



## 2. Brackets

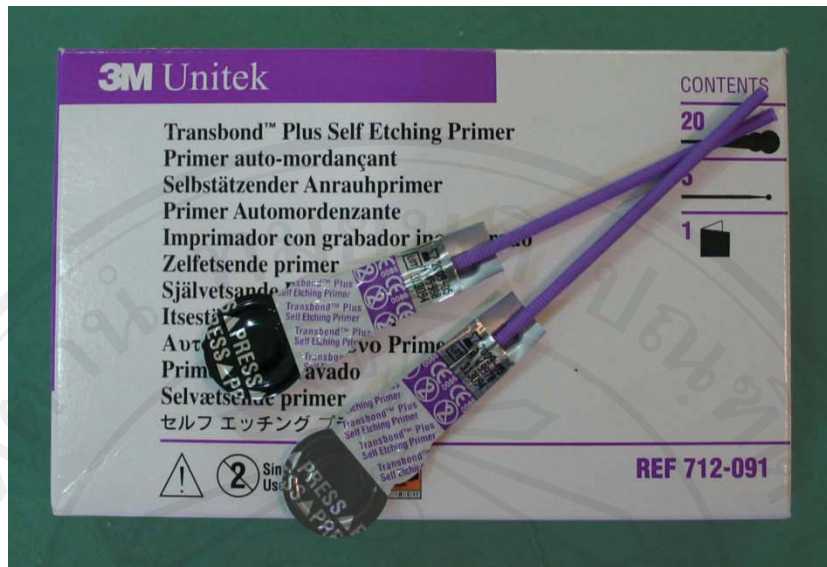
The brackets used had the following characteristics: a) pre-angulated 17-4 stainless steel bracket with a 0.022 x 0.028 inch slot, b) Roth prescription for upper first and second premolar teeth, and c) pre-coated with Transbond XT adhesives (APC™ II Gemini Twin brackets, batch No. 3119-735, 3M Unitek, Monrovia, California, USA). Each bracket base incorporates a mesh. The area of each bracket base was 10.61 square millimeters<sup>31, 32</sup> (Figure 5: a, b and c).



**Figures 5 a.** Adhesive coated premolar bracket, **b.** Base of bracket, **c.** Bracket base pre-coated with adhesive

## 3. Primers

Primers used for this investigation were one-step self-etching primer (Transbond™ Plus Self Etching Primer, 3M Unitek). The primer is an etchant. It is also a primer which is combined into one product in a three-well single-patient-use foil pack (Figure 6).



**Figure 6** Transbond™ Plus Self Etching Primer

#### 4. Distilled water

Distilled water was de-ionized and prepared by the Dental Materials Laboratory Unit, Faculty of Dentistry, Chiang Mai University (Figure 7).



**Figure 7** Distilled water

## 5. Thymol solution

A 0.1% (weight/volume) thymol solution was prepared by dissolving 10 g of thymol in 100 ml of de-ionized water.

## II. INSTRUMENTS

### 1. The high-power light-emitting diode curing unit

A curing unit (Mini LED™, Satelec® Acteon Group, Merignac, France) provided light intensity at 1,250 Mw/cm<sup>2</sup> (Figure 8).



**Figure 8** Mini LED™ light-curing unit

### 2. Conventional halogen lamp

A curing unit (Spectrum 800, Dentsply/Caulk, Milford, Delaware, USA) was set to provide light intensity at 300 Mw/cm<sup>2</sup> (Figure 9).



**Figure 9** Spectrum 800 conventional halogen curing unit

### 3. Incubator

The temperature of this incubator (Model 200, Memmert Corporation, Schwabach, Germany) was maintained at  $37 \pm 1^\circ \text{C}$  at the time of the experiment (Figure 10).



**Figure 10** Incubator

#### 4. Thermocycling machine

A thermocycling machine consisted of two water baths, cold and hot, (Model TC 301 with baths of cold and hot water, models CWB332R and HWB332R respectively, Medical and Environment Equipment Research Laboratory, King Mongkut's Institute Of Technology Ladkrabang, Bangkok, Thailand). The temperature for cold and hot water was set at 5 ° and 55 ° C, respectively, at the time of the experiment (Figure 11).



**Figure 11** Thermocycling machine

#### 5. Instron® universal testing machine

The machine (Model number 5566, Instron Calibration Laboratory, Norwood, Massachusetts, USA) was used with a load cell of 1 kilo-Newton, and the data was analyzed with Bluehill software, CAT No. 2603-080 (Bluehills Software Company, Whitstable, Kent, England) (Figure 12: a and b).



a.



b.

**Figure 12 a.** Instron<sup>®</sup> universal testing machine, **b.** One kilo-Newton load cell

## 6. De-bonding plate

This instrument was designed to fit under the bracket wing to ensure a vertical force application between the bracket base and the enamel surface (Figure 13).



**Figure 13** De-bonding plate

## 7. Mounting jig

This instrument was designed to hold the tooth in position in an acrylic block with the bracket base parallel to the direction of force (Figure 14).



**Figure 14** Mounting jig



### III. METHODS

The experiments were divided into three parts.

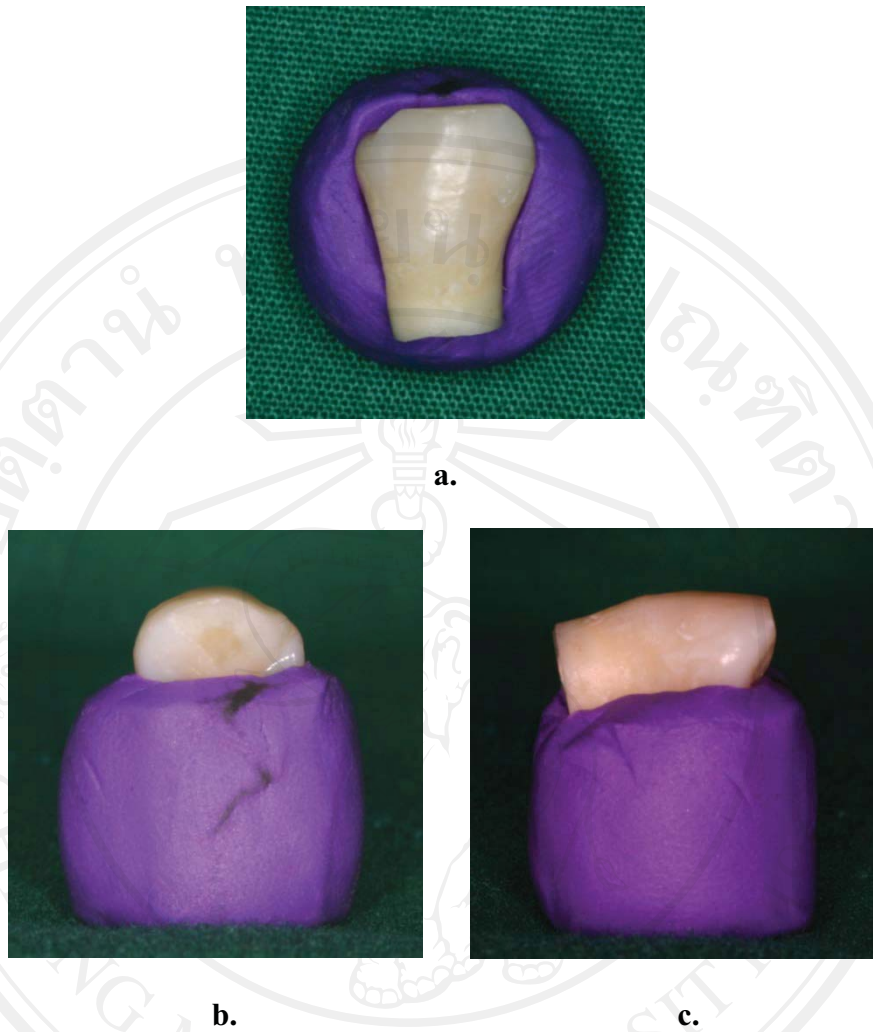
1. Shear bond strength testing
2. Adhesive remnant evaluation
3. Statistical analysis

#### 1. Shear bond strength testing

All teeth were prepared by sectioning with carborundum discs 2 to 3 mm apical to the cemento-enamel junction (Figure 15). The buccal surface of each crown was lightly polished with fluoride-free pumice and a rubber cup for 10 seconds, and washed with tap water. The excess water was removed from the surface with an oil-free compressed air stream, but the surface was not allowed to dry completely. Samples were then carefully placed in clay blocks as a stabilizer during the bonding process. The lingual halves of the samples were embedded in clay blocks. The Long axes of the samples were laid as parallel as possible to the base of the block (Figure 16: a, b and c).

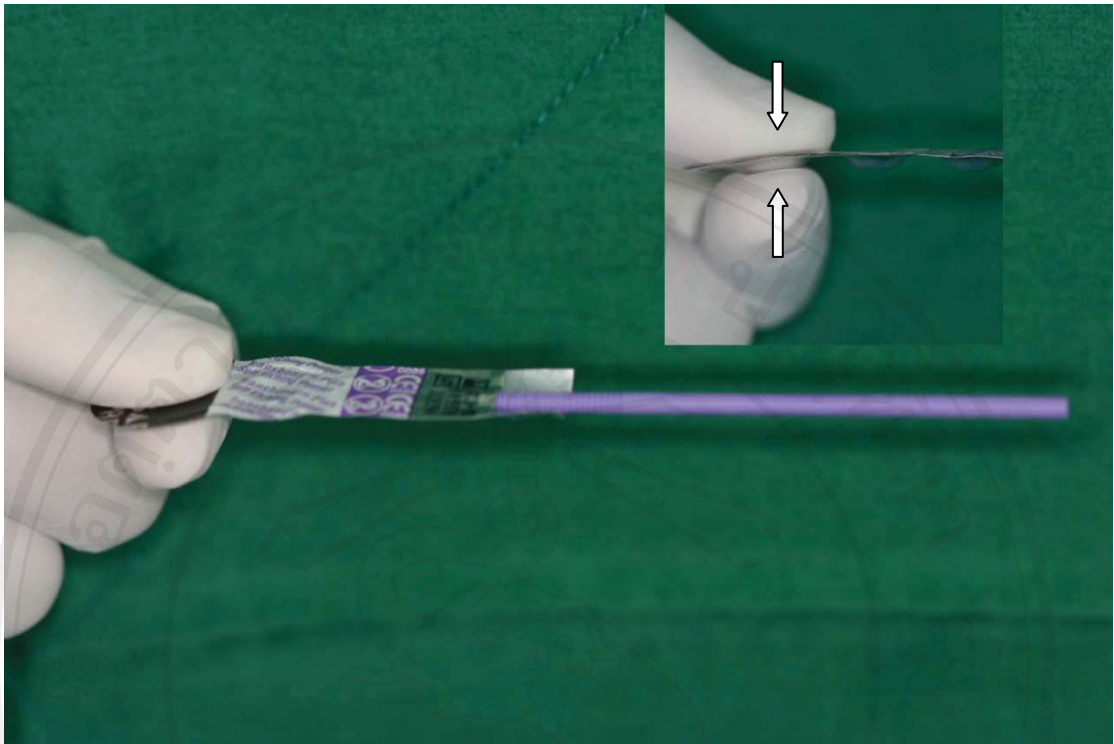


**Figure 15** Each tooth was sectioned to separate the root from the crown

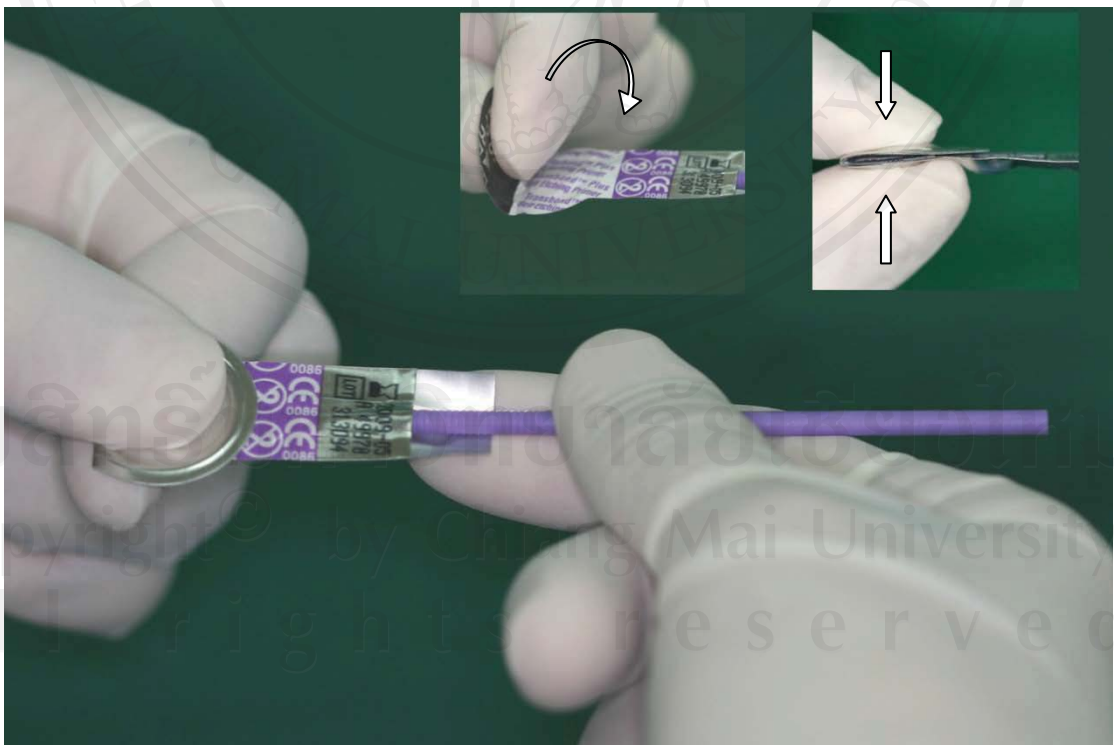


**Figure 16** Tooth sample embedded in clay block

All samples were bonded with adhesive pre-coated stainless steel premolar brackets and Transbond™ Plus Self Etching Primer (3M Unitek). For the one-step light-cured adhesives with self-etching primers, the liquid in the first primer reservoir of the package was completely squeezed into the second reservoir, toward the applicator, as recommended by the manufacturer (Figure 17). After the first reservoir was emptied, it was folded at the folding line between the first and second reservoir (Figure 18). Again, the mixed liquid in the second reservoir was squeezed into the last (smallest) reservoir of the package.

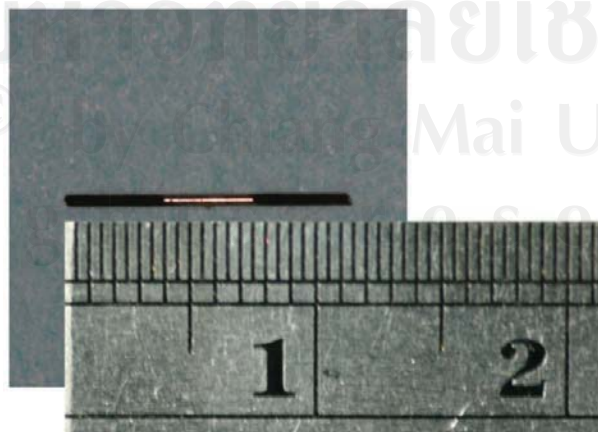


**Figure 17** Pressing the first reservoir of the adhesive package

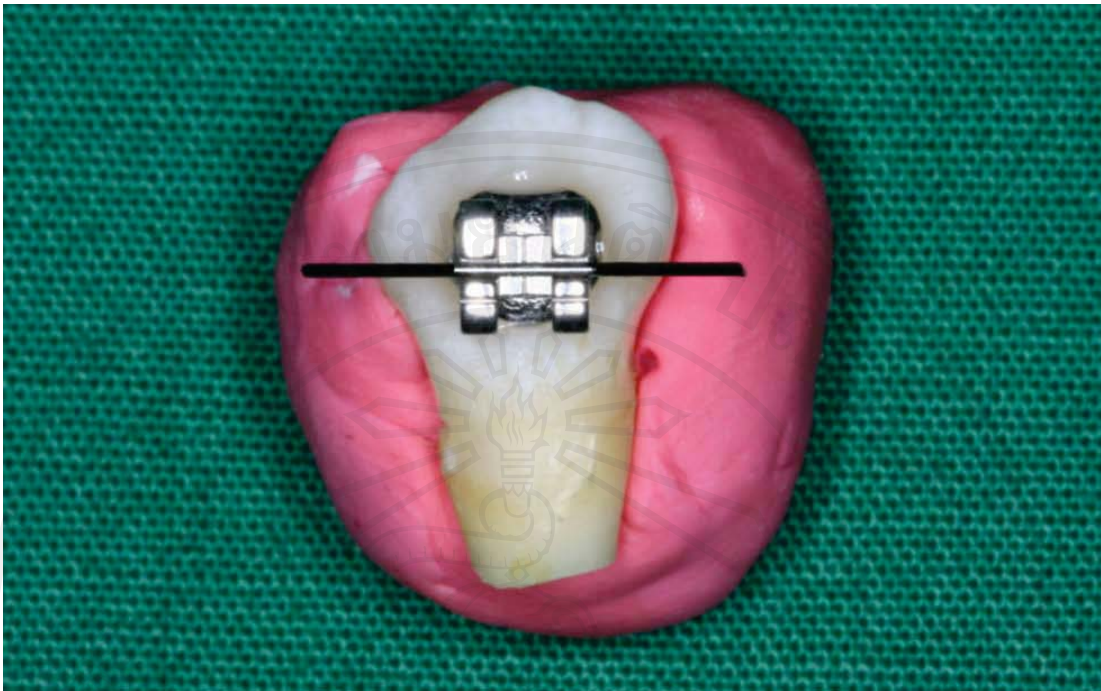


**Figure 18** Folding the first reservoir, and pressing the first and second reservoirs

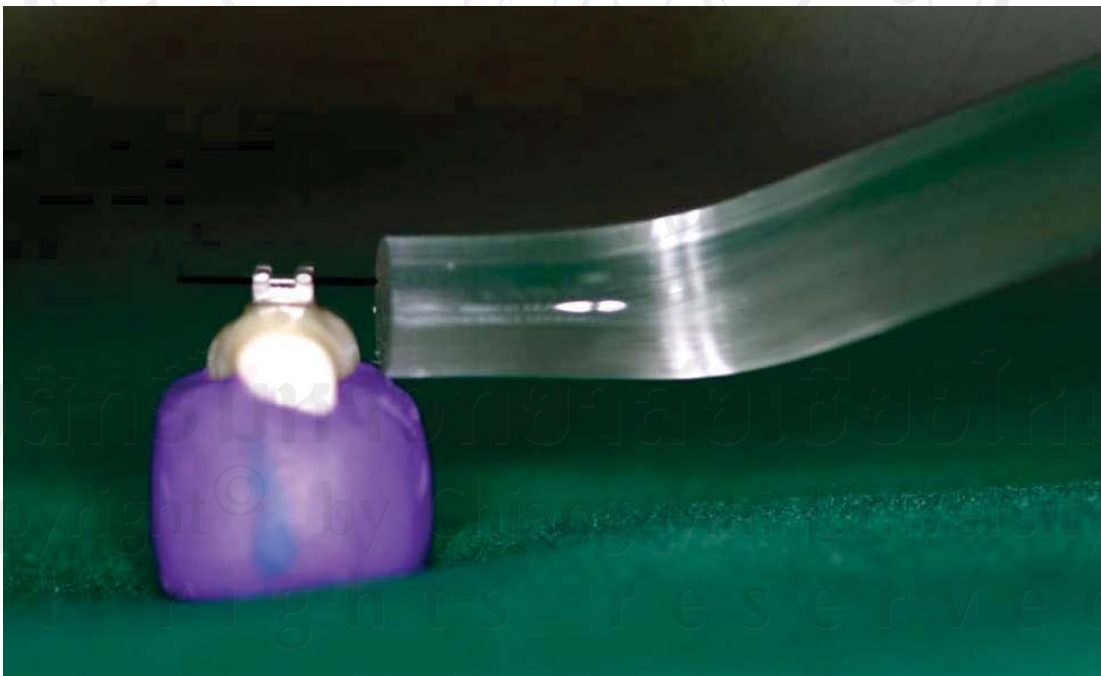
After the liquid was transferred into the last reservoir, the applicator was churned and swirled inside the last reservoir for 5 seconds to completely mix the chemicals and coat the tip of the applicator. After finishing the mixing, the applicator was removed from the reservoir. The buccal surface of the first sample was rubbed with the primer-saturated tip of the applicator, while applying some pressure for a minimum of 3 to 5 seconds per surface. Before applying the primer to the each of the other samples, the applicator was re-dipped into the reservoir to saturate the tip. After each tooth was primed, the primer was dried into a thin film with an oil- and moisture-free air source to deliver a gentle air burst for 1 to 2 seconds to each surface. A bracket was firmly placed on the middle of the buccal surface of each tooth. Any excess adhesive was removed with an orthodontic sickle. The space between the bracket and the light guide-tip was determined by placing a 0.020 inch stainless steel wire in the bracket slot before light curing so that 4 mm protruded beyond the bracket on both ends. The steel wire protrusions were marked with black ink for ease of illustration (Figures 19 and 20). The light tip was placed at the end of the wire, away from the mesial and distal edges of the bracket base and with its face parallel to the lateral edge of the bracket (Figure 21).



**Figure 19** Stainless steel wire with 4 mm protrusions marked in black ink



**Figure 20** Marked stainless steel wire in bracket slot



**Figure 21** Placing light guide-tip using marked wire

Prior to each activation, the light intensity was checked using the built-in radiometer. The Mini LED™ curing unit was set in the fast mode, which emitted constant light intensity at 1,250 Mw/cm<sup>2</sup>. The light guide-tip was placed in the aperture located on the front part of the base (Figure 22). The indicator light, located at the front of the base, would show green or red light for evaluating the performance of this device. The indicator light must show green before activating the adhesives, to ensure that the correct light intensity was achieved. A light intensity of 300 Mw/cm<sup>2</sup> was chosen for the Spectrum 800 curing unit to activate the adhesive in this study. The light guide-tip was placed on the radiometer's aperture at the base. The light was adjusted until it achieved the desired intensity, which was shown on the hand-piece screen (Figure 23 a and b).



**Figure 22** Checking light intensity for Mini L.E.D™ light curing unit



**Figure 23** a. Checking light intensity for Spectrum 800 halogen curing unit, b. The screen showed intensity of the light

All samples were light-cured equally on both mesial and distal surfaces of the tooth for the times described in Table 2.

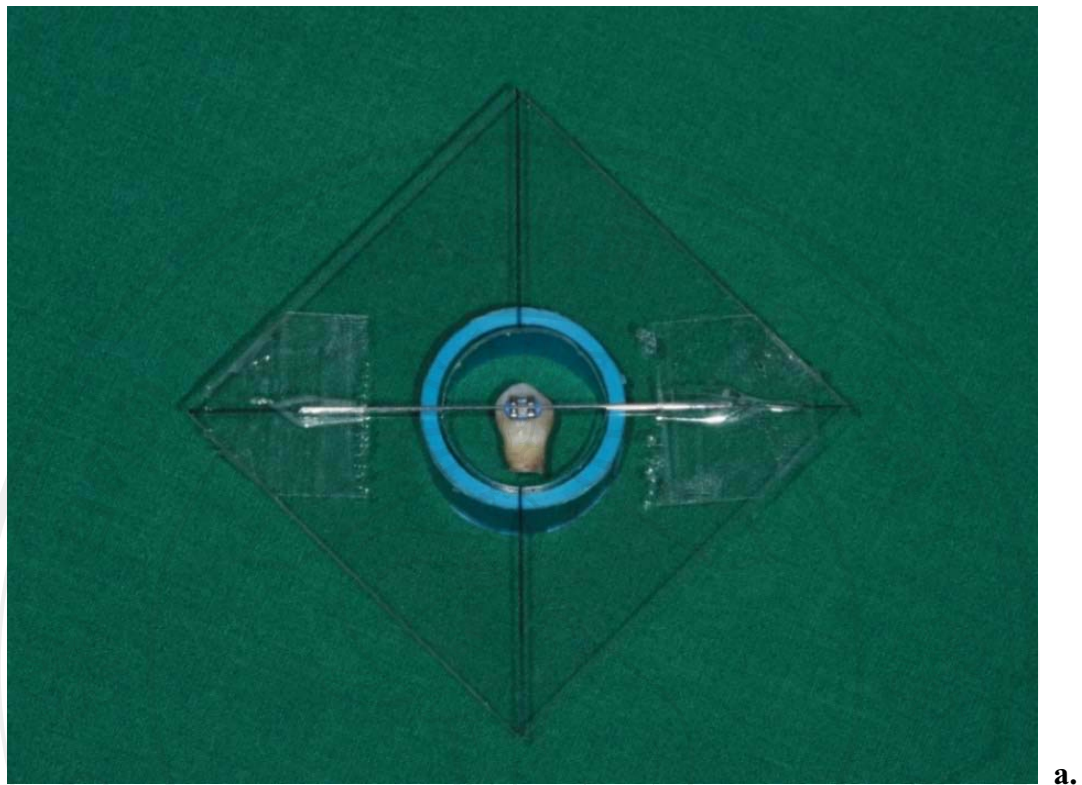
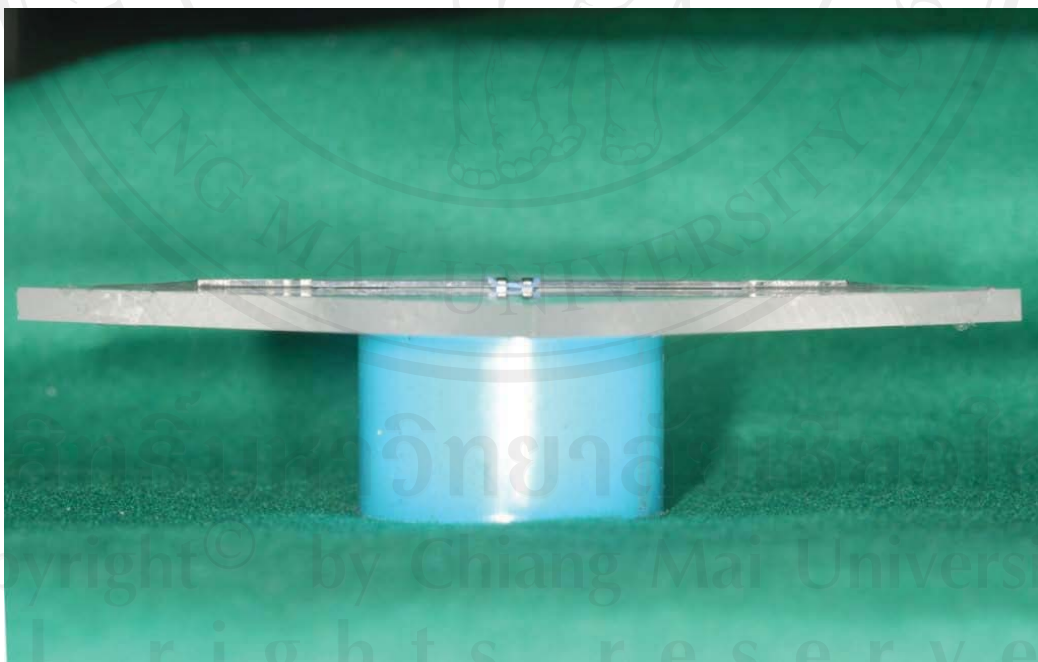
**Table 2** Curing times for each group

Group		LED ( $I = 1,250 \text{ Mw/cm}^2$ )						Halogen ( $I = 300 \text{ Mw/cm}^2$ )
		1	2	3	4	5	6	7 (control)
Curing time	seconds/tooth	2	4	6	8	10	12	40
	seconds/surface	1	2	3	4	5	6	20

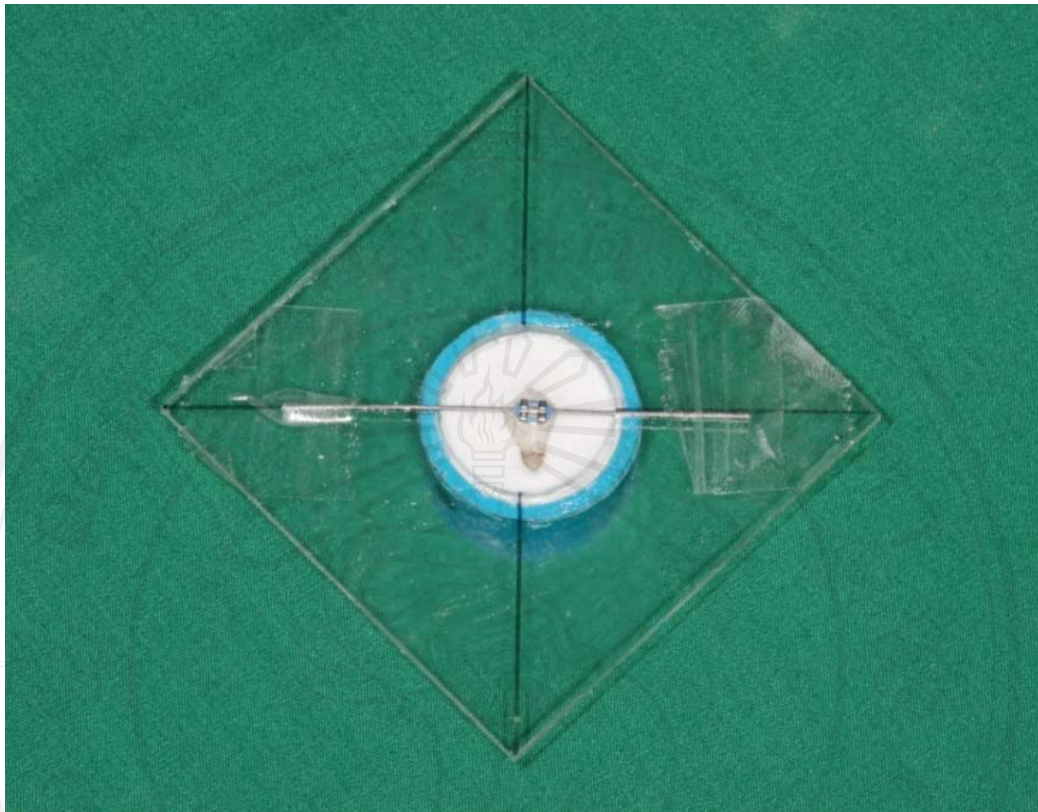
After the bonding process, all samples were prepared for embedding in blocks, made from cylindrical polyvinylchloride rings. Each ring was 25 mm in diameter, 17-mm high, and 1-mm thick. The clay block was removed from the teeth. A 0.016 x 0.022 inch straight stainless steel wire was placed in each bracket and tied with an elastomeric ligature. The 0.022 inch side of the wire was fully seated in the bracket slot in order to control the angulation of the mounted tooth in the polyvinylchloride ring and the direction of the force applied to each sample.

Each sample with a wire was placed in an aperture in the center of a flat 2-mm thick plastic plate. The 0.022 inch side of the wire was placed on and parallel to the surface of the plate. The tooth was hung in the middle of the aperture. The wire was fixed with adhesive tape to control the angulation of the wire on the plate. The whole assembly of the tooth with wire and plastic plate was placed on top of a polyvinylchloride ring (Figure 24 a and b). The space in the polyvinylchloride ring was filled with self-cured acrylic resin so that the lingual part of the tooth was embedded in the center of the ring. Only the surface of the tooth-bracket assembly was exposed (Figure 25). All samples were left until complete curing of the acrylic resin was achieved. The elastomeric ligatures, wires and plastic plates were then removed. All samples were incubated in distilled water at 37° C for 24 hours in the incubator. A thermocycling procedure was performed in water baths at 5° and 55° C for 30 seconds per bath using the thermocycling machine and with a transfer time of 10 seconds, for 1,000 cycles.



**a.****b.**

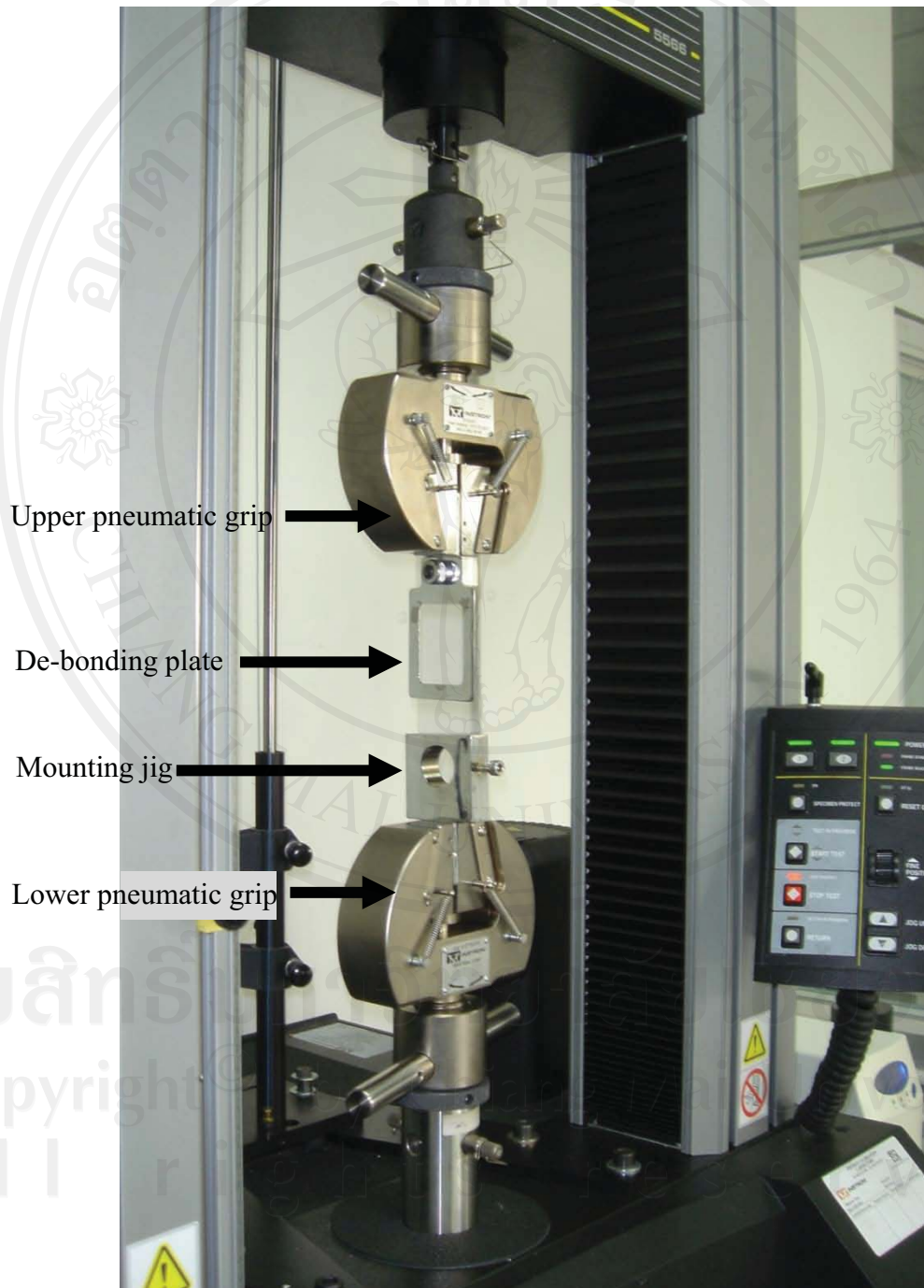
**Figure 24** Sample held in a polyvinylchloride ring with a 0.016 x 0.022 inch stainless steel wire, tied with an elastomeric ligature on a 2-mm plastic plate and fixed with adhesive tape; **a.** top view, **b.** side view



**Figure 25** Tooth embedded in polyvinylchloride ring with self-cured acrylic resin

Brackets were de-bonded after thermocycling was completed, using a universal testing machine (Figure 26). The de-bonding plate was fixed into the upper pneumatic grip, while the mounting jig was attached to the lower pneumatic grip. The polyvinylchloride ring was mounted to the mounting jig (Figure 27). The de-bonding plate was vertically adjusted and fully engaged at the bottom of the bracket wing at the time of testing (Figure 28). Brackets were de-bonded from the tooth surfaces by the Instron<sup>®</sup> testing machine at a cross head speed of 0.5 mm per minute and a load cell of 1 kilo-Newton. The force direction was occluso-gingival, parallel to the buccal tooth surface (Figure 29). Force was applied until the bracket was dislodged from the tooth surface. The force values in Newtons were divided by the area of the bracket

base, which was 10.61 square mm. The bond strength, interpreted as the force required to removing the brackets, was recorded in Mega Pascals (MPa). All bonding and de-bonding procedures were carried out by one operator.



**Figure 26** Composition and position of Instron<sup>®</sup> universal testing machine during shear bond strength testing



**Figure 27** Polyvinylchloride ring mounted into mounting jig attached to the lower pneumatic grip with de-bonding plate attached to the upper pneumatic grip



**Figure 28** Position of de-bonding plate and tooth-bracket assembly in acrylic block ready for shear bond strength testing

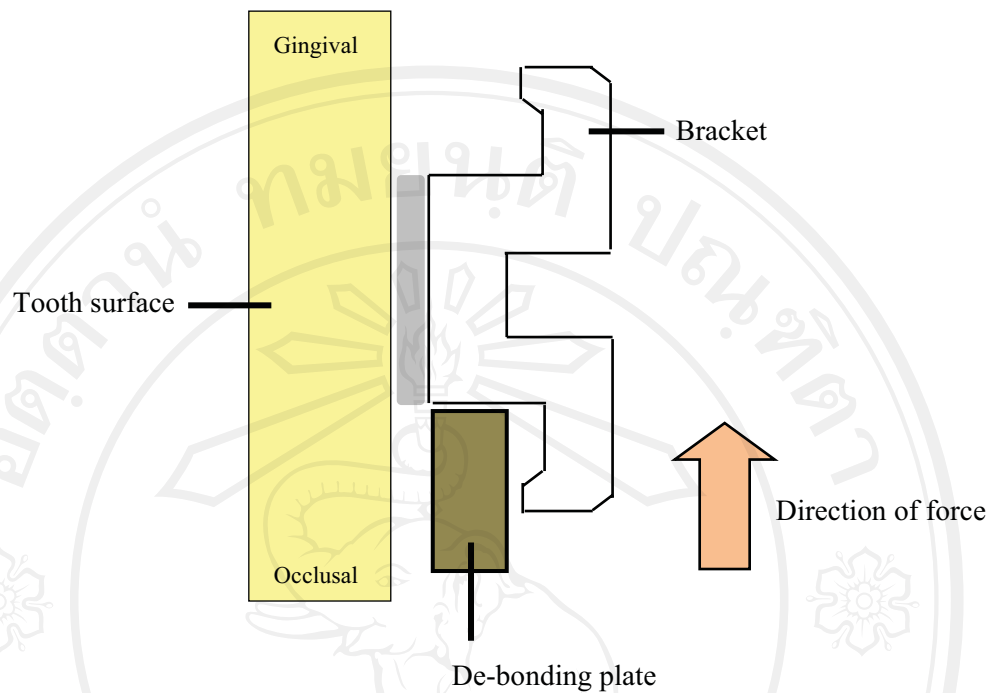


Figure 29 Schematic diagram of force direction for shear bond strength testing

## 2. Adhesive remnant evaluation

Adhesive remnants on enamel surfaces were evaluated. The remnant on the bracket base was measured, and was then converted to the amount of remnant left on the enamel surface. A digital single-lens reflex camera (Canon 300D, Canon Incorporated, Tokyo, Japan) and a Canon macro lens (Canon EF 100 mm f/2.8 MACRO USM) at 1 x magnification was used to make photographs of the adhesive remnant which was left on each bracket base. The amount of adhesive left on the bracket base was measured by superimposing a photograph of the bracket base on a computer-generated grid (Figure 30) using Adobe Photoshop CS2 version 9.0 software (Adobe Systems Incorporated, San Jose, California, USA). The area of the adhesive left on the bracket base and the area free of adhesive were calculated. The

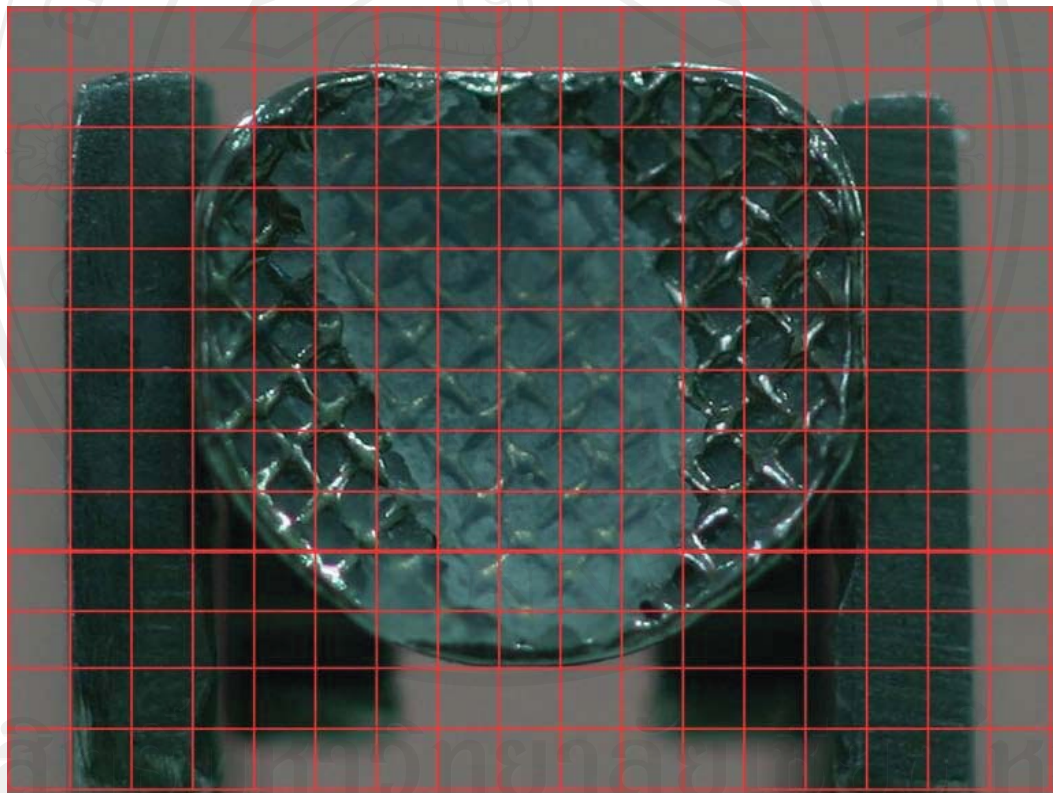
ratio of adhesive left on the bracket base was then classified as the Adhesive Remnant Index Score, (ARI score)<sup>33</sup> as follows:

‘0’ = No adhesive left on the tooth

‘1’ = Less than half of the adhesive left on the tooth

‘2’ = More than half of the adhesive left on the tooth

‘3’ = All the adhesive left on the tooth, with distinct impression of the bracket mesh



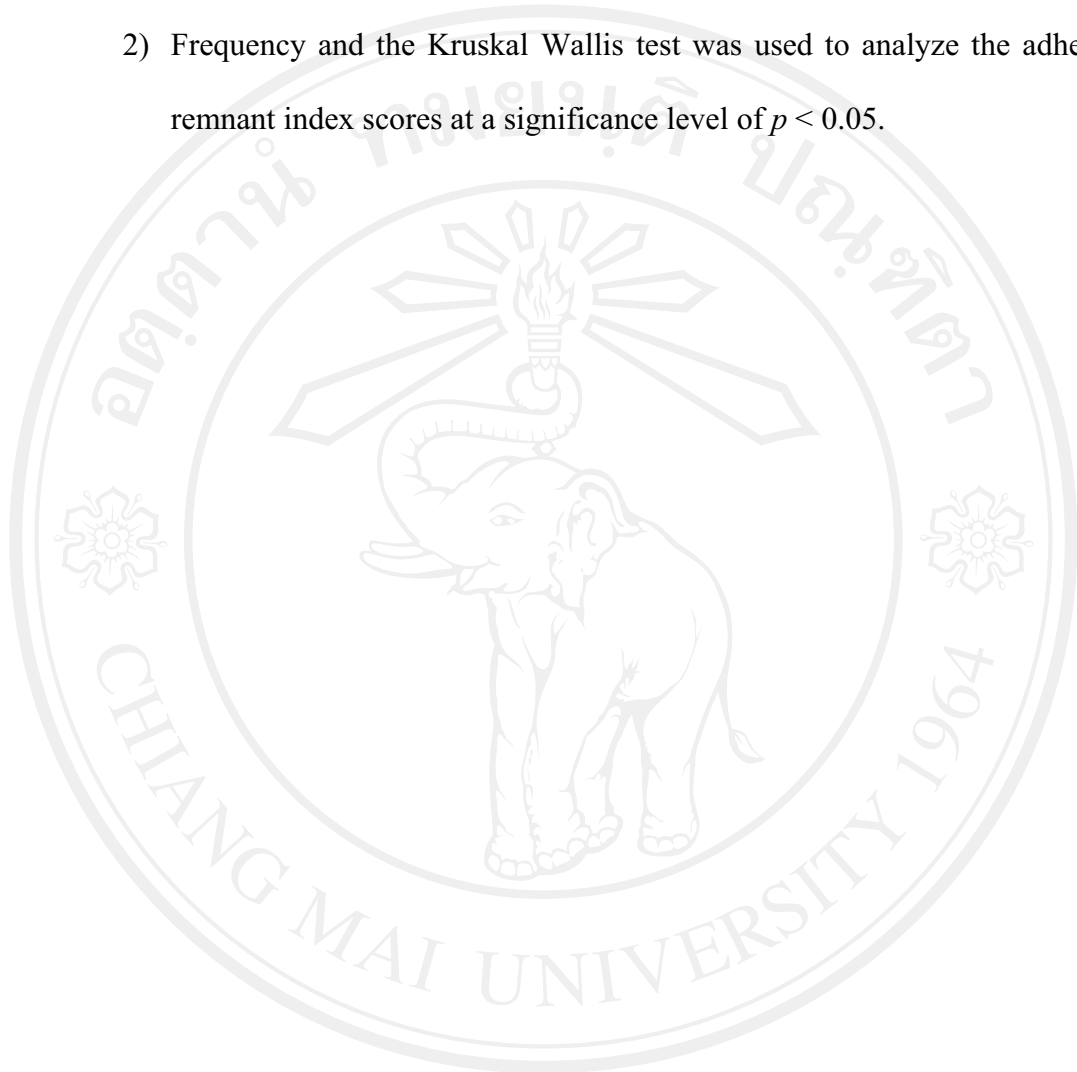
**Figure 30** Superimposition of the bracket base on a grid

### 3. Statistical analysis

- 1) Analysis of Variance (one-way ANOVA) was used to compare the means of shear bond strength in each group, followed by a multiple comparisons (Tukey's) test. If one-way analysis of variance showed significant

difference among groups at  $p < 0.05$ , Tukey's test was used to analyze the differences at the same significance level.

- 2) Frequency and the Kruskal Wallis test was used to analyze the adhesive remnant index scores at a significance level of  $p < 0.05$ .



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