CHAPTER III
MATERIALS AND METHODS

Materials

Materials being used in this experimental process were as followed:

1. **The orthodontic magnets** (Ormco, U.S.A., Part No. 671-0001).

The orthodontic magnets were cylindrical samarium-cobalt magnets. The company recommended them for molar distalization. They were coated with a biocompatible polymer to avoid leach out products. The size of orthodontic magnets were 3.7 millimeters ϕx2.0 millimeters (diameter x thickness). The twelve samples of orthodontic magnets were used in this investigation (Figure 3.1).

2. **The commercial magnets**

The available magnets can attract the orthodontic bracket with strong forces and can be made in various shapes. However, their composition and force generated are unidentified. There were supplied in four sizes, 3.28x3.28x2.0 millimeters, 6.0x6.0x2.0 millimeters, 8.0x8.0x2.0 millimeters, 10.0x10.0x2.0 millimeters. There were twenty samples in each size (Figure 3.2).

3. **The metal bracket** (Ormco, batch No. 350-0331, Ormco Corporation, U.S.A.).

Full metal bracket of 0.018"x0.025" slot standard edgewise cuspid bracket, minidiamond type. There were two components in these foil/mesh backed bracket: body and base. 17-4 stainless steel bracket body was joined with 316 stainless steel foil/mesh base by brazing with gold alloy. There was only one bracket used in this investigation.

Figure 3.1 The orthodontic magnets (Ormco, U.S.A., Part No. 671-0001).

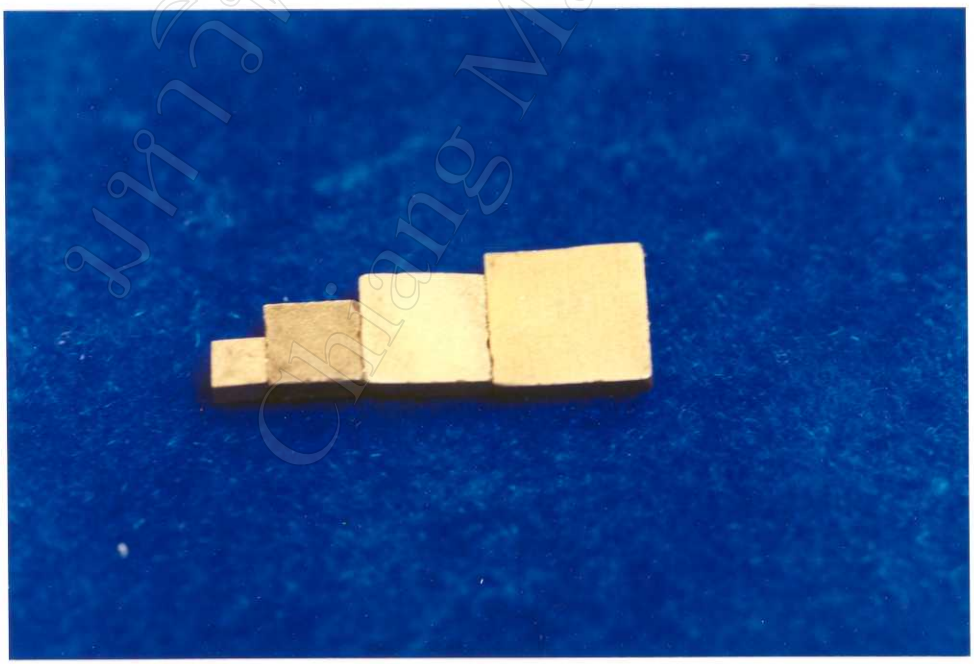

Figure 3.2 The commercial magnets

Instruments

1. **The universal testing machine** (LLOYD instruments, model LRX, Intro enterprise Co, LTD) with one hundred Newtons load cell and computer connection, was used to obtain force–distance curve from each group of magnets and to measure the magnetic force of magnet attract to orthodontic bracket in various distances (Figure 3.3).

Figure 3.3 Universal testing machine (LLOYD instruments, model LRX, Intro enterprise Co, LTD)

2. **Mounting jigs** – upper mounting jig was designed to hold bracket and lower mounting jig was designed to hold magnets in the centric position to control the direction of force (Figure 3.4).

3. **Electronic digital caliper (KEIBA)** was used for digital measuring the size of magnets to the nearest 0.01 millimeter (Figure 3.5).

Figure 3.4 Mounting jigs – 1) upper mounting jig 2) lower mounting jig

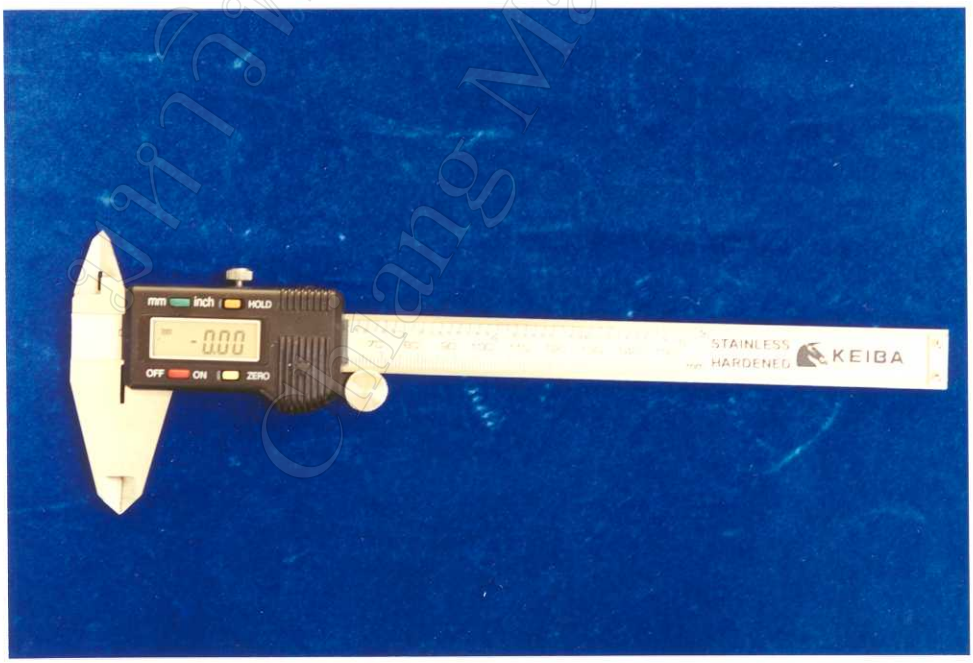

Figure 3.5 Electronic digital caliper (KEIBA)

4. Scanning electron microscope (SEM), model JSM – 840A (JEOL Co, LTD) was used for analyze the morphology of the commercial and orthodontic magnets (Figure 3.6).

5. X-ray microanalysis, Link QX-2000 (OXFORD Co, LTD) attached to scanning electron microscope (SEM), model JSM – 840A (JEOL Co, LTD) was used for analyze the composition of the commercial and orthodontic magnets(Figure 3.7).

6. X- ray diffractometer (XRD), model JDX-8030 (JEOL Co, LTD) was used for analyze the crystal structure of the commercial and orthodontic magnets(Figure 3.8).

Figure 3.6 Scanning electron microscope (SEM), model JSM – 840A (JEOL Co, LTD)

Figure 3.7 X-ray microanalysis, Link QX-2000 (OXFORD Co, LTD)

Figure 3.8 X- ray diffractometer (XRD), model JDX-8030 (JEOL Co, LTD)

Methods

The methods were divided into two parts.

Part I: The forces generated by the commercial and the orthodontic magnets.

Part II: The composition and crystal structure of the commercial and the orthodontic magnets.

Part I: The forces generated by the commercial and the orthodontic magnets.

The samples for force testing comprised of orthodontic and commercial magnets. There were twelve samples of orthodontic magnets in 3.7 millimetersϕx2.0 millimeters (diameter x thickness). The commercial magnets were prepared in four sizes, 3.28x3.28x2.0 millimeters, 6.0x6.0x2.0 millimeters, 8.0x8.0x2.0 millimeters, 10.0x10.0x2.0 millimeters. There were twenty samples for each size. The size of all magnets were measured by electronic digital caliper which could measure to the nearest 0.01 millimeter. The size of the magnets could be deviated $\pm$0.2 millimeters There were five groups of sample as follows:

Table 3.1 Samples distribution in the five groups.

Group	Types of magnet	Sizes of magnet (millimeters)	Numbers of magnet
1	Orthodontic	3.7ϕx 2.0	12
2	Commercial	3.28x3.28x2.0	20
3	Commercial	6.0x6.0x2.0	20
4	Commercial	8.0x8.0x2.0	20
5	Commercial	10.0x10.0x2.0	20

The commercial magnets 3.28x3.28x2.0 millimeters were used for comparing the magnetic force with orthodontic magnets 3.7 millimetersϕ x2.0 millimeters (diameter x thickness). The volume of commercial magnet 3.28x3.28x2.0 millimeters and

orthodontic magnet 3.7 millimetersϕ x2.0 millimeters (diameter x thickness) are relatively equal. The commercial magnets 6.0x6.0x2.0 millimeters, 8.0x8.0x2.0 millimeters, 10.0x10.0x2.0 millimeters were used to analyze the correlations between size and generated force of commercial magnet to orthodontic bracket.

All magnets were fixed in the acrylic resin block at centric position in embedding blocks. The embedding blocks were made from polyvinylchloride (PVC) tube which diameter, height and thickness are 11.0x8.0x1.0 millimeters. These blocks were fitted in the lower mounting jigs. All magnets were embedded in self cure acrylic resin in the centric position of the block. Only superior surface of the magnets were exposed. Orthodontic bracket was embedded in self cure acrylic resin in centric position of upper mounting jig. Only the bracket base were embedded in the resin.

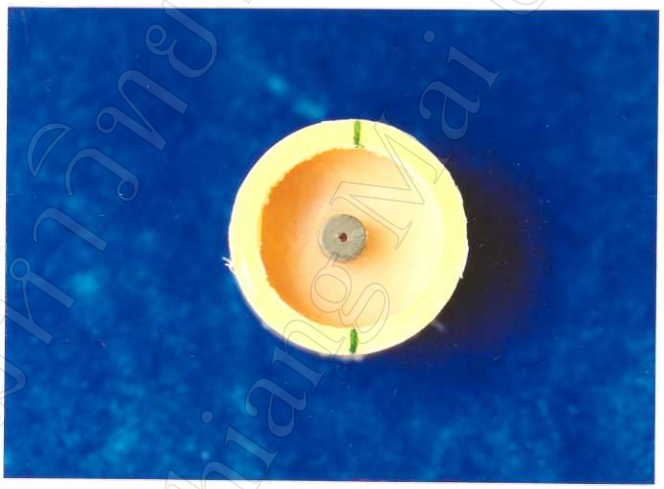

Figure 3.9 The orthodontic magnet embedded in polyvinylchloride tube

Figure 3.10 Four sizes of commercial magnets embedded in polyvinylchloride tubes

Figure 3.11 Orthodontic bracket embedded in centric position of upper mounting jig

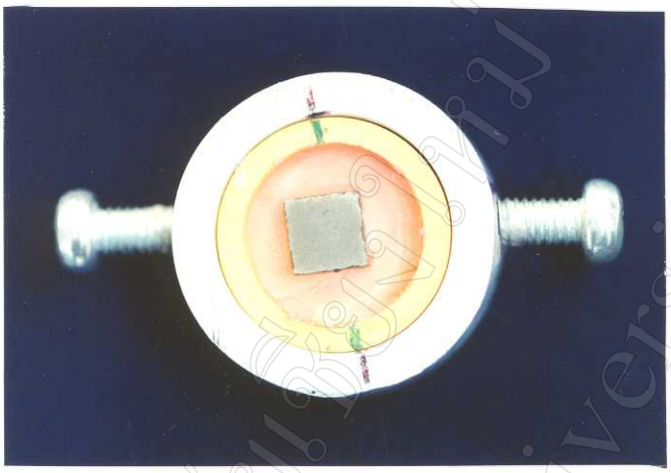

Figure 3.12 Magnet embedded in polyvinylchloride tube fitted in the lower mounting jig

Figure 3.13 The upper and lower mounting jigs were fitted into the upper and lower pneumatic grips

The generated forces of the magnets were determined by using the universal testing machine (LRX, Intro Enterprise Co,) with one hundred Newtons load cell. The upper mounting jig with orthodontic bracket was fitted into the upper pneumatic grip. The lower mounting jig with magnet embedded in acrylic block was fitted into the lower pneumatic grip.

In the initial position, bracket and magnet were contact in centric position. The force was measured by moving the upper pneumatic grip in upward direction employing a crosshead speed of 2.5 millimeters per minute (mm./min) (Fraunhofer *et al.* 1992). The upper pneumatic grip was moved upward until the space between bracket and magnet was equal to 6.0 millimeters. Magnetic force of magnets attracting to orthodontic bracket were recorded digitally and graphically. The magnetic force and distance were recorded in gram and millimeter. The magnetic force versus distance curve of each sample was shown on the computer monitor. The magnetic force of magnet attracting to orthodontic bracket in each distance was shown when selectively marked at the magnetic force versus distance curve in each distance.

Part II : The composition and crystal structure of the commercial and the orthodontic magnets .

The composition of orthodontic and commercial magnets was analyzed by energy dispersive X-ray microanalysis (EDX) and the morphology of orthodontic and commercial magnets was confirmed by scanning electron microscope (SEM). X-ray diffraction analysis (XRD) were used to determine the crystal structures of the magnets.

A. The composition of the commercial and the orthodontic magnets

One sample from group 1 (orthodontic magnet) and group 2 (commercial magnet) were used to investigate the composition. All of magnets being analysed used in this part were removed from the embedded blocks and demagnetized by being burned at 800^{0}C for one hour. All specimens were mounted on aluminum stubs and prepared for SEM by coating with carbon. Photomicrographs were made at a magnification of x450 using a JEOL JSM-840A scanning electron microscope, operated

at 20 kV. After all specimens were photographed, their compositions were analyzed by energy-dispersive analysis. X-ray microanalysis, Link QX-2000 attached to a JEOL JSM – 840A scanning electron microscope was used in this process. The system was operated at 20 kV. The analysis areas were selected by scanning images. Output from energy dispersive analysis was shown by graph of intensity of each component. However, amount of each component could not be detected.

B Crystal structure of the commercial and orthodontic magnets

After their compositions were analyzed, all specimens were removed from aluminum stubs and used to determine the crystal structure by X–ray diffraction analysis (XRD). The specimens were fixed in mounting plate. The crystal structure was analyzed by X-ray diffractometer model JDX-8030. The system was operated at 30 kV, 200 mA. Output from X–ray diffraction analysis (XRD) was shown by graph between intensity (Y axis) and 2θ (X axis). The graph of diffraction data were shown by the peak of d-spacing value. Series of the d-spacing value of each specimen from high to low intensity were used to find out the crystal structure of the magnets by compared with series of the d-spacing value from data base of JCPDS file.

Statistical analysis

1. Descriptive analysis was used to determine means and standard deviations and estimation of means of magnetic forces of magnets attracting to orthodontic bracket in various types and sizes of magnets at ten various distances from magnets to bracket: initial 0.25, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0 millimeters.

2. T-test analysis was used to compare the magnetic forces of the commercial magnets (3.28x3.28x2.0 millimeters) and orthodontic magnets (3.7 millimetersϕx2.0 millimeters) attracting to orthodontic bracket in ten various distances: initial, 0.25, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0 millimeters.

3. One way analysis of variance (ANOVA) was used to analyze the correlations between magnetic force of commercial magnets attracting to orthodontic bracket and

size of commercial magnets by compare the magnetic force among three sizes of commercial magnets (6.0x6.0x2.0 millimeters, 8.0x8.0x2.0 millimeters, 10.0x10.0x2.0 millimeters) in ten various distances: initial, 0.25, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0 millimeters.