CHAPTER III

MATERIALS AND METHODS

Materials

1. Samples

The samples were maxillary and mandibular normal premolar teeth (TF score 0) and fluorotic premolar teeth extracted from patients for orthodontic reasons (TF score 3 to 5) TF scores of 3 to 5 were selected because it has been shown that the shear bond strength between brackets and teeth with mild fluorosis is not significantly different from that between brackets and normal teeth.[39] Moreover, in severe fluorosis, the outer enamel, is lost and not available for bonding. The samples consisted of 120 teeth. All teeth were free from caries, cracks and buccal restorations. All teeth were investigated within 6 months after extraction. Each tooth was stored at room temperature in 0.1% (weight/volume) thymol, and antimicrobial agent to inhibit bacterial growth.

2. Brackets

All brackets used were metal standard edgewise premolar brackets 0.018" x 0.025" slot, minidiamond type (Figure 3.1). In these foil/mesh backed brackets, there were two components: body and base. Each stainless steel bracket body was joined with a stainless steel foil/mesh base by brazing with gold alloy. The total area of each bracket was 8.0 square millimeters and the foil mesh had one hundred interlock holes per inch (Ormco Corporation, Glendora, California, USA).

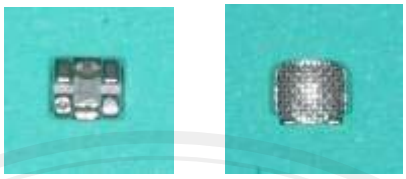

Figure 3.1 Standard edgewise premolar brackets 0.018” x 0.025” slot

3. Adhesive Systems

3.1 Conventional phosphoric acid etching system

Conventional phosphoric acid etching system with nomix self cured composite resin , System^{TM}1+ (Ormco Coporation) and Unite^{TM} (3M Unitek, Monrovia, California, USA)

3.2 Adhesive resin cement

Superbond C&B , self cured resin cement (Sun Medical Co. Ltd., Shiga, Japan)

The adhesive systems used in this study are shown in Figures 3.2-3.4

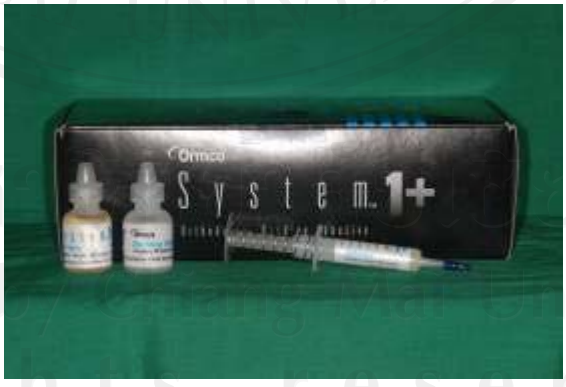

Figure 3.2 Conventional phosphoric acid etching system (System^{TM} 1+)

Figure 3.3 Conventional phosphoric acid etching system (Unite™)

Figure 3.4 Adhesive resin cement (Superbond C&B)

4. Supplies

4.1 Carborundam discs

4.2 Fluoride-free pumice

4.3 0.018” x0.025” Stainless steel archwires.

4.4 Cylindrical polyvinylchloride rings, whose diameter, height and thickness were 22 millimeters, 16 millimeters, and 1.5 millimeters, respectively, were sealed at base with acrylic resin (Figure3.5).

4.5 Elastic ligatures

4.6 2.0 millimeters thickness of hardened plastic sheath

4.7 Acrylic resin and monomer

Figure 3.5 Cylindrical polyvinylchloride ring

5. Instruments

5.1 An Instron 5566 Universal testing machine (Instron Calibration Laboratory, Northwood, Massachusetts, USA) was used for measuring shear bond strength (Figure 3.6)

Figure 3.6 Universal testing machine

5.2 A de-bonding plate was designed to fit under the bracket wing to ensure vertical force application between the bracket base and the enamel surface (Figure 3.7)

Figure 3.7 De-bonding plate

5.3 A mounting jig was designed to hold the tooth in a vertical position with the bracket base parallel to the direction of force (Figure 3.8)

Figure 3.8 Mounting jig

The de-bonding plate , mounting jig and tooth with bracket are shown in Figure 3.9

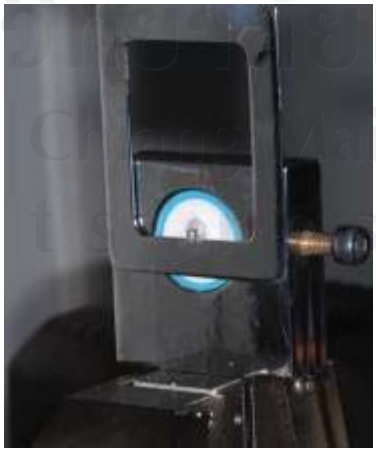

Figure 3.9 De-bonding plate , mounting jig and tooth with bracket

5.4 A Memmert Model 200 Incubator (Memmert GmbH+Co.KG, Schwabach, Germany) was maintained at 37°C (Figure 3.10)

Figure 3.10 Incubator

5.5 Thermocycling device (HW B332R, TC 301, CW B332R, Medical & Environment Equipment Research Laboratory, King Mongkut's Institute of Technology Lad Krabang, Thailand) (Figure 3.11)

Figure 3.11 Thermocycling device.

5.6 A computer-generated transparent grid was used for determining the amount of residual adhesive on the de-bonded bracket in each photograph (Figure 3.12)

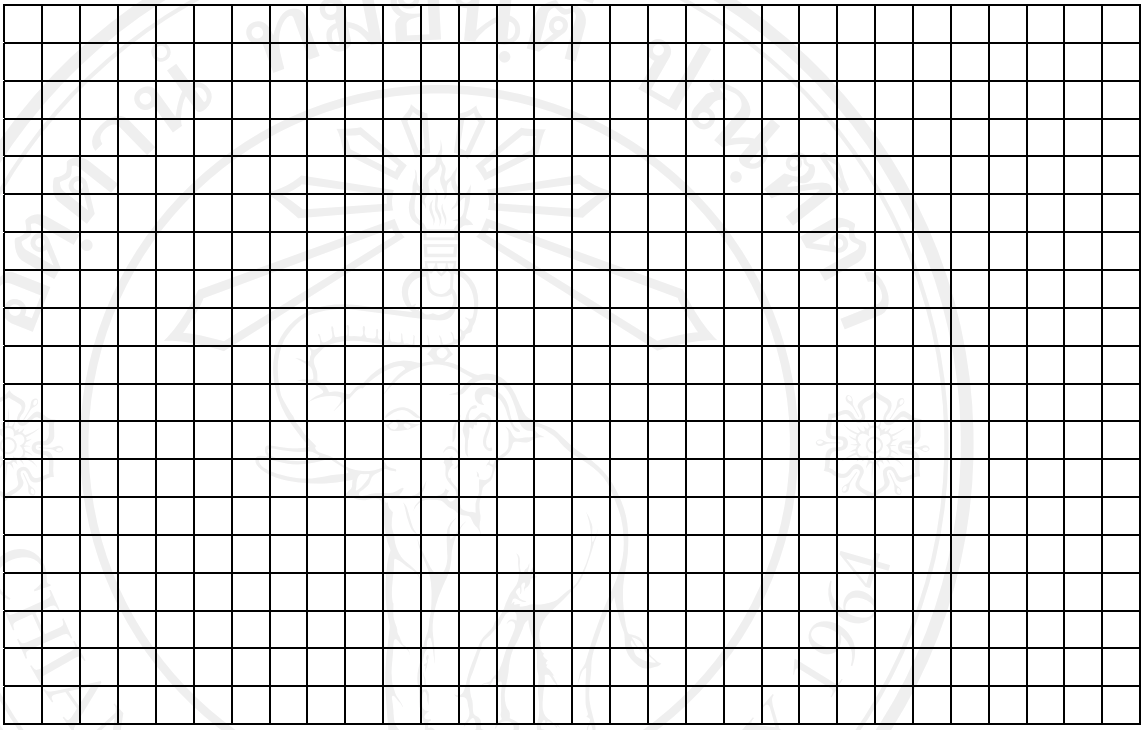

Figure 3.12 Computer-generated transparent grid

Methods

The experimental process was divided into four parts as follows:

I. Tooth allocation for experimental groups

II. Tooth preparation

III. Shear bond strength testing

IV. Examination of the failure modes

I. Tooth allocation for experimental groups

Three adhesive systems were used: System^{TM} 1+ and Unite^{TM}, each of which uses 37% phosphoric acid as the etching system, and Superbond C&B, which uses 65% phosphoric acid gel for etching. The compositions of the adhesive components, including their liquid activators, are proprietary.

Four extracted premolar teeth from each patient, consisting of teeth 14 or 15, 24 or 25, 34 or 35, 44 or 45, were randomly allocated to each experimental group to avoid a selection bias. A table of random digits was the choice for the allocations.

II. Tooth preparation

1. All teeth were prepared horizontal sectioning of the roots with carborundum discs about 2 mm. below the CEJ (Figure 3.13). The buccal surfaces were cleaned with fluoride-free pumice and water for 10 seconds, rinsed with distilled water and then dried with a stream of oil-free compressed air

Figure 3.13 shows sectioning of the roots with carborundum discs.

2. Each tooth was bonded with one adhesive system, following the manufacturer's instructions. Standard brackets were used for bonding each tooth. The long axis of

the bracket was aligned parallel to the long axis of the tooth and the bracket was positioned in the center of the clinical crown.

Each sample was bonded by one of the following procedures:

2.1 Group1: (normal teeth) and Group 4 (fluorotic teeth): Conventional phosphoric acid etching system with self- cured composite resin, System^{TM} 1^{+} (Ormco Coporation)

Each buccal enamel surface was etched with a 37% phosphoric acid solution for 30 seconds, rinsed with water for 20 seconds and dried thoroughly with a stream of oil-free compressed air. A 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 enamel surface and excessive resin was removed from the enamel surface with an explorer.

2.2 Group 2 (normal teeth) and Group 5 (fluorotic teeth): Conventional phosphoric acid etching system with self-cured composite resin, Unite^{TM} (3M Unitek)

Each buccal surface was etched with a 37% phosphoric acid solution for 15 seconds, rinsed with water for 20 seconds and dried thoroughly with a stream oil-free compressed air. A 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 enamel surface and excessive resin was removed from the enamel surface with an explorer

2.3 Group 3 (normal teeth) and Group 6 (fluorotic teeth): Adhesive resin cement (Superbond C&B, Sun Medical Co. Ltd.)

Each buccal surface was etched with red activator (65 %phosphoric acid gel), which was in the Superbond C&B kit, for 30 seconds, washed for 20 seconds and air dried. The catalyst, a partly oxidized TBB initiator, was added to the monomer mixture of 4-META and MMA to prepare an activated polymerized monomer liquid. Then, the polymer powder and the activated monomer liquid was mixed and used to bond the metal brackets to the treated enamel surface using the brush-dip technique. The study group characteristics are shown in Table 3.1

Table 3.1 Study group characteristics

Group	Tooth	Adhesive
1	Normal	37%phosphoric acid and System^{TM} 1+
2	Normal	37%phosphoric acid and Unite^{TM}
3	Normal	65 %phosphoric acid and Superbond C&B
4	Fluorotic	37%phosphoric acid and System^{TM} 1+
5	Fluorotic	37%phosphoric acid and Unite^{TM}
6	Fluorotic	65 %phosphoric acid and Superbond C&B

A 0.018” x 0.025” stainless steel wire was placed in the bracket slot of the tooth-bracket complex and elastic was used to ligate the body of the bracket perpendicular to the shear force. The assembly was attached to the hardened plastic sheath by transparent tape so that the tooth-bracket complex protruded downwards through a 2 cm. diameter aperture in the centre of the sheath. so that the buccal enamel, the wire and the bracket protruded above the rim of the ring. The whole assembly was then

placed onto a cylindrical polyvinylchloride ring (Figure 3.14) so that the buccal enamel, the wire and the bracket protruded above the rim of the ring.

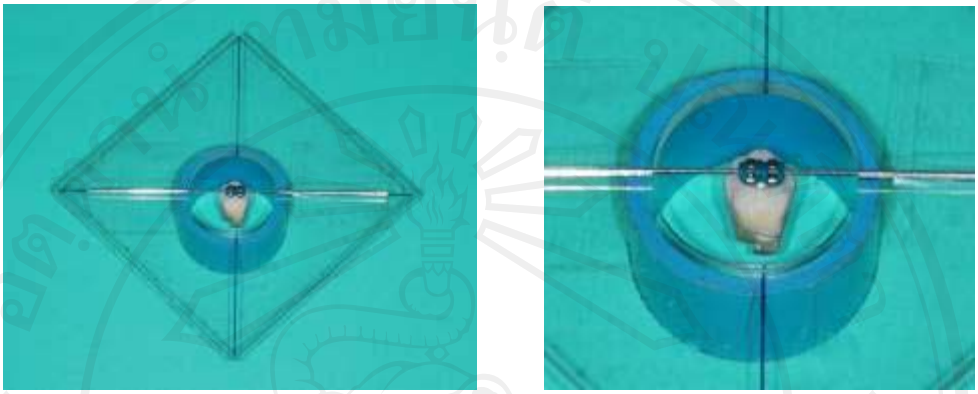

Figure 3.14 A 0.018” x 0.025” stainless steel wire was placed on the bracket slot by ligating the elastic ligature to the body of the bracket

Mixed acrylic monomer (Figure 3.15) was poured into the ring, leaving only the buccal tooth surface and attached orthodontic bracket exposed.

Figure 3.15 The acrylic resin that was used in this study

After the acrylic resin was cured, the elastic ligature, the wire, and the plastic sheath were removed (Figure 3.16).

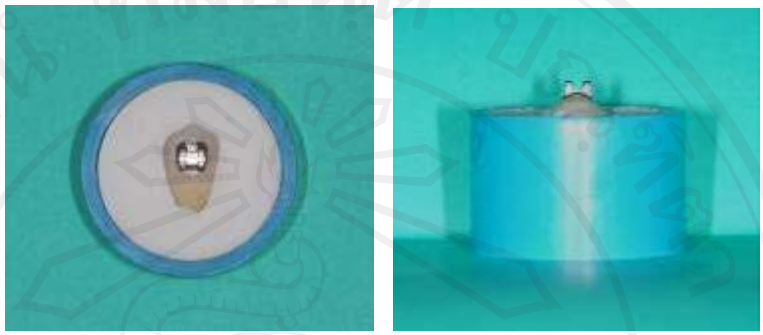

Figure 3.16 Tooth embedded in polyvinylchloride ring after removal of the elastic ligature, the wire, and the plastic sheath. A: Top view, B: Side view.

All specimens were submerged in distilled water at 37°C for 24 hours in an incubator to achieve maximum bond strength before testing. After submersion, all specimens were thermocycled between 5°C and 55°C for 1,000 cycles of 30 seconds each, using a thermocycling instrument containing two water baths. The transfer time between the two baths was 10 seconds.[52] (The thermocycling test simulates the hot and cool conditions in the oral cavity and the water exposure simulates saliva and other ingested liquids. Differences in coefficients of thermal expansion among brackets, adhesives and tooth, and repetitive contraction and expansion stresses are produced. These stresses may affect bond strength and bond failure. The decrease in bond strength after thermocycling can be affected by increasing water absorption and thermal stresses.)

III. Shear bond strength testing

Shear bond strength was determined by using a universal testing machine (Instron®) at a crosshead speed of 0.5 mm per minute and a 500-newton load cell (Figure 3.17). The 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. The force was applied in the gingivo-occlusal direction until the bracket was dislodged from the tooth surface and the shear bond strength values at bond failure were recorded (Figures 3.18 and 3.19).

Figure 3.17 500-newton load cell

Figure3. 18 The de-bonding plate was fixed into the upper pneumatic grip and the mounting jig was fixed into the lower pneumatic grip.

Figure3. 19 The apparatus assembled for testing shear bond strength.

IV. Examination of the failure modes

Failure modes were examined at the failure sites and by evaluating the amounts of residual adhesive on de-bonded bracket bases.

Examination of the failure sites

After de-bonding, failure sites were evaluated by examination of photographs of the de-bonded bracket bases from a Nikon D80 camera at F 20 and an 11 centimeter camera to bracket base distance.

The failure sites were divided into four locations according to the method of Artun and Bergland,[53] which used the Adhesive Remnant Index as follows:

0 = no adhesive remains on the tooth surface

1 = less than half of the adhesive remains on the tooth surface

2 = more than half of the adhesive remains on the tooth surface

3 = all the adhesive remains on the tooth surface with a distinct impression of the bracket base

Examination of amount of residual adhesives on de-bonded enamel surfaces

The amounts of residual adhesives on the de-bonded bracket surfaces were determined from photographs taken from the Nikon D80 camera (Figure 3.20). The image of the computer-generated transparent grid was superimposed on the image of each digital photograph (Figure 3.21) and 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.

Figure 3. 20 Photograph of de-bonded bracket surface area

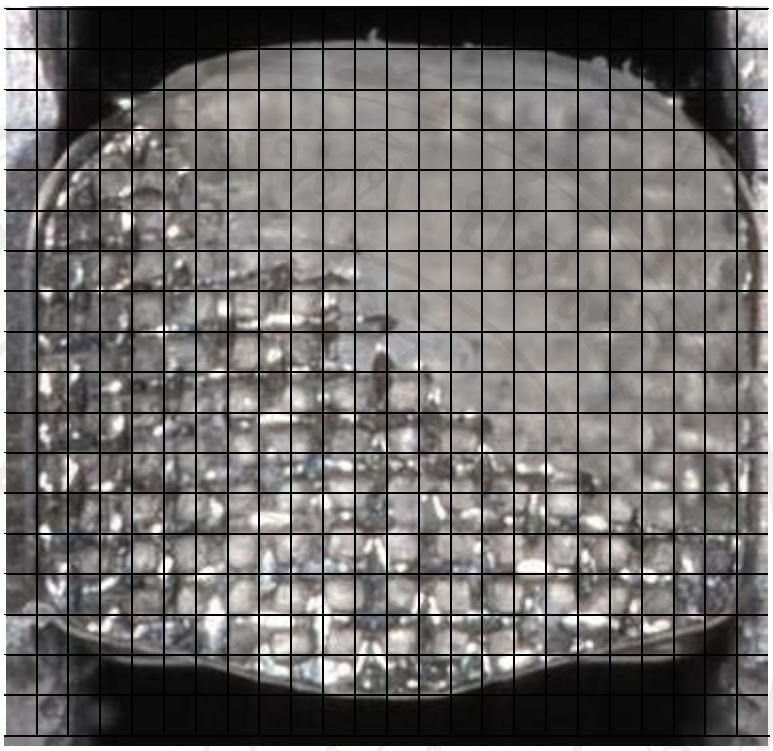

Figure 3.21 Amounts of residual adhesive on the de-bonded bracket surfaces were determined by using a computer-generated transparent grid.

Statistical analyses

The SPSS for Windows Release 15.0 program was used to calculate the following analysis:

1. The Shear bond strength values of three different adhesive systems were described by means, standard deviations.
2. Two way analysis of variance (ANOVA) was used to compare the mean shear bond strength values among three different adhesive systems on normal and fluorotic teeth.

3. A multiple comparisons test (Tukey's test) was used to identify which groups were different when there was a significant difference in the mean shear bond strength values among three different adhesive systems in orthodontic bracket placement as determined by the two-way analysis of variance (ANOVA).

4. Descriptive statistics were used to describe the percentage of failure modes (ARI score)

The 95% confidence interval was used to determine significance.