

# กำลังยึดติดแบบเฉือนของระบบสารยึดติดที่ต่างชนิดกัน เมื่อใช้ยึดแบร็กเกตจัดฟันกับฟันผิวฟันตกกระ Shear Bond Strength of Different Adhesive Systems for Bonding Orthodontic Brackets to Fluorotic Teeth

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## บทคัดย่อ

วัตถุประสงค์ของการศึกษาเพื่อวัดและเปรียบเทียบค่าเฉลี่ยกำลังยึดติดแบบเฉือนของระบบสารยึดติดต่างชนิดกันสำหรับการยึดแบร็กเกตทางทันตกรรมจัดฟันบนฟันตกกระและฟันปกติ การศึกษานี้ใช้ฟันกรามน้อยจำนวน 120 ซี่ แบ่งฟันออกเป็น 6 กลุ่ม กลุ่มละ 20 ซี่ กลุ่มที่ 1 กลุ่มที่ 2 และ 3 ยึดแบร็กเกต บนฟันปกติด้วยระบบสารยึดติดซิสเทมวันพลัส ยูไนท์และซูเปอร์บอนด์ซีแอนด์บี ตามลำดับ กลุ่มที่ 4 กลุ่มที่ 5 และ 6 ยึดแบร็กเกตบนฟันตกกระด้วยระบบสารยึดติดซิสเทมวันพลัส ยูไนท์และซูเปอร์บอนด์ซีแอนด์บี ตามลำดับ นำทุกกลุ่มทดลองผ่านกระบวนการเปลี่ยนแปลง

## Abstract

The aims of this study were to measure and compare the mean shear bond strength of different adhesive systems for bonding orthodontic brackets to fluorotic and normal teeth. One hundred and twenty premolar teeth were divided into six groups (N=20), brackets were bonded and fixed to the sample teeth. Groups 1, 2 and 3 contained normal teeth bonded with System<sup>TM</sup>1+, Unite<sup>TM</sup> and Super-Bond C&B. Groups 4, 5 and 6 contained fluorotic teeth bonded with System<sup>TM</sup>1+, Unite<sup>TM</sup> and Super-

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อุณหภูมิที่อุณหภูมิระหว่าง 5±2 และ 55±2 องศาเซลเซียส จำนวน 1000 รอบ จากนั้นนำไปทดสอบกำลังยึดติดแบบเชื่อมด้วยเครื่องทดสอบแบบสากล นำข้อมูลมาวิเคราะห์ทางสถิติด้วยการวิเคราะห์ความแปรปรวนแบบสองทาง ผลการศึกษาพบว่าค่าเฉลี่ยกำลังยึดติดแบบเชื่อมของระบบสารยึดติดทุกชนิดที่ใช้ยึดติดบนฟันปกติ สูงกว่าค่าเฉลี่ยกำลังยึดติดแบบเชื่อมของระบบสารยึดติดกับฟันตกกระอย่างมีนัยสำคัญทางสถิติ ( $p<0.05$ ) ในกลุ่มฟันปกติและฟันตกกระ พบว่าค่าเฉลี่ยกำลังยึดติดแบบเชื่อมของระบบสารยึดติดซูเปอร์บอนด์ซีแอนด์บีมากกว่า ซิสเทมวันพลัสและยูไนท์อย่างมีนัยสำคัญทางสถิติ ( $p<0.05$ )

**คำสำคัญ:** กำลังยึดติดแบบ ระบบสารยึดติด ฟันตกกระ

Bond C&B. Thermocycling was performed at 5±2°C and 55±2°C for 1,000 cycles. Shear bond strength was measured using an universal testing machine at a crosshead speed of 0.5 mm/min. The data were analyzed using two-way ANOVA. The results indicated the mean shear bond strength values of all adhesives used on normal teeth were significantly greater than those used on fluorotic teeth ( $p<0.05$ ). With both normal and fluorotic teeth, the mean shear bond strength value of Super-Bond C&B was significantly greater than the mean shear bond strength values of System™1+and Unite™ ( $p<0.05$ ).

**Keywords:** shear bond strength, adhesive systems, fluorotic teeth

## Introduction

Excessive fluoride ingestion during tooth formation leads to dental fluorosis.<sup>(1)</sup> The features of fluorosis vary from white lines in the enamel to chalky, pitted and discolored enamel.<sup>(1-2)</sup> Endemic fluorosis results from the ingestion of excessive quantities of fluoride, usually in drinking water. There are well known endemic areas in India, Kenya, Tanzania, Uganda, Ethiopia, Sudan, China, Japan, Argentina, Saudi Arabia, United States of America, Canada and Europe.<sup>(3-4)</sup> The geological features of Northern Thailand include mountains and forests with abundant mineral resources, including fluoride. So, a high prevalence of dental fluorosis is found in this area.<sup>(5-6)</sup> Some people in this area need orthodontic treatment. There are reports that it is difficult to bond fluorotic teeth and there is a notable clinical failure rate for bonding to such teeth.<sup>(7)</sup> It should be beneficial to find an alternative adhesive system for teeth with

fluorosis. The aims of this study were to measure and compare the mean shear bond strength of different adhesive systems for bonding orthodontic brackets to fluorotic and normal teeth.

## Materials and Methods

One hundred and twenty (60 fluorotic and 60 non-fluorotic, age range 12-19 years) non-carious, human permanent premolar teeth, freshly extracted for orthodontic reasons without any visible defects, were used in this study. The study was approved by the Human experimentation Committee, Faculty of Dentistry, Chiang Mai University. The fluorotic teeth were selected according to the Thylstrup and Fejerskov (TF) index<sup>(8)</sup>, which is based on the clinical changes in fluorotic teeth. Teeth with a TFI score of 3, 4 or 5 were used in this study. The specific features of teeth with a TFI score of 3 are: merging of white lines and cloudy areas of opacity spread over many parts of the surface. The specific

features of teeth with a TFI score of 4 are: a marked opacity and a chalky white appearance on the entire surface. The specific features of teeth with a TFI score of 5 are: an opaque surface with round pits that are less than 2 mm in diameter. The roots were removed from the crowns with carborundum discs, approximately 2 mm below the cemento-enamel junction. Each tooth was individually embedded in autopolymerizing acrylic resin in a polyvinylchloride ring. Each tooth was embedded in the center of the ring. The setting time of acrylic resin was 10 minutes. The specimens were kept in distilled water except during the bonding and testing procedures. The 60 fluorotic teeth were divided into three adhesive groups. In the same way, the non-fluorotic teeth were also assigned to three groups (Table 1). The adhesives used in this study were: System™1+ (Ormco Corp., Glendora, California, USA), Unite™ (3M Unitek, Monrovia, California, USA) and Super-Bond C&B (Sun Medical Co. Ltd., Shiga, Japan) (Table 2).

**Table 1** Study group characteristics

**ตารางที่ 1** ลักษณะของกลุ่มที่ทำการศึกษา

Group	Tooth	Adhesive
1	Normal	37%phosphoric acid and System™ 1+
2	Normal	37%phosphoric acid and Unite™
3	Normal	65 %phosphoric acid and Super-Bond C&B
4	Fluorotic	37%phosphoric acid and System™ 1+
5	Fluorotic	37%phosphoric acid and Unite™
6	Fluorotic	65 %phosphoric acid and Super-Bond C&B

**Bonding with System™1+ (Group 1 & Group 4)**

Before bonding, the facial surfaces of the teeth were cleaned with a mixture of water and pumice. The teeth were rinsed thoroughly with water and dried with oil- and moisture-free compressed air. Each tooth was etched with 37 percent phosphoric acid gel for 30 seconds and rinsed with a water/spray combination for 20 seconds. Minidiamond

**Table 2** Adhesive systems used in the study

**ตารางที่ 2** ระบบสารยึดติดที่ใช้ในการทดลอง

Material	Application	Composition	Lot No.	Manufacture
System™ 1+ Self-cured (no-mix)	Paste-primer formulation. Application of liquid component on enamel and bracket base. No mixing is involved.	Etching agent 37% phosphoric acid Urethane modified dimethacrylate	7E1 080612 080709	Ormco Corp (Glendora, California, USA)
Unite™ Self-cured (no-mix)	Paste-primer formulation. Application of liquid component on enamel and bracket base. No mixing is involved.	Etching agent 37% phosphoric acid Bis-GMA/ Triethyleneglycol dimethacrylate	7E1 7D1 7E1	3M Unitek (Monrovia, California, USA)
Super-Bond C&B Self-cured adhesive resin cement	Mix liquid and power with brush-dip technique. Apply on bracket base.	Etching agent 65% phosphoric acid Polymethyl methacrylate 4-META, MMA Partly oxidized TBB	SK1  SG1 SL1 SF32	Sun Medical Co. Ltd. (Shiga, Japan)

(Ormco Corp., Glendora, California, USA) premolar metal brackets with an 8.0 mm<sup>2</sup> surface area were used. System™ 1+ (Ormco Corp.) was used as the orthodontic adhesive. The liquid activator was applied to the etched enamel surface and the bracket base. Then, the composite resin was applied to the bracket base. After that, the bracket was firmly placed on the center of the enamel surface and excessive resin was removed from the enamel surface with an explorer before setting.

**Bonding with Unite™ (Group 2 & Group 5)**

Each tooth was etched with 37 percent phosphoric acid gel for 15 seconds, rinsed with a water/spray combination for 20 seconds. Unite™ (3M Unitek, Monrovia, California, USA) was used as the orthodontic adhesive. The liquid activator was applied to the etched enamel surface and the bracket base. Then, the composite resin was applied to the bracket base. After that, the bracket was firmly placed on the center of the enamel surface and excessive resin was removed from the enamel surface with an explorer.

**Bonding with Super-Bond C&B (Group 3 & Group 6)**

Each buccal surface was etched with red activator (65 %phosphoric acid gel), which was in the Super-Bond C&B kit (Sun Medical Co. Ltd., Shiga, Japan), for 30 seconds, washed for 20 seconds and air dried. Super-Bond C&B was used as the orthodontic adhesive. The catalyst, a partly oxidized Tri-*n*-Butyl borane (TBB) initiator, was added to the monomer mixture of 4-methacryloxyethyl trimellitate anhydride (4-META) and methyl-methacrylate (MMA) to prepare an activated polymerized monomer liquid. Then, the polymer powder and the activated monomer liquid were mixed and used to bond the metal brackets to

the center of the treated enamel surface using the brush-dip technique.

All specimens were stored in distilled water at 37°C for 24 hours and thermocycled for 1,000 cycles between 5±2 and 55±2°C, using a dwell time of 30 seconds and transfer time of 10 seconds.

**Shear bond strength testing**

The shear bond strength value was determined by using a universal testing machine (Instron® 5566, Instron Calibration Laboratory, Norwood, Massachusetts, USA) at a crosshead speed of 0.5 mm per minute. The polyvinylchloride ring was mounted into the jig of the testing machine, which was mounted into the lower part of the instrument. The de-bonding plate was fixed into the upper part of the instrument (Figure 1). The force was applied in the gingivo-occlusal direction until the bracket was dislodged from the tooth surface and the shear bond strength at bond failure was recorded.



**Figure 1** De-bonding plate, mounting jig and tooth with bracket

**รูปที่ 1** แผ่นทำลายการยึดติด แนววัดตั้งและฟันซึ่งมีแปรีกเกตยึดอยู่

**Failure mode evaluation**

After de-bonding, failure sites were determined by examination of the de-bonded bracket bases on photographs from a Nikon D80 camera (NIKS (Thailand) Co, Ltd., Bangkok, Thailand ), with a 105 F2.8 EX DG MACRO lens (Sigma Corporation, Kuriki Asao-Ku Kawasaki-shi, Kanagawa, Japan) at F 20, a distance between the camera and bracket bases of 11 cm and an exposure time of 1/125 seconds. The percentages of residual adhesive per total de-bonded bracket surface area were calculated and converted to residual adhesive per total de-bonded enamel surface area.

The failure sites were divided into four locations according to the method of Artun and Bergland<sup>(9)</sup> as follows:

The failure sites were divided into four locations according to the method of Artun and Bergland,<sup>9</sup> which used the Adhesive Remnant Index (ARI) as follows:

- 0 = no adhesive remains on the enamel
- 1 = less than half of the adhesive remains on the tooth surface
- 2 = more than half of the adhesive remains on the tooth
- 3 = all the adhesive remains on the tooth with a distinct impression of the bracket base

**Statistical analysis**

Descriptive statistics, including the means and standard deviations, were calculated for each of the test groups. Two-way analysis of variance (ANOVA) and Tukey multiple comparison tests were used to compare the shear bond strength values in the groups. Significance for all statistical tests was predetermined at  $p < 0.05$ . The Kruskal-Wallis test was used to analyze the ARI score.

**Results**

**Shear bond strength**

The mean shear bond strength values in Groups 1 to 6 were 10.25±2, 11.59±2.32, 13.86±1.65, 6.51±3.57, 7.51±4.79, and 12.29±2.91 MPa, respectively (Table 3). Two-way ANOVA revealed that the interaction of two factors, adhesive system and type of enamel, was not significant ( $p > 0.05$ ). So, shear bond strength was influenced by two factors: type of enamel ( $p < 0.05$ ) and adhesive system ( $p < 0.05$ ). The mean shear bond strength values of all adhesives used on normal teeth were significantly greater than those of all adhesives used on fluorotic teeth ( $p < 0.05$ ). The multiple comparisons test showed that for both normal and fluorotic teeth, the mean shear bond strength values of System™ 1+ and Unite™ were not significantly different, but were significantly

**Table 3 Mean shear bond strength and standard deviations for the adhesive systems**

**ตารางที่ 3 ค่าเฉลี่ยและส่วนเบี่ยงเบนมาตรฐานของกำลังยึดติดแบบเหนียวของระบบสารยึดติด**

Group	Tooth	Adhesive	Mean shear bond strength ( MPa )
1	Normal	37%phosphoric acid and System™ 1+	10.25 <sup>b</sup> ±2.00
2	Normal	37%phosphoric acid and Unite™	11.5 <sup>9b</sup> ±2.32
3	Normal	65 %phosphoric acid and Super-Bond C&B	13.86 <sup>d</sup> ±1.65
4	Fluorotic	37%phosphoric acid and System™ 1+	6.51 <sup>a</sup> ±3.57
5	Fluorotic	37%phosphoric acid and Unite™	7.51 <sup>a</sup> ±4.79
6	Fluorotic	65 %phosphoric acid and Super-Bond C&B	12.29 <sup>c</sup> ±2.91

\* mean shear bond strength values with the same letter superscripts are not significantly different ( $p > 0.05$ )

\* ค่าเฉลี่ยของกำลังยึดติดแบบเหนียวที่อักษรตัวเดียวกันไม่มีความแตกต่างกันอย่างมีนัยสำคัญทางสถิติ ( $p > 0.05$ )

different from those of Super-Bond C&B ( $p=0.000$ ).

### Failure mode

The modes of failure following the shear bond strength test are summarized in Table 4. The Kruskal-Wallis test revealed that the mean rank of ARI scores of the six groups were significantly different ( $p<0.000$ ). In normal teeth, the commonest site of failure for System™ 1+ was at the enamel/adhesive interface which suffered adhesive and cohesive failures. For Unite™, the sites of failure were found at the enamel/adhesive interface as well as at the adhesive/bracket interface, both of which suffered adhesive and cohesive failures. The commonest site of failure for Super-Bond C&B was found at the adhesive/bracket interface, which suffered only adhesive failures. In fluorotic teeth, the commonest site of failure for System™ 1+ and Unite™ was at the enamel/adhesive interface where both adhesive and cohesive failures were found. The commonest site of failure for Super-Bond C&B was at the adhesive/bracket interface, where only adhesive failures were found.

### Discussion

In this study, the mean shear bond strength of normal teeth ranged from 10.25 to 13.86 MPa; that of fluorotic teeth ranged from 6.51 to 12.29 MPa.

The findings demonstrate that fluorotic teeth significantly reduced the shear bond strength of brackets bonded to enamel. This effect may be due to the acid-resistant outer layer of the fluorosed enamel.<sup>(10)</sup> Fluorotic teeth have the highest concentration of fluoride in the outer 200 µm of enamel. The concentration of fluoride in this region increases with increasing Thylstrup and Fejerskov's (TF) score.<sup>(11)</sup> The hypermineralized surface layer of fluorotic enamel is difficult to etch, resulting in less irregularity of the enamel surface after enamel etching than in normal enamel.<sup>(7,12-13)</sup>

Our findings are consistent with those of Adanir et al.<sup>(10,14)</sup> and Gungör et al.,<sup>(15)</sup> who reported that fluorotic teeth significantly decreased the bond strength of orthodontic brackets. However, the bond strength with fluorotic teeth is more than the minimum 6 to 8 MPa that is sufficient for clinical orthodontic bonding.<sup>(10,14-15)</sup> Ertugrul et al.<sup>(16)</sup> studied shear bond strength using three different bonding strategies with normal and moderately fluorotic enamel. They found that the bonding effectiveness to enamel was lower in fluorotic teeth than in normal teeth for all the adhesives tested. Besides, another report showed that routine acid etching of fluorotic teeth produced shear bond strength that was less sufficient than that required for clinical orthodontic bonding.<sup>(17)</sup> Weerasinghe et al.<sup>(18)</sup> reported that the degree of fluorosis in fluorotic teeth affected the

**Table 4** The numbers and percentages of ARI scores of adhesive systems

**ตารางที่ 4** จำนวนและค่าร้อยละของคะแนนเออาร์ไอของระบบสารยึดติด

Group	Tooth	Adhesive	ARI				Total
			0	1	2	3	
1	Normal	System™ 1+	-	10 (50%)	9 (45%)	1 (5%)	20
2	Normal	Unite™	-	10 (50%)	10 (50%)	-	20
3	Normal	Super-Bond C&B	-	-	5 (25%)	15 (75%)	20
4	Fluorotic	System™ 1+	5 (25%)	13 (65%)	2 (10%)	-	20
5	Fluorotic	Unite™	3 (15%)	12 (60%)	5 (25%)	-	20
6	Fluorotic	Super-Bond C&B	-	4 (20%)	6 (30%)	10 (50%)	20

micro-shear bond strengths of a self-etching bonding system to fluorosed enamel, and Shida et al.<sup>(19)</sup> reported that fluorotic teeth demonstrated significantly lower bond strengths than normal teeth. Ermis et al.<sup>(20)</sup> found that the micro-shear bond strength of fluorotic teeth was significantly lower than that of normal teeth.

In contrast to the present study, Ng'ang'a et al.<sup>(21)</sup> studied tensile bond strength in teeth with mild to moderate dental fluorosis. Their study found no significant difference in mean bond strength between fluorotic and normal teeth. Ateyah and Akapata<sup>(22)</sup> reported that the degree of fluorosis had no significant effect on shear bond strength of composite resin bonded to enamel. These findings are consistent with those of Ratnaweera et al.,<sup>(23)</sup> who reported that micro-shear bond strength was not affected the degree of fluorosis.

Studies vary in the results they have reported in bond strength values between fluorotic and normal teeth.<sup>(10,21-22)</sup> Some findings demonstrated that fluorotic teeth significantly reduced the shear bond strength of adhesives used to bond brackets to enamel.<sup>(10)</sup> The decrease may be due to the acid resistant outer layer of the fluorosed enamel. Two studies demonstrated that the bond strength of adhesives used with fluorotic teeth was not significantly different from that with normal teeth.<sup>(21-22)</sup> The lack of difference may have resulted from the fact that one study<sup>(21)</sup> used teeth with mild fluosis and in the other<sup>(22)</sup> the outer hypermineralized acid resistant outer layer was ground before testing.

The mean shear bond strength value of Super-Bond C&B was significantly greater than those of System™ 1+ and Unite™ on both normal and fluorotic teeth. It has been reported that the variation of the concentration of phosphoric acid from 20% (wt) to 65% (wt) did not produce

different bond strength between 4-META/MMA-TBB resin and etched enamel, although demineralization decreased with increasing concentration of phosphoric acid.<sup>(24)</sup> Thus, manufacturers recommend pre-etching the enamel surface with 65% (wt) phosphoric acid for tight adhesion of the 4-META/MMA-TBB resin to enamel in order to minimize the enamel loss. Several investigators have reported that there were statistical differences in bond strength between etching times of 15 and 30 second.<sup>(25-27)</sup> The increase bond strengths achieved with Super-Bond C&B were most likely a result of its being an unfilled acrylic material containing 4-META monomer. 4-META is a difunctional monomer presenting a hydrophobic methacrylate group and a hydrophilic aromatic anhydride group. Functionally, the hydrophobic methacrylate group is able to combine with resins in composite/acrylic adhesives, while the hydrophilic aromatic anhydride group is able to promote adhesion to the tooth surface. The hydrophilic aromatic anhydride group facilitates the infiltration of resin into the etched enamel by wetting the etched surface, reduces interfacial porosity and, therefore, increases adhesion, achieving greater bond strength through polymerization.<sup>(28)</sup> Tri-n-Butyl borane initiates the graft polymerization of MMA and its infiltration into the tooth substrates, and good adhesion to the tooth is, therefore, obtained.<sup>(29)</sup> It is thought that increased bond strength is achieved through the ability of 4-META to enhance diffusion into enamel.<sup>(30)</sup>

Of primary concern to the clinician is the maintenance of a sound, unblemished enamel surface after removal of the bracket; yet bracket failure at each of these two interfaces has its own advantages and disadvantages. As an example, bracket failure at the bracket/adhesive interface is advantageous because it leaves the enamel surface relatively intact. However, considerable chair time

is needed to remove the residual adhesive, with the added possibility of damaging the enamel surface during the removal process. Conversely, when brackets fail at the enamel/adhesive interface, less residual adhesive remains, but the enamel surface can be damaged when failure occurs in this mode.<sup>(31)</sup> In the present study, the ARI scores indicated that brackets bonded with either System™ 1+ or Unite™ showed a similar range of bond failure modes. The most common site of failure of both adhesives was found with adhesive and cohesive failures at the enamel/adhesive interface. Super-Bond C&B significantly increased the bond strength of brackets bonded to fluorotic teeth; it also resulted in the most common site of failure at the adhesive/bracket interface and left the enamel relatively intact.

## Conclusions

1. The mean shear bond strength values in Groups 1 to 6 were 10.25±2, 11.59±2.32, 13.86±1.65, 6.51±3.57, 7.51±4.79, and 12.29±2.91 MPa, respectively.

2. The bonding effectiveness to enamel was lower in fluorotic teeth than in normal teeth for all the adhesives tested.

3. In normal and fluorotic teeth, adhesive and cohesive failures at the enamel/adhesive interface were most common with System™ 1+ and Unite™, whereas with Super-Bond C&B the commonest site of failure was the adhesive/bracket interface.

4. This study suggests Super-Bond C&B (Japan) for clinical use in orthodontic placement of brackets on fluorotic teeth because Super-Bond C&B significantly increased the bond strength of brackets bonded to fluorotic teeth.

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