

Initial Tensile Bond Strength among Various Types of Orthodontic Adhesives

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Abstract

The purpose of this study was to evaluate the initial tensile bond strengths of three light-cured adhesives (Enlight, Transbond XT, and Transbond XT + Transbond Plus Self Etching Primer) compared with a self-cured adhesive (Unite). Three hundred and six new stainless steel brackets were bonded to extracted human premolars. Tensile bond strength of three light-cured adhesives were tested at five time intervals, immediately (representing initial bond strength), 2 minutes, 3 minutes, 4 minutes and 24 hours after light activation. As a control, Unite (self-cured adhesive) was tested at 4 minutes for initial bond strength and 24 hours after bracket bonding. The results indicated that the initial tensile bond strengths of three light-cured adhesives, Enlight (3.30 ± 0.57 MPa), Transbond XT (3.04 ± 0.71 MPa), and Transbond XT + Transbond Plus Self Etching Primer (3.29 ± 0.85 MPa), were significantly lower than that of Unite self-cured adhesive (4.34 ± 1.36 MPa) at p -value < 0.05 . However, the tensile bond strength at 2 minutes after light activation of all light-cured adhesives were not significantly different from initial bond strength of Unite. In addition, tensile bond strength of light-cured adhesives tested at 2, 3, and 4 minutes were not significantly different. The results from this study suggest that when using light-cured adhesives, clinicians should wait at least 2 minutes after light activation in order to minimize bond failure.

Key words: initial tensile bond strength; light-cured adhesive

Introduction

Since Buonocore introduced the acid etch bonding technique in 1955, the concept of bonding various resins to enamel has developed in every field of dentistry including the bonding of orthodontic brackets.^{1,2} After this introduction, various bonding agents were developed. The first and the most popular bonding resin was chemical-curing bonding system.³ Its only drawback is the inability of the practitioner to control the setting time.⁴

The use of light-cured materials for orthodontic bonding was first described by Tavus and Watts in 1979⁵ and they have become increasingly popular since then.⁶ A rapid polymerization occurs when visible light is applied, producing a "command set" that is of the great advantage. This setting on demand results in more accurate bracket placement and the ability to engage the bracket immediately after light activation.^{3,7,8} Most companies have recommended that the self-cured adhesive should be allowed to set for 4-5 minutes before inserting arch wire. For light-cured adhesives, the archwire could be placed immediately after bonding.

From this advantage, clinicians could potentially save chairtime. Moreover, in the straight wire concept introduced by Andrews, where exact positioning of brackets is of utmost importance, light activated systems may be preferred to chemically cured systems as clinicians have more working time.^{7,8,9,10} Light-cured adhesives may eventually replace chemically cured adhesives just as chemically cured adhesives replaced banding.^{5,6}

However, in everyday clinical situations, some dislodgement of the brackets after fully engaging archwire still exists even though the manufacturer recommendations of both light-cured and self-cured adhesives have been followed. Furthermore, some previous studies showed that bond strengths of visible-light-cured adhesives were insufficient after initial setting.^{2,6,11,12} Analyzing in terms of sufficient bond strength, previous studies presented that the successful clinical bonding may be obtained with the bond strength of 5.9 to 7.8 MPa.^{2,13,14} In addition to offering good bond strength, an orthodontic adhesive should enable easy debonding and cleanup procedure without causing enamel damage.¹⁵ The adhesive which had optimal bond strength, and a failure site at the enamel/adhesive interface would be desirable because debonding and subsequent polishing would become much easier without damaging the enamel surface.^{3,16}

Bond strengths of adhesives have been evaluated by many methods. The most common method is to measure the brackets tensile or shear peel forces.¹⁷ Nevertheless, during the initial phase, the tensile force plays the major role because it is a normal force while seating and ligating an arch wire into bracket slots.^{18,19}

Because of the dislodgement problem, this study focuses on finding out whether the light-cured adhesive really sets and reaches the ultimate bond strength immediately after light activation or if it requires a certain amount of setting time before the archwire can be tied in. A comparative study of the initial tensile bond strength between light-cured and self-cured adhesive was evaluated. The effect of time on the tensile bond strength of various orthodontic adhesives was also investigated. In addition, the sites of bond failure were examined.

Materials and Methods

Three hundred and six extracted human premolar teeth with sound buccal enamel surface (free from carious lesion, restoration, enamel crack, enamel hypoplasia or abnormal buccal surface anatomy that might affect the strength of the enamel) were collected and stored in a solution of 0.1% (weight/volume) thymol to prevent dehydration and bacterial growth.

All brackets used in this experiment were 0.022" x 0.028" slot pre-torque, pre-angulated edgewise stainless steel premolar brackets (Minidiamond, Ormish[®], Ormco Corporation, CA, USA.) that had 17-4 stainless steel bracket body and 316 stainless steel foil/mesh base type. Total surface area of the base of each bracket was 10.5 mm² (3 x 3.5 mm).

Four commercially available orthodontic adhesives chosen for bracket attachment in this study comprised 3 light-cured and 1 self-cured adhesives.

These materials were

1. Unite (3M Unitek, USA)
a chemically cured dimethacrylate resin
2. Enlight (Ormco Corporation, Glendale, California, USA)
a low viscosity light-cured dimethacrylate resin
3. Transbond XT (3M Unitek, Monrovia, California, USA)
a light-cured highly-filled dimethacrylate resin
4. Transbond XT + Transbond Plus Self Etching Primer (3M Unitek, USA)
a light-cured dimethacrylate resin with self etching primer

Two thirds of the root of each premolar tooth was cut off and then the lingual surface was grooved to aid retention. Each tooth was mounted with the buccal surface uppermost in a PVC mounting ring with self-cured acrylic resin. The buccal surface of the tooth was adjusted and aligned parallel to the bottom of the mold and perpendicular to the force during the tensile strength test. The specimens were kept in distilled water until testing time.

The 306 specimens were randomly divided into 17 groups. Each group contained 18 specimens for bonding. Two groups were tested with self-cured adhesives at two time intervals; 4 minutes and 24 hours. The other fifteen groups were tested with light-cured adhesives at five time intervals; immediate (within 1 minute), 2 minutes, 3 minutes, 4 minutes and 24 hours for each type of adhesive.

Before bonding, the enamel surfaces of each tooth were polished for 15 seconds using rubber cup and slurry of nonfluoridated

pumice. The bonding protocols followed the manufacturer instructions of each adhesive.

The light source used in this study was Ortholux XT Light Curing unit (3M, Unitek, Monrovia, CA, USA). The light bulb used was a 75 watt tungsten halogen bulb with a light intensity of 400 mW/cm². Before light activation of each sample, the light curing unit was tested for a minimum output of 400 mW/cm². Light-curing time was 40 seconds, dividing 20 seconds on mesial and 20 seconds on distal sides.

The bonding of all brackets was performed by the same operator to standardized technique as in clinical situation. After initial polymerization, the brackets were debonded with a Universal Testing Machine (LF Plus, Lloyd) and tensile bond strength was recorded. Each specimen was inserted and tightly locked in the special mounting jig which was fixed in the lower part of the Universal Testing Machine and the specimens were then loaded to debond the brackets at a crosshead speed of 0.5 mm per minute and loading cell of 1000 Newton (Fig. 1). The tensile force was measured in Megapascals (MPa) and recorded at the point which bond failure occurred.

After bracket dislodging, each tooth and the bracket base were inspected visually under a light microscope, Nikon Eclipse E400POL, at x10 magnification. The area of failure was determined by examining where the majority of adhesive remained.

The means, standard deviations, and ranges for all groups tested were calculated and analyzed by SPSS software for MS WINDOWS, version 12. Descriptive statistics including the mean and standard deviations of the initial tensile bond strength were also evaluated. The data of the initial tensile bond strength were tested for normality with the Kolmogorov-Smirnov method.

1. The differences in initial tensile bond strength among 3 light-cured and 1 self-cured orthodontic adhesives were evaluated. The initial tensile bond strength of Unite tested at 4 minutes was used as a comparative value for clinical use. The nonparametric Kruskal-Wallis test was used to determine whether significant differences existed among 4 types of adhesives. Then the Mann-Whitney test was used to compare and identify which groups were different.

2. The differences in tensile bond strength among different time intervals of each orthodontic adhesive were evaluated. The nonparametric Kruskal-Wallis test was used to determine whether significant differences existed among the 5 time intervals. Then the Mann-Whitney test was used to compare and identify which groups were different.

3. For failure site analysis, descriptive statistics were used to explain the site of bond failure among various types of orthodontic adhesives at different time intervals.

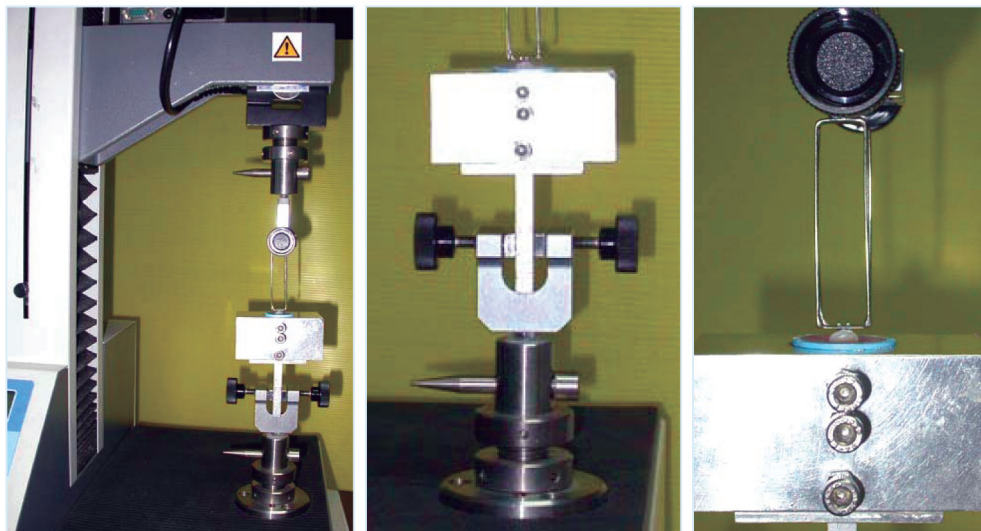


Fig. 1 Specimen was inserted and tightly locked in the special mounting jig which was fixed in the lower part of the Universal Testing Machine.

The overall test was interpreted for significant difference at p -value less than 0.05.

Results

The mean values and the standard deviations (SD) for tensile bond strength of various orthodontic adhesives in Megapascals (MPa) at each time interval are shown in Table 1. Initial tensile bond strengths were measured immediately after light activation (within 1 minute) for light-cured adhesives and at 4 minutes for self-cured adhesive according to manufacturer instructions for archwire placement. Maximum tensile bond strength for every adhesive was measured at 24 hours.

The means of tensile bond strength increased with time in every adhesive group. At initial polymerization, all light-cured adhesives had very low tensile bond strength and increased in consequence with time. Bond strength of Unite, self-cured adhesive, at initial polymerization was also lower than at 24 hours.

The Kruskal-Wallis one-way analysis of variance and subsequent statistical analysis Mann-Whitney U Test were performed on all groups. The statistical results are also shown in Table 1. The letters a, b, c represents the statistical analysis results of tensile bond strength. Identical letters indicate no statistically significant differences were found at p -value > 0.05.

The statistical result in Table 1 shows that the initial bond strength of Unite (4.34 MPa) was significantly higher than the initial bond strength of Enlight (3.30 MPa), Transbond (3.04 MPa), and Self Etch (3.29 MPa) at p -value < 0.05. Whereas, the tensile bond strength at 2, 3, and 4 minutes of light-cured groups was not significantly different from initial bond strength of Unite. Furthermore, the tensile bond

strength of all adhesives tested at 24 hours was not significantly different among different types of adhesives. The initial tensile bond strength of Unite self-cured adhesive was 65.07% of the maximum strength at 24 hours and significantly different between the two time intervals.

For Enlight, Transbond, and Self Etch, the tensile bond strengths at different five time intervals were compared. Significant differences were found between the initial tensile bond strength and that of 2, 3, 4 minutes and 24 hours. The bond strength at 2, 3, and 4 minutes was also significantly different from 24 hours, whereas the bond strength among 2, 3, and 4 minutes was not significantly different. The initial bond strength was 55.56% of maximum strength for Enlight, 46.70% for Transbond, and 47.68% for Self Etch.

The result indicated that bond strength increases with time from initial to that of 24 hours by increasing significantly at the beginning up to 2 minutes then gradually increasing at a slower pace after that.

Site of Bond Failure

Fig. 2 shows the frequency distribution of the failure sites divided into 3 locations according to previous studies.^{3,16}

- EA = enamel/adhesive interface
(0-25% of the adhesive left on the tooth)
- CO = cohesive failure, within the adhesive
(25-75% of the adhesive left on the tooth)
- BA = bracket/adhesive interface
(75-100% of the adhesive left on the tooth)

Table 1 Means and standard deviations of tensile bond strength (MPa)

Time	Immediate (within 1 minute)	2 minutes	3 minutes	4 minutes	24 hours
Adhesive	Mean \pm SD	Mean \pm SD	Mean \pm SD	Mean \pm SD	Mean \pm SD
Unite				4.34 ^b \pm 1.36	6.67 ^c \pm 1.76
Enlight	3.30 ^a \pm 0.57	4.05 ^b \pm 1.04	4.52 ^b \pm 1.94	4.79 ^b \pm 1.47	5.94 ^c \pm 1.18
Transbond	3.04 ^a \pm 0.71	4.06 ^b \pm 0.98	4.52 ^b \pm 1.02	4.78 ^b \pm 1.26	6.51 ^c \pm 1.93
Self Etch	3.29 ^a \pm 0.85	4.20 ^b \pm 1.02	4.46 ^b \pm 1.22	4.84 ^b \pm 1.13	6.90 ^c \pm 1.41

Means followed by the same letter are not significantly different, $p > 0.05$

The frequency distribution of failure sites indicated that the failure site of the light-cured adhesives mostly occurred at BA particularly at the initial time up to 4 minutes after light-activation. Failure sites of light cured adhesives at 24 hours varied among 3 locations except for Enlight, which were still at BA. Failure sites of Unite self-cured adhesive equally occurred at BA and CO at both initial time (4 minutes) and 24 hours.

Discussion

Most clinicians are interested in learning about the properties of the adhesive systems they use in order to optimize their ability to handle them properly and efficiently.¹⁹ Numerous studies have been

performed to find the best orthodontic adhesives.

Bond strength tests can be made using different modes of load application; shear, tension, or torsion. Use of shear loading has been very popular due to the relative simplicity of the experimental configuration and the presumably increased reliability of simulating debonding that occurs from occlusal forces during treatment.^{9,20} However, the tensile bond strength test was used in this study to compare the results with the adequate bond strength suggested by Reynolds²¹ in 1975 and numerous other studies which reported by the mode of tensile strength.^{9,10,11} The adequate strength suggested by Reynolds to withstand orthodontic forces was 5.9-7.8 MPa for clinical performances and 4-5 MPa for laboratory performances. These numbers were widely accepted and have been cited by several studies.^{22,23}

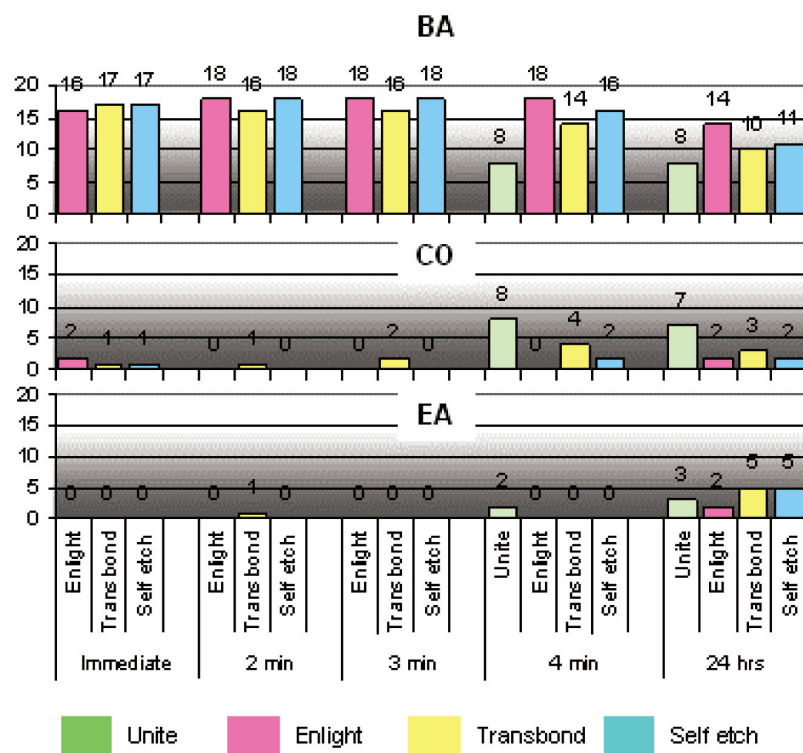


Fig. 2 Frequency distribution of the failure site

In addition, the subcommittee on testing methods of the International Association for Dental Research in 1967 recommended a tensile test for measuring bond strength of resins to dental hard tissues.^{24,25}

This study aimed to evaluate and compare the initial tensile bond strength of three light-cured adhesives measured within 1 minute and one self-cured adhesive measured at 4 minutes. These different time intervals were chosen to represent initial bond strength for each group according to the time intervals recommended by the manufacturers for archwire placement. Previous laboratory studies usually measured bond strength at 24 hours. However, archwires were clinically ligated only minutes after the brackets were bonded.²⁶ From Table 1, the initial tensile bond strength of Unite was significantly higher than those of Transbond, Enlight, and Self etch group. In addition, Unite was the self-cured adhesive that had higher levels of bond strength than many adhesives in previous studies.^{11,27,28} If the bond strength of different self-cured adhesives were compared, the results might be different.

Some studies suggested that the lower tensile bond strength obtained in the light-cured composite resin may be caused by incomplete polymerization.^{3,9,21,27,29} The current literature was inconsistent in terms of a setting time required before stressing a light-cured bond.⁶ In addition, Greenlaw et al. found that the bond strength of visible-light-cured adhesives was insufficient after initial curing.¹² Delaying archwire insertion for 24 hours was recommended by Bishara et al.² However, this recommendation may not be practical for routine clinical work. On the other hand, Read stated that all adhesive under the bracket were polymerized after initial exposure to visible light, so a force could be immediately placed on the bracket after curing.³⁰

The polymerization process of the self-cured adhesive resin occurs when primer and adhesive paste are in contact, whereas the polymerization process of the light-cured resin occurs when it is activated by a visible light curing unit. The light curing unit is usually directed to interproximal surfaces of the brackets but the light may not pass through stainless steel brackets, so it is possible that adhesive resin under the center of the brackets may not be exposed to the light which may lead to incomplete polymerization.^{31,32,33}

The bond strength of light-cured groups at 2 minutes after light activation increased significantly from initial polymerization and reached a level that was not significantly different from the initial bond strength of Unite. This result could be explained by the increasing polymerization reaction of light-cured adhesives indicating that the polymerization process (the chain reaction where methacrylate monomers are networked by splitting the double bonds) continues after the exposure

to light has ceased.³⁴ Composite resin polymerization occurs by the conversion of the monomer molecules into a polymer network, accompanied by a closer packing of the molecules. When more intense light energy is used to cure a resin composite, more photons reach the camphorquinone photoinitiator molecules within the resin and more photoinitiator molecules are activated and raised to the excited state. In this excited state, camphorquinone collides with amine. Then a free radical is formed, which can later react with carbon and form a carbon double bond (C=C) of a monomer molecule and initiate polymerization.³⁵ After initiated molecules are activated by light to form free radicals, initiation and propagation steps of chain growth will occur.^{32,33} These events may result in the increase of the bond strength after light-activation.

From Table 1, even though the initial tensile bond strength increased with time, the values were still less than the adequate bond strength at 5.9–7.8 MPa as recommended by Reynolds.²¹ However, these initial bond strengths are adequate to resist archwire seating force. A recent study by Iwasaki et al. attempted to quantify the archwire seating forces by a combination of direct measurement and mathematical calculation.¹⁹ The overall mean archwire seating force values for the tight stainless steel (SS) ligation samples and the loose SS ligation samples were 14.7 ± 9.2 and 6.2 ± 5.8 N, respectively. The mean archwire seating force was found to be 16.0 ± 1.7 N for the elastomeric modules.¹⁸ In this study, the initial tensile bond strengths of Unite, Enlight, Transbond and Transbond + Self Etching Primer groups were 4.34, 3.30, 3.04 and 3.29 MPa, respectively. Based on the equation^{15,36,37,38} that $1 \text{ MPa} = 1 \text{ N/mm}^2$, the bond strength over the bracket base area of 10.5 mm^2 for all these materials should be 45.57, 34.65, 31.92 and 34.55 N, respectively. These results suggest that the tensile bond strengths of every adhesive used in this study had fulfilled the requirement and were sufficient to withstand the calculated archwire seating forces from the initial setting time. Furthermore, at 24 hours all these adhesives had significantly increased tensile bond strength. The means of tensile bond strengths at this time were within the recommended range of 5.9–7.8 MPa as suggested by Reynolds.²¹

These findings indicate that, clinically, we could follow manufacturer instruction and wait at least 4 minutes after bonding before placing orthodontic arch wire when using Unite, as opposed to the use of Enlight, Transbond, and Transbond + Transbond Plus Self Etching Primer, which permits putting the orthodontic system into action at 2 minutes. Using light-cured adhesive thereby saves chairtime, particularly when bonding just a few brackets.

The Site of Bond Failure

At 24 hours the bond failure sites were more various than that of the initial setting time. This suggested that there was a weak bond between metal bracket and light-cured composite resin particularly from the initial time up to 4 minutes after light-activation. On the other hand, the failure sites of Unite self-cured adhesive were mostly at bracket/adhesive interface and within the adhesive at the initial time and 24 hours. The cohesive failure and failure at enamel/adhesive site were higher when measured at 24 hours than at initial time in every light-cured adhesive tested. This result may be due to the increasing polymerization process of the adhesive. Oesterle et al.⁶ suggested that stronger bonds may not only be caused by increasing polymerization of the overall adhesive, but due specifically to the mesh of the metal bracket. This resulted in a stronger mechanical interlocking of the bracket to the adhesive and, hence, increased fractures at other sites than at the bracket/adhesive interface.⁶ In addition, the difference in the site of bond failure between light-cured and self-cured adhesives may be due to the effect of resin consistency.¹¹ Both Enlight and Transbond light-cured adhesive are more viscous than Unite self-cured adhesive; thus, Unite flows more easily facilitating resin penetration into foil-mesh than both Transbond and Enlight.¹¹

The differences in the site of bond failure among different adhesives and different time intervals may be due to type and composition of each adhesive, resin consistency, polymerization reaction, and time of test.

Nonparametric statistical tests were used in this study as the results were non-homogeneous and not normally distributed according to the tests for normality (Kolmogorov-Smirnov). Further study testing *in vivo* or using more types of adhesives and brackets such as the new generation of orthodontic adhesives and other types of bracket base design is recommended.

Conclusion

1. A statistically significant difference was found in initial tensile bond strength between light-cured adhesives (within 1 minute) and self-cured adhesive (4 minutes). The initial tensile bond strengths of the light-cured groups were significantly lower than that of self-cured adhesive.

2. A statistically significant difference was found between initial tensile bond strength and bond strength at 2 minutes after light activation in all light-cured adhesives.

3. At 2 minutes, the tensile bond strengths of all light-cured

adhesives increased significantly and achieved values not significantly different from initial bond strength of self-cured adhesive. In addition, the tensile bond strength of light-cured adhesives at 2, 3, and 4 minutes were not significantly different.

4. The bond strengths of all adhesives did not reach maximum level at initial set, but increased subsequently. Increasing of the polymerization time after curing/bonding also increased the tensile bond strength in both light-cured and self-cured orthodontic adhesives. The tensile bond strength tested at 24 hours was significantly higher than at other time intervals and there were no significant differences between light-cured and self-cured adhesives.

5. The site of bond failure depended on the type of adhesive. The failure site of the light-cured adhesives mostly occurred at BA at the initial 1-4 minutes after light-activation with a greater variety of failure sites at 24 hours. The failure sites of Unite self-cured adhesive were mostly at BA and CO both at the initial time and at 24 hours.

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บทความวิชาการ

การศึกษาแรงยึดติดแรกเริ่มในวัสดุยึดทางทันตกรรมจัดฟันต่างชนิด

พาสันศิริ นิสาลักษณ์

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บทคัดย่อ

วัตถุประสงค์ของการศึกษาค้นคว้าครั้งนี้เพื่อศึกษาค่าแรงยึดติดแรกเริ่ม ในวัสดุยึดทางทันตกรรมจัดฟันที่แข็งตัวด้วยแสง 3 ชนิด ได้แก่ เอ็นไลต์ (Enlight) ทรานสบอนด์-เอ็กซ์ที (Transbond XT) และทรานสบอนด์เอ็กซ์ทีที่ร่วมกับทรานสบอนด์พลัสเซลฟ์เอ็ท-ซิงไพรเมอร์ (Transbond XT + Transbond Plus Self Etching Primer) โดยเปรียบเทียบกับวัสดุชนิดแข็งตัวด้วยตัวเอง ได้แก่ ยูไนต์ (Unite) กลุ่มตัวอย่างประกอบด้วยแบร็กเก็ตใหม่ 306 ชิ้น ซึ่งถูกนำมายึดติดกับฟันกรามน้อยที่ถูกถอนมาแล้ว ค่าแรงยึดติดของวัสดุชนิดแข็งตัวด้วยแสงทั้ง 3 ชนิด ถูกนำมาทดสอบที่ 5 ช่วงเวลา ได้แก่ หลังการฉายแสงทันที (แรงยึดติดแรกเริ่ม) 2 นาที 3 นาที 4 นาที และ 24 ชั่วโมง ในขณะที่ ยูไนต์ ซึ่งเป็นวัสดุชนิดแข็งตัวด้วยตัวเอง ที่นำมาเป็นกลุ่มเปรียบเทียบ จะทดสอบภายหลังทำการยึดติดแบร็กเก็ตแล้ว 4 นาที (แรงยึดติดแรกเริ่ม) และ 24 ชั่วโมง ผลจากการศึกษาพบว่าค่าแรงยึดติดแรกเริ่มในวัสดุยึดที่แข็งตัวด้วยแสงทั้ง 3 ชนิด ได้แก่ เอ็นไลต์ (3.30 ± 0.57 MPa) ทรานสบอนด์เอ็กซ์ที (3.04 ± 0.71 MPa) และทรานสบอนด์เอ็กซ์ทีที่ร่วมกับทรานสบอนด์พลัสเซลฟ์เอ็ทซิงไพรเมอร์ (3.29 ± 0.85 MPa) ต่ำกว่าอย่างมีนัยสำคัญทางสถิติ เมื่อเปรียบเทียบกับแรงยึดติดแรกเริ่มของวัสดุยึด ยูไนต์ (4.34 ± 1.36 MPa) ที่ ค่า $p < 0.05$ แต่อย่างไรก็ตามพบว่า ค่าแรงยึดติดที่เวลา 2 นาที หลังการฉายแสงของวัสดุชนิดแข็งตัวด้วยแสงทั้ง 3 กลุ่มนั้น มีค่าแรงยึดติดเพิ่มขึ้น เทียบเท่ากับแรงยึดติดแรกเริ่มของ ยูไนต์ และยังพบว่าค่าแรงยึดติดของวัสดุยึดชนิดแข็งตัวด้วยแสงที่เวลา 2 3 และ 4 นาที นั้นไม่มีความแตกต่างกันอย่างมีนัยสำคัญทางสถิติ ซึ่งผลจากการวิจัยครั้งนี้แนะนำว่า เมื่อใช้วัสดุยึดชนิดแข็งตัวด้วยแสง ควรรออย่างน้อย 2 นาที หลังฉายแสง เพื่อลดโอกาสในการหลุดของแบร็กเก็ต