CHAPTER 3

MATERIAL AND METHODS

This chapter describes the details of processing of the composites, standard specimen size and the experimental procedures followed by their mechanical characterization.

3.1 MATERIAL

The following material is selected for experimental work

1. Coconut fiber

3.1.1 Coconut Fiber

Natural fibers such as jute, hemp, sisal, pineapple, abaca and coir have been studied as reinforcement and filler in composites. Taking into account the availability, growing attention is being paid to coconut fiber. The coconut husk is available in large quantities as residue from coconut production in many areas, which is yielding the coarse coir fiber. Figure 3.1 shows the cross-section of coconut.
Coir is a ligno-cellulosic natural fiber. It is a seed hair fiber obtained from the outer cell, or husk of the coconut. The total world coir production is 2,50,000 tones per annum. The coir fiber industries are particularly important in some areas of the developing world. Over 50% of the coir fiber produced annually through-out the world is consumed by the countries of origin, mainly India. Brown coir is harvested from fully ripened coconuts. It is thick, strong and has high abrasion resistance.

It is typically used in mats, brushes and sacking. Mature brown coir fibers contain more lignin and less cellulose than fibers such as flax and cotton and so stronger but so flexible. White coir fibers are harvested from the coconuts before they are ripe. These fibers are white or light brown in color and are smoother and finer, but also weaker. They are generally spun to make yarn that is used in rope. The coir fiber is relatively water proof and is one of the few natural fibers resistant to damage by salt water.

Figure 3.1 Cross section of coconut
The other benefits of coir fiber are:

1. 100% organic, bio-degradable, renewable resources.
2. High water and nutrition holding capacity, expands to hold up to 5 times its weight in water.
3. Strong air porosity for excellent aeration.
4. pH 5.8 to 6.4.
5. Low electrical conductivity
6. Low density of around 1.15 - 1.33 kg/m$^3$

Ripe coconut husks were collected from rural areas of Erode and were soaked in the tap water container for 5 months. This process is called as retting process, which can partially decompose the pulp on the shell, allowing the fiber to be removed from the husk easily. Figure 3.2 shows the retting process.

![Figure 3.2 Retting process](image)

After the retting process, the husks were beaten with a hammer. Coir fibers were removed from the shell and separated with a comb. After drying in the room temperature, the coir fibers were combed in a carding
frame to further separate the fibers into an individual state, or as close to that as possible. Figure 3.3 shows the separation of coir fiber from the husk.

![Image](image.png)

**Figure 3.3 Segregation of coir fiber**

Then a sifter was employed to remove the shorter fibers and impurities. The derived fibers are shown in Figure 3.4.

![Image](image.png)

**Figure 3.4 Coconut coir fibers**

### 3.1.2 Sodium Hydroxide (NaOH)

Sodium Hydroxide, also known as lye or caustic soda has the molecular formula NaOH and is of caustic metallic base. It is a white solid available in pellets, flakes and granules, which is shown in Figure 3.5. NaOH is soluble in water, ethanol and methanol. This alkali is deliquescent and
readily absorbs moisture and carbon dioxide in air. NaOH is used in many industries, mostly as a strong chemical base in the manufacture of pulp and paper, textiles, drinking water, soaps and detergents and as a drain cleaner.

Figure 3.5 Sodium hydroxide pellets

Properties of NaOH

NaOH is predominantly ionic; containing sodium cations and hydroxide anions making it a strong base which reacts with acids to form water and corresponding salts. The properties of NaOH are shown in Table 3.1.

Table 3.1 Properties of NaOH

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular formula</td>
<td>NaOH</td>
</tr>
<tr>
<td>Molar mass</td>
<td>39.9971 g/mol</td>
</tr>
<tr>
<td>Melting point</td>
<td>318°C</td>
</tr>
<tr>
<td>Density</td>
<td>2.13 g/cm³</td>
</tr>
<tr>
<td>Standard enthalpy</td>
<td>-734.95 KJ/mol</td>
</tr>
<tr>
<td>pH</td>
<td>14</td>
</tr>
<tr>
<td>Appearance</td>
<td>White</td>
</tr>
<tr>
<td>Refractive Index</td>
<td>1.3576</td>
</tr>
</tbody>
</table>
3.1.3 Sodium Lauryl Sulfate

SLS is an organic compound with the formula $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$. SLS which is shown in Figure 3.6 is commonly used as a detergent, wetting agent, and emulsifying agent in many of the products that we use on a daily basis. It is used as an ingredient in shampoos, conditioners, dish soap, hand soap, and even toothpaste. SLS is a crystal like salt derived from sulfated lauryl alcohol. This chemical is widely used for its relatively low in cost, long shelf life and for its multi-use. It is used in cosmetic cleaners and floor cleaners, but in varying concentrations. For example, a floor cleaner with SLS will contain a more powerful concentration of the chemical than a bubble bath solution.

![Figure 3.6 Sodium Lauryl Sulfate](image)

SLS has a drying effect and takes out moisture and can cause your skin to become extremely dry. Those with sensitive skin may develop a rash or redness. Others can develop eczema or dandruff if they have an allergy or allergic reaction to the chemical. Table 3.2 shows the properties of SLS.
### Table 3.2 Properties of SLS

<table>
<thead>
<tr>
<th>Appearance</th>
<th>White</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular formula</td>
<td>NaC\textsubscript{12} H\textsubscript{25} So\textsubscript{4}</td>
</tr>
<tr>
<td>Molar mass</td>
<td>288.38 g/mol</td>
</tr>
<tr>
<td>Density</td>
<td>1.01 g/cm\textsuperscript{3}</td>
</tr>
<tr>
<td>Melting point</td>
<td>206 °C</td>
</tr>
<tr>
<td>Refractive Index</td>
<td>1.461</td>
</tr>
<tr>
<td>pH</td>
<td>9-10</td>
</tr>
</tbody>
</table>

#### 3.1.4 Epoxy Resin LY556

Epoxy resins are widely used in filament-wound composites. They are reasonably stable to chemical attacks and are excellent adherents having slow shrinkage during curing and no emission of volatile gases. Starting materials for epoxy matrix are low-molecular weight organic liquid resins containing a number of epoxide groups, which are three-member rings of one oxygen atom and two carbon atoms:

![Figure 3.7 DGEBA molecule (Starting Material of epoxy)](image)

A common starting material is Di-Glycidyl-Ether of Bisphenol A (DGEBA), which contains two epoxide groups, one at each end of the molecule (Figure 3.7). Other ingredients that may be mixed with the starting
liquid are diluents to reduce its viscosity and flexibilizers to improve the impact strength of the cured epoxy matrix. Epoxy matrix, as a class, has the following advantages over other thermo-set matrices:

1. Wide variety of properties, since a large number of starting materials, curing agents, and modifiers are available
2. Absence of volatile matters during cure
3. Low shrinkage during cure
2. Excellent resistance to chemicals and solvents
3. Excellent adhesion to a wide variety of fillers, fibers, and other substrates

The principal disadvantages are its relatively high cost and long curing time.

3.1.5 Hardener HY951

A hardener is a substance or mixture added to a plastic composition to take part in and promote or control the curing action and also to control the degree of hardness of the cured film. In general, uncured epoxy resins have only poor mechanical, chemical and heat resistance properties. However, good properties are obtained by reacting the linear epoxy resins with suitable reactive to form three dimensional cross-linked thermo-set structures. This process is commonly referred to as curing. Curing of epoxy resins is an exothermic reaction and in some cases produces sufficient heat to cause thermal degradation if not controlled. Hardeners which show only low or limited reactivity at ambient temperature, but which react with epoxy resins at elevated temperature are referred to as latent hardener. The epoxy resin and hardener may be mixed and stored for some time prior to use, which is advantageous for many industrial processes.
3.2 METHODOLOGY

The following steps are involved in the fabrication of the composite plate.

1. Fiber surface modification by alkali treatment
2. Preparation of Epoxy and Hardener
3. Mould preparation
4. Specimen preparation as per ASTM standard

3.2.1 Fiber Surface Modification By Alkali Treatment

The two types of alkali treatments were conducted using the alkali solutions namely NaOH and SLS. The coir fibers were treated in the NaOH and SLS solutions at room temperature (28°C) with densities of 2%, 4%, 6%, 8% and 10% separately for 10 days respectively. The alkali treated fibers were immersed in the distilled water after the treatment for 1hr to remove the residual NaOH and SLS. Figures 3.8 (a), (b) and (c) shows some examples of the untreated and treated coir fibers with NaOH and SLS Solutions.

(a) SEM image of raw coir fiber  
(b) SEM image of 6% NaOH treated fibers
(c) SEM image of 6% SLS treated coir fiber

Figure 3.8 (a,b and c) SEM image of single coir fiber

3.2.2 Preparation of Epoxy and Hardener

Epoxy LY556 resin of density 1.15 – 1.20 g/cm³, chemically belonging to the epoxide family was used as a matrix and mixed with the corresponding hardener HY951 of density 0.97 - 0.99 g/cm³ and it was used to prepare the composite plate. The solution was mixed with 10:1 by weight percentage. The resin and hardener were purchased from Covai Seenu and Company, Coimbatore, Tamil Nadu, India.

3.2.3 Mould Preparation

The coir fiber reinforced epoxy composite plates were manufactured through a Metal mould of size 300 x 300 x 3mm. The fabrication of the composite material was carried out through hand – layup technique. The top and bottom plate surfaces of the mould and the walls were coated with remover and allowed to dry. Figure 3.9 shows the mould box without assembly.
The treated and untreated coir fibers were cut into 10, 20 and 30mm length for molding the composites. The epoxy resin properly mixed with coir fiber was transferred to the mould and the mould closed and then it was pressed in the compression testing machine and left for 24hrs for curing. Figure 3.10 shows the assembly view of the mould box.
3.2.4 Specimen Preparation as per ASTM Standards

After the curing process, the coir fiber reinforced epoxy composite plate was taken from the mould box. Figure 3.11 shows the fabrication arrangement in compression testing machine. Figure 3.12 shows the preparation of coir fiber composite plate. Figures 3.13 and 3.14 shows some of the coir fiber reinforced epoxy composite plates prepared by this method. Figure 3.15 shows the mould plate for pure epoxy resin.

![Fabrication arrangement](image1)

**Figure 3.11 Fabrication arrangement**

![Preparation of coir fiber composite plate](image2)

**Figure 3.12 Preparation of coir fiber composite plate**
Figure 3.13 2% NaOH treated 10 mm long coir fiber reinforced epoxy composite

Figure 3.14 2% SLS treated 20 mm long coir fiber reinforced epoxy composite

Figure 3.15 Pure epoxy plate
After fabrication of the treated and untreated coir fiber reinforced epoxy composites the plates were cut into required size as per ASTM standards. The tensile test was performed on flat specimens. A uni-axial load was applied through both the ends. The tensile test was carried out on standard specimen as per ASTM D3039 (Figure 3.16). The flexural test was carried out on standard specimen as per ASTM D648-06 (Figure 3.17). Figure 3.18 shows ASTM D256 standard specimen used for impact test. The charpy impact testing machine was used for measuring the impact strength.

Figure 3.16  ASTM Standard specimen for Tensile test

Figure 3.17 ASTM Standard specimen for Flexural test
Figures 3.19 (a), (b) and (c) show the NaOH treated coir fiber reinforced epoxy composite specimens for impact, flexural and tensile tests as per ASTM standards. Figures 3.20 (a), (b) and (c) show the SLS treated coir fiber reinforced epoxy composite specimens prepared as per ASTM standards for impact, flexural and tensile tests. Figure 3.21 shows the pure epoxy specimen for tensile, flexural and impact tests as per ASTM standards.
Figure 3.20  ASTM standard coir epoxy composite Specimen
(a) Specimen for Impact Test (4% SLS with 20mm long fiber)
(b) Specimen for Flexural Test (4% SLS with 20mm long fiber)
(c) Specimen for Tensile Test (4% SLS with 20mm long fiber)

Figure 3.21 ASTM standard Pure epoxy

(a) Specimen for Tensile Test
(b) Specimen for Flexural Test
(c) Specimen for Impact Test