

CHAPTER III EXPERIMENTAL



3.1 Materials

3.1.1 Biodegradable Polymers

3.1.1.1 *Tapioca Starch*

Tapioca starch was kindly supplied by Siam Modified Starch Co.,Ltd.

3.1.1.2 *Polyvinyl alcohol*

Poly (vinyl alcohol) was purchased from Fluka, the degree of hydrolysis was reported to be 97.5-99.5 mol%.

3.1.1.3 *Polycaprolactone*

Polycaprolactone was supplied as a courtesy from Daiseru Chemical Co. Ltd., Japan.

3.1.1.4 *Polylactide*

Polylactide was supplied as a courtesy from Daiseru Chemical Co. Ltd., Japan.

3.1.2 Plasticizers

Ammonium chloride, glycerol, and urea, used as plasticizers, was purchased from Carlo Erba.

3.1.3 Additives

3.1.3.1 *Guar gum*

Guar gum was purchased from Sigma Chemical.

3.1.3.2 *Magnesium stearate*

Magnesium stearate was kindly supplied by Coin Chemical (Thailand) Co.,Ltd.

3.1.4 Diethyl Ether

Diethyl ether ($C_2H_5OC_2H_5$), used as a non-solvent for measuring specific gravity of tapioca starch by the pycnometric technique, was purchased from J.T. Baker.

3.1.5 Salts

The salts used in this study (with corresponding relative humidities at 25°C) were indicated in table 3.1

Table 3.1 Relative humidity of saturated salt solutions at 25 °C.

Salt	Source	Relative Humidity (%)
Lithium Chloride	UNILAB	11
Magnesium Chloride	UNILAB	32
Potassium Carbonate	UNILAB	42
Magnesium nitrate	UNILAB	52
Copper(II)Chloride	AnalaR	67
Sodium Chloride	CARLO ERBA	75

3.1.6 Enzyme

Termamyl 120 α -amylase (120 KNU/g) used in this study was kindly supplied by East Asiatic, Co.,Ltd.

3.2 Experimental Procedures

3.2.1 Preparation of Starch-Based Composite Foam

3.2.1.1 *Tapioca Starch Preparation*

Tapioca starch was dried in an oven at 110 °C for 24 hours to remove moisture. After that, it was stored in a sealed containers until use.

3.2.1.2 Preparation of Starch Batter

Tapioca starch, guar gum (1% by weight of starch) and magnesium stearate (2% by weight of starch) were mixed in the dry state using a Kitchen Aid mixer with a wire whisk attachment. Distilled water was then added to the mixture and the batter was mixed on the medium speed setting for 20 min. The types and amounts of polymers (poly(vinyl alcohol), polycaprolactone, and polylactide) and plasticizers (glycerol, urea, and ammonium) used were also mixed in the starch batter.

3.1.1.3 Compression Molding

Seventy-five grams of starch batter were applied to a mould. Polyethyleneterephthalate-foil was used to cover the sample on both sides to facilitate mould release. The mould was placed in a compression molding machine, the temperature of which was pre-set at 220 °C. After a 2 min holding period, the mould was cooled to room temperature at a rate of 20 ° C/min and the mould content was released.

3.3 Humidity Testing

Starch-based composite foams were equilibrated at 11, 32, 42, 52, 67, and 75 % relative humidity (R.H.) at 25 °C for 9 days prior to mechanical testing. The salts used to control the humidity were LiCl₂ (11%), MgCl₂ (33%), K₂CO₃ (43%), Mg(NO₃)₂ (52%), CuCl₂ (67%), NaCl (75%) and KBr (83%). These choices were made in accordance with the recommendations of the method based on equilibrium sorption rate.

3.4 Mechanical Properties of Starch-Based Composite Foams

3.4.1 Tensile Properties Testing

Tensile strength and percent elongation of the starch-based composite foam were determined by Lloyd tensile tester, LRX series, with the maximum load of 2500 N.

The specimens were cut in to dumbbell-shaped form, according to Type I specified in ASTM D 1623. Tests were carried out using the crosshead speed of 5.2 mm/min and the gauge length of 118 mm. Five specimens were measured for each sample and the results were averaged.

3.4.2 Flexural Properties Testing

Flexural strength and %Elongation of the starch-based composite foam was determined by Lloyd Instrument LRX series of Lloyd tensile tester with the maximum load of 2500 N.

The specimens were cut in rectangular form, according to type I, ASTM D 790-92, using the three-point bending method. Measurements were done using 1.3 mm./min crosshead speed and 50 mm. of support span. Five specimens were measured for each sample and the results were averaged to obtain a mean value.

3.5 Microstructure Characterization

The morphology of the starch-based composite foam was examined by secondary electron images, using a JEOL scanning electron microscope (SEM) Model JSM 5200. The operating voltages were in the range of 1-10 kV. The fractured samples obtained after impact testing were cut about 2 mm below the fractured surface and stuck to aluminium stubs. Prior to examination, the surface of the specimen was coated with a thin evaporated

layer of gold under vacuum for 3 minutes in order to improve conductivity and prevent electron charging on the surface.

3.6 Moisture Determination

The starch-based composite foams was equilibrated at 11, 32, 42, 52, 67, and 75% relative humidity at 25°C for 9 days prior to weight gain tests. The weight of the specimen was recorded by first weighing after kept in humidity chamber. And then the test specimens were conditioned by drying in an oven for 24 hours at 50°C. After that the test specimens were reweighed. The weight gain was calculated as the weight difference and reported as the percent increase of the initial weight. Five specimens of each sample were tested and the results were reported as an average value.

3.7 Water Absorption

Water absorption tests were performed by first weighing a foam equilibrated to 50% RH, adding 100 mL of distilled water to the foams at 25°C, waiting for 10 min, pouring off the water and reweighing the foam.

3.8 Enzymatic Degradation

Ten specimens of each sample were cut into 15.0×50.7 mm squares and weighed (each specimen weighed approximately 0.10-0.20 g) before being placed in a vial. A reaction mixture consisting of 25 ml of 0.05 M acetate buffer (pH 6.0), 1 ml of Termamyl 120 α -amylase (120 KNU/g) and 54 mM $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ was added to the vial which was then heated and shaken in a water bath at 60°C for 3 hours. Specimens were taken out from the vial and collect at 5, 10, 15, 20, 30, 40, 50, 60, 120, and 180 minutes. Then specimens were later washed with distilled water and dried in a hot-air oven at 60 °C for 5 hours to remove traces of moisture before testing. Specimens were then weighed to determine the percentage of weight loss of weight loss.