CHAPTER-3

MATERIALS AND METHODS

3.1 Materials

In the present work, a mixture of polyester resin and styrene monomer was used as matrix. Styrene was used as hardener. Commercially available polyesters are syrup liquids with viscosities varying from 200 to 2000 Centistrokes and with the color varying from pale straw to almost colorless clarity.

Unsaturated polyesters are chemically reactive and relatively unstable compounds having a limited storage life. They tend to gel even in the absence of catalyst particularly in an elevated temperature in sunlight. Stabilizers are, therefore, added to the resins to give them a reasonable self-life and the commercial polyesters are stable for more than six months when stored in a cool place away from sunlight.

In order to convert the resin to a hard and infusible solid within a reasonably short time so as to make their molding a commercial possibility, catalyst/accelerators were added to the resin shortly before the use. For cold curing system i.e. for curing the material at room temperature an accelerator has to be used in addition to the catalyst. An accelerator by itself has influence on the polymerization, its role being to increase the effect of the catalyst at a given temperature or to make the catalyst effective at low temperature.

Two types of catalyst/accelerator systems are in use depending upon whether the final curing of the resin takes place with or without the application of external heat.

For the hot curing system i.e. where external heat is applied to the molding in the range of 80 -130°C a peroxide catalyst such as benzoyl peroxide of external heat. In
present work, cold curing catalyst/accelerator system employed is methyl ethyl ketone peroxide in conjunction with cobalt naphthanate.

The choice of catalyst and the quantity of catalyst used in a formulation are very important. The curing of unsaturated polyester resins is accompanied by evolution of considerable heat of copolymerization. The reaction is thus said to be exothermic. The rate at which heat is liberated depends upon such features as the size of the mix, ambient temperature, composition and concentration of the catalyst/accelerator. The rate of cure of the resin will influence the properties of the final cured resin, such as, freedom from cracking and other defects. The amount of catalyst and accelerator used for most general-purpose work varies from 0.5 to 3.1 on weight of the resin used. While casting the sheets for tensile and flexural tests, 1 ml of catalyst and 1 ml of accelerator were added. At 30°C gelation time was decreased by approximately 30 percent.

One of important factor that is considered by composite designer and builder is the amount of shrinkage that occurs in a resin during and following its cure period. Shrinkage is due to the resin molecules rearranging and re-orientating themselves in the liquid and semi-gelled phase. Polyester requires considerable molecular rearrangement to reach their cured state and can show shrinkage of up to 8%.

3.1.1 Matrix

A mixture of polyester resin and styrene monomer was used as matrix. Styrene was used as hardener. As polyester resin was cheap, it was selected as matrix material for present work. Some of the mechanical properties of polyester resin are given in the table 3.1.
Table 3.1 Mechanical Properties of Polyester Resin

<table>
<thead>
<tr>
<th></th>
<th>Tensile Strength</th>
<th>Flexural Strength</th>
<th>Impact Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyester Resin</td>
<td>49.6 MPa</td>
<td>40.60 MPa</td>
<td>0.10 J/cm²</td>
</tr>
</tbody>
</table>

3.1.2 Mould Releasing Agents

Mould releasing agents were applied to the mould surface to facilitate easy release of the molding from the mould. The most widely used material is a solution of polyvinyl alcohol in water. Hard wax such as carnauba is also used on polished metal mould.

In present experimentation Acetone (Dabade, 2005) was used as releasing agent. Hard wax that was obtained from naturally available honeycomb was also applied on the surfaces of the mold for easy removal of the casted sheet. Alternate layers of hard wax and Acetone were applied and allowed to dry for 20 minutes of time.

3.1.3 Fibers

Four types of natural fibers were extracted from natural resources (Plants) and were used for the present purpose. Methods of extracting the fibers from the respective plants are explained in the next paragraphs. Tensile properties of these single fibers were also obtained through experiments. Some of the properties of Natural fibers are given in the table 3.2.

3.1.3.1 Sun Hemp fiber and Extraction of Sun hemp fiber

Fibre from the sun hemp was extracted from the stem of the legume, crotolaria juncea, indigenous to India and known as sun hemp, brown hemp or Indian
hemp (Punmia, 1998). Crotalaria is a genus of the papilionacea. India is the largest producer of this fiber followed by Bangladesh and Brazil. Sun hemp is an erect herbaceous shrub growing to a height of 1-5 m with a thin cylindrical branch free stem. The plant is harvested in the seedpod stage. After hand cutting or peeling, it was left in the field for defoliation. The stems were water rotted for three weeks and washed and dried. These fibers are hackled, dressed and graded based on their color, length, strength, fineness, uniformity and percentage of extraneous matter for export. This fiber is traditionally used for making ropes. Fig 3.1 shows a photograph of sun hemp plant.

3.1.3.4 Sisal Fiber and Extraction of Sisal Fiber

In Mexican language sisal means cold water. The Indians grew Agave plants. They use to separate fibers by hands and used for making ropes, carpets and clothing. This use later spread throughout the world in the 19th century and the beginning of 20th century. After 1898, Bbahmas produced sisal in large scale.

There are two methods for separating the fiber from the leaves. In one method; leaves are fed through sets of crushing rollers. The crushed leaves are held firmly at their centers and both ends are passed between pairs of metal drums, on which blades are mounted to scrape away the pulp. The centers are also scraped similarly. The fine strands are washed and dried in the air.

In the second method, leaves, which are obtained, from the matured plant are water rotted for three weeks. In due course all the pulp will decompose in the presence of water. Soft brushes are used to further separate the decomposed pulp from the fibers. Typical sisal leaves, which are obtained, have 5% fiber and 95% sisal pulp waste and water. Figure 3.2 shows a photograph of sisal plant. The second method of extracting fibers was adopted for present study.
3.1.3.2 Palmyra Fiber and Extraction of Palmyra fibers

Palmyra fibers were obtained from mature leaves and trunk of palm tree. In India it is found in dry land as well as in coastal areas. Palm trunk bears a crown of 30-40 fans like leaves. The leaves contain stalks of 6-12 in length, which yields strong fiber. The stalks of palmyra leaves were torn apart into strips. By gently blowing with wooden mallet, fibers get separated. Further these fibers were cleaned and dried under sun. Fibers, which are extracted nearer to the epidermis, are soft and fibers from core portion of the stalk are harder. Figure 3.3 shows a photograph of palmyra tree.

3.1.3.3 Banana Fiber and Extraction of Banana fibers

Fruit, leaf sheath as well as pseudo stem of banana plant are the source for fiber. It is obtained by decortications and chemical treatment and its yield is about 1% of the weight of the green trunk. India is one of the major sources for the banana fiber. Banana fiber is traditionally used in making ropes, hats, and coarse cloth. stamped along their length by heavy rectangular block of wood, which will have rounded surfaces. During stamping the inner soft material is removed. Further this pithy material and epidermis is scraped with hand. The fibers obtained from the central core and fruit stalk are less valuable than those extracted from the sheaths. Fibers from fruit stack were obtained by retting the same in water for 3-4 weeks and cleaning by nylon brushes.
Figure 3.1 Sun hemp Plant
Figure 3.2 Sisal Shrubs
Figure 3.3 Palmyra Tree
Figure 3.4 Banana Plant
One of importance to the composite designer and builder is the amount of shrinkage that occurs in a resin during and following its cure period. Shrinkage is due to the resin molecules rearranging and re-orientating themselves in the liquid and semi-gelled phase. Polyester requires considerable molecular rearrangement to reach their cured state and can show shrinkage of up to 8%. Figure 3.4 shows a photograph of banana plant.

Table 3.2 Properties of Natural Fibers

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Density (g/cm³)</th>
<th>Tensile Strength (MPa)</th>
<th>% of Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hemp</td>
<td>0.673</td>
<td>690</td>
<td>1.6</td>
</tr>
<tr>
<td>Sisal</td>
<td>1.5</td>
<td>573</td>
<td>2.3</td>
</tr>
<tr>
<td>Banana</td>
<td>1.35</td>
<td>500</td>
<td>7.0</td>
</tr>
<tr>
<td>Palmyra</td>
<td>1.14</td>
<td>280</td>
<td>4.6</td>
</tr>
</tbody>
</table>
Figure 3.5 Two different Views of the Mould cavity used for making Specimen Plates
3.2 Methods

3.2.1 Fabrication

In order to make the test specimens for mechanical strength testing, sheets were prepared by impregnating the fibers in matrix in open molds. Molds were specially prepared for making tensile, flexural and impact test specimens. Design of these moulds is discussed in the following paragraphs.

3.2.2.1 Mold for making sample plate tensile test

A wooden mold (seasoned teak wood) with 120 × 120 × 3 mm mould cavity was designed. The bottom surface of the cavity and the walls were lined with good surface quality decolam sheet. This decolam lining ensured smooth finishing for the sheet after curing. Similar type of mould cavity can be prepared using glass sheets, which produces good surface finish on the specimen sheet. (Figure 3.1)

3.2.2.2 Mold for making sample plate flexural test

A wooden mold (seasoned teak wood) with 120 × 120 × 3 mm mould cavity was designed. The bottom surface of the cavity and the walls were lined with good surface quality decolam sheet. This decolam lining ensured smooth finishing for the sheet after curing.

3.2.2.3 Mold for making Impact test specimen

A good quality teak wood was used to make a mold cavity of 70 × 10 × 10 mm. The cavity was coated with varnish and further it was coated with hard wax obtained from honeycomb.
3.2.2 Tensile Test

The tensile strength of the specimen was measured by means of tensometer. The tensometer used for present work is capable of applying a force of 20 kN (2000 kgf) on a test specimen held in chucks. The maximum force of 20 kN is sufficient to stress a metric size B (20 mm²) test piece to 1000 MN/m² (100 kg/mm²). It was observed that this instrument gives repeated values and is most convenient.

Disc wire chucks were used with tensile test is to be performed on a specimen with a diameter up to 0.8 mm. They were treaded through a hole in the clamping screw and gripped between aluminum washers. For very soft wires, standard washers are replaced by leather or lead washers.

Eccentric roller grips are another variety of grips available to hold flat tensile specimens. They are self-tightening grips using spring loaded rollers for waisted or dumbbell shaped test pieces of rubber, plastic etc., up to 25 mm wide. They can bear a load of 220 kg. They are made in accordance to BS 903: Part A2, ASTM D 412 DIN 52504 standards.

3.2.2.1 Method of determining Tensile Strength and Young's Modulus and Stiffness

Tensile test was performed to evaluate the ultimate tensile strength. A specimen of dumbbell shape with standard specimen (described in the appropriate chapter) was cut from the composite plate already made. The cut tensile specimen was held in eccentric roller grips and the load was applied on the specimen gently and the mercury ran in the glass column from zero point. The load at the break was noted from the scale at the time of failure. The same procedure was repeated for other specimen cut from the same composite plate. Later, the average load at break was
noted and tensile strength is calculated on the basis of the following formula (Punmia, 1998).

\[
\text{Tensile Strength} = \frac{\text{Average Load at Break ( kg )}}{\text{Cross-Sectional Area in Gauge length portion ( mm}^2\text{ )}}
\]

The tensile strength of fiber reinforced polyester composite was determined by holding the specimen in eccentric roller grips. Minimums of 10 test specimens were tested and their average value of tensile strength is found.

\[
\text{Young's modulus of Elasticity} = \frac{\text{Stress}}{\text{Strain}}
\]

From the stress Vs strain curves, it is observed that these curves are similar to the curves of a brittle material. For brittle materials Young's modulus of Elasticity is calculated by the below shown formula. Ultimate tensile strength is considered for calculation of Young's modulus.

\[
\text{Young's Modulus of Elasticity for any Composite (Punmia, 1998)} \quad = \frac{\text{Ultimate Tensile Strength}}{\text{Strain}}
\]

Stiffness if defined as Change in load for unit deflection. A graph is plotted between load applied and the displacement. Stiffness is calculated from this graph.
3.2.3 Flexural Test

A flexural test was conducted on the plain resin and composite samples to determine Flexural Modulus of the material. For this purpose, Flexural test set up was fabricated in the laboratory. Minimums of 10 test specimens were tested and their average value of the required output is found.

3.2.3.1 Fabrication of equipment

An I-section with a central length of 400 mm and two ends with a length of 200 mm each was prepared. Further, two round mild steel solid bars 450 mm in length and 25 mm diameter were taken and one end of both bars was machined to knife edge using shaping machine. The other end of both bars was placed vertically and welded to the flat I base. The distance between the knife edges was kept at 200 mm.

3.2.3.2 Determining Flexural Modulus

A flat sample was made using the wooden mould. The sample was placed on the knife edges of the equipment and the system acts as a simply supported beam. Now a pan for loading weights was hung at the center of the sample. The specimen was loaded in a uniform fashion by adding weights in an ascending order. As the load increased, deflection was observed. This deflection was measured using a precision dial gauge with an accuracy of 0.001 mm. This gauge was on a stand with magnetic base and placed at \( \frac{1}{2} \) of the length of the test specimen, and deflection was noted for every increase in weight. The test was repeated in the descending order and the average deflection was noted. The Flexural Modulus were calculated using the formulae (Punmia, 2005)
\[ E = \frac{11 \times WL^3}{768 Y I} \]

Where \( E \) = Flexural Modulus,
\( W \) = Load applied ( kg ),
\( L \) = Span Length of Beam ( mm ),
\( I \) = Moment of Inertia of Beam.

The moment of inertia for the rectangular cross section is given by (Bansal, 1991)
\[ I = \frac{bd^3}{12} \]

Where \( b \) and \( d \) are the breadth and thickness of the test specimen in mm respectively.

### 3.2.4 Impact Test

The principle used in impact testing machines was that a notched specimen with a standard blow from a pendulum hammer absorbs certain amount of energy before it before its impact strength. If the material is tough, it absorbs more energy and so it requires more energy in order to fracture. If it is brittle it absorbs less energy and so it breaks more easily. Minimums of 10 test specimens are tested and their average value of the required output is found.