA at  $\alpha = 0.05$ .

**ตารางที่ 3** การเปรียบเทียบค่ากำลังแรงยึดแบบเหนียวโดยการวิเคราะห์ความแปรปรวนแบบสองทางที่ระดับนัยสำคัญทางสถิติ 95%

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	281a	7	0.094	3.160	0.027
Intercept	74.978	1	74.978	2.534E3	0.000
Adhesive	0.267	3	0.267	9.034	0.003*
Primer	0.010	1	0.010	0.353	0.553
Adhesive Primer	0.003	3	0.003	0.093	0.761
Error	3.432	112	0.030		
Total	78.691	120			
Corrected Total	3.713	119			

R Squared = 0.076 (Adjusted R Squared = 0.052)

**Table 4** Comparison of mean bond strength values between adhesive types.

**ตารางที่ 4** การเปรียบเทียบค่าเฉลี่ยกำลังแรงยึดแบบเหนียวระหว่างชนิดแอตชีฟเรซิน

Levene's test for equality of variances		t-test for equality of means			Mean diff.	Std. error diff.
F	Sig.	t	df	Sig.(2-tailed)		
0.666	0.416	3.026	118	0.003*	1.244	1.074

At  $\alpha = 0.05$ , the t-test to compare the bond strength between both adhesive resins found that the Transbond™ XT adhesive provided statistically significantly higher mean bond strength ( $6.88 \pm 1.48$  MPa) than did the Enlight® adhesive ( $5.54 \pm 1.48$  MPa), as shown in Table 4.

The ARI scores to identify the bond failure mode after de-bonding are shown in Table 5.

According to Artun and Bergland<sup>(12)</sup>, score 1 means that there was no adhesive left on the tooth surface. Score 2 means that there was less than 50% of the adhesive left on the tooth surface. Score 3 means that there was more than 50% of the adhesive left on the tooth surface. Finally, score 4 means that all of the adhesive was left on the tooth surface.

**Table 5** Frequency and percentage of ARI scores for each bonding approach.

**ตารางที่ 5** ความถี่ของคะแนน ARI (ร้อยละแสดงในวงเล็บ) สำหรับแต่ละกลุ่ม

Bonding approach		ARI Score: Count (%)				Total
Adhesive	Primer	0	1	2	3	
Transbond™ XT	Transbond™ XT	0	3 (10)	13 (43.33)	14 (46.67)	30 (100)
Transbond™ XT	Ortho Solo™	0	1 (3.33)	12 (40)	17 (56.67)	30 (100)
Total 1		0	4 (6.67)	25 (41.67)	31 (51.66)	60 (100)
Enlight®	Transbond™ XT	0	10 (33.33)	10 (33.33)	10 (33.34)	30 (100)
Enlight®	Ortho Solo™	0	3 (10)	19 (63.3)	8 (26.7)	30 (100)
Total 2		0	13 (21.67)	29 (48.33)	18 (30)	60 (100)



More than 85% of the specimens indicated ARI scores of 2 or 3. This implies that more than half of the adhesive resin remained on the tooth surfaces after de-bonding. However, there was an increase in the number of specimens with ARI scores of 1 with the Enlight<sup>®</sup> adhesive (21.67%) compared to the Transbond<sup>™</sup> XT adhesive (6.67%). There was no cohesive failure either on tooth surfaces or bracket bases.

## Discussion

This study compared the shear bond strength of two light-cured adhesive resins commonly used in the Orthodontic Clinic, Faculty of Dentistry, Chiang Mai University, and also compared the bond strength when the primers and the adhesives were alternated. For each bonding brand, the Transbond<sup>™</sup> XT system provided statistically significantly higher bond strength than did the Enlight<sup>®</sup> bonding system. This finding is the same as that of another study.<sup>(13)</sup> Some studies found similar mean bond strength values comparing these two adhesives.<sup>(5,14-16)</sup> However, alternating between primers and adhesive did not show a statistically significant difference in bond strength among each group.

Within the same adhesives, whichever the primer was, the mean shear bond strength values were not significantly different, as shown in Table 3. It is implied that the primer factor is not significant. In contrast, the study showed that the adhesive factor is more important than the primer factor. The pure light-cured adhesive (Transbond<sup>™</sup> XT) provided significantly higher bond strength than did the commercially-claimed light and self-cured adhesive (Enlight<sup>®</sup>).

The bonding attachment to enamel is primarily due to micromechanical adhesion.<sup>(6)</sup> The removed inorganic tooth material is replaced by resin monomers that become interlocked in the retentive

tags upon curing. Adhesion-promoting constituents, particularly resin components, enhance the attraction to enamel and metallic substrates and create a chemical bond.<sup>(6)</sup> In this way a combination of mechanical interlocking with chemical bonding increase bond integrity favorably. Adhesion-promoting monomers differ among each adhesive, each thus producing its own specific attraction or reactivity toward certain substrates.<sup>(6)</sup> However, these monomers function similarly; they are, typically, bifunctional molecules having at least one methacrylate group that can participate in the polymerization (setting) reaction, and at least one reactive group that can create a chemical bond to enamel and the metallic appliance.<sup>(6)</sup> The filler may also contain reactive species that may chemically interact with the substrate.<sup>(6)</sup> This organic or semi-organic constituent, used as a minor component of the filler matrix, has little or no significant contribution to the cohesive strength of the composite, but offers an added chemical bond enhancement to the substrate.

In this study comparing two primers, Ortho Solo<sup>™</sup> and Transbond<sup>™</sup> XT, there was no significant difference in mean bond strength. Ortho Solo<sup>™</sup> contains the hydrophilic acrylic monomer HEMA, which is used in general dentistry for bonding to enamel and dentin.<sup>(17)</sup> This hydrophilic monomer displaces moisture during bonding.<sup>(18)</sup> Transbond<sup>™</sup> XT primer contains Bis-GMA and TEGDMA as monomers. Both primers contain ethanol as a solvent to wet the surface, promote penetration of the monomers into the demineralized surface, and absorb any water present.<sup>(6,19)</sup> Transbond<sup>™</sup> XT primers contain DL-CQ and hydroquinone as photo-initiators. Ortho Solo<sup>™</sup> contains disodium hexafluorosilicate as an initiator which can also release fluoride. Ortho Solo<sup>™</sup> also contains a small amount of submicron

silica which imparts additional strength and viscosity control.<sup>(19)</sup> Moreover, only Ortho Solo™ contains 4 MEHQ as an inhibitor which extends shelf-life of the monomer.<sup>(6)</sup>

This study found that a significant difference in mean bond strength values was found between adhesive types. Selection of the adhesive is a concern in bonding. Transbond™ XT adhesive contains two forms of Bis-GMA to enhance chemical bonding.<sup>(8)</sup> There are small amounts of uncured methacrylated ester monomers in Enlight® adhesive. Each adhesive contains fillers to control the viscosity and reduce sliding of the attachment.<sup>(6)</sup> Transbond™ XT adhesive contains silane-treated silica and quartz to allow for chemical bonding between the filler and resin matrix.<sup>(20)</sup> This could be the reason for significantly higher shear bond strength of Transbond™ XT adhesive than Enlight® adhesive. In addition, silane coupling protects the adhesive resin against premature degradation and improves the stress transmission between the resin matrix and filler particles.<sup>(20)</sup> The activator in Enlight® adhesive may represent the chemically cured behavior which renders it a dual-cured adhesive. Its polymerization is initiated rapidly through exposure to light, and polymerization in the material occurs by a chemical curing process with the expectation of improved properties of bulk materials.<sup>(2)</sup>

An ideal orthodontic adhesive should have adequate bond strength while maintaining unblemished enamel.<sup>(21)</sup> ARI determination shows the cohesive or adhesive nature of orthodontic bonds. In this study, cohesive failure, either at the enamel or bracket base surface, was not found. Comparing both adhesives in this study, there was an increase in the number of specimens with ARI scores of 1 for Enlight® adhesive (from 6.67% to 21.67%), which implies that less than 50% of the adhesive

resin remained on the tooth surface, as shown in Table 5. It is accepted that reduced adhesive remaining on the tooth decreases the time required to clean the enamel surface.<sup>(22)</sup> This would be the advantage of Enlight® adhesive.

Orthodontic adhesives are intricate mixtures of ingredients.<sup>(23)</sup> Each ingredient has some specific effect on the bond strength, bond efficiency, bond durability, and the shelf life and biocompatibility of the adhesive system. The chemical composition of contemporary adhesives determines their clinical success. This study concerned only two bonding systems or brands commonly used in our orthodontic clinic. It is possible to state that Transbond™ XT adhesive provided higher bond strength than did Enlight® adhesive, whichever primer was applied, in bonding metal brackets on the enamel surface. Reynolds suggested that a minimum bond strength of 5-8 MPa was adequate for orthodontic bonding.<sup>(24)</sup> In this study, the bond strengths of all groups were acceptable according to Reynolds' suggestion. However, this was a laboratory study and some more materials need to be investigated.

## Conclusions

The results of this study suggest the followings:

1. At  $p < 0.05$ , the Transbond™ XT bonding system (Transbond™ XT primer and Transbond™ XT adhesive) possessed significantly greater shear bond strength than did the Enlight® bonding system (Ortho Solo™ and Enlight® adhesive).
2. The primer and adhesive resin of each bonding brand or system can be alternated.
3. At  $p < 0.05$ , Transbond™ XT adhesive can be applied satisfactorily with both adhesive primers, while Enlight® adhesive can also be utilized the same way but with significantly less shear bond strength than with Transbond™ XT.



4. Future investigations should focus on the other adhesive systems available commercially to identify more cost-beneficial bonding systems. More profound knowledge of these ingredients is one key to better understanding the behavior of adhesives in the laboratory and in the clinic.

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