CHAPTER III

EXPERIMENTAL

3.1 Chemicals and Materials

3.1.1 Bone cement powder

Three types of the powder supplied by Bonar Polymers Ltd. were used in the present study. The characteristic was shown in Table 3.1.

Table 3.1 Characteristic of powders and reinforcements

Powder	Molecular weight	Residual BPO (%)	Mean particle size (μm)	Density (g/ml)
PMMA (a)	500,000	2.8	15.15 (c)	1.27
PMMA-Co-PEMA (a)	500,000	0.6	12.82 (c)	1.36
$BaSO_4$ (b)	233.34	---	< 2 (d)	3.55
Hydroxyapatite (b)	1004.8	---	< 0.5 (d)	2.99

(a), (b) Supplied by Bonar Polymers Ltd. and Plasma Biotal respectively

(c), (d) By scanning electron microscopy JEOL model TSM-T20 and HITACHI model S-2300, as shown in Figure 4.1-4.4.

3.1.2 Bone cement Liquid

MMA monomer, containing 21.50 ml of liquid, consists of 21.22 ml (approximately 98.70 % by volume) monomethyl methacrylate, 0.21 ml (approximately 1.00 % by volume) N,N-dimethyl-p-totuidine, and 0.065 ml (approximately 0.30 % by volume) hydroquinone which has average of molecular weight distribution as 100.10 .

3.1.3 Silane Coupling Agent

The silane coupling agent used in the present was 3-trimethoxysilylpropylmethacrylate (A-174). It was supplied by Union Carbide Co., Ltd.

3.1.4 Reinforcement

$BaSO_4$, and hydroxyapatite, were used as reinforcements in the present study. They were supplied by Bonar Polymers Ltd. and the Plasma Biotal, respectively. Particle size and particle size distribution curve were shown in Figure 3.1 and 3.2, respectively.

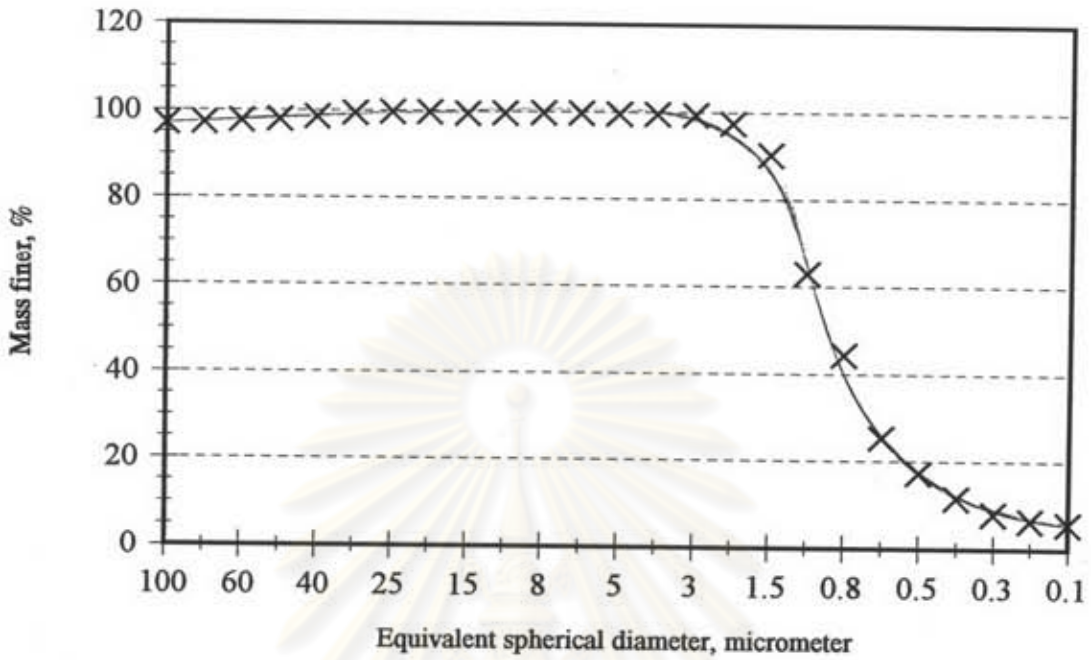

Figure 3.1 A size distribution curve of $BaSO_4$

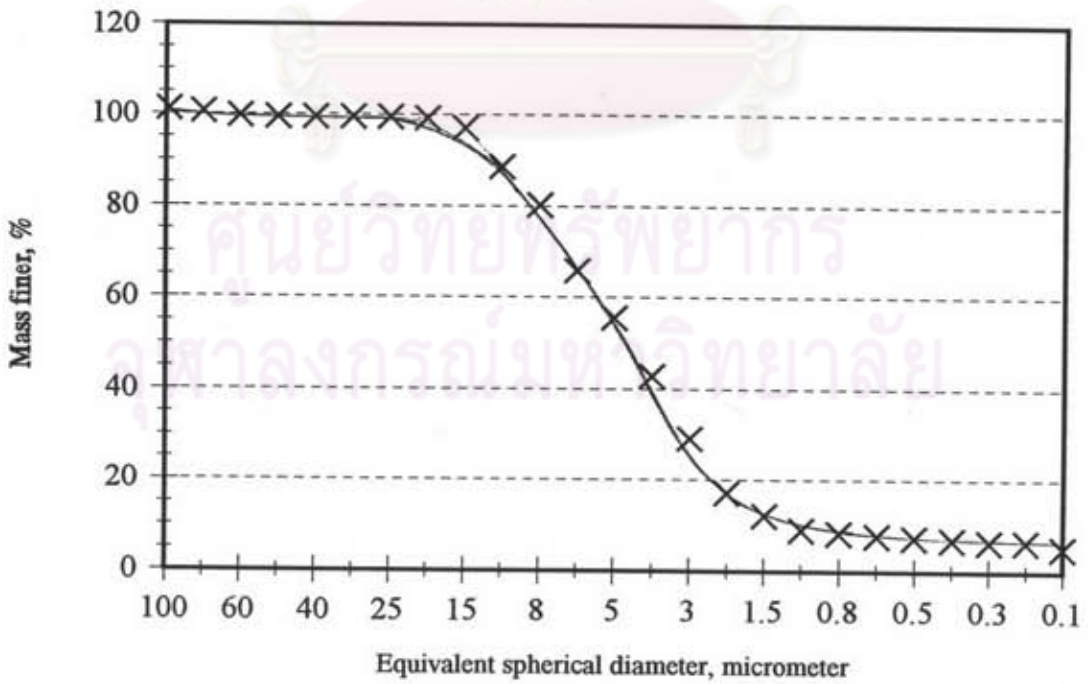

Figure 3.2 A size distribution curve of hydroxyapatite

3.2 Apparatus

Mould for tensile testing has two types of mould as shown in Figure 3.3 and 3.4, were used in the present study. One was the mould for tensile specimens conformed to ASTM D638 having cavity for six specimens. The specimens had 3 mm thickness, and 75 mm total length, while the parallel test sections in the center had a reduced width of 4.5 mm and a length of 20 mm. The other was the mould for tensile specimens conformed to ISO/DIS 6239/1 standard having cavity for 8 specimens. The specimens had 3 mm thickness and 75 mm total length, while the parallel test sections in the center had a reduced width of 4.5 mm and a length of 40 mm.

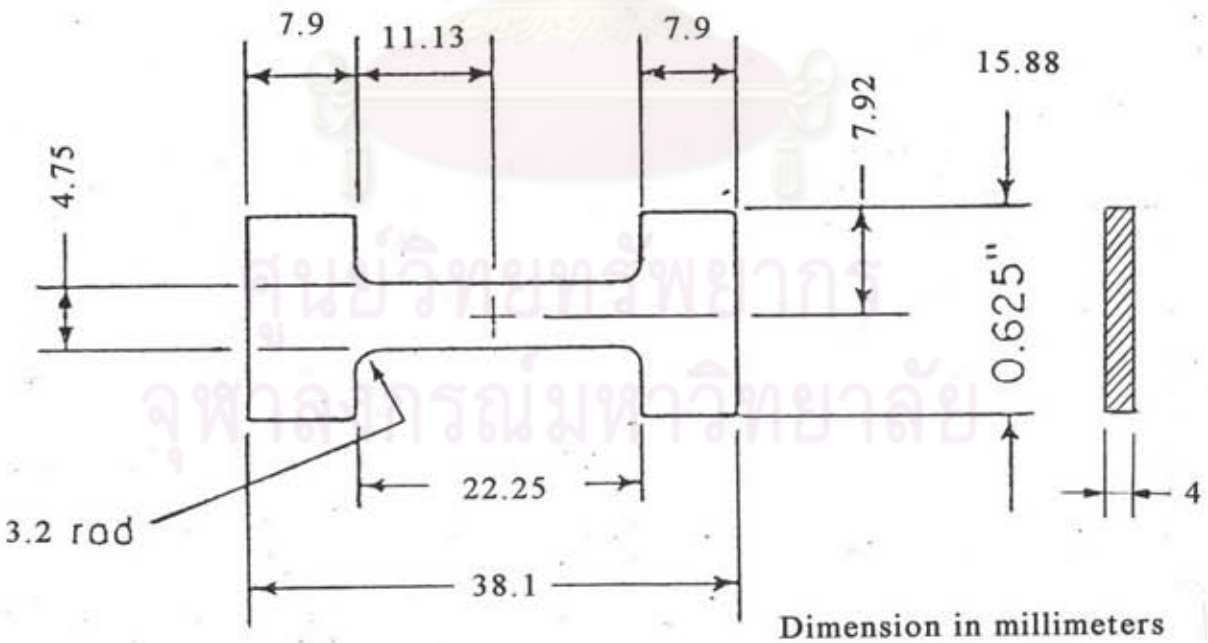

Figure 3.3 Standard dimensions mould of ASTM D638 type V (microtension) designed for tensile test.

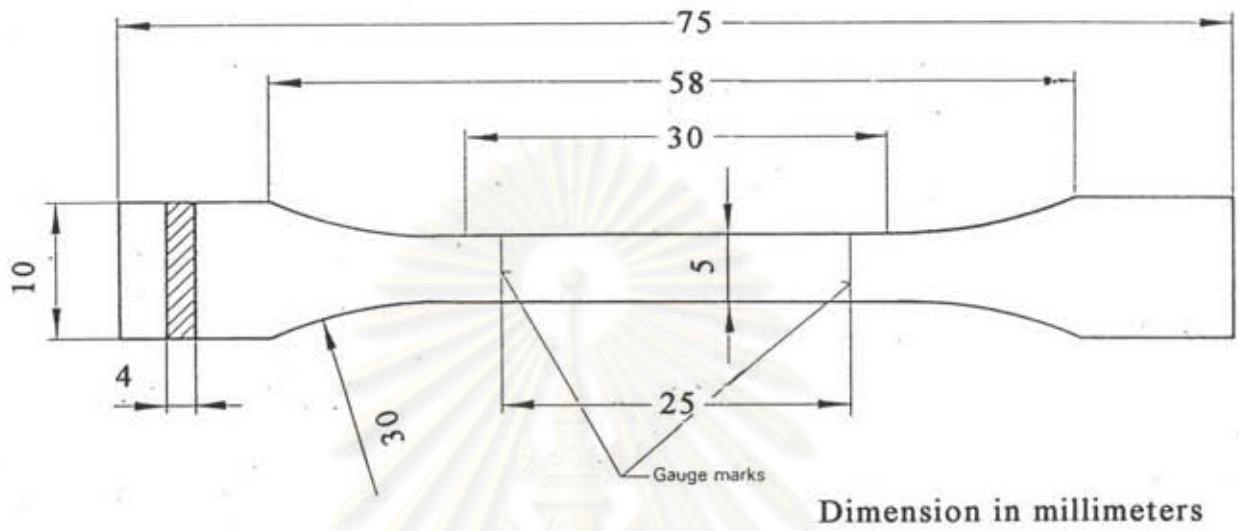

Figure 3.4 Standard dimensions mould of ISO/DIS 6239/1 tensile test specimen

3.3 Machines and Instruments

3.3.1 Particle size analyzer

The Micromeritics model SediGraph 5100 was used for determinated size of reinforcements.

3.3.2 Scanning electorn microscopy (SEM)

The JEOL model JSM-T220A was used for studied shape and distribution of reinforcements, polymers, and composites.

3.3.3 FT-IR spectrophotometer

The Perkin Elmer 1760x was used in for characterized silane coupling agent on hydroxyapatite.

3.3.4 Universal Testing machine

Two models of the machine were used in the present studied. One was Instron model 4206-006 with extensometer 2603-070 having 250 travel. The other was J.J. Lloyd model 500 with extensometer V/25.

3.4 Sample Preparation and Testing Procedure

3.4.1 Particle Size Analysis

Particle size distribution of $BaSO_4$ and hydroxyapatite was determined with Sedigraph 5100 (see also appendix C), using 0.2 % aqeous solution of calgon anionic (sodium hexametaphosphate) as dispersing agent and homogenized with ultrasonic for 30 minutes.

3.4.2 Microstructural Analysis

After tensile testing, the samples were cut at a cross-section part of breaking, sticked on a stub and sputter coated with gold film. Then microstructure analyzed by scanning electron microscope (see

also appendix C) in order to observe distribution of hydroxyapatite in composite.

Shape and size of hydroxyapatite of the bone cement was also determined by SEM analysis.

3.4.3 Silane surface treatment on hydroxyapatite

A portion of hydroxyapatite was surface treated with various percentages of silane coupling agent by dissolving the silane in a 30/70 water/acetone mixture and the resultant liquid with the hydroxyapatite to obtain a slurry. The acetone and water are then remove from the slurry at 100 °C and finally the silane is condensed on the surface of the reinforcements by heat treatment, at 125 °C for 2 hours with rotary evaporator. The coated reinforcements is filtered, washed, dried and sieved through a 60 mesh screen(102). Silane on the hydroxyapatite surface was confirmed by FT-IR spectra.

3.4.4 Silane content determination

Silane-treated on hydroxyapatite sample was weighted accurately about 0.5 g of into a porcelain crucible. The sample heated at 110 °C for 3 hours to determine moisture content and then ignite it at 750 °C for 3 hours and then determine the silane content from the weight loss.

3.4.5 Preparation of Tensile specimens

A composition range of reinforced copolymer bone cements was obtained by using 80/20 PMMA/PEMA copolymer as starting material. Prior to mixing, a known quantity of the copolymer powder containing 1.5 % by weight of benzoylperoxide was replaced and physically blended with an equal weight of $BaSO_4$ or hydroxyapatite. In this way a range of reinforecement concentrations in the powder was investigated. Forty grams of copolymer powder (either with or without reinforcement) and 20 g of monomer (ratio 2:1) containing 2.5 % by volume of N,N-dimethyl-p-toluidine and 10 ppm of hydroquinone were mixed in a beaker until the dough state was reached subsequently transferred into standard tensile test specimen moulds under a compression pressure of 100 psi at room temperature for 25 min. The specimens, then, were removed from the mould and trimmed with emery paper. Every care has been taken to avoid crack initiation. For powder reinforces with various of reinforcement was shown in Table 3.2.

Table 3.2 Chemical composition of bone cement

% Reinforcement base on powder weight	Powder (g)	Reinforcement (g)	Monomer (ml)	% Reinforcement base on total weight
0	40	0	21.5	0.00
5	38	2	21.5	3.33
10	36	4	21.5	6.67
20	32	8	21.5	13.33
30	28	12	21.5	20.00
40	24	16	21.5	26.67

3.4.6 Tensile Testing

Tensile test specimens were carried out on a Instron Universal Tester at room temperature by using an initial grip separation distance of 50 mm. Six specimens of ASTM D638 were deformed to fracture at a cross head speed of 5 mm/min and determined the elongation to fracture (%) or strain at break and the ultimate tensile strength or stress at maximum load by using load cell 100 KN. The tensile Young's modulus was calculated from the linear portion of the stress-strain curve. For strain was measured directly from the specimen with a clip-on extensometer model 2603-070, which was a sutiable with long extension sample. As specimen size and extensometer were not suitable for the bone cement specimens, only tendency of the result could be achieved.

Thus, all tensile test specimens were again carried out on model Lloyd by using an initial grip separation distance of 70 mm and

prepared from the ISO/DIS 6239/1 moulded design. Eight specimens were deformed to fracture with load cell 2.5 KN which was more suitable than the 100 KN because all specimens had a maximum load approximately 2 KN. Strain was measured with a short travel clip-on extensometer.

Both Instron universal testing machine and Lloyd universal testing machine are a same principle to move grip during deforms specimen to fracture. By bottom grip is fixed with a base and only top grip moves up from a base. In addition, both are connected with computer, it can calculate and record other values more accurately in statistics. The data of Instron universal testing machine shown in the Tables were mean value and standard deviation, as shown in Appendix A.