CHAPTER 4

Unsaturated Polyester Composites:
Experimental

SOLUTION BLENDS AND SISAL/GLASS HYBRID COMPOSITES OF UNSATURATED POLYESTER RESIN
This chapter deals with materials and methods used for the present research work. The classification of materials has been made in terms of resin, catalyst, accelerator, reinforcements, and chemical reagents. The methods part deals with the equipment used for the moulding of composites, the moulds, the test specimens, and the testing equipment.

4.1 MATERIALS

4.1.1 Matrix

4.1.1.1 Unsaturated Polyester Resin (UPR)

In the present work, the unsaturated polyester resin supplied by M/s. Rishabh Polymers Pvt. Ltd., India is used as matrix for the fabrication of hybrid composites. This resin is viscous, pale yellow coloured of low degree of polymerization. It has got properties like excellent dimensional stability, chemical and heat resistance. Some of the properties of UPR are given in Table 4.1.

Unsaturated polyesters are one of the most important and widely used matrix resins for fiber reinforced composites. These are obtained by the polycondensation of unsaturated and saturated acids or anhydrides with diols. The unsaturated polyester
resin (GP) supplied was a copolymerization product of propylene glycol, maleic anhydride and phthalic anhydride.

A segment of the polyester obtained from propylene glycol, maleic anhydride and phthalic anhydride might have the following structure:

\[
\begin{align*}
\text{CH}_3 & \quad \text{CH}_3 \\
\text{O} & \\
\text{CO-CH=CH-CO-O-CH}_2\text{O-OC} & \quad \text{CO-CH=CH-CO-O-CH}_2\text{O-OC} \\
\end{align*}
\]

Table 4.1 Properties of Unsaturated Polyester Resin

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific Gravity at 25°C</td>
<td>1.14</td>
</tr>
<tr>
<td>Gel time at 25°C</td>
<td>20-25'</td>
</tr>
<tr>
<td>HDT</td>
<td>60°C</td>
</tr>
<tr>
<td>Tensile strength (Kg/cm²)</td>
<td>500</td>
</tr>
<tr>
<td>Flexural strength (Kg/cm²)</td>
<td>1000</td>
</tr>
</tbody>
</table>

Some of the advantages of UPR over other matrices include excellent chemical resistance, moderate cost, smooth surfaces and not highly viscous.

4.1.1.2 Catalyst and Accelerator

In order to convert the resin into hard and infusible solid within a reasonably short time so as to make their moulding a commercial possibility, catalysts/accelerators are added to the resin shortly before the use. 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., when external heat is applied to the moulding in the range of 80-130°C, a peroxide catalyst such as benzoyl peroxide is widely used. 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 low temperature. The most widely used cold curing catalyst/accelerator system employs methyl ethyl ketone peroxide in conjunction with cobalt naphthenate.

The author, in the present work, employed cold curing method to develop the hybrid composites and used methyl ethyl ketone peroxide as catalyst and cobalt naphthenate as accelerator.

4.1.2 Reinforcements

In the present study, sisal and glass fibers (short, randomly oriented) have been used as reinforcing materials for the unsaturated polyester based hybrid composites.

4.1.2.1 Sisal fibers

Sisal (Agave veracruz) fiber was obtained from local sources. The physical and mechanical properties of sisal fibers are presented in Table 4.1.
<table>
<thead>
<tr>
<th>Fiber diameter (µm)</th>
<th>Density (g/cm³)</th>
<th>Lignin content (%)</th>
<th>Cellulose content (%)</th>
<th>Tensile strength (Mpa)</th>
<th>Tensile modulus (Gpa)</th>
<th>Elongation at break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100-300</td>
<td>1.1-1.45</td>
<td>4-5</td>
<td>85-88</td>
<td>400-700</td>
<td>9-20</td>
<td>2-14</td>
</tr>
</tbody>
</table>

**Fiber Treatment:**

The sisal fibers were taken in a glass tray. A 2% solution of NaOH was added into the tray and the fibers were allowed to soak in the solution for 1 hour. The fibers were then washed thoroughly with water to remove the excess of NaOH sticking to the fibers. Final washings were carried out with distilled water and the fibers were then dried in hot air oven at 70°C for 3 hours. The fibers were chopped into short fibers of length 10mm for moulding the composites.

**4.1.2.2 Glass Fibers**

Glass fiber is commercially a part of the plastics and electrical industry as it is used either as reinforcement in reinforced plastics or as yarn/fabric in electrical insulation. E-glass is lime-alumina borosilicate glass first developed for producing continuous fibers, was designed for electrical applications. It proved effective in a variety of processes and is now the standard glass reinforcement. It has high surface electrical resistivity as well as excellent fiber forming characteristics. E-glass fibers obtained in the form of chopped strands, chopped strand mat, rovings, woven rovings are commonly used as reinforcements in the manufacture of glass reinforced plastics.
Chopped strand mat of glass fibers was used for the present work. It consists of about five centimetre long fiber glass strands randomly distributed and bonded together with an adhesive “binder” chosen for its high performance with the range of resins likely to be used. The mat was chopped into short fibers of 10-15mm length for processing.

The physical and mechanical properties of glass fibers are presented in Table 4.2.

Table 4.2 Physical and Mechanical Properties of Glass Fibers [2,3]

<table>
<thead>
<tr>
<th>Fiber diameter (µm)</th>
<th>Density (g/cm³)</th>
<th>Cellulose/Lignin (wt%)</th>
<th>Tensile strength (Mpa)</th>
<th>Tensile modulus (Gpa)</th>
<th>Elongation at break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.5-10</td>
<td>2.55</td>
<td>Nil</td>
<td>800-1200</td>
<td>68-96</td>
<td>2-5</td>
</tr>
</tbody>
</table>

Some of the characteristics of glass fiber are very high tensile strength to weight ratio, high thermal conductivity, low thermal expansion and retention of strength at high temperatures, excellent moisture resistance, outstanding dimensional stability and excellent resistance to chemical reagents.

4.1.3 Chemical Reagents

4.1.3.1 Solvents, acids and bases

The following chemical reagents have been used for the chemical resistance test of the sisal/glass hybrid composites:
Carbon tetrachloride, benzene, toluene, distilled water, sodium hydroxide, sodium carbonate, ammonium hydroxide, acetic acid, hydrochloric acid and nitric acid.

4.1.3.2 Mould releasing agent

Poly(vinyl alcohol) (PVA) was used as the mould releasing agent in the composite processing. The mould cavity was coated with a thin layer of aqueous solution of PVA for the easy removal of the sheet from the mould and for a smooth surface finishing.

4.1.3.3 Coupling agent

An alkoxy silane (trimethoxy silane) has been used as the coupling agent in order to enhance the adhesion characteristics between the fiber and the matrix.

Most of the coupling agents can be represented by the following formula:

\[ R - (\text{CH}_2)_n - \text{Si(OR')}_3 \]

Where \( n = 0-3 \), OR' is the hydroxylyzable alkoxy group, and R the functional organic group.

4.2 METHODS

4.2.1 Moulding

For mechanical testing, castings of different thickness were made from resin. Unsaturated polyester and styrene are mixed in the ratio 100:25 parts by weight
respectively. Later, 1 wt% methyl ethyl ketone peroxide and 1 wt% cobalt naphthenate were added and mixed thoroughly. This system can be processed by different methods like injection, hand lay-up, winding, and pressure moulding, etc. In the present work, hand lay-up technique was used for making test specimens. In order to make the test specimens, the matrix system is poured into a mould made of glass plates. Excess resin was removed by a blade, and a glass plate was placed on top. The castings were allowed to cure for 24 hours at room temperature, post-cured at 80°C for 4 hours, and cut into test specimens of different shapes. The dimensions for these test specimens are given in section 4.2.2.

4.2.2 Moulds and Test specimens

A glass mould with 280mm x 150mm x 30mm was made. To ensure the smooth surface of sheet after curing, a good finish by wax polish was given on the mould. From the sheet, tensile specimens with dimensions 100 mm x 15mm x 3mm were cut as described elsewhere [4]. For flexural tests, specimens with dimensions 100mm x 12.7mm x 3mm were cut as per ASTM D 618 specifications from the same sheet. The impact test samples were made with 63.5mm x 12.7mm x 12.7mm dimensions using moulds having dimensions 200mm x 12.7mm x 12.7mm and the notch was made as per ASTM D 256 specifications. Compression and hardness samples were made as per ASTM D 3410/695 and ASTM D 785 specifications respectively using appropriate moulds.
4.2.3 Tensile Test

The tensile strength of the plain matrix and the composites was measured by employing an Universal Testing Machine (Hounsfield - model: 4201, UK).

Tensile test was performed to evaluate the ultimate tensile strength. A specimen with standard shape and specifications (described earlier in this chapter) was cut from the composite plate already made. The cut tensile specimen was held in the grips and the load was applied on the specimen gently and as the load increased, fracture occurred in the gauge length portion of the test specimen. The load at the break was noted from the graph at the time of failure. The same procedure was repeated for other specimens cut from the same composite. Later, the average load at break was noted and tensile strength was calculated on the basis of the following formula [5].

\[
\text{Tensile Strength} = \frac{\text{Average load at break (kg)}}{\text{Cross-sectional area in gauge length portion (mm}^2\text{)}}
\]

4.2.4 Flexural Test

A flexural test was conducted on the matrix and composite samples to determine the stiffness. The specimens were made as per ASTM Standards. The flexural tester used by the author was supplied by M/s. PSI Sales (P) Ltd., New Delhi. Five samples were tested in each case and the average value was reported. The flexural strength (\(\sigma_f\)) and flexural modulus (\(E_f\)) of the composites measured using three-point bending tests are calculated as [6,7]:
Flexural Strength \((\sigma_f)\) = \(\frac{3PL}{2bd^2}\)

Flexural Modulus \((E_f)\) = \(\frac{L^3 \Delta P}{4bd^3 \Delta m}\)

where \(P\) is the applied fracture force, \(L\) is the distance between the supports, \(b\) is the width of the specimen, \(d\) is the thickness of the specimen, \(\Delta P\) is the change in fracture force in the linear portion of the load-deflection curve, and \(\Delta m\) is the change in deflection corresponding to \(\Delta P\). The flexural strength and modulus were represented in units of MPa and GPa respectively.

4.2.5 Compression Test

The compressive strength of the plain matrix and the composite was measured by employing the tensometer by using a precision compression cage. Five specimens were tested in each case, and their average load at first deformation was noted. The compressive strength was calculated using the following equation:

\[
\text{Compressive Strength} = \frac{\text{Compressive load at first deformation (kg)}}{\text{Area of cross-section (cm}^2\text{)}}
\]

4.2.6 Izod Impact Test

The samples were made as per ASTM Standards (ASTM D 256) and notch was made. The izod impact tester used by the author was supplied by M/s. PSI Sales.
(P) Ltd., New Delhi. Five samples were tested in each case and the average value is reported.

4.2.7 Hardness Test

The hardness of the samples was measured using a Rockwell hardness tester supplied by M/s. PSI Sales (P) Ltd., New Delhi. The diameter of the ball indentor used was ¼" and the maximum load applied was 60 kg as per the standard of L-scale of the tester. The test was repeated five times for each sample and their average value is reported.

4.2.8 Chemical Resistance Test

To study the chemical resistance of the composite, the test method ASTM D 543-87 [8] was employed. This method covers the testing of the resistance of all plastic materials including cast, hot moulded, cold moulded, and laminated resinous products as sheet materials to chemical reagents. In this test method, there is a provision for reporting changes in weight, dimensions, appearance and strength properties. Standard reagents are specified to establish results on a comparable basis. There is provision for various exposure times and exposure to reagents at elevated temperatures.

The author in the present work, conducted chemical resistance tests on sisal/glass hybrid fibers reinforced in matrix composites. The author judiciously selected three acids, three bases and three solvents for this purpose. Glacial acetic acid, nitric acid, hydrochloric acid, ammonium hydroxide, aqueous sodium carbonate,
aqueous sodium hydroxide, carbon tetrachloride, benzene and toluene were used after purification.

In each case, the samples were pre-weighed in a precision electrical balance and dipped in the respective chemical reagents for 24 hours. They were then removed and immediately washed in distilled water and dried by pressing them on both sides with a filter paper at room temperature as described elsewhere [9]. The treated samples were then re-weighed and the percentage loss/gain was determined using the following equation:

\[
\frac{\text{Original weight} - \text{Final weight}}{\text{Original weight}} \times 100
\]

% weight loss or gain of the sample
References


