

CHAPTER III EXPERIMENTAL

3.1 Materials

Tapioca starch was obtained from Siam Modified Starch Co., Ltd. (Pathum-thani, Thailand). The inherent moisture content was 12 to 13% by weight (wt%). High ammonia content natural rubber latex (60% DRC) was kindly supported from Assoc.Prof. Rathanawan Magaraphan. Cellulose pulp as a reinforcing filler which 10-20 micron in width and 1 mm. in length was kindly supported from advanced agro Advance Agro Public Co., Ltd (Thailand). Guar gum was purchased from Sigma Chemicals (Saint Louis, Missouri, USA). Magnesium stearate was donated from Coin Chemical Co., Ltd. (Bangkok, Thailand). Guar gum was used as a thickening agent, while magnesium stearate was used as a mold-releasing agent. The salts used (with their corresponding relative humidity (RH) levels at 25°C were LiCl (11.3%), MgCl₂ (32.8%), K₂CO₃ (43.2%), Mg(NO₃)₂ (52.9%), and NaCl (75.3%). These salts were purchased from Ajax Chemicals (New South Wales, Australia) and Fisher Chemicals (Loughborough, England). Terix X10 and termamyl 120 α-amylase (120 KNU/g) was supplied as a courtesy by East Asiatic Co., Ltd (Thailand).

3.2 Methodology

3.2.1 Preparation of Diluted Natural Rubber Latex (NRL)

High ammonia content natural rubber latex with 60% dry rubber content (DRC) was diluted into expected ratio by adding with distilled water. In order to prevent the coagulation of natural rubber latex while mixing with other ingredient, Terix X-10, stabilizer was required to add. The amount of added stabilizer was function as the added distilled water.

3.2.2 Preparation of Starch-Based Batters

Native tapioca starch or Octyl tapioca starch, guar gum and magnesium stearate were first mixed in the dry state. Required amount of diluted natural rubber latex was then added to the mixture and the batter was further mixed until the mixture was homogeneous. Finally, glycerol, plasticizer was then added to the mixture and further mixed until the mixture was homogeneous. In order to evaluate the effect of natural rubber content on mechanical properties of starch-based foam, the batter formulation was varied as summarized in Table 3.1

Table 3.1 Batter formulations for preparing starch-based and starch-based foams

Component	Weight(g)			
	SF ^a	10%NR/SCF ^b	30%NR/SCF ^b	50%NR/SCF ^b
Tapioca starch	100	100	100	100
Natural rubber latex	-	17.33	54	90
Distilled water	85	78.068	63.4	49
Glycerol	5	5	5	5
Magnesium stearate	2	2	2	2
Guar gum	1	1	1	1
Terix X10	-	0.132	0.108	0.084
Baking time(S)	150	170	190	210

^adenotes starch-based foam

^bdenotes Natural rubber/tapioca starch composite foams and Natural rubber content calculated based on 60% dried rubber content of natural rubber latex.

In order to evaluate the effect of cellulose pulp content on properties of as prepared foam, the amount of cellulose pulp were dispersed in diluted natural

rubber latex prior to mix with other ingredients and the batter ratio each component were summarized in Table 3.2

Table 3.2 Batter formulations for preparing cellulose pulp reinforced natural rubber/tapioca starch composite foams

Component	Weight(g)		
	30%NR/SCF with 1% fiber	30%NR/SCF with 3%fiber	30%NR/SCF with 5% fiber
Tapioca starch	100	100	100
Natural rubber latex	54	54	54
Distilled water	63.4	63.4	63.4
Glycerol	5	5	5
Magnesium stearate	2	2	2
Guar gum	1	1	1
Terix X10	0.108	0.108	0.108
Cellulose pulp	1	3	5
Baking time(S)	200	210	220

3.2.3 Baking process

80 g of the as-prepared starch-based batters (see Table 3.1 and 3.2) was applied to a picture-frame mold. The size of the mold cavity was 167.3 mm in length, 130.1 mm in width, and 3.1 mm in depth. Clean poly(ethylene terephthalate) sheets were used to cover the sample on both sides to facilitate mould release. A compression press (Wabash, V50H) was used to prepare starch-based foam from the as-prepared starch-based batters. Each molding was placed between the platens, the temperature of which was fixed at 220°C. Slight pressure was applied just to close the mold. After 150-210 seconds, the molding was cooled down to 50°C at a cooling

rate that was fitted well with an exponential decay with a time constant of around 7 min and the as-prepared starch-based foam was demolded.

3.3 Determination of Moisture Content

Conditioning jars having specific RH levels of 11.3, 32.8, 43.2, 52.9, and 75.3% were prepared by filling the jars with saturated, aqueous solutions of LiCl, MgCl₂, K₂CO₃, Mg(NO₃)₂, and NaCl salts, respectively (according to ASTM E104-85 standard test method). Pre-dried starch-based foam specimens (13*70*3.1 mm.) were then conditioned on wire grids over these solutions in these jars at a fixed temperature of 25°C for a total observation period of 1 week. The weight of each specimen was recorded after being conditioned in a respective conditioning jar for a specified observation time and the initial weight, divided by the initial weight and multiplied by 100. The results were reported as the average value from measurements of at least five specimens.

3.4 Physical and Mechanical Property Measurements

The specimens used to investigate the effects of moisture content, natural rubber content and cellulose pulp content on their physical and mechanical properties were conditioned at 11.3, 32.8, 43.2, 52.9, or 75.3%RH for at least 7 days. Bulk density starch-based foam specimens were calculated from the ratio between weight and volume. Flexural properties for starch-based foams were determined using a universal testing machine (Lloyd, LRX series), with the maximum load of 500 N and the span of 50 mm. The specimens were rectangular in shape, cut according to ASTM D790-92 standard test method. The probe was lowered onto each specimen until a load of 0.5 N was reached and then was further lowered at a speed of 1.3 mm/min. Specific flexural strength, Maximum flexural strain, and Specific flexural modulus were determined. The results were reported as the average value from measurements of at least five specimens.

3.5 Microstructures Observation

The morphology of the starch-based foam was examined using a scanning electron microscope (SEM) (JEOL, JSM 520-2AE). The operating voltage used was 10 kV, magnification = 35X, 350X. Selected, fractured specimens obtained after mechanical property measurement were cut about 2 mm below the fractured surface and mounted on aluminum stubs. Prior to examination, the surface of the specimen was coated with a thin layer of gold under for 3 minutes in order to improve the conductivity and to prevent electron charging on the surface.

3.6 Enzymatic Degradation

Selected pure starch foam and cellulose pulp reinforced natural rubber/ tapioca starch composite foams were also tested for their enzymatic degradability (Allenza, Schollmeyer, & Rohrbach, 1990). Each foam specimen was weighed before being placed in a test vial. A reaction mixture containing 25 ml of 0.05 M acetate buffer (pH 6.0), 1 ml of Termamyl α -amylase (120 KNU/g), and 54 mM of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ was added to the vial, which was then warmed at 60 °C, while mechanically shaken, in a shaking water bath for 3 h. Specimens were collected after different reaction time periods, ranging from 5 to 180 min, prior to being washed with distilled water and dried at 60 °C for 5 h. Their weights were then recorded and used to determine the percentage of weight loss. It should be noted that the specimens used in the enzymatic degradation tests were of equal size (i.e. cut into rectangular pieces of $1 \times 2 \text{ cm}^2$, with the thickness being fixed at 3.1 mm).

$$\frac{W_f - W_i}{W_i} \times 100 = \text{Weight loss}$$

Where:

W_f = Weight of specimen after condition.

W_i = Weight of specimen prior condition.