Chapter 2

Materials and Experimental

This chapter gives a brief description about the materials and methods used for the preparation of composites. The chemicals used for the various fibre treatments and preparation of different hybrid composites have been discussed. A brief description about the different analytical techniques used for the characterization of fibres and composites are also given in this chapter.
2.1. Materials

2.1.1. Banana fibres
Banana fibres obtained from Sheeba fibres and Handicrafts, Poovancode, Tamilnadu was used for the study. Important characteristics of the banana fibre are given in Table 2.1.

Table 2.1. Chemical characteristics of banana fibres

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Cellulose</td>
<td>63-64 %</td>
</tr>
<tr>
<td>Hemi cellulose</td>
<td>19 %</td>
</tr>
<tr>
<td>Lignin</td>
<td>5 %</td>
</tr>
<tr>
<td>Moisture</td>
<td>10-11 %</td>
</tr>
</tbody>
</table>

2.1.2. Glass fibres
E glass fibre rovings obtained from Hitech fibres, Bangalore, were used for the study. They are high silicate materials having high strength to weight ratio.

2.1.3. Phenol Formaldehyde resin
Phenol formaldehyde resole type resin obtained from M/S West Coast Polymers Pvt. Ltd., Kannur, Kerala was used as matrix. Important characteristics of phenol formaldehyde resins are given in Table 2.2.

Table 2.2. Characteristics of phenol formaldehyde resole

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Deep brown color</td>
</tr>
<tr>
<td>Viscosity (CPS)</td>
<td>18-22</td>
</tr>
<tr>
<td>Water tolerance</td>
<td>1:18</td>
</tr>
<tr>
<td>Solid content %</td>
<td>50</td>
</tr>
</tbody>
</table>
2.1.4. Chemicals
Sodium hydroxide, glacial acetic acid and acrylonitrile were used in reagent grade. Triethoxyvinyl silane and 3-aminopropyltriethoxy silane were used as silane coupling agents. Natural rubber latex (dry rubber content 60%) was also used for latex modification of fibre.

2.2. Fibre modifications

2.2.1. NaOH treatment
Fibres were immersed in 4 \% NaOH solution for two hours, washed many times with distilled water and finally washed with water containing little acid and dried.

2.2.2. Silane treatments
The NaOH pretreated fibres were dipped in alcohol water mixture (60:40) containing triethoxyvinyl silane and 3-aminopropyltriethoxy silane as coupling agents. The fibres were washed in distilled water and dried.

2.2.3. Heat treatment
Banana fibres were heated at 150\(^\circ\)C in an air-circulating oven for 4hrs. They were then cooled to room temperature and dried.

2.2.4. Cyanoethylation
Banana fibres were immersed in a 4\% solution of NaOH saturated with sodium thiocyanate for 30 minutes. Subsequently the fibre was pressed to drain off the excess water and transferred quickly into a round-bottomed flask allowing a reaction with acrylonitrile (banana fibre to AN ratio 1:3) for 1hr at 40-45\(^\circ\)C. After the reaction, a solution of acetic acid was used to neutralize the alkali catalyst. The product was washed first with water and then with alcohol respectively.
2.2.5. Acetylation

The NaOH pretreated fibres were neutralized washed with water and air-dried. The fibre was soaked in a 50% solution of acetic acid for 5 minutes, washed and air-dried.

2.2.6. Latex treatment

The pretreated fibres with NaOH were given a latex coating by dipping it into natural rubber latex having 10% dry rubber content.

2.3. Preparation of composites

Prepreg route was followed for the preparation of the composites. Hand lay-up method followed by compression moulding was adopted for composite fabrication. Mats of uniform thickness were prepared from chopped banana fibres of particular fibre length. The mats were impregnated in PF resin and the prepreg was kept at room temperature up to semicured stage. They were then pressed at 100°C in a mould having dimensions 150mmx150mmx3mm to get a three dimensionally crosslinked network. Different composite sheets were prepared by varying the fibre length (keeping fibre volume fraction constant) and volume fractions (keeping the fibre length constant). Glass fibre reinforced PF composites were also prepared by varying fibre length and loading.

For preparing hybrid composites, mats of uniform thickness were prepared from chopped banana fibres (30mm) and glass fibers (40mm). The mats were impregnated in PF resin and composites were prepared as mentioned earlier. Hybrid composites of different hybrid ratios were prepared. For this intimately mixed composites were prepared by varying the banana: glass ratio as 100:0, 91:9, 12:88, 26:74, 35:65, 50:50, 75:25, 0:100, keeping the total volume fraction of the fibres (0.3) constant. Hybrid composites were also prepared by varying the layering pattern of the fibres, keeping the total volume fraction of the fibres
(0.3) and hybrid ratio (banana: glass ratio, 0.75:0.25). Different layering patterns used in this study are given in Figure 2.1.

Figure 2.1. Schematic diagram showing different layering patterns used for the preparation of hybrid composites

2.4. Scanning electron microscopy

The SEM photographs of fibre surfaces and cross sections of untreated and treated fibres were taken using a scanning electron microscope of model JSM-
Chapter 2

5600 LV. The surface of the fractured specimens after tensile and impact tests were examined using SEM to study the fracture mechanisms and the fibre/matrix interface in composites.

2.5. Thermogravimetric analysis
Thermogravimetric tests were performed using universal V 2.5H TA Instruments. The measurements were carried out in nitrogen atmosphere from 0 to 600°C at a heating rate of 20°C/minute.

2.6. Mechanical tests
2.6.1. ISS measurements
Single fibre pull out specimen was prepared as shown in the Figure 2.2. Here a single fibre is partially embedded in resin kept in a box like container. The embedded length, the length of fibre embedded in the matrix is kept as 5mm and fibre free length as 20mm. After curing, the upright fibre was pulled out from the block of resin using universal testing machine at a crosshead speed of 10mm/min [1,2,3]. The load and displacement were recorded during the test.

![Figure 2.2. Single fibre pull out specimen](image)

2.6.2. Tensile testing of fibres
Tensile testing of banana fibre and glass fibre was carried out in a FIE universal testing machine at a crosshead speed of 10mm/min. Specimens
were prepared by mounting single fibre on a stiff cardboard piece with a 50mm window. The ends of fibres were fixed on the cardboard. Twenty-five fibre samples were tested in each case and the average value has been reported. The diameters of the fibre specimens were measured microscopically and average of sixty readings were taken for diameter determination.

2.6.3. Tensile testing of composites
Test specimens were cut from composite sheets. Tensile testing was carried out in a FIE universal tensile testing machine TNE-500 according to ASTM D 638-76. The three point flexural properties were determined by same machine according to ASTM D 790. The load displacement curves were obtained and flexural strength and modulus were calculated. Izod impact test was done on notched specimen with an impact speed of 3.46 m/s and incident energy of 2.75 joules according to ASTM D 256. Minimum of four samples were tested in each case and the average value is reported.

2.7. Dynamic mechanical analysis
A dynamic mechanical thermal analyzer of Polymer Laboratories (Model PL-MK II) was employed for dynamic mechanical property evaluation of the composites. Samples of dimension 50x10x3 mm³ were used for testing. The testing temperature ranged from 20 to 250°C and the experiment was carried out at frequencies 0.1, 1 and 10Hz. The experiment was performed under tensile mode.
2.8. Water absorption Studies

Samples of approximate dimensions 10X10X3 mm (length X breadth X thickness) were used for the measurement of water absorption. The corners of the samples were curved to avoid non-uniform water diffusion. The thickness and weight of the sample were measured. Samples were immersed in water at different temperatures 27, 50, 70 and 90 in a thermostatically controlled air oven. The specimens were periodically taken out of water, surface dried with absorbent paper and reweighed. This process was continued till equilibrium was reached. The mole percent uptake, $Q_t$ for water by 100 g of polymer was plotted against square root of time. The $Q_t$ values were calculated using the equation,

$$Qt = \frac{M(w)}{M_i} \times 100$$

where, $M(w)$ is the mass of water at time t, $M_r(w)$ the relative molecular mass of water, ie, 18, $M_i(s)$ is the initial mass of the sample. When equilibrium was reached, $Q_e$ was taken as the mole percent uptake at infinite time ($Q_{\infty}$).

2.9. Aging studies

2.9.1 Cold water aging

Composite samples were cut in specified dimensions according to ASTM standard for mechanical testing. Preweighed sample was kept in distilled water at 27°C for two weeks to attain the saturation level. The water adhering to the surface of the composite was wiped off, the sample was weighed in an electronic balance and tensile test was carried out.
2.9.2. Thermal aging

Composite samples were cut into specified dimensions according to ASTM standard for mechanical testing. It was then placed in an air oven at 100°C, for three days. Samples were then allowed to cool to room temperature and tensile test was carried out.

2.9.3. Boiling water aging

Samples were kept in boiling water for 2 hours. After wiping off the water adhering to the surface of the samples, it was weighed in an electronic balance and tensile test was carried out.

2.9.4. Soil burial test

Prewighted samples were completely buried in soil having 50% moisture content. The samples were in constant contact with soil and buried vertically in jars at a temperature of 27°C. To ensure adequate supply of oxygen the lid of jar was not closed and moisture content was kept constant. The samples were taken out after 12 months, removed the soil using a brush, dried in an air oven at 50°C for 5 hours and weighed. The tensile testing of the composite was also done.

2.9.5. Outdoor weathering studies

Weathering evaluation of the composites was carried out for a period of twelve months by natural weathering. For this the samples were cut in specified dimensions according to ASTM standard for tensile testing. They were exposed to outdoor to get rainfall and solar radiation and all conditions of local climate. After one year of exposure, they were taken out, dried in an air oven at 50°C for 5 hours, weighed and tensile test was carried out.
2.10. Electrical property evaluation

The capacitance, resistance and dissipation factor were measured directly using Hoiki 3532 LCR Hitester version 1.03 by varying frequencies (10 Hz to 5 MHz). Square samples of thickness 3 mm, length 10 mm and breadth 10 mm were used for the study. The test samples were fixed between two electrodes.

The volume resistivity ($\rho$) was calculated from the resistance using the equation,

$$\rho = RA/t$$

where $R$ is the resistance, $A$ is the area of cross section of the sample, and $t$ is the thickness of the sample.

The dielectric constant $\varepsilon$ was calculated from the capacitance using the equation

$$\varepsilon = CT/\varepsilon_0 A$$

where $\varepsilon$ is the dielectric constant of the material, $\varepsilon_0$ is the permittivity of air ($8.85 \times 10^{-12}$ F m$^{-1}$), $C$ is the capacitance, $A$ is the area of cross section of the sample, and $t$ is the thickness of the sample.

2.11. Extraction of microfibrils and preparation of micro composites

The banana fibre was extracted with 2% NaOH at 190°C for 3 hours in a hot air oven and washed. This process allows the solubilization of pectins and hemi celluloses, which will be removed by filtration. The resulting insoluble residue was stirred using a mechanical stirrer with NaClO$_2$ solution at 80°C. At this stage, the different cell walls would get individualized. The insoluble and bleached out pulp was then suspended in
distilled water followed by disintegration using a grinder. The resulting suspension is a constituent of cellulose microfibrils.

Microcomposites were prepared by the incorporation of these microfibrils in phenol formaldehyde resin matrix. The microfibrils were mixed with phenol formaldehyde resin by high speed mechanical stirring to get good dispersion and prepreg was prepared from the mix. Composites were prepared by the compression molding of the prepreg at 100°C.

References