CHAPTER 2
RESEARCH DESIGN AND METHODOLOGY

This study was divided into three main parts:

2.1 Bonding efficiency between the plasma-treated FRCP and the composite resin core build-up material after treating the posts with different gas plasma was evaluated. Also the evaluation of surface roughness of the posts in each plasma treatment group was performed.

2.2 The best plasma treatment from 1 was utilized in this part, the suitable parameters of treatment involving the gas pressure, discharge power, and treatment time which induce the most bonding efficiency were determined, and

2.3 Within the best condition of plasma treatment, the stability of the established bonding efficiency between the plasma treated-FRCP and the composite resin core build-up material was evaluated through hydrothermal stress.

2.1 Part 1: The studies of bonding efficiency between the plasma treated FRCP and the composite resin core build-up material and the evaluation of surface roughness of the posts of each plasma treatment group.

The objective of this study was to evaluate the tensile-shear bond strength between a composite core build-up material and two different types of FRCPs, with and without the four types of plasma treatments. The null hypothesis of the study was that either the post or the plasma treatment has no significant influence on the tensile-shear bond strength between the FRCP and the composite core build-up material.

Materials and methods

2.1.1 Materials used

The materials investigated, including their chemical compositions, manufacturers, and sizes, are listed in table 1 and their chemical structures displayed in fig. 7. Two commercial FRCPs were selected: one was methacrylate-based (FRC Postec, Ivoclar Vivadent, Schaan, Liechtenstein; hereafter FRC), the other was epoxy resin-based (DT Light-Post, RTD, St. Egreve, France; hereafter DT). These posts were composed of unidirectional quartz fiber (DT) and fiberglass (FRC) embedded in different resin matrices. Both were taper-shaped posts, of which 30% of the coronal portion was parallel.

2.1.2 Plasma treatment groups

FRC and DT posts (n=45 each) were randomly selected and divided into five groups (n=9 per group); a control group (C), an oxygen plasma treatment group (O_2), an argon plasma treatment group (Ar), a nitrogen plasma treatment group (N_2), and a group treated with a mixture of helium (20% vol) with nitrogen (80% vol) plasma ($He+N_2$).

For each plasma treatment group, nine posts were attached to one specimen holder which was placed in the center of the cylindrical quartz chamber of a plasma-generating machine developed by the Plasma and beam Physics research facility unit (Department of Physics and Material Sciences, Chiang Mai University, Chiang Mai, Thailand) (fig. 8). Under a vacuum with a base pressure of 2.4 Pa, the experimental

gas was fed into this chamber at a flow rate of 6 cm^3/min and kept at a constant pressure of 13.3 Pa throughout this experiment. The experimental gas was ionized to plasma by inductive coupling discharge, using a radiofrequency generator (Dressler CESAR, Advanced Energy, Stolberg, Germany) at 13.56 MHz, 50 W for 10 minutes.

Table 1 List of investigated materials.

Material	Composition	Manufacturer	Lot no.	Size
DT Light-Post	Quartz fibers: 60% vol. Epoxy resin: 40% vol.	Recherches Techniques Dentaires, St. Egreve, France	093730810	#2, ∅ = 1.8 mm Length = 20 mm
FRC Postec-post	Glass fiber: 61.5% vol. Triethylene-glycol-dimethacrylate (TGDMA) and urethane-dimethacrylate (UDMA) monomer: 38.5% vol.	Ivoclar Vivadent, Schaan, Liechtenstein	L35052 L47590	#3, ∅ = 2 mm Length = 20 mm
MultiCore Flow	Bisphenol A glycol dimethacrylate (Bis-GMA), TGDMA, UDMA 29 wt% inorganic fillers, Ytterbiumtrifluoride, initiator, stabilizer and pigment	Ivoclar Vivadent, Schaan, Liechtenstein	K 34799 K 44769	

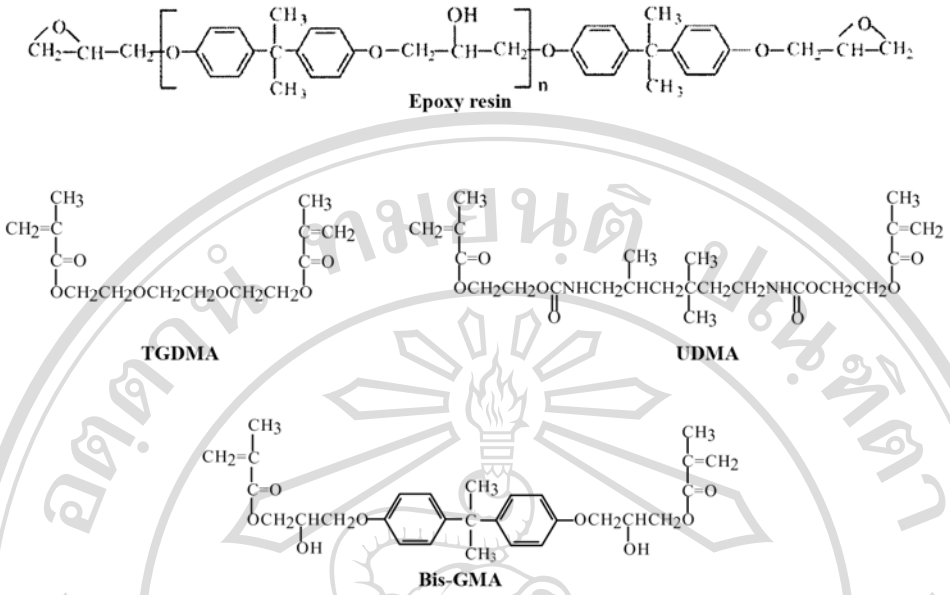

Figure 7 Chemical structures of the investigated materials.

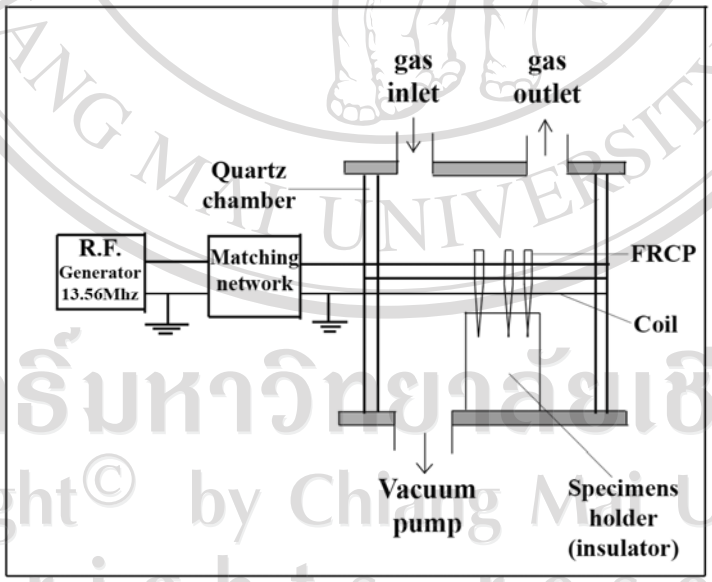

Figure 8 Schematic illustration of low pressure plasma generator with fiber-reinforced composite post (FRCP) on the specimen holder at the center of quartz chamber.

2.1.3 Specimen preparation for pull-out test

After plasma treatment, each post was kept in a glass vial sealed with a rubber cap at atmospheric pressure and room temperature (25 ^{o}C). Six posts in each group were selected and prepared for a pull-out test within 12 hours of plasma treatment. Each post was placed in the upright position in a special apparatus, consisting of a split, metal cylinder with a cylindrical mold in the center of one end. Dimensions of the cylindrical mold were 8 mm in diameter and 2 mm in height (fig. 9a).

In this upright position, 2 mm of the parallel portion of the coronal end of the tapered post was exposed outside the cylindrical mold. A composite core build-up material (MultiCore Flow, Ivoclar Vivadent, Schaan, Liechtenstein) was directly syringed into the mold and around the post to form a core for the pull-out test. Then, the top of the core material was light-activated using a light curing unit (Elipar Trilight, 3M/ESPE, Seefeld, Germany) for 40 seconds with a light intensity of 600 mW/cm^2.

After complete polymerization, the post was removed from the mold. Excess core build-up material on top of the specimen was removed with abrasive paper until the height of this core was exactly 2.0 mm (fig. 9b). A thin metal ring was placed under the core to support the bottom surface of the core (fig. 9c).

The prepared post was then placed upside down in the center of a new split metal cylinder with a cylindrical mold in the center of the upper half. Dimension of the cylinder mold were 18 mm in diameter and 5 mm in height, so that the apical 8 mm of the post was in the center of this mold (fig. 9d). A polyvinyl chloride (PVC) tube with a diameter of 18 mm and a height of 10 mm was inserted into the this mold (fig. 9e). An autopolymerized resin (Instant tray mix, Lang Dental Manufacturing Co, Wheeling, Ill., USA) was mixed and poured into the tube until it reached the top of the tube. After complete polymerization, the specimen was detached from the mold (fig. 9f) and kept in a glass vial sealed with a rubber cap at atmospheric pressure and room temperature for 24 hours before the pull-out test.

For the pull-out test procedure, the PVC tube was attached to a special jig holder of a universal testing machine (Lloyd, LR10K, Fareham, UK). The thin metal ring under the composite core was supported by a special upper jig holder**,** fixed in the

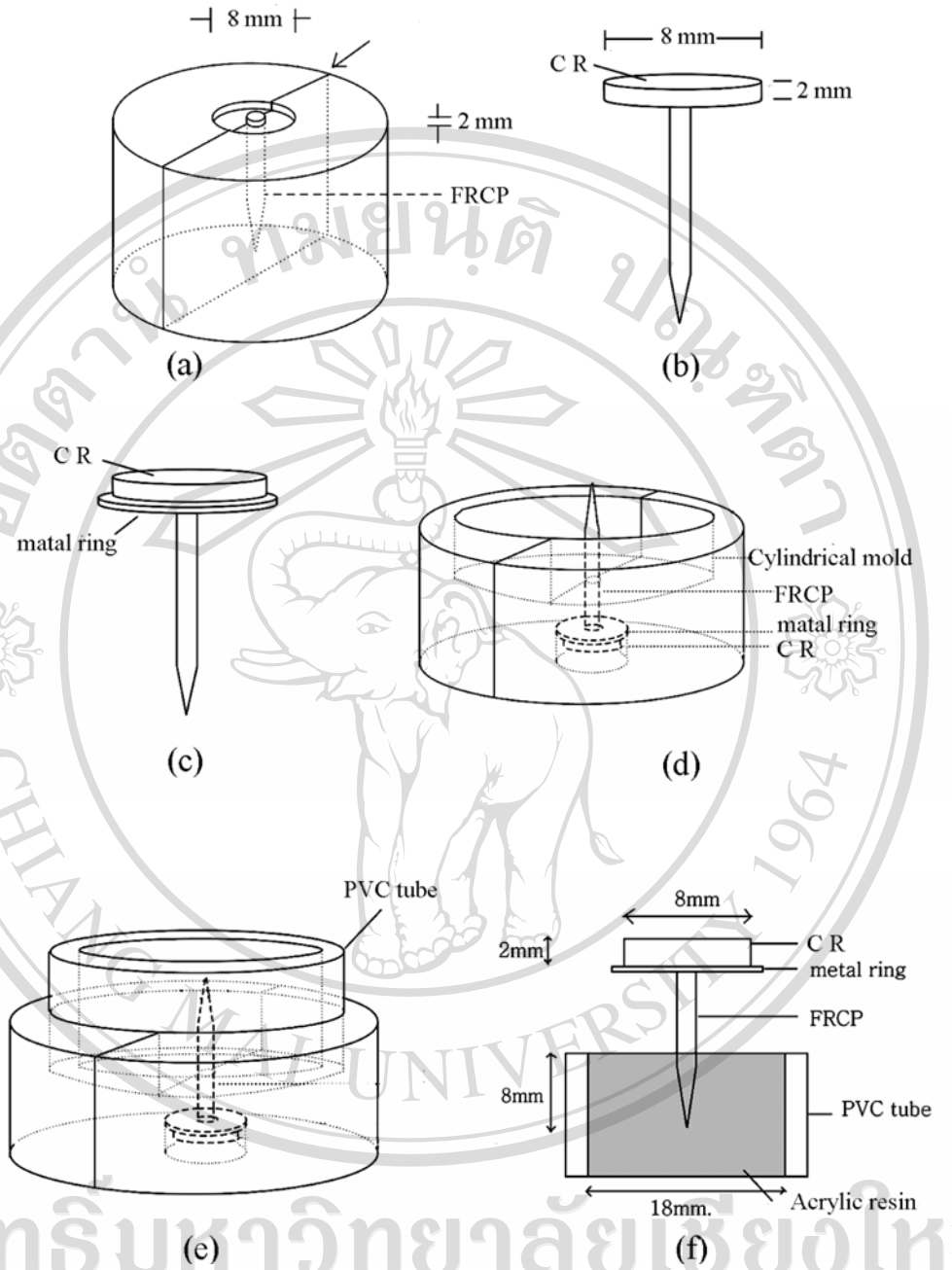

Figure 9 Schematic illustration of specimen preparation for pull-out test: (a) fiber-reinforced composite post (FRCP) in the special apparatus; (b) composite resin (C R) on FRCP; (c) metal ring supporting the bottom surface of the composite core; (d) prepared post placed in the center of a new split metal cylinder; (e) the post positioned in the center of the polyvinyl chloride (PVC) tube; (f) the prepared specimen.

tensile device of the machine's moving part. Each specimen was loaded in tension at a crosshead speed of 1mm/min until the composite resin core was separated from the post. The maximum load (N) was divided by the bonding area (mm^2) and then recorded in MPa for statistical analysis. After the pull-out test, all de-bonded interfaces between the post and the composite core build-up material were observed using a light microscope (Meiji Techno Co., Ltd, Tokyo, Japan) at 15x magnification.

2.1.4 Surface roughness measurement

The remaining three posts in each group were evaluated for surface roughness by a profilometer (Tylyscan 150, Taylor Hobson, Leicester, UK), using a scan field of 2 x 0.5 mm on three randomly selected areas for each post. The average surface roughness (S_a) of the posts in each group was expressed in μm.

2.1.5 Statistical analysis

Statistical analyses of tensile-shear bond strength and surface roughness were performed respectively using two-way ANOVA. Tukey's test was used for *post hoc* multiple comparisons to detect significantly different pairs. In all the tests, the level of significance was set at α=0.05, and calculations were performed using a statistical software (SPSS ver 13.0, SPSS Inc., Chicago, Ill., USA).

The most effective gas for plasma treatment obtained in this study will be used in part 2.

2.2 Part 2: The study on the suitable parameters of plasma treatment involving the gas pressure, discharge power, and plasma treatment time which induce the optimal bonding efficiency.

The objective of this study was to find out the suitable parameters of the most effective gas from part 1 that induced the optimal bonding between FRCP and composite core build-up material. The null hypothesis of this study was that each of the above-mentioned parameters has no significant influence on the tensile-shear bond strength between the FRCP and the composite core build-up material. This study was divided into 3 steps.

2.2.1 Step 1 Gas pressure

The gas pressure of 2.7, 6.7, 13.3, 26.7 and 40 Pa were used. FRC and DT posts were randomly divided into 5 group each (n= 6) according to the gas pressure used in the treatments. The most effective gas from part 1 was used for plasma treatment for 10 minutes by varying gas pressure as above-mentioned in each group. The specimens for pull-out test were prepared in the same manner as mentioned in part1. Because the resin matrices of FRC and DT post were absolutely different in their chemical compositions, then the statistical analysis was performed separately. Statistical analysis of tensile-shear bond strength of each post was performed using 1-way ANOVA. Tukey's test was used for *post hoc* multiple comparisons to detect significantly different pairs and the level of significance was set at α=0.05. The corresponding gas pressure which provided the highest tensile-shear bond strength would be selected for the experiment in step 2.

2.2.2 Step 2 Discharge power

The most effective gas pressure from step 1 was used in this part. The discharge power at 25W, 50W, and 75W were selected in this part. Six FRC and six DT posts were treated by the most effective gas from part 1 for 10 minutes for each discharge power and then the specimens for pull-out test were prepared as mentioned in part 1. Statistical analysis for tensile-shear bond strength was performed using 1-way ANOVA for FRC and DT post separately. Tukey's test was used for *post hoc* multiple comparisons to detect significantly different pairs and the level of

significance was set at α=0.05. The most effective discharge power would be selected for the experiment in step 3.

2.2.3 Step 3 Plasma treatment time

Under the most effective gas pressure from step 1 and the most suitable discharge power from step 2, six FRC and six DT posts were treated by plasma with the treatment time of 3, 5, 10, 15 and 30 minutes for each experimental group. Specimen preparation and pull-out test was performed in the same way as mentioned in part 1. One way-ANOVA was used for statistical analysis of tensile-shear bond strength for the FRC and DT posts separately. Tukey's test was used for *post hoc* multiple comparisons to detect significantly different pairs and the level of significance was set at α=0.05.

2.3 Fourier transform infrared spectroscopy (FTIR) for chemical analysis

To understand the mechanism of bonding enhancement between the treated-post to composite core build-up material by the most effective plasma treatment, the surface of the untreated posts and the plasma-treated posts using the best condition as obtained from the study of step 1- 3 was analyzed by FTIR (Nicolet 6700, Bruker, Germany) in attenuated total reflected (ATR) mode. The spectra were collected by averaging 64 scan at the resolution of 4 cm^{-1} from 500-4000 cm^{-1}. Element components of untreated post and plasma-treated post were determined using energy dispersive X-ray spectroscopy (EDX, INCA PentaFETx3 model6647, Oxford, England) provided in the scanning electron microscope (JEOL model JSM-5140LV, Tokyo, Japan).

2.4 Part 3: The study of hydrothermal effect on bonding stability between the FRCP and composite core build-up material.

According to the pilot study on bond strength between the plasma-treated FRCP and composite core build-up material, it was demonstrated that storage the specimens in 37 ^{o}C deionized water for 7 days (hydrothermal condition) significantly decreased the tensile-shear bond strength between the FRCP and composite core build-up material from 29.37 MPa to 13.58 MPa. The decreasing in tensile-shear bond strength may influence by the effect of water or by an increasing in temperature or both of them. The other factor that may influence the bond strength was the treatment time, since optimum treatment time in room temperature may exceed the optimum treatment time in hydrothermal condition, prolong treatment time may implied a decomposition or degradation of the post surface.

The objective of this study was to evaluate the effect of plasma treatment time and the hydrothermal conditions on the tensile-shear bond strength between the FRCP and composite core build-up material. The null hypothesis of the study was that either the plasma treatment time or the hydrothermal condition has no significant influence on the tensile-shear bond strength between the FRCP and the composite core build-up material. This study was divided into 2 main divisions.

2.4.1 Division 1: Effect of plasma treatment time and hydrothermal storage condition

In this division, the most effective plasma treatment obtained from the study in part 2 was used. Both FRC and DT posts were randomly divided into eight treatment groups, including; non plasma treatment group (control), the groups of plasma treatment for 30 seconds, 1, 3, 5, 10, 15, and 30 minutes (n=12 each). After plasma treatment, each post was prepared for the specimens for pull-out test as mentioned in part 1 within 12 hours of plasma treatment. Six specimens in each group were stored in two different storage conditions, one was in the closed vial at room temperature for 24 hours, and the other was in the 37 ^{o}C deionized water for 7 days (fig.10). Pull-out test was performed as mentioned in part 1. Statistical analysis for tensile-shear bond strength was performed using 2-way ANOVA for the FRC and DT post separately.

Tukey's test was used for *post hoc* multiple comparisons to detect significantly different pairs and the level of significance was set at α=0.05.

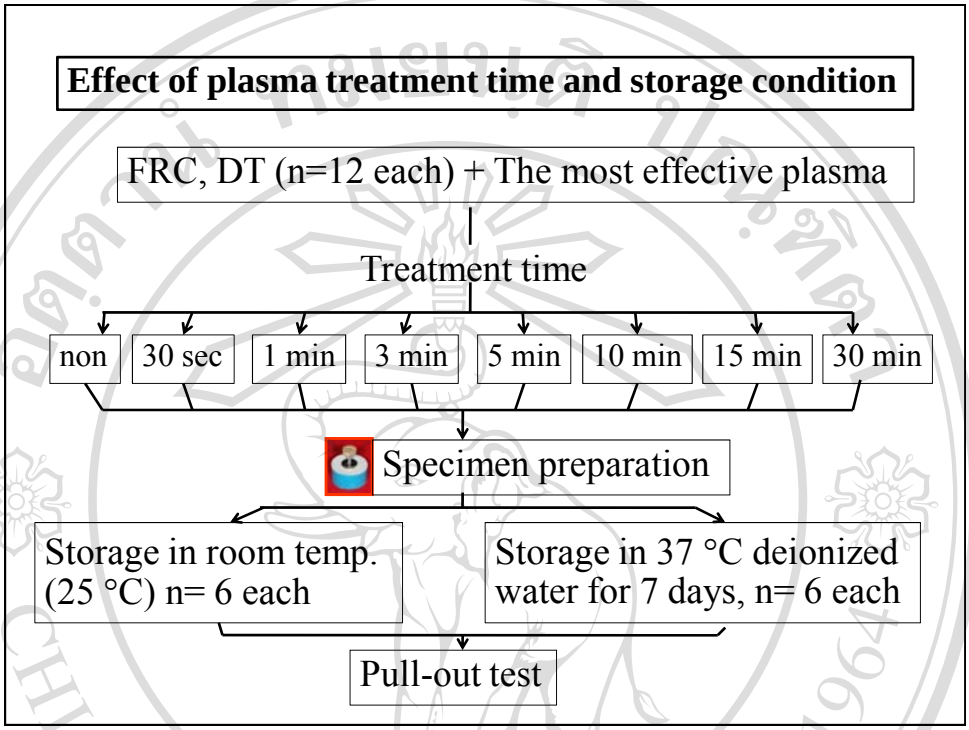

Figure 10 Flow-chart of the experimental procedures in division 1: Effect of plasma treatment time and hydrothermal storage condition.

As earlier-mentioned that the decreasing in tensile-shear bond strength between the FRCP and the composite core build-up material may influence by the effect of water or by an increasing in temperature or both of them. To clarify the main factor of the hydrothermal condition, the study in division 2 was performed.

2.4.2 Division 2: Main factor of hydrothermal condition

In this division, the study was divided into 2 sections.

2.4.2.1 Section 1: Thermal effect.

Both types of the posts were randomly divided into 3 groups according to the method of plasma treatment including; the first group: non plasma treatment (control group) (n=12 each), the second group: the FRC posts and the DT posts (n=12 each)

were treated with the most effective plasma treatment, and the third group: both types of the posts (n=12 each) were treated as the same way as performed in the second group but the treatment time was 30 minutes. These posts were prepared for the specimens for pull-out test in the same way as done in part 1, then the specimens in each group were divided into 2 subgroups (n=6 each) according to the storage temperature. The first subgroup was storage the specimens in the closed vial at room temperature (25 ^{o}C), the second subgroup was storage the specimens in the chamber at 37 ^{o}C for 7 days (dry storage condition) (fig.11). After pull-out test, statistical analysis for tensile-shear bond strength was performed using 2-way ANOVA for FRC and DT post separately. Tukey's test was used for *post hoc* multiple comparisons to detect significantly different pairs and the level of significance was set at α=0.05.

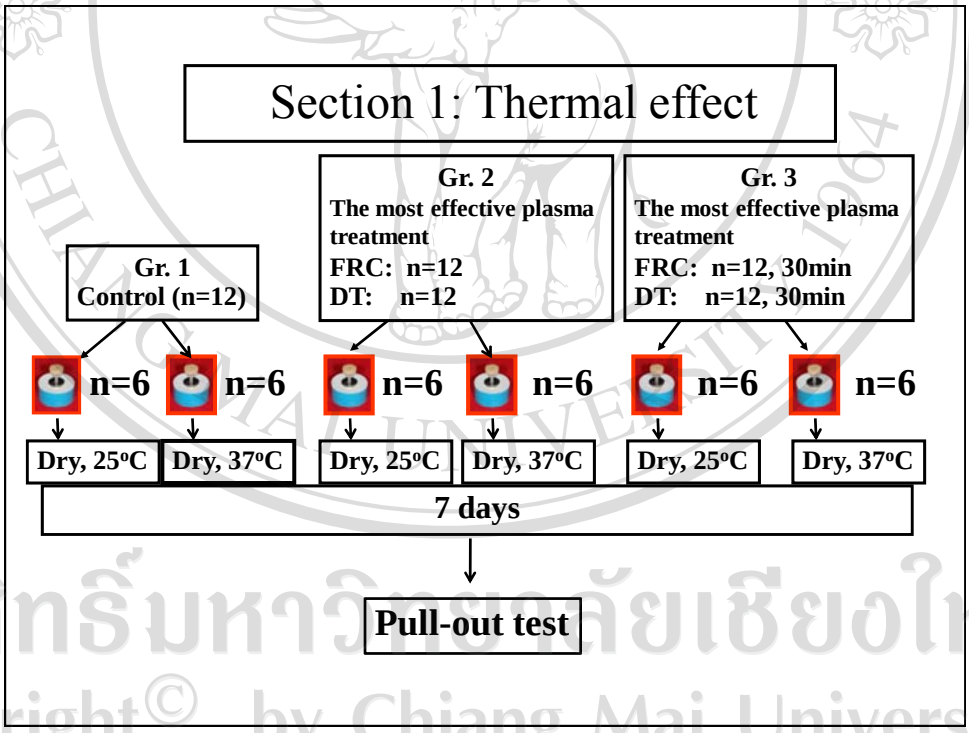

Figure 11 Flow-chart of the experimental procedures in section 1: Thermal effect.

2.4.2.2 Section 2: Hydration effect.

Both types of posts (n=12 each) were randomly divided into 2 groups (n=6) and the posts in each group were treated in the same ways as mentioned in the second and the third groups in section 1, then the posts were prepared for the specimens for pull-out test. All of the specimens were stored in 37 °C deionized water for 7 days (wet storage condition) (fig. 12). After pull-out test, the data in this part was pooled with the data in section 1 (the second group and the third group). Statistical analysis for tensile-shear bond strength was performed using 2-way ANOVA for FRC and DT post separately. Tukey's test or Tamhane's test was used for *post hoc* multiple comparisons to detect significantly different pairs and the level of significance was set at α=0.05.

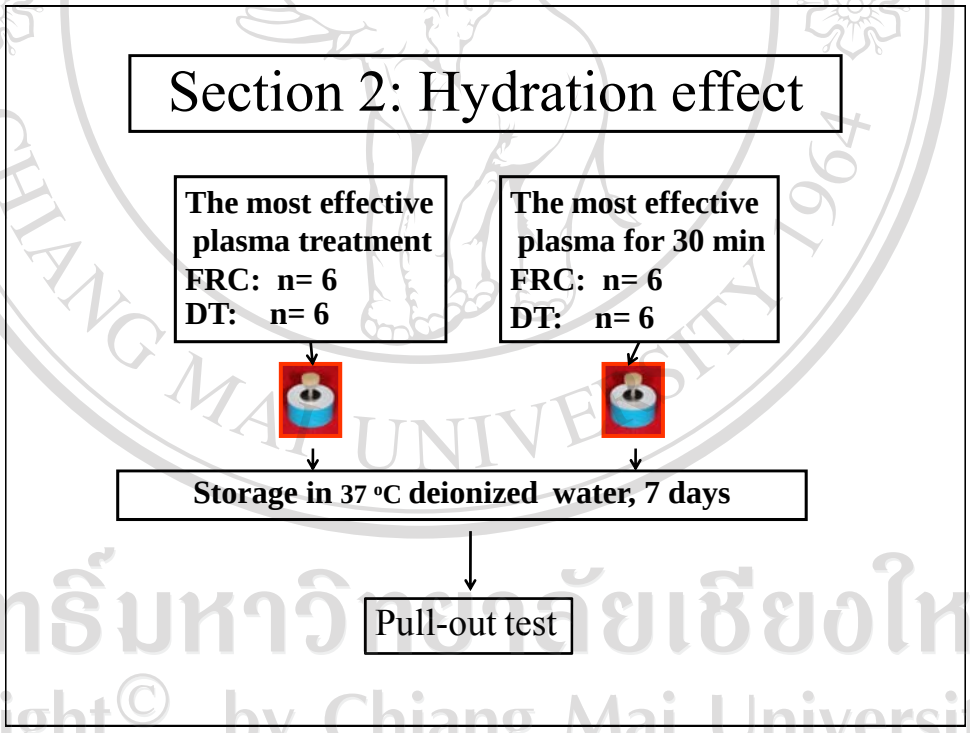

Figure 12 Flow-chart of the experimental procedures in section 2: Hydration effect.