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

2.1.1. Isora fibre

Isora fibre was separated from the bark of Helicteres isora plant by retting in water for 30 days. The average length of the fibre is approximately 75-100 cm. The physical and chemical characteristics of isora fibre are given in Chapter 1. (1.7).

2.1.2. Unsaturated polyester resin

General Purpose grade Unsaturated polyester resin (HSR 8113M), was obtained from M/s Bakelite Hylam, Hyderabad. methyl ethyl ketone peroxide (MEKP) and cobalt napthenate were commercial grade supplied by M/s Sharon Engineering Enterprises, Cochin.

2.1.3. Epoxy resin

Commercial grade Epoxy Resin 103, and the room temperature amine hardener 301(polyamine) were supplied by M/s Sharon Engineering Enterprises, Cochin.

2.1.4. Chemicals for fibre modification

Sodium hydroxide, acetic anhydride, glacial acetic acid, benzoyl chloride, Triton (Iso octyl phenoxy poly ethoxy ethanol) were used for fibre surface modification was of reagent grade. Coupling agent vinyl tri ethoxy silane used was of analytical grade procured from M/s Union carbide Co, Montreal, Canada.

2.2. Chemical composition of isora fibre

Chemical composition of isora fibre was estimated according to ASTM procedures. Lignin-ASTM D 1106, Cellulose- ASTM D 1104, Ash content- ASTM D 1102, Alcohol benzene solubility -ASTM D 1107, Ether solubility- ASTM D 1108, 1% NaOH solubility -ASTM D 1109, Water solubility -ASTM D 1110.

2.3. Fibre preparation and surface modification

Isora fibre was separated from the bark of Helicteres isora plant by retting in running water for 30 days and dried. Then it was chopped to different lengths 10, 20,
30, 40 and 50mm for preparing short fibre composites. For the preparation of oriented long fibre composites, fibre in the tape form of length 150mm was used.

2.3.1. Mercerization

a. For preparing randomly oriented fibre composites, fibres of average length 30 mm were treated with NaOH solutions of various concentrations for different time intervals, viz. 2, 4, 6, 8 and 12 hours at room temperature. Finally the fibres were repeatedly washed with water containing a little acetic acid and then dried in an air oven at about 70°C.

b. For preparing long fibre composites, fibres of length 150 mm in tape form were treated with 1% alkali solution for 4 hours. Finally the fibres were repeatedly washed with water containing a little acetic acid and then dried in an air oven at about 70°C.

Mercerization is one of the oldest methods proposed by John Mercer in 1842, which has been used for treating natural fibres [1]. The process involves the conversion of native cellulose (cellulose I) into cellulose II, when the former is swelled in NaOH followed by the removal of alkali by washing with water. This process causes significant changes in the molecular orientation, crystal structure and degree of crystallinity and morphology of the fibres [2]. This led to the formation of more flexible micro fibrils than that present in the native cellulose due to the less crystallinity in the resulting cellulose II. The natural impurities are also removed from the surface as a result of mercerization. There are three chemically active –OH groups on each anhydro glucose units of the untreated cellulose. Even though these groups are not much reactive under normal conditions, these become more reactive on mercerization.

2.3.2. Silane treatment

Alkali treated fibres were dipped in alcohol/water mixture (60:40) containing 1% vinyl triethoxy silane coupling agent. The pH of the solution was maintained between 3.5 and 4. Fibres were washed in distilled water and dried.
2.3.3. Acetylation

Alkali treated fibres were soaked in glacial acetic acid for one hour, decanted and then soaked in acetic anhydride containing two drops of concentrated sulphuric acid for five minutes. They were then filtered, washed and dried in an air oven at 70°C.

2.3.4. Benzoylation

Alkali treated fibres were suspended in 10% sodium hydroxide and agitated with 50ml benzoyl chloride for one hour, filtered, washed with water and dried. They were then soaked in ethanol for one hour to remove the unreacted benzoyl chloride, washed with water and finally dried in an air oven at 70°C.

2.3.5. Detergent treatment

Isora fibres of length 150mm in tape form was treated with 5% solution of commercially available detergent solution for 4 hours with occasional stirring. The treated fibres were washed thoroughly with water and dried in an air oven at 70°C.

2.3.6. Ultrasonication

Isora fibres of length 150mm in tape form were suspended in an ultrasonicating bath and subjected to ultrasonication for 40 minutes. The fibres were dried in an air oven at 70°C.

2.3.7. Triton treatment

Isora fibres of length 150mm in tape form was treated with 1% alkali solution for 4 hours, washed in water containing a little acetic acid and dried. These fibres were then treated with 5% triton (iso octyl phenoxy poly ethoxy ethanol) solution for 2 hours. Finally the fibres were washed in water and dried in an air oven at 70°C.

2.4. Preparation of composites and test specimens

2.4.1. Short fibre reinforced composites

Randomly oriented isora fibre-polyester composites containing fibres of varying length and fibre volume fractions were prepared by hand lay up method
using a three-piece stainless steel mould having dimensions \(200 \times 150 \times 30\text{mm}^3\) [Figure 2.1]. Prior to the composite preparation, the mould surface was polished well and a mould-releasing agent (waxpol) was applied on the surface of the mould. Unsaturated polyester resin (GP) was mixed well with 1% by wt. Cobalt naphthenate accelerator and 1% by wt. MEKP catalyst. The fibre mat was placed in the mould and the resin mixture was poured evenly on it. Using a metallic roller, the air bubbles were carefully removed and the mat was allowed to wet completely. The mould was closed and the excess resin was allowed to flow out as ‘flash’ by pressing in a hydraulic press. The pressure was held constant during the curing process at room temperature for 24 hours. The composite sheet was post cured at 80°C for 4 hours. Test specimens, according to ASTM standards, were cut from the sheet.

**Figure 2.1.** Three-piece steel mould used for preparing randomly oriented fibre composites

### 2.4.2. Oriented long fibre reinforced composites

Oriented long isora fibre reinforced composites were prepared by hand lay up technique using a three-piece stainless steel mould having dimensions \(150 \times 70 \times 2\text{mm}^3\) [Figure 2.2]. Prior to the composite preparation, the mould surface was polished well and a mould-releasing agent (waxpol) was applied on the surface. A weighed amount of the polyester resin was thoroughly mixed with
0.5% by wt. Cobalt naphthenate accelerator and 0.5% by wt. MEKP catalyst. Using a brush, the resin was applied evenly on the fibre lamina and were stacked one above the other in the mould and the mould was closed. The mould was pressed in a hydraulic press at a pressure of 20 kg m$^{-2}$ at room temperature and the excess resin was allowed to flow out as 'flash'. The pressure was held constant during curing. Mylar film was placed on both sides of the stack for easy release and to obtain superior surface finish of the pressed laminates. The composite sheet was post cured at 80°C for 4 hours. Test specimens, according to ASTM standards, were cut from the sheet.

![Figure 2.2. Three-piece steel mould used for preparing oriented long isora fibre composites](image)

2.5. Characterisation of fibre and composites

2.5.1. Scanning electron microscopy

The SEM photograph of the fibre surface and cross section of the untreated and treated fibres and fracture surface of the composites were taken using JEOL 35C Model scanning electron microscope. The principle of which was detailed by Johar et al.[3]. The fracture surfaces of the composites were carefully cut from the failed test specimens without touching the surface. To avoid electron-charging effects, samples were sputter coated with gold within 24 hours in a Polaron SEM coating unit SC 515. The fractured
specimens and gold-coated samples were stored in a desiccator till SEM observations were made. The SEM photographs of the fractured surfaces give information regarding fracture mechanism and interface adhesion of the composites.

2.5.2. Infrared spectroscopy

Infrared spectra of raw and chemically modified isora fibre were obtained with Schimadzu model IR 470 infrared spectrophotometer, Schimadzu, Japan by using solid KBr pellet technique. Fibre samples were cut into small pieces and ground well before mixing it with KBr.

2.5.3. Optical microscopy

An optical stereomicroscope of Leitz metallux 3, Germany was used for observing the impact fracture surface of the composites.

2.5.4. Thermal Analysis

*Dynamic Mechanical Analysis (DMA)*

The dynamic storage modulus (E'), loss modulus (E'') and the loss factor (tan δ) of the composites were measured using a dual cantilever, and samples with dimensions $60 \times 10 \times 3$ mm$^3$, as function of temperature at a frequency of 1Hz on a Dynamic Mechanical Thermal Analyzer, DMA Q 800 TA instruments. The properties were determined over a temperature range of 30 to 130°C at a heating rate of 3°C min$^{-1}$.

2.6. Mechanical property measurements

The standard mechanical properties are determined by the procedures found in ASTM standards for plastics. The mechanical properties studied are tensile strength, Young’s modulus, elongation at break (EB) %, flexural strength, flexural modulus and impact strength.

2.6.1. Tensile properties

The tensile properties were tested on a Shimadzu Autograph (AG-150 kN) Universal Testing Machine at a constant rate of traverse of the moving grip of
5 mm min\(^{-1}\) for randomly oriented fibre composites (ASTM D 638-99) and 20 mm min\(^{-1}\) for oriented fibre composites (ASTM D 5083). The test specimens were rectangular in shape with dimensions 150 × 15 × 3 mm\(^3\) for randomly oriented fibre composites and 150 × 10 × 2 mm\(^3\) for oriented fibre composites. The sides of test specimens were polished using emery paper prior to testing. One grip is attached to a fixed and the other to a movable (power-driven) member so that they will move freely into alignment as soon as any load is applied. The test specimen was held tight by the two grips, the lower grip being fixed. The output data in the form of stress-strain graph and elongation, modulus and energy absorbed at various stages of the test directly appears on the console of the microprocessor and as a print out.

2.6.2. Flexural properties

The flexural properties were tested on a Shimadzu Autograph (AG-150 kN) Universal Testing Machine (ASTM D 790-99) at a constant rate of traverse of the moving grip of 5 mm min\(^{-1}\) [Figure 2.3] for randomly oriented fibre composites and 2 mm min\(^{-1}\) for oriented fibre composites. The test specimens were rectangular in shape with dimensions 100 × 15 × 3 mm\(^3\) for randomly oriented fibre composites and 75 × 20 × 2 mm\(^3\) for oriented fibre composites. The specimens were polished using emery paper prior to testing. The depth and width of the specimen was measured nearest to 0.01 mm. The support span was 16 times the depth of the specimen. The specimen was centred on the supports with the long axis of the specimen perpendicular to the loading nose and supports. The load was applied to the specimen and flexural strength and modulus were recorded. The load-deflection curve was also obtained. It is calculated at any point on the stress strain curve by the following equation

\[
S = \frac{3PL}{2bd^2}
\]  

.................................2.1
where \( S = \) stress in the outer fibres at midpoint (MPa), \( P = \) Load at any point on the load–elongation curve (N), \( L = \) support span (mm), \( b = \) width of specimen tested (mm), \( d = \) depth of specimen (mm).

![Figure 2.3. Three-point bending](image)

Flexural modulus is the ratio of stress to corresponding strain and is expressed in MPa. It is calculated by drawing a tangent to the steepest initial straight-line portion of the load-deflection curve and using the equation

\[
E_B = \frac{L'm}{4db^3}
\]

Where \( E_B = \) modulus of elasticity in bending (MPa), \( L = \) support span (mm), \( b = \) width of specimen tested (mm), \( d = \) depth of specimen (mm), \( m = \) slope of the tangent to the initial straight line portion of the load – deflection curve (N mm\(^{-1}\) of deflection)

2.6.3. Impact strength

Izod impact strength of unnotched samples of the composite was measured on a Tinius Olsen Model 503 Impact Tester, according to ASTM D256-97 specifications [Figure 2.4]. Impact strength is a measure of the energy required to
break a specimen by means of a sharp impact and is also a measure of the shock resistance of a material. The impact tests involve striking a standard shaped specimen with a swinging pendulum. The distance of upswing of the break pendulum after breaking the specimen is compared to the distance travelled when no specimen is present. This gives a measure of the energy required to break the specimen.

![Diagram of pendulum impact tests](image)

**Figure 2.4. Pendulum impact tests**

2.7. Water absorption

Samples of approximate dimensions $76 \times 25 \times 3 \text{ mm}^3$ and $76 \times 25 \times 2 \text{ mm}^3$ for randomly oriented and oriented fibre composites respectively were used for the measurement of water absorption (ASTM D 570-98). The corners of the samples were curved to avoid non-uniform water diffusion. The water absorption test samples were directly placed in a temperature-controlled oven. The temperature was kept constant at $50^\circ\text{C}$ for 24 hours. The samples were taken out and cooled in a desiccator and the thickness and weight of the samples were measured. The randomly oriented fibre composite samples were immersed in
water for 24 hours at room temperature and at 50°C (in a thermostatically
temperature controlled air oven). The specimens were removed, wiped dry with absorbent
paper and immediately weighed. The increase in weight was found out. The
oriented long fibre composite samples were immersed in water at room
temperature. The specimens were periodically taken out of water, surface of the
samples dried with absorbent paper and reweighed. This process was continued
till equilibrium was reached. The mole percent uptake, Q_t, of water by 100g. of
polymer was plotted against time. The Q_t values were calculated using the
equation,

$$Q_t = \frac{M_{(w)}}{M_{r(w)}} \times 100$$  ......................................................2.3

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

2.8. Aging studies

Prolonged exposure to extreme conditions like heat affects the properties of
the composites. The samples were heated in an air-circulating oven at 100°C for
24, 48 and 72 hours. After cooling down and conditioning at room temperature
the tensile and flexural properties of these samples were measured.

References


[3] Johar O., Samudra A.V., Characterisation of solid surface, Kane P.F.,