CHAPTER III

MATERIALS AND METHODS

3.1 Materials

The materials used in this study were as follows:

1. One hundred and twenty maxillary premolar teeth (Figure 9) were extracted for orthodontic treatment purposes. Selection criteria were as follows: intact surface, free from caries, no dental fluorosis, no restorations or cracks on the labial surface, and no contamination by any chemical agents that effect the strength of the enamel.

Figure 9 An extracted maxillary premolar tooth

2. Sixty uncoated upper premolar ceramic brackets (Clarity™, 3M Unitek, Monrovia, California, USA) (Figure 10) and sixty precoated upper premolar ceramic brackets (APC Plus Clarity™ 3M Unitek,) were used in the study (Figure 11). The average surface area of the Clarity™ and APC Plus Clarity™ bracket bases was 10.59 square millimeters.[79]

Figure 10 Uncoated ceramic bracket

Figure 11 Precoated ceramic bracket

3. Three types of adhesives systems were used with Transbond XT™ composite.

   3.1 Total-etching adhesive system (37%phosphoric acid, Ormco, Glendora, California, USA (Figure 12) and Transbond XT™, 3M Unitek) (Figure 13)

Figure 12 37%phosphoric acid

Figure 13 Transbond XT™

3.2 Two-step self-etching adhesive system (Clearfil SE bond, Kuraray Dental, Osaka, Japan) (Figure 14)

Figure 14 Clearfil SE bond

3.3 One-step self-etching adhesive system (Clearfil S^3 bond, Kuraray Dental) (Figure 15)

Figure 15 Clearfil S^3 bond

4. Self-curing acrylic resin (Figure 16)

Figure 16 Self-curing acrylic resin

5. Elastomeric ligatures and rectangular stainless steel wire (Figure 17)

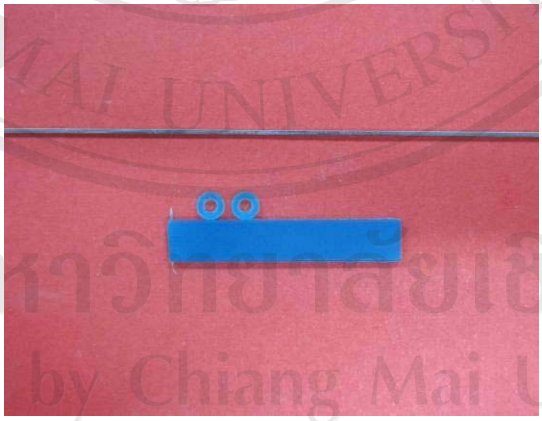

Figure 17 Elastomeric ligatures and rectangular stainless steel wire

6. 50 x 50 x 1.5 mm plastic sheath with a center hole with diameter size of 15 mm (Figure 18)

Figure 18 Plastic sheath

7. Stainless steel ring with diameter, height and thickness of 19, 10 and 1.3 mm respectively (Figure 19)

Figure 19 Stainless steel ring

3.2 Instruments

1. Mini LED™ (1250 mW/cm^2) (Satelec® Acteon, France) (Figure 20)

Figure 20 Mini LED™

2. A Memmert Model 200 incubator (Memmert GmbH + Co. KG, Schwabach, Germany) (Figure 21)

Figure 21 Incubator

3. Model HWB332R, TC301, CWB332R Thermocycling device (Medical & Environmental Equipment Research Laboratory King Mongkut's Institute of Technology Ladkrabang, Thailand) (Figure 22)

Figure 22 Thermocycling equipment

4. Instron® 5566 universal testing machine with load cell of 500 newtons (Instron Calibration Laboratory, Norwood, Massachusetts, USA) (Figure 23)

Figure 23 Instron® universal testing machine

5. Digital single-lens reflex camera (Canon kiss X, Japan) (Figure 24)

Figure 24 Digital single-lens reflex camera

6. Computer-generated transparent grid (Figure 25)

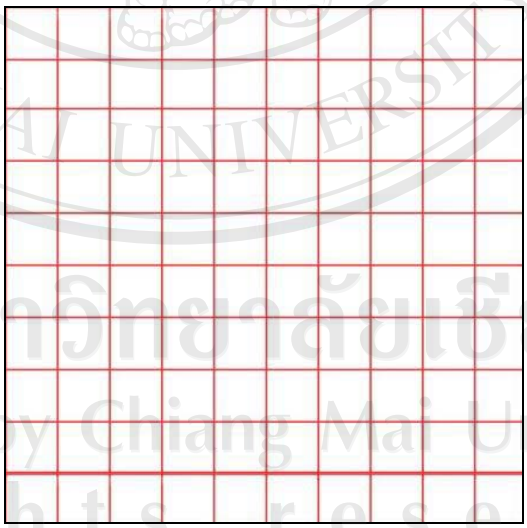

Figure 25 Computer-generated transparent grid

3.3 Methods

1. One hundred and twenty maxillary premolar teeth were collected, cleaned and stored in 0.1% thymol solution (dissolved 0.1 g of thymol in 100 ml deionized water) at room temperature (25˚C). The teeth were used within 6 months after extraction.

2. All teeth were randomly separated into 6 groups (Table 3).

3. All teeth were prepared for mounting by sectioning of the teeth about 2 millimeters below cementoenamel junction by using carborundum disc (Figure 26).

Figure 26 Sectioning of the tooth by using carborundum disc.

4. The buccal surface of each tooth was cleaned and polished with fluoride-free pumice using a low-speed handpiece for 10 seconds, rinsed with water for 10 seconds, and dried with oil-free air for 10 seconds.

Table 3 The teeth were divided into six groups according to the types of adhesives and brackets to be used.

Group	Adhesive	Paste	Bracket	N
1 Control	37%phosphoric acid and Transbond XT™ primer	Transbond XT™	Uncoated ceramic bracket (Clarity™)	20
2	37%phosphoric acid and Transbond XT™ primer	-	Precoated ceramic bracket (APC Plus Clarity™)	20
3	Clearfil SE bond	Transbond XT™	Uncoated ceramic bracket (Clarity™)	20
4	Clearfil SE bond	-	Precoated ceramic bracket (APC Plus Clarity™)	20
5	Clearfil S^3 bond	Transbond XT™	Uncoated ceramic bracket (Clarity™)	20
6	Clearfil S^3 bond	-	Precoated ceramic bracket (APC Plus Clarity™)	20

5. Six bonding procedures were used, one for each group. All brackets were bonded to the buccal surfaces of maxillary premolar teeth. The vertical axis of the bracket was parallel to long axis of the teeth, and the slot of bracket was at the center of the long axis of the clinical crown. Brackets in all groups were bonded as recommended by the manufacturer's directions.

Group 1: In the first step, buccal tooth surfaces were etched with 37% phosphoric acid solution for 30 seconds, rinsed with water for 15 seconds, and dried with light oil-free compressed air until a frosted enamel appearance was achieved. In the second step, Transbond XT™ primer was applied to the etched surface with a micro-brush and cured for 10 seconds. In the third step, Transbond XT™ paste was applied on the Clarity™ bracket base. The brackets were placed with firm pressure. The excess resin composite was removed with an explorer. In the fourth step, the mini LED™ was used to cure the adhesive at the midbracket position for 5 seconds. The distance between the light tip of the mini LED™ and the midbracket area was 2 mm.

Group 2: First, buccal tooth surfaces were etched, and second, primer was applied as described for Group1. Third, APC Plus Clarity™ brackets were placed. The excess resin composite was removed. Fourth, the resin paste was cured as described for Group 1.

Group 3: Buccal tooth surfaces were first applied with Clearfil SE primer, which includes etchant and primer together by agitation with a micro-brush for 5 seconds, and lightly dried by using oil-free compressed air. Then, bonding agent was applied, dried gently and light-cured with a mini LED™ for 10 seconds. Transbond XT™

paste was then applied on the Clarity™ bracket bases. Finally, the brackets were placed and cured as described for Group 1.

Group 4: Buccal tooth surfaces were etched and primed with Clearfil SE primer as in the first step, and bonded as in the second step as described for Group 3. APC Plus Clarity™ brackets were placed. The excess resin composite was removed. The resin paste was cured as described for Group 1.

Group 5: In the first step, buccal tooth surfaces were applied with Clearfil S^3 Bond containing etchant, primer and bonding agent in a single solution by agitation with a micro-brush for 5 seconds, dried using high-pressure oil-free compressed air and light-cured with a mini LED™ for 10 seconds. In the second step, Transbond XT™ paste was applied on the Clarity™ bracket bases. The brackets were placed. The excess resin composite was removed, and in the third step, the resin paste was cured as described for Group 1.

Group 6: Buccal tooth surfaces were etched, primed and bonded with Clearfil S^3 bond in a single step as described for Group 5. APC Plus Clarity™ brackets were placed. The excess resin composite was removed, and the resin paste was cured as described for Group 1.

6. On each tooth, a stainless steel rectangular wire was tied to the bracket using an elastomeric ligature. Acrylic resin was mixed and poured into the stainless steel ring. The rectangular wires were placed on the 1.5 mm-thick plastic sheath covering the ring. The lingual surface of the tooth was embedded in self-cured acrylic resin. The resin was left for self-polymerization (Figure 27).

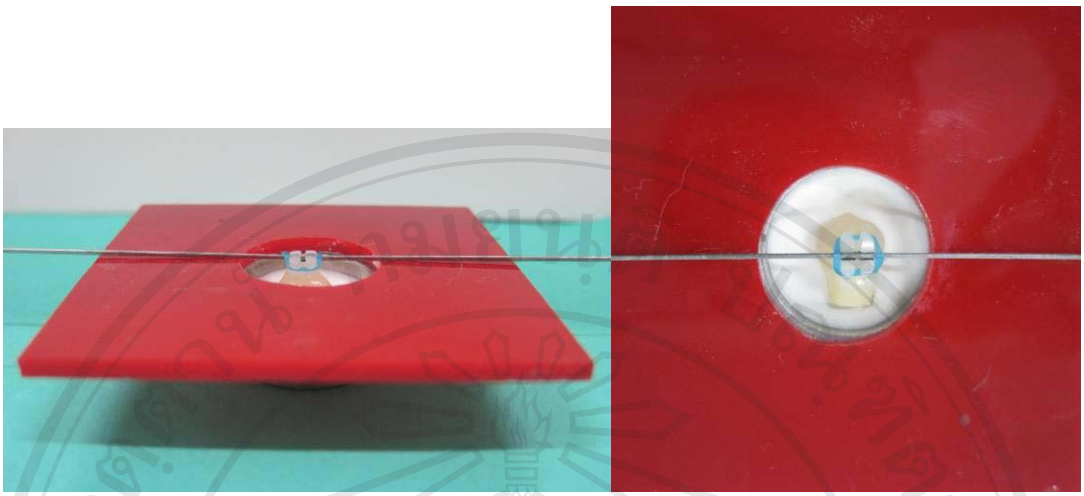

Figure 27 Tooth embedded in self-cured acrylic resin.

7. The elastomeric ligature, the wire and the plastic sheath were removed (Figure 28).

Figure 28 Specimen in stainless steel ring

8. All specimens were submerged in distilled water at 37±1˚C for 24 hours in an incubator to achieve maximum bond strength prior to shear bond strength testing.

9. After storage, all specimens were thermocycled by using the thermocycling equipment (MODEL TC301) in water baths at 5˚C and 55˚C for 1,000 cycles. The exposure to each bath was 30 seconds, and the transfer time between the two baths was 10 seconds.

10. Shear bond strength testing

The stainless steel rings were mounted into the lower part of the jig of the testing machine. The de-bonding blade was fixed into the upper part of the machine. Shear bond strength were tested by using the Instron® universal testing machine with a 500 Newtons load cell (Figure 29). The cross head speed was 0.5 mm/minute. The force was applied in a gingivo-occlusal direction between the enamel surface and the bracket base until the bracket dislodged from the tooth surface. The shear bond strengths at bond failure were recorded. (Figure 30). The force was directly recorded in Newtons and converted into megapascals (MPa) with the equation: shear force (MPa) is equal to de-bonding force (Newton) divided by surface area (mm^2). The surface areas of both Clarity™ and APC Plus Clarity™ ceramic brackets were 10.59 mm^2.

Figure 29 500 Newtons load cell of the Instron® universal testing machine

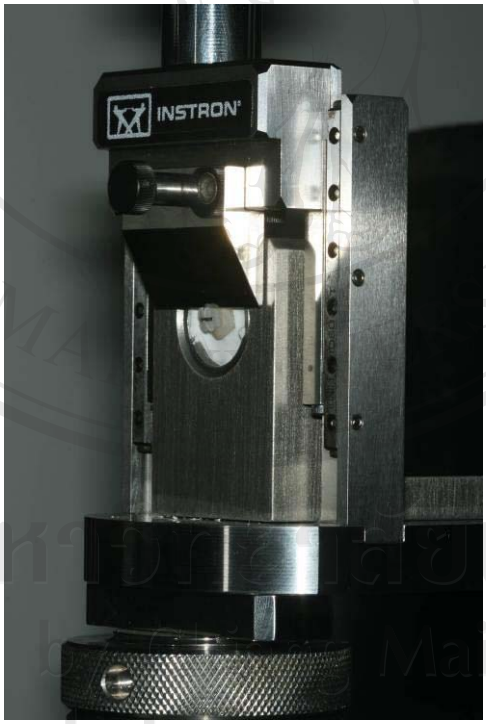

Figure 30 The force from Instron® universal testing machine was applied in a gingivo-occlusal direction.

11. Assessment of the amount of residual adhesives on de-bonded bracket base

After de-bonding of brackets, images of the bracket bases were recorded using a Canon kiss X digital single-lens reflex camera (Figure 31). The distance from bracket base to digital single-lens reflex camera was the same for each specimen. The images were used to assess the amounts of residual adhesives on the de-bonded bracket bases. The computer-generated transparent grid was superimposed on the digital photograph of the bracket base in order to assess the amount of residual adhesive (Figure 32). The percentages of residual adhesive on the bracket bases were subtracted from 100, the result representing the percentages of residual adhesives on the enamel surface. The residual adhesive amounts were scored using the Adhesive Remnant Index (ARI score) as follows.[80]

Score '0' = No adhesive left on the tooth

Score '1' = Less than half of the adhesive left on the tooth

Score '2' = More than half of the adhesive left on the tooth

Score '3' = All of the adhesive left on the tooth

Figure 31 Photograph of de-bonded bracket base from digital single-lens reflex camera

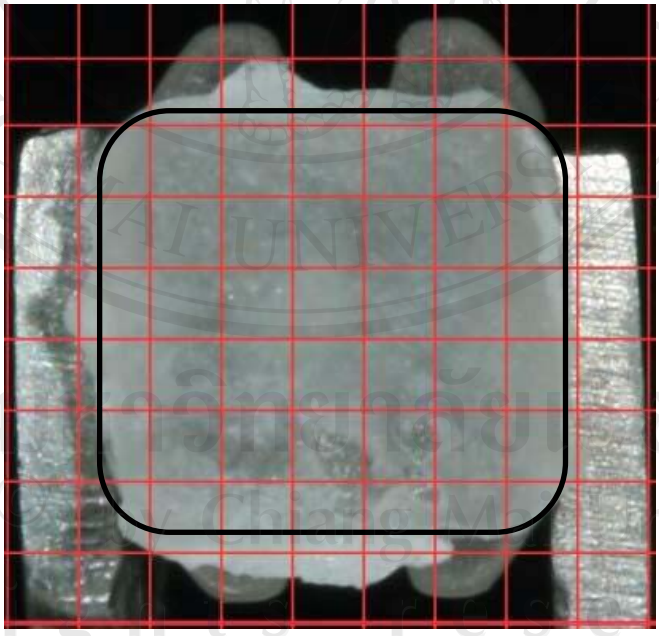

Figure 32 Residual adhesive on the de-bonded bracket base was calculated using a computer-generated transparent grid.

12. Statistical analysis

1. The shear bond strengths of two self-etching adhesive systems and a total-etching system were described by means, standard deviations, maximum and minimum.

2. Two-way analysis of variance (Two-way ANOVA) was used to test the differences among the shear bond strengths of the adhesive systems and the differences between precoated and uncoated ceramic brackets.

3. A multiple comparisons test (Tukey's test) was used to identify significant differences in the mean shear bond strength values between adhesive systems.

4. The failure modes (ARI score) described by descriptive statistics.

Statistical analysis was performed using SPSS for Windows version 14.0.