

CHAPTER III

MATERIALS AND METHODS

Materials

Materials used in this investigation were as follows:

1.1 One hundred and twenty maxillary premolar teeth were extracted for orthodontic treatment purposes from patients with the age between 10 to 20 years and stored in distilled water. All teeth were free from buccal restoration, carious lesion, or abnormal labial surface anatomy that might effect the strength of the enamel. The teeth were used within 1 year after extraction. The sexes of the patients were not considered.

1.2 All metal brackets were 0.018" x 0.022" slot premolar standard edgewise brackets, Minidiamond type. In these foil/mesh backed brackets, there were two components: body and base. 17-4 Stainless steel bracket bodys were joined with 316 stainless steel foil/mesh bases by brazing with gold alloy. Total area of each bracket was 0.084 squre centimeters and foil mesh had 100 interlock holes per inch (Ormco, batch NO. 350-0506, Ormco Glendora, California).

1.3 Three types of composite resins were used

Type I Chemically cured composite resin which had one group of System 1+.

Type II Visible light cured composite resin which had one group of Transbond.

Type III Dual cured composite resins which had two groups, Sequence and Enlight.

Figure 5-8 showed the materials used in this experiment.



Figure 5 System 1+

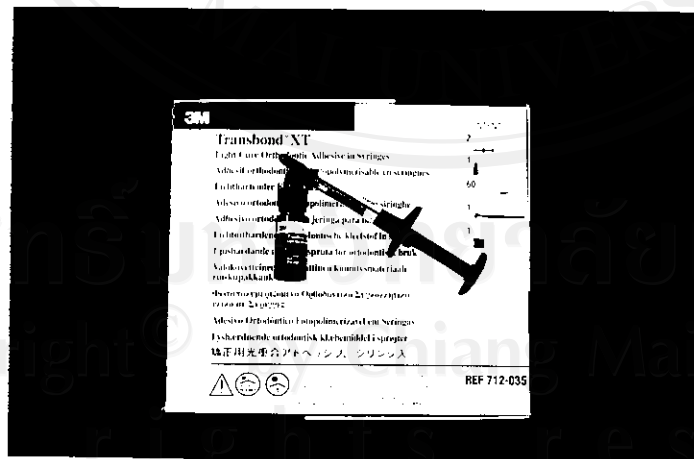


Figure 6 Transbond

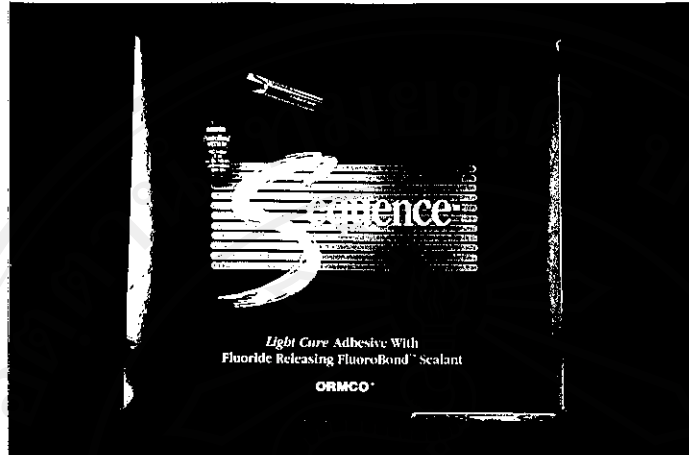


Figure 7 Sequence

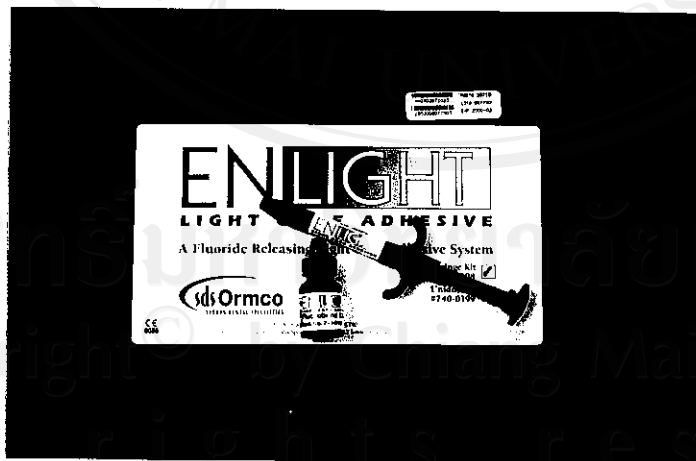


Figure 8 Enlight

Instruments

2.1 Instron® universal testing machine (Figure 9), Model 5565 with load cell five kilonewtons (Instron Corporation, England)



Figure 9 Instron® universal testing machine

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2.2 Debonding plate designed to fit under the bracket wing to ensure a vertical force application between the bracket base and the enamel surface (Figure 10).

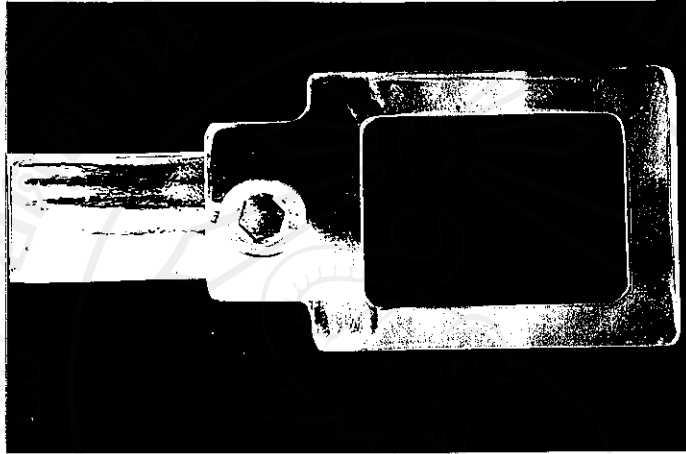


Figure 10 Debonding plate

2.3 Mounting jig designed to hold the tooth in vertical position with the bracket base parallel to the direction of force (Figure 11).

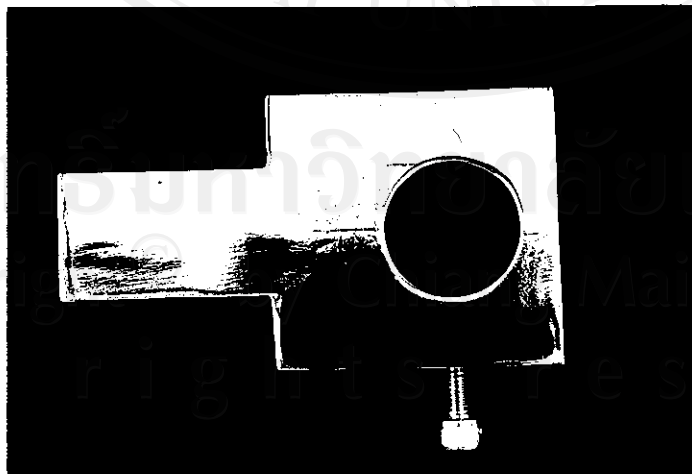


Figure 11 Mounting jig

2.4 Incubator (Mettmert model 200, Mettmert Corporation, Germany) was maintained at $37 \pm 1^\circ\text{C}$ (Figure 12).



Figure 12 Incubator

2.5 Nikon stereozoom microscope (SMZ-U zoom, Japan) used for examining and determining the resins on the debonded bracket bases and tooth surfaces (Figure 13).



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Figure 13 Nikon stereozoom microscope

2.6 One hundred-scale grid which was made from clear plastic had ten rows and ten columns. The grid area was equally to the debonded bracket base and tooth surface area except the cervical parts (Figure 14).

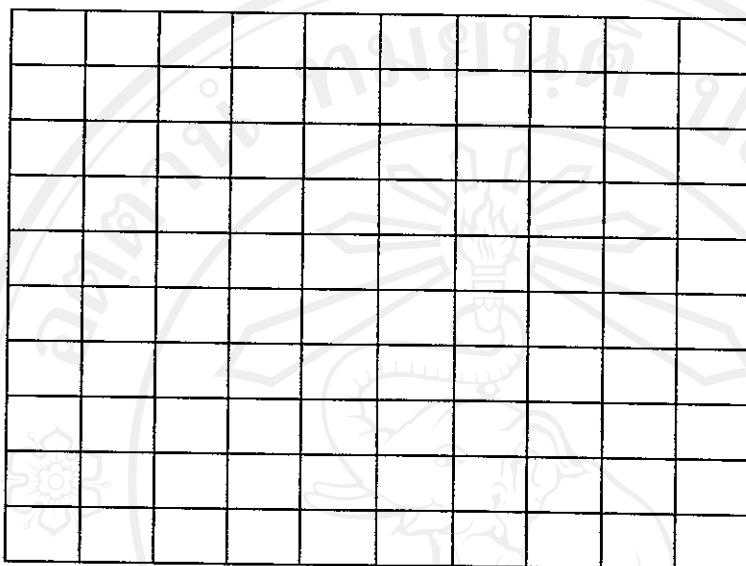


Figure 14 One hundred-scale grid

Methods

The 120 collected teeth were divided into 4 groups, 30 in each adhesive, to test the shear bond strength.

1 Tooth preparation for bond strength testing

All teeth were prepared for mounting by sectioning of the roots with tapered diamond bur. The buccal surfaces were cleaned with fluoride free pumice and water paste for 15 seconds, then rinsed with water, dried in a stream oil-free compressed air. Standardized area was obtained by masking off the enamel surface with transparent tape in which a hole, 5 millimeters in diameter covering bonding area, was punched. The center of the hole was far from the tip of buccal cusp 4-4.5 millimeters. Each buccal surface was etched with 37 percent phosphoric acid solution (Ormco, Ormco Glendora, California, Figure 15) for 60 seconds, rinsed for 20 seconds, and dried thoroughly.



Figure 15 Phosphoric acid solution

2 Bonding procedure for bond strength testing

Each tooth was bonded with the bracket position: the long axis of the bracket was the long axis of the tooth and the slot of the bracket was the center of the area.

After tooth preparation, 30 samples in each adhesive were carefully bonded with the following procedures:

Procedure 1 System 1+ (Ormco, Ormco Corporation, USA)

The liquid activator was painted on the etched enamel surface and the bracket base followed by a resin applied to the bracket base. The bracket with activator and adhesive was placed onto the activated tooth. After applying the bonding agents, the brackets were firmly pushed on the enamel surfaces with a finger tip pressure as equally as possible. Excessive resin was removed with an explorer.

Procedure 2 Transbond (3M Unitek, U.S.A.)

Light cure adhesive primer was applied onto each tooth surface and the bracket base followed by a resin applied to the bracket base. After applying the adhesive, the bracket was placed onto tooth surface. The bracket was

adjusted into the final position and then firmly pressed. The excess adhesive was removed. Finally, curing around the bracket base with visible light was completed by using a Visilux® II lamp for 20 seconds on mesial and distal sides.

Procedure 3 Sequence (Ormco, Ormco Corporation, USA)

Sequence consisted of two components, a resin sealant, FluoroBond with BF_3 and a 'no-mix' composite paste. It was marketed as having a 'dark cure' mechanism to enable complete polymerization below metal brackets. FluoroBond was applied to the etched enamel and the bracket base followed by a resin applied to the bracket base. The bracket was firmly placed on the tooth and the excess resin was removed. Finally, curing around the bracket base with visible light was completed by using a Visilux® II lamp for 20 seconds on mesial and distal sides.

Procedure 4 Enlight (Ormco, Ormco Corporation, USA)

Enlight was performed in a moist field due to its hydrophilic-like fluoroBond XM sealant and provided caries protection with its BF_3 fluoride release. Enlight contained a dark cure mechanism for continuing curing in the absence of light. FluoroBond was applied to the etched enamel and the bracket base followed by a resin applied to the bracket base. The bracket was firmly placed on the tooth and the excess resin was removed. Finally, curing around the bracket base with visible light was completed by using a Visilux® II lamp for 20 seconds on mesial and distal sides.

The composite resins were allowed to bench cure at least 10 minutes and then the adhesive tape was removed from each tooth.

3 Tooth embedding

On each tooth, a 0.018" x 0.022" stainless steel wire was ligated to the bracket by an elastic ligature for the body of the bracket perpendicular to the shear force.

The cylindrical polyvinylchloride ring of which diameter, height and thickness were 25 millimeters, 17 millimeters and 1 millimeter respectively was sealed at the base of the ring with green stone (Figure 16).

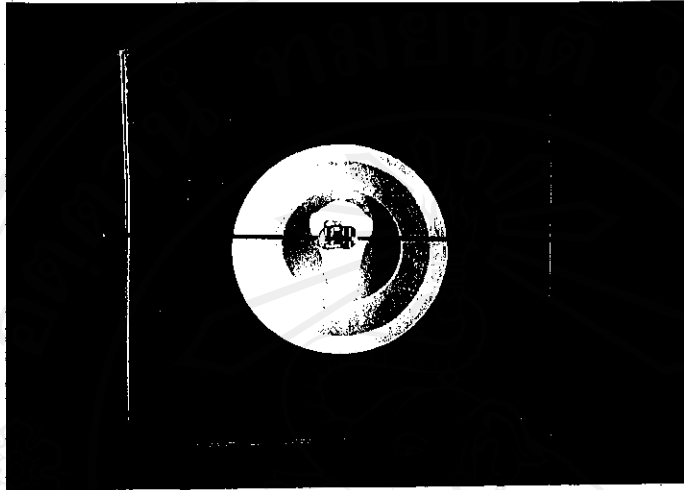


Figure 16 The wire attached to the tooth-bracket was placed above the ring by the plastic for embedding of the tooth.

Mixed acrylic-monomer was poured into the ring. The wire which was attached to the tooth-bracket was placed above the rim of the ring by the 2 millimeters thickness of plastic sheath and the tooth was embedded in self curing acrylic resin. The resin was left to harden. Only the buccal tooth surface and the attached orthodontic bracket were exposed. The elastic ligature, the wire and the plastic sheath were eliminated (Figure 17).



Figure 17 Tooth embedded in polyvinylchloride ring

The specimens were immersed in distilled water at 37°C for 24 hours in an incubator (Figure 12) to achieve maximum bond strength prior to testing.

4 Shear bond strength testing

Shear bond strength was determined by using an Instron® testing machine at 0.1 millimeter per minute in crosshead speed and five kilonewtons load cell. The ring was mounted into the jig which was fixed into the lower pneumatic grip. The debonding plate was fixed into the upper pneumatic grip of the machine. The Instron machine was activated, and the shear bond strength at bond failure was recorded (Figure 18, 19 and 20).



Figure 18 The debonding plate was fixed into the upper pneumatic grip and the mounting jig was fixed into the lower pneumatic grip.



Figure 19 An apparatus assembled for testing shear bond strength

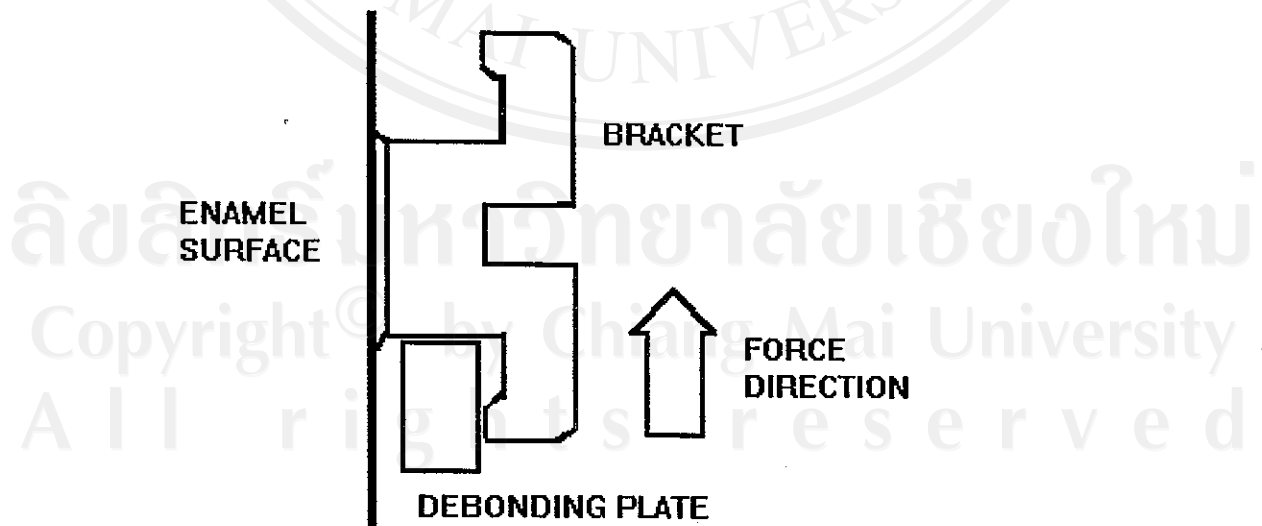


Figure 20 Schematic diagram of force direction for shear bond strength testing

5 Examining the bond failure

Each debonded bracket and tooth were immersed in 2 percent methylene blue for three days. They were washed with tap water and then examined under a Nikon stereozoom microscope. Photographs were taken to identify and categorize the modes of bond failure (Figure 21a and b). The calculation of percent resin was carried out by placing a one hundred-scale grid onto each photograph (Figure 22a and b). Each scale was subjectively observed having resin materials only if the resin occupied more than fifty percent of the area. These scales were summed up and calculated into percent resin. Remnants of composite resin that were observed on the mesh of the brackets had fractures at bracket-composite interface. The calculations of adhesive and cohesive failures were as follows:

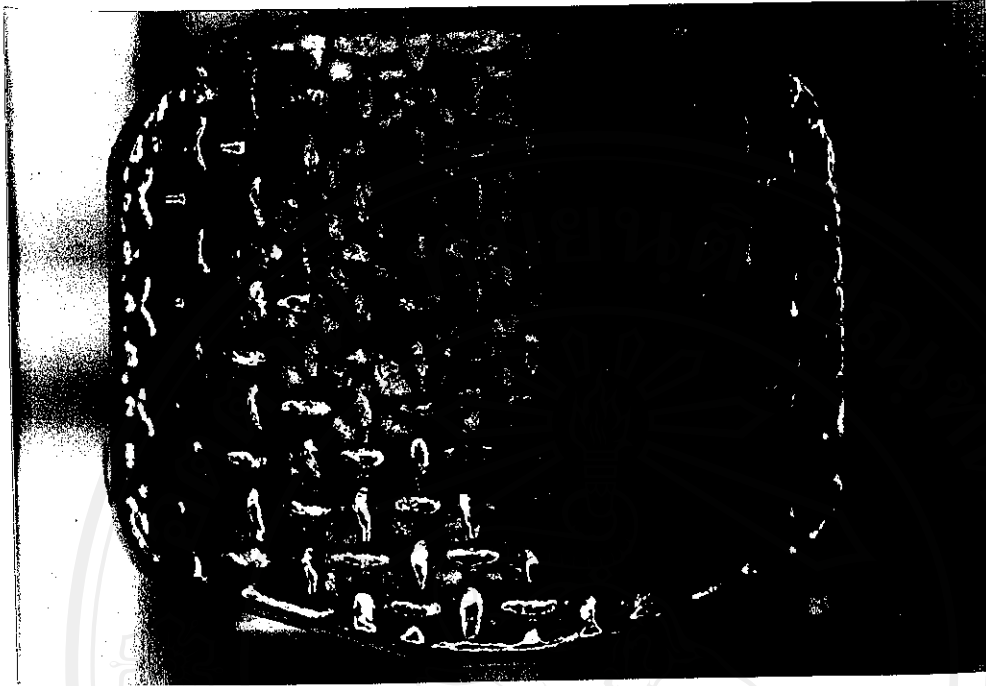
$$\% \text{cohesive failure} = \% \text{resin at B} + \% \text{resin at E} - 100\%$$

$$\% \text{adhesive failure at B} = \% \text{resin at E} - \% \text{cohesive failure}$$

$$\% \text{adhesive failure at E} = \% \text{resin at B} - \% \text{cohesive failure}$$

where B and E were bracket base and enamel surface respectively.

Figure 21 and 22 showed the photographs of sample number 25 of System 1+. The bracket base had 57 percent resin and the enamel surface had 60 percent resin. Then the cohesive failure was 17 percent, the adhesive failure at bracket-composite interface was 43 percent and at enamel-composite interface was 40 percent.

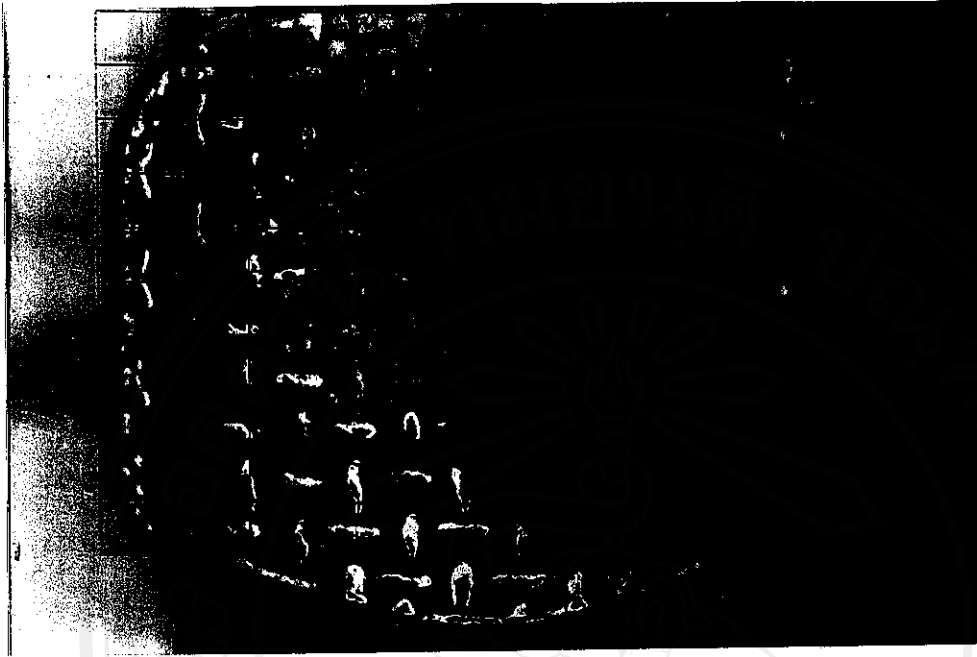


a) at bracket base



b) at enamel surface

Figure 21 Photographs of debonding failure



a) at bracket base



b) at enamel surface

Figure 22 One hundred-scale grid placed onto photographs

Statistical analysis

INDEPENDENT VARIABLE = The types of composite resins

DEPENDENT VARIABLE = The shear bond strength

1. The shear bond strengths of four composite resins were described by means, standard deviations, and range.
2. One way analysis of variance (ANOVA) was used for comparison of the mean values of shear bond strength of four composite resins.
3. The percentage of failure modes were described by means, standard deviations, and ranges in terms of adhesive and cohesive failure.

Statistical analysis was done by SPSS for Window Released 6.0 program.