CHAPTER III
EXPERIMENTAL

3.1 Materials

Tapioca starch was the unmodified grade obtained from Siam Modified Starch Co., Ltd. (Pathumthani, Thailand). The inherent moisture content was 12 to 13% by weight (wt%). Jute and flax fibers were purchased locally and used as-received. Guar gum was purchased from Sigma Chemicals (Saint Louis, Missouri, USA). Magnesium stearate was donated from Coin Chemical Co., Ltd. (Bangkok, Thailand). The salts used (with their corresponding relative humidity (RH) levels at 25°C) were LiCl (11.3%), $MgCl_2$ (32.8%), K_2CO_3 (43.2%), $Mg(NO_3)_2$ (52.9%), and NaCl (75.3%). These salts were purchased from Ajax Chemicals (New South Wales, Australia) and Fisher Chemicals (Loughborough, England).

3.2 Methodology

3.2.1 Preparation of Fibers

Natural cellulose fibers, jute and flax, were characterized for their diameter by microscopic technique. The average fiber diameter was calculated from measurements of at least 100 individual fibers. The fibers were then cut into staples of specific aspect ratios (i.e., specific length-to-diameter ratio). The density and the tensile properties of the as-received fibers were investigated based on ASTM D1577-90 and ASTM D3822-95a standard test methods, respectively. Table 3.1 summarizes the characteristic information of the as-received jute and flax fibers.

3.2.2 Preparation of Starch-Based Batters

Unmodified tapioca starch, guar gum, magnesium stearate, and cellulose fibers were first mixed in the dry state. Distilled water was then added to the mixture and the batter was further mixed until the mixture was well-dispersed. In order to evaluate the effect of fiber content on mechanical properties of SCFs, the batter formulation was varied as summarized in Table 3.2; in order to evaluate the

effect of fiber aspect ratio on mechanical properties of SCFs, the length of the fibers was varied as summarized in Table 3.1 (for a fixed fiber content of 10 wt%); and finally, in order to evaluate the effect of fiber orientation on mechanical properties of SCFs, continuous flax fibers were pre-oriented in the longitudinal, random, and transverse directions in the mold before the application of the batter (for a fixed fiber content of 10 wt%).

Table 3.1 Physical and mechanical properties of jute and flax fibers used

Fiber type	Density (g/cm^3)	Tensile strength (MPa)	Elongation (%)	Modulus (MPa)	Average diameter (μm)	Length (mm)	Aspect ratio (L/D)
Jute	0.268	425.3	1.9	2.3×10^5	69. 6	2 10 20	28.75 143.76 287.52
Flax	0.294	663.0	5.0	1.6×10^5	210.3	6 30 60	28.53 142.67 285.33

Table 3.2 Batter formulations for preparing starch-based and starch-based composite foams

Component	Weight (g)			
	SF[1]	SCF[2] with 1% fibers	SCF[2] with 5% fibers	SCF[2] with 10% fibers
Tapioca starch	100	100	100	100
Natural fibers	-	1	5	10
Distilled water	85	85	85	85
Magnesium stearate	2	2	2	2
Guar gum	1	1	1	1

[1] denotes starch-based foam

[2] denotes starch-based composite foam

3.2.3 Baking process

85 g of the as-prepared starch-based batters (see Table 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 SCFs from the as-prepared starch-based batters. Each molding was placed between the platens, the temperature of which was fixed at 220°C. After 150 seconds, the molding was cooled down to 50°C at a cooling rate of ca. 20°C·min^{-1} and the as-prepared SCF 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, $MgCl_2$, K_2CO_3, $Mg(NO_3)_2$, and NaCl salts, respectively (according to ASTM E104-85 standard test method). Pre-dried SCF specimens were then conditioned 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 every 24-hour period. The moisture content (in percent) of SCFs was then taken as the difference between the weight of the specimen 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 SCF specimens used to investigate the effects of moisture content and fiber content on their physical and mechanical properties were conditioned at 11.3, 32.8, 43.2, 52.9, or 75.3 %RH for 3 days, while the SCF specimens used to investigate the effects of fiber aspect ratio and fiber orientation on their physical and mechanical properties were conditioned at 43.2%RH for 3 days. Bulk density of

SCF specimens was calculated from the ratio between weight and volume. Flexural properties for SCF specimens were determined using a mechanical 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. Flexural strength, flexural strain at maximum force, and flexural modulus of elasticity were determined. The results were reported as the average value from measurements of at least five specimens.

3.5 Microstructural Observation

The morphology of the SCFs was examined using a scanning electron microscope (SEM) (JEOL, JSM 520-2AE). The operating voltage used was 10 kV. 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 vacuum for 3 minutes in order to improve the conductivity and to prevent electron charging on the surface.