



CHAPTER II

EXPERIMENT

2.1 Synthesis of 2-diallylamino-4,6-dichloro-1,3,5-triazine as Coupling agent

Chemical reagents

<u>Chemical</u>	<u>M.W.</u>	<u>Manufacturer</u>	<u>Grade</u>
Cyanuric chloride	184.41	E. Merck	AR. Grade
Diallylamine	97.16	E. Merck	AR. Grade
Potassium carbonate	138.21	E. Merck	AR. Grade
Acetone	58.08	E. Merck	AR. Grade
Carbon tetrachloride	153.82	Univar	AR. Grade

2.1.1 Preparation of Starting Materials

Cyanuric chloride and diallylamine employed as starting materials were purified by recrystallisation from carbon tetrachloride and by distillation over potassium hydroxide. Organic solvents were distilled and dried over molecular sieve 5A before use.

The weighing of all materials used in the preparation of coupling agents was carried out with a Mettler model H 43 digital analytical balance with a precision of 0.1 mg. Melting points were determined with an Electrothermal melting points apparatus.

2.1.2 Preparation of 2-diallylamino-4,6-dichloro-1,3,5-triazine, the Coupling agent

Two synthesis methods namely, Thurston method (12) and

Modified method were used in this experiment.

2.1.2.1 The Thurston Method (12)

To a fine slurry of cyanuric chloride, prepared by pouring a thin stream of a hot solution of cyanuric chloride (184.5 g., 1 mole) in 400 ml of hot acetone into 600 ml of well-stirred ice-bath, diallylamine (97.16 g., 1 mole), sodium hydroxide (40 g., 1 mole) and anhydrous potassium carbonate (138.2 g., 1 mole) were added. The mixture was then stirred at 0-5°C for 2 hours. The product was filtered, washed with cold water and dried by suction. The crude product was recrystallized from acetone-water to yield the compound as the colorless crystal. The yield of this synthesis was 66.1 %.

2.1.2.2 The Modified Method

A stirred mixture of cyanuric chloride (3.8 g. 0.02 mole) and anhydrous potassium carbonate (2.80 g., 0.02 mole) in 20 ml dried acetone was placed in a 100 ml two-necked flask connected with a dropping funnel and a calcium chloride guard tube. The mixture was added dropwise at room temperature to a solution of diallylamine (2.0 g., 0.02 mole) in 15 ml dried acetone through a dropping funnel over a period of 0.5 hour. The resulting mixture was continuously stirred for 3 hours and was then poured into 400 ml of iced-water to precipitate the product. After the ice being melted the crude product was collected by filtration, washed with cold water, and dried by suction to give the product with white crystal, the yield of method was 97.3 %, having melting point of 38°C.

2.1.3 Purification of the product

The crude product of method 2.1.2.2 was placed in a

conical flask and a small quantity of acetone was added. More acetone was added dropwise with stirring until a clear solution was obtained. The solution was filtered and the filtrate was then poured into cold water without stirring to give the precipitate as colorless needles (4.67 g., 92.6%), which was collected by filtration and dried in a desiccator, the product has the melting point of 41-42 °C (9).

2.2 Chemical Treatment of Coir Fibers

2.2.1 Materials

The coir fibers used in this experiment were obtained from Prachuabkirikhan province. The fibers come in the form of coarse fiber rope. After loosening the rope, the properties of dark-brown fibers were found appropriate by using Instron 1026 Testing Machine and Vernier and the results of which are as follow.

a. fiber length	10 -20 cm
b. fiber diameter	0.28 +/- 0.24 mm
c. tensile strength	158 +/- 78 N/mm ²
d. elongation at break	18 +/- 15 %

2.2.2 Procedure

Ten grams of coir fibers in the chopped strand form of length about 1-1.5 cm were soaked in a solution of the coupling agent in 2%, 5%, 8% aqueous acetone solution for 5 minutes. After drying for 15 minutes at 30 °C in an oven with air circulation, the coir fibers were soaked in an aqueous solution of 0.1 M sodium hydroxide for 5 minutes. The coir fibers were then heated for 1 hour at 50 °C in a vacuum oven and rinsed several times with distilled water and acetone. Finally, the coir fibers were Soxhlet-extracted with acetone for 20 hours to remove the compound not covalently bonded to the fibers and then dried in vacuum to constant weight.

2.3 Preparation of Composites

2.3.1 Material

Short-cut coir fibers treated with the coupling agent
(1 cm length)

Short-cut untreated coir fibers (1 cm length)

Polylite F-H 123 (unsaturated polyester) obtained from
Siam Chemical Industry Co., Ltd.

Glass fiber (chopped strand mat made up of glass fiber
weighed 450 g/m^2)

Silicone mold-releasing agent

Methyl ethyl ketone peroxide

2.3.2 Procedure

2.3.2.1 Preparation of Neat Polyester Sheets

The preparation was carried out in a press with a metal frame of 100 mm in length, 100 mm in width and 5 mm in depth. A mixture of unsaturated polyester resin (100 g.) and methyl ethyl ketone peroxide (1.0 g.) was poured into a metal frame. The mixture was then cured at room temperature for 24 hours and then put in an oven for post curing for 2 hours at 100°C . After being cooled to room temperature, the polyester sheet was removed from the metal frame.

2.3.2.2 Preparation of Coir Fiber Composite Sheets

Chopped coir fibers (30 g.) were uniformly spread on the metal frame and placed on a flat platen press at a pressure of 2000 psi for 10 minutes. After removing from the press, a mixture of unsaturated polyester resin (100 g.) and methyl ethyl

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ketone peroxide (1.0 g.) was poured on the prepared coir fibers the metal frame. After gelling, left 24 hours at room temperature and then put in an oven for post curing for 2 hours at 100 °c to obtain the composite. The preparation of the composite sheets with treated coir fibers was carried out in a similar manner.

2.3.2.3 Preparation of fiber glass sheets

The fiber glass composite was prepared by the same method as for the coir fiber composite except the fiber glass-made chopped strand mat of 4 plies in a dimension of 100 mm x 100 mm.

2.4 Measurement of Mechanical Properties

2.4.1 Flexural strength

Flexural strength of the neat polyester and composites was determined according to ASTM D 790-71, Method I (A-three point loading), using a T/M Avery testing machine and a cross-head speed of 5 mm/min. The rectangular specimens have the dimension of 13 mm width, 4.8 mm thickness, and 100 mm length. The testing was performed with the span-to-depth ratio of 16:1.

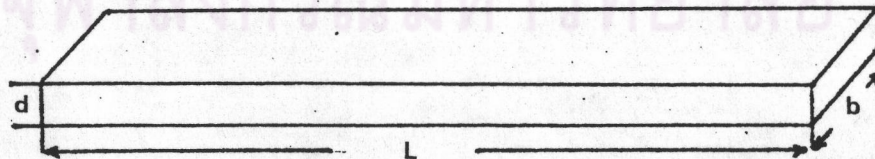


Figure II Specimen for flexural testing.

2.4.1.1 Procedure

Measure the width and depth of the specimen to the nearest 0.02 mm at the center of support span. Set the machine to the specified rate of crosshead motion, or as near as possible. Center the specimen on the supports, with the long axis of the specimen perpendicular to the loading and supports. Apply the load to the specimen at the specified crosshead rate, until the specimen was rupture.

Therefore, the Flexural strength can be represented in terms of the following equation :

$$\text{Flexural strength} = 3PL / 2bd^2$$

where P = maximum load at rupture, lbf
 L = span length, inch
 b = width of specimen, inch
 d = depth of specimen, inch

2.4.2 Tensile strength

The tensile strength of the neat polyester and composites was determined according to ASTM D 638-77a using the dumbbell-shaped specimens as shown in Figure III.

A T/M Avery testing machine of the constant-rate-of-crosshead-movement type, a crosshead speed of 50 mm/min.

2.4.2.1 Procedure

A specimen was gripped at the ends and pulled by the machine until fracture took place. The ultimate tensile

strength was calculated from the applied load at a breaking point and the cross-sectional area of the specimen.

$$\text{Tensile Strength} = \text{load at break (lbf)} / \text{area (square inch)}$$

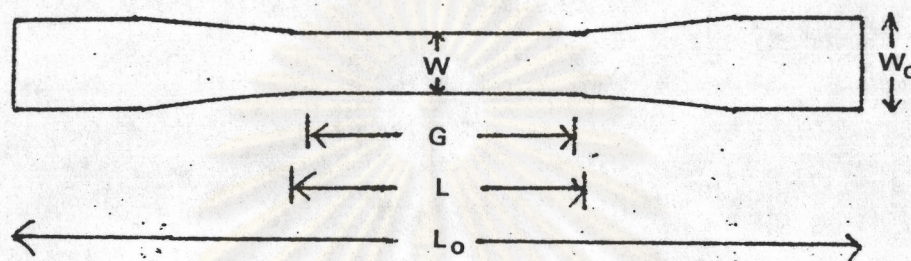


Figure III Specimen for tensile testing.

where	W = width of narrow section	= 13 mm
	L = length of narrow section	= 57 mm
	W _o = width of over-all	= 19 mm
	L _o = length of over-all	= 165 mm
	G = gauge length	= 50 mm
	R = radius of fillet	= 76 mm

2.4.3 Impact strength

The impact strength of the neat polyester and composites were determined according to ASTM D 256-78 using the rectangular specimens as shown in Figure IV.

The testings were performed by a Izod impact testing machine, T/M Avery.

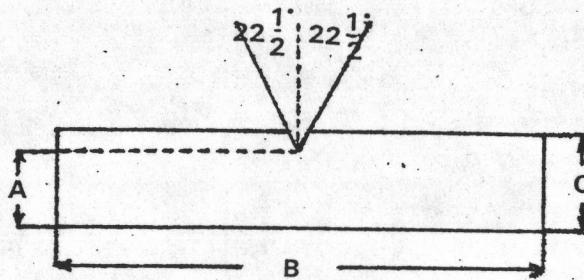


Figure IV Specimen for impact testing.

where A = depth of the specimen, 10.16 ± 0.05 mm
 B = length of the specimen, 63 mm
 C = width of the specimen, 12.70 ± 0.15 mm

2.4.3.1 Procedure

Position the specimen precisely and rigidly but not too tightly clamped in the vise. Release the pendulum from its normal starting position and record the excess energy in the pendulum after breaking the specimen. The impact strength was calculated as follows :

$$\text{Izod impact strength} = IE / b$$

IE = Impact Energy, foot-pound

b = width of the specimen, inch

2.4.4 Compressive strength

Compressive strength of the neat polyester and composites was determined according to ASTM D 695-77 using a Shimadzu

Model UMH-50 testing machine and a standard test specimens in a prismatic form with a size of 12.7 mm in width, 25.4 mm in length and 5 mm in depth.

2.4.4.1 Procedure

Measure the width and thickness of the specimen to the nearest 0.01 mm at several points along its length. Calculate and record the minimum value of the cross-sectional area. Measure the length of the specimen and record the value. Place the specimen between the surfaces of the compression tool, taking care to align the center line of its along axis with the center line of the plunger and to ensure that the ends of the specimen are parallel with the surface of the compression tool. Adjust the crosshead of the testing machine until it just contacts the top of the compression tool plunger. The testing was carried out by allowing the testing machine to operate at a speed of 5 mm/min until the specimen was broken.

The value of the compressive strength was calculated from the maximum compressive strength loaded and the original minimum cross-sectional area of the specimen.

Compressive Strength, (pound/inch²) = maximum compressive load/area