

## CHAPTER III

### EXPERIMENTAL

#### 3.1 Materials

Table 3.1 Chemicals

Chemicals	Grade	Source
Acetone	RPE	AnalytiCals
Acetonitrile	AR	BDH
Benzoyl peroxide	GR	MERCK
Cyanuric chloride	purum	Fluka Chemika
Diallylamine	purum	Fluka Chemika
Methanol	RPE	AnalytiCals
Potassium carbonate	AR	BDH
Potassium iodide	RPE	AnalytiCals
Potassium permanganate	GPR	BDH
Sodium hydroxide	GR	MERCK
Sodium thiosulfate	GR	MERCK
Starch (Corn flour)	-	CPC/AJI(THAILAND)LTD.
Styrene monomer	Commercial	SRC
Sulfuric acid	R	'BAKER ANALYZED'
Toluene	GR	MERCK

#### 3.2 Apparatus and Instruments

FT-IR Spectrophotometer, Perkin-Elmer, 1760x

Grinder Machine, Retsh, Type S1

Hydraulic Compression Moulding Machine, Labtech, LP 20, 20 MT

Impact Testing Machine, Zwick/Materialprufung

Universal Testing Machine, Instron, 4206-006, 100 KN

Scanning Electron Microscope, Jeol, JSM T220

Siever Machine, Retsh, Type Vibro  
 Thermal Analysis Interments, Shimadzu, DT-30

### 3.3 Methods

#### 3.3.1 Extraction of the Coir Fiber

Normally, dehusking of nuts is done manually at coconut estates or at fiber mills. Separation of the fiber from the husk is facilitated by sodium hydroxide solution treatment called "retting" and then beating the husk. The unfibrous parts of husk were decomposed so that the extraction could be performed easily. The process consists of soaking the husk in 0.1 M NaOH solution for 1 week and beating the husks to extract the fiber from the husk.

#### 3.3.2 Delignification of the Coir fiber

Lignin was damaged and removed from the coir fiber by treating with 0.1 M NaOH solution at 100°C. The batch consisted of 200 g of the coir fiber impregnated in 0.1 M sodium hydroxide solution of 1.5 dm<sup>3</sup> in a 2 dm<sup>3</sup> beaker. The residual lignin content of the coir fiber was treated at 5 levels by changing the time of treating the coir fiber from 1 to 5 hours. The procedure was performed according to the following Table.

Table 3.2 Time of Treatment of the Coir Fiber

Sample	Time to treat (hour(s))
1	1
2	2
3	3
4	4
5	5



### 3.3.3 Determination of the Residual Lignin Content of the Coir Fiber

The Kappa Number of pulp could be applied to determine the relative degree of delignification of the coir fiber. The Kappa Number is the volume (in milliliters) of 0.1 N potassium permanganate solution consumed by one gram of moisture-free pulp under the conditions specified in TAPPI T 236 cm-85. This procedure consisted of:

1 Prior to weighing the test samples, it was conditioned for at least 20 min in the atmosphere near the balance.

2 The pulp was weighed to the nearest 0.0001 g so that the amount of pulp would consume approximately 50% of the potassium permanganate solution. The permanganate consumption must be between 30 and 70%. At the same time, a second sample was weighed and it was determined its moisture content in accordance with TAPPI T 210 "Sampling and Testing Wood Pulp Shipments for Moisture".

3 The test specimen was disintegrated in 500 cm<sup>3</sup> or less of distilled water until free of fiber clots and undispersed fiber bundles. Avoid methods of disintegration which extensively cut the fiber.

4 The disintegrated test specimen was transferred to a 2 dm<sup>3</sup> reaction beaker which was rinsed with enough distilled water to bring the total volume to 795 cm<sup>3</sup>. The temperature of distilled water should be at least at 25.0±0.2°C.

5 The beaker was placed in a constant temperature bath adjusted so that the reaction temperature stayed at 25.0±0.2°C during the entire reaction. The suspension was continuously stirred so as to produce a vortex about 25 mm deep but not fast enough to introduce air into the mixture.

6 100.0±0.1 cm<sup>3</sup> of potassium permanganate solution was pipetted and 100 cm<sup>3</sup> of the sulfuric acid solution was added into a 250 cm<sup>3</sup> beaker. While this mixture was controlled at 25°C, it was immediately added to the disintegrated test specimen, a stopwatch was simultaneously started. The beaker was rinsed by using not more than 5 cm<sup>3</sup> of distilled water, and the washing was added to the reaction mixture. The total volume of the reaction should be 1000±5 cm<sup>3</sup>.

7 At the end of reaction time of exactly 10.0 min, The reaction was stopped by adding 20 cm<sup>3</sup> of potassium iodine solution from a graduated cylinder.

8 Immediately after the addition of KI solution, the free iodine was titrated with the sodium thiosulfate solution, a few drops of starch indicator was added towards the end of the reaction to determine the end point.

9 A blank determination was carried out exactly the same method as described above only without the pulp.

### 3.3.4 Correlation of Tensile Properties and Residual Lignin Content of the Coir Fiber

The 5 levels of residual lignin content of the coir fiber were determined to obtain the tensile strength, tensile strain and Young's modulus according to ASTM D 638-89<sup>E1</sup>. Tensile properties of ten specimens of each group at each level of the residual lignin content were determined. The closest diameter of each coir fiber in this procedure was strictly chosen so as to accomplish the confidence result.

### 3.3.5 Synthesis of 2-Diallylamino-4,6-dichloro-s-triazine, the Coupling Agent

A fine slurry of cyanuric chloride was prepared by running a thin stream of a hot solution of one mole (184.5 g) of cyanuric chloride in 400 cm<sup>3</sup> of hot acetone into 600 cm<sup>3</sup> of well stirred ice-water. One mole (97 g) of diallylamine and one equivalent (69.16 g) of potassium carbonate was slowly add into the above solution at 0-5°C and the mixture was stirred with a mechanical stirrer for one hour. The product was filtered, washed to be free of chloride and sodium ions with cold water, and it was pressed as dry as possible in a pressure filter. Recrystallization of the product was obtained in acetonitrile-water solution for the more purified coupling agent. The product could be dried in vacuum oven for 24 hours. The IR spectrophotometry was performed to confirm the product.



### 3.3.6 The Coupling Reaction of Coupling Agent with the Coir Fiber

The coir fiber was milled by a grinder, Retch type S1, at 800 rpm for 30 minutes and sieved to obtain 200 mesh powder. Such coir powder was soaked in 8% coupling agent-acetone solution for 5 minutes. After the coir powder was dried in the room atmosphere, it was soaked again with 0.1 M NaOH solution for 1 minute. The coir powder was then dried at 50°C for 1 hour in a vacuum oven, after that the treated coir powder was rinsed several times with distilled water and acetone. Finally, the treated coir powder was soxhlet extracted with acetone for 24 hours to remove the coupling agent not covalently bonded onto the powder surface and was dried in a vacuum oven. The product was verified by a Diffuse Reflectance Technique in an FT-IR spectrophotometry (50 scans) to determine the peaks of the coupling agent.

### 3.3.7 Copolymerization of the Coupling Agent and Styrene Monomer

A 100 cm<sup>3</sup> round bottom flask was placed with 0.05 mole (12.25 g) of the coupling agent dissolved in 25 cm<sup>3</sup> of toluene and mixed with 0.05 mole (5.74 cm<sup>3</sup>) of styrene monomer dissolved in 10 cm<sup>3</sup> of toluene. The reaction flask was equipped with a condenser and was heated to 50°C. The solution of benzoyl peroxide (0.074 g in 15 cm<sup>3</sup> of toluene) was quickly added to the reaction mixture. The N<sub>2</sub> gas was purged above the reaction mixture at all time of the reaction which required 5 hours. The product was precipitated by adding 10 cm<sup>3</sup> of MeOH, collected and rinsed several times with MeOH to remove the unreacted monomer. The copolymer product was soxhlet extracted for 24 hours with toluene to remove polystyrene homopolymer from the copolymer. The copolymer was confirmed by FT-IR.

### 3.3.8 Graft Copolymerization of Styrene Monomer onto Coir Powder Treated with the Coupling Agent

A mixture of 1.0 g treated coir powder, 5.75 cm<sup>3</sup> of styrene monomer, 0.0745 g of benzoyl peroxide and 50 cm<sup>3</sup> of toluene was placed in a round-bottom flask equipped with a condenser. Graft copolymerization reaction took place at 50°C under the N<sub>2</sub> atmosphere. After 5 hours, the reaction was stopped by an addition of MeOH. The product was collected and rinsed several times with acetone. The homopolymer (ungrafted one) was removed by soxhlet extraction with toluene for 24 hours. Finally the grafted product was dried in a vacuum oven to constant weight.

The product was confirmed by a Diffuse Reflectance Technique in the FT-IR (50 scans) Spectrophotometry.

### 3.3.9 Treatment of the Coupling Agent on the Coir Fiber Surface

The coir fiber used in this experiment was obtained from Kanchanaburi province. Chopped strand coir fiber at any desirable length was soaked in a solution of the coupling agent at 2%, 5% and 8% acetone solution for 5 minutes. After being dried for 15 minutes in the room atmosphere, the coir fiber was impregnated in an aqueous solution of 0.1 M NaOH for 1 minute. The coir fiber was then dried for 1 hour at 50°C in a vacuum oven. After the reaction was completed, then the treated coir fiber was rinsed several times with distilled water and acetone, respectively, to remove NaOH and the coupling agent not covalently bonded to the fiber surface and then it was dried in the room atmosphere.

### 3.3.10 Preparation of the Composites

The chopped coir fiber of 1 cm length was randomly spread on the metal frame sandwiched with metal flat plates (the mould). The mould was placed on a platen and was pressed at a pressure of 2,000 psi for 1 minute. After removing the plates from the press, a mixture of the unsaturated polyester resin and the hardener (methyl ethyl ketone peroxide) was poured on the prepared coir fiber in the metal frame. The mould was pressed again at 2,000 psi and 50°C for 30 minutes to achieve the gelling composite. The composite was removed from the



mould and it was left for a post-curing for 48 hours at the room temperature. The preparation of the composite sheets with other lengths of coir fiber in the presence and absence of the coupling agent was carried out in a similar manner. The load amount of the coir fiber was performed at 20% (wt/wt) at all conditions of the coupling agent concentration and coir fiber lengths, and at 10%, 30%, 40% and 50% for 8% treated coir fiber and 1 cm coir fiber length only. Table 3.3 shows various conditions of composite sheets carried out in this work.

Table 3.3 Conditions of the Composite Sheets

		Coir Fiber Length			Glass Fiber Length
untreated	treated	[Coupling Agent]			
		2%	5%	8%	
1 cm	1 cm	√	√	√	1 cm
5 cm	5 cm	√	√	√	5 cm
10 cm	10 cm	√	√	√	10 cm
ordinary	ordinary	√	√	√	continuous

### 3.3.11 Differential Thermal Analysis of the Composites

The specimens consisted of pure UP, untreated coir fiber-UP composite and 8%-treated coir fiber-UP composite were investigated with a Shimadzu Thermal Analysis Instrument DT-30, operated on Differential Thermal Analysis (DTA) mode from room temperature to 280°C, heating rate at 10°C/min, sensitivity at +25 μV, under N<sub>2</sub> atmosphere and in the sealed aluminum cell.

### 3.3.12 Scanning Electron Microscopy of the Composites

Impact fracture surface of the specimens consisted of untreated coir fiber- and 8%-treated coir fiber-UP composite were investigated

with a Jeol JSM T220 Scanning Electron Microscope, operated at 20 eV at a tilt angle of 30°. The specimens were coated with gold to prevent electrical charging.

### 3.3.13 Measurement of the Mechanical Properties

#### 3.3.13.1 Tensile Properties

The tensile strength of the neat polyester and composites was determined according to ASTM D638-89<sup>E1</sup> using the dumbbell shape specimen as illustrated in Figure 3.1. The testing machine is an INSTRON model 4206-006. The tensile testing conditions were as follows:

Temperature:	23±2°C
Relative humidity:	50±5 %
Speed of testing:	5±25% mm/mm
Distance between grips:	115 mm
Gage length	50 mm

The tensile stress at yield was calculated from the applied load at the yield point and the minimum cross-sectional area of the specimen was computed as follows:

$$\text{Tensile Strength} = \text{Load at Yield (N)} / \text{Area (m}^2\text{)} \text{-----(3.1)}$$

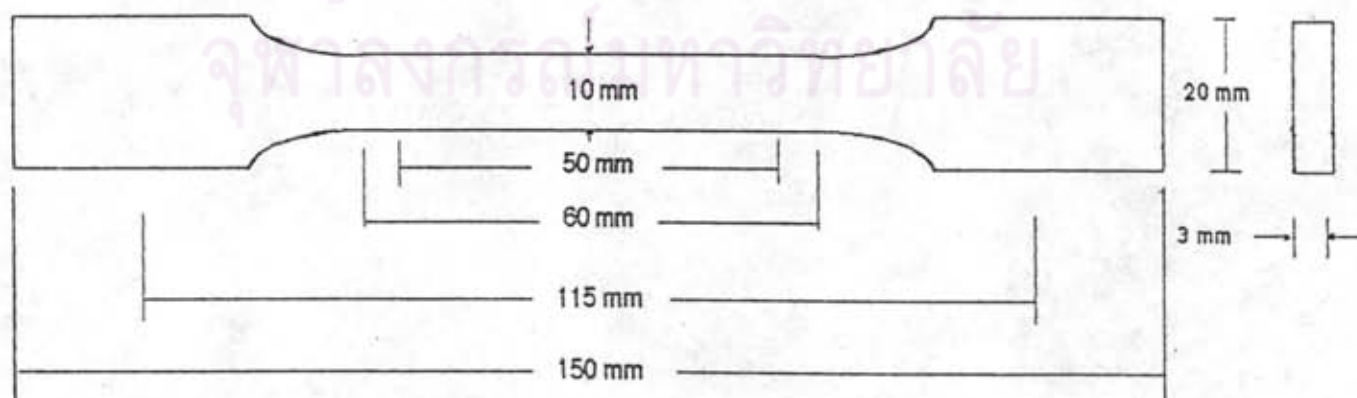


Figure 3.1 Dimension of tensile test specimen



The tensile strain was calculated from the increment of displacement divided by the specific gage length (50 mm) and the Young's modulus was calculated from stress divided by strain at an appropriate interval.

### 3.3.13.2 Compressive Properties

The compressive strength, strain and modulus of the neat polyester and composites were determined according to ASTM D 695M-90 by using an INSTRON Universal Testing Machine and the standard test specimens were in a dumbbells shape as illustrated in Figure 3.2. The machine parameters of the test were as follows:

Temperature:	23±2°C
Relative humidity:	50±5 %
Speed of testing:	1±25% mm/mm
Platen separation:	80 mm
Gage length	80 mm

The compressive strength can be calculated by the following equation:

$$\text{Compressive Strength (N/m}^2\text{)} = \frac{\text{Maximum Compressive Load (N)}}{\text{Area(m}^2\text{)}} \quad \text{---(3.5)}$$

$$\text{The compressive strain (mm/mm)} = (l-l_0)/l_0 \quad \text{-----(3.6)}$$

where

$l$  = the distance between the gage marks, and  
 $l_0$  = the original distance between the gage marks.

### 3.3.13.3 Flexural Properties

The flexural strength, strain and modulus of the neat polyester and composites were determined according to ASTM D 790-90. The rectangular specimens have an average dimension of 25 mm width, 3.2 mm thickness and 80 mm length as illustrated in Figure 3.3. The test was performed with the span-to-depth ratio of 16:1. The machine parameters

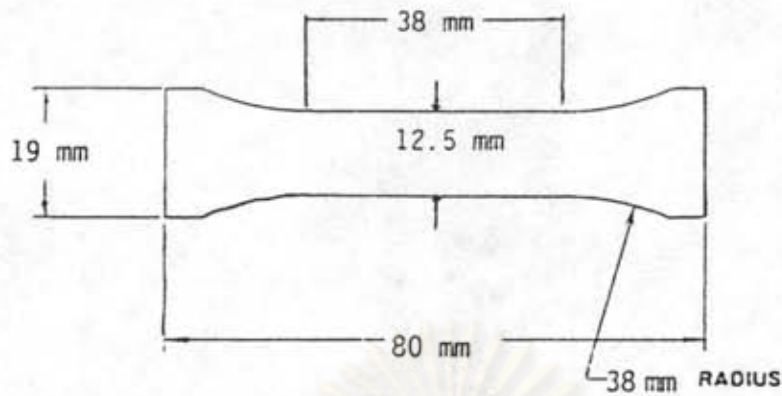


Figure 3.2 Dimension of compressive test specimen

for the test were as follows:

Temperature:	$23 \pm 2^{\circ}\text{C}$
Relative humidity:	$50 \pm 5\%$
Speed of testing:	$1.3 \pm 25\%$ mm/mm
Span length	50 mm

The flexural strength can be calculated by the following equation:

$$\text{Flexural Strength} = 3PL/2bd^2 \quad \text{-----}(3.2)$$

where

- P = maximum load at rupture, N,
- L = span length, m,
- b = width of specimen, m, and
- d = depth of specimen, m.

The flexural strain was calculated from the following equation:

$$r = 6Dd/L^2 \quad \text{-----}(3.3)$$

where

- r = maximum strain in the outer fiber, mm/mm,
- D = Maximum deflection of the center of the beam, mm,
- L = support span, mm, and
- d = depth, mm.



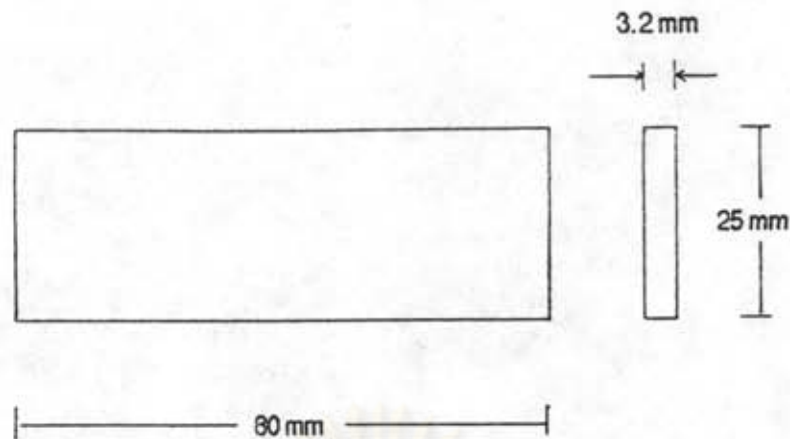


Figure 3.3 Dimension of flexural test specimen

#### 3.3.13.4 Impact Strength

The impact strength was determined according to ASTM D 256-90b using the rectangular specimens as illustrated in Figure 3.4. The testing was performed by an Izod impact testing machine. The machine parameters of the impact test were listed below:

Pendulum capacity	=	12.7 J
Depth of specimen	=	10.16 mm

The impact strength was calculated by the equation below:

$$\text{Izod impact strength} = \frac{\text{Impact energy(J)}}{\text{Width of specimen(m)}} \quad \text{-----}(3.7)$$

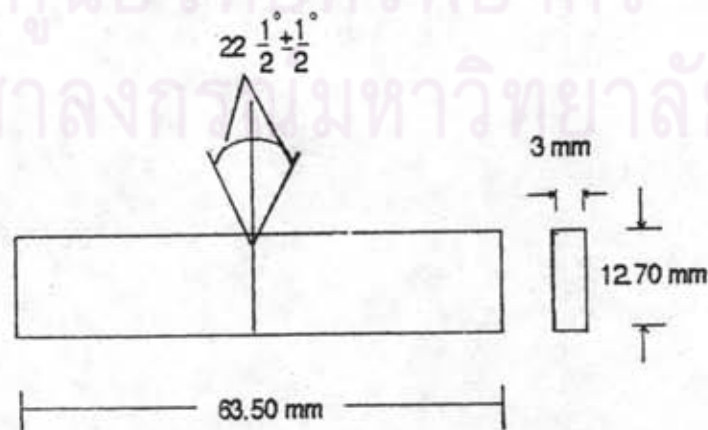


Figure 3.4 Dimension of izod impact test type specimen