CHAPTER 3

MATERIALS AND EXPERIMENTS

3.1 EXTRACTION OF FIBER

The matured Borassus fruits were collected from Borassus fruit trees and immersed in water for a week. The flush which was bonded with the fibers absorbed water and the retting of the same started. The flush lost its bonding strength at this stage. Now the fruits were taken out of immersion tank and thoroughly washed in for several times. During the washing process the fruits were gently pressed for the removal of the retted flush. The fruits were then immersed in water for three days and the process was repeated for the removal of the remaining flush. The fibers were taken out and allowed to dry in the shade for a couple of days. The fibers were then dried in sunlight for half an hour and extracted. The fibers were gently rammed to remove the unwanted short fibers and dry flush particulates from them (Figure 3.1).

Figure 3.1 Borassus fruit fiber
3.2 ALKALI TREATMENT

The alkali treatment of the fibers removed the hemicellulose, lignin and other impurities from the fiber surface and made the fiber surface rough. This led to better fiber matrix interface, fiber wetting characteristics and bonding. The Borassus fruit fibers were washed in water to remove any short fibers and particulates. They were dried at room temperature for 48 hours. The fibers were soaked in 5%, 10% and 15% Sodium hydroxide (NaOH) solution for half an hour at room temperature-29 ºC (Figure 3.2). The fibers were neutralized with 2.5% Hydro Chloric acid (HCl) solution. The fibers were washed with distilled water and dried at room temperature for 24 hours.

Figure 3.2 Fibers soaked with 5%, 10% and 15% NaOH solution

Figure 3.3 Alkali treated chopped fibers
3.3 PHYSICAL, CHEMICAL AND MECHANICAL TESTS OF BORASSUS FRUIT FIBER

3.3.1 Fiber Density Test

The water displacement method was employed to find the density of the Borassus fruit fibers. The weighed quantity of fibers was completely immersed in water and the volumetric displacement was observed. The weight to volume ratio yielded the density value.

3.3.2 Fiber Diameter Test

The air wedge shearing interferometer was used to measure the diameter of the Borassus fruit fibers. The two optical glass wedges which were tied together at one end were separated by the placement of the Borassus fruit fiber at the other end with a thin air wedge gap. The sodium lamp acts as monochromatic light for this experiment and the interference was observed using the microscope. The dark and bright alternative fringes were taken as reference to find the bandwidth and the fiber diameter was calculated by Equation (3.1).

\[ d = \frac{l \lambda}{2 \beta} \]  

(3.1)

where,  
\( d \) - Diameter of the fiber (mm),  
\( l \) - Length from the tied end of the glass plate to the fiber (mm)  
\( \lambda \) - Wave length for the sodium monochromatic light (mm)  
\( \beta \) - Bandwidth (mm).
3.3.3 Water Absorption Test

Water absorption properties of raw and alkali treated fibers were determined by placing the weighed quantity of the sample in a distilled water bath at room temperature (29 ºC) and measuring the water uptake percentage for different swelling time of 15, 45, 90, 150 and 225 minutes respectively. The natural fibers absorb water because the cell wall polymers have certain hydroxyl and other oxygenated groups that draw moisture during hydrogen bonding. The hemicellulose is mainly responsible for water absorption in the natural fiber. A comparison of the moisture absorption of raw and alkali treated samples was done. The Borassus fruit fiber was dried at room temperature for three days before testing. The increase in weight of the fiber was measured at various time intervals as mentioned above. After removing the samples from water, the excess surface water from the fibers were carefully removed by tissue paper and the samples were weighed. The percentage of water uptake is estimated by the Equation (3.2),

\[
W_u = \left( \frac{W_{as} - W_{bs}}{W_{bs}} \right) \times 100\% \tag{3.2}
\]

where, 
- \( W_u \) - The percentage of water uptake (g)
- \( W_{bs} \) - The weight of the sample before swelling (g)
- \( W_{as} \) - The weight of the sample after swelling (g)

3.3.4 Cellulose Content Test

The weighed quantity of Borassus fruit fibers were immersed in a mixture of sodium chloride 1.72 % and 3 drops of sulphuric acid in water for a period of 1 hour. Then the excess fluid was taken away by the suction process and ammonia was added. The residue was washed with distilled
water, dried at room temperature and weighed. The percentage of cellulose was noted by the ratio of the residue weight to the dry sample weight. The cellulose content was measured by Equation (3.3) for raw, 5% NaOH, 10% NaOH and 15% NaOH treated fibers.

\[
\text{Cellulose content in \%} = C = \frac{R}{D} \times 100
\]

(3.3)

where,

- \( C \) = Cellulose content in \%
- \( R \) = Weight of residue (g)
- \( D \) = Dry weight of the sample (g)

### 3.3.5 Lignin Content Test

The weighed quantity of the Borassus fruit fibers sample were immersed in a mixture of sulphuric acid 12.5 ml and water 300 ml at room temperature (29 °C). Two hours reflux time was provided. The solvents were removed and the residue was weighed. The residue weight was noted as the lignin content.

### 3.3.6 Hemi Cellulose Content Test

The dry Borassus fruit fibers of weighed quantity was immersed in a mixture of 5% NaOH solution at room temperature (29 °C) for 0.5 hours and then neutralized with Hydro chloric acid. The fibers were then dried in an oven, weighed and the weight difference accounts for the presence of hemi cellulose content.
3.3.7 Wax Content Test

The wax content was measured with the help of sox lot apparatus. Petroleum benzene liquid was heated to 70°C and one gram of Borassus fruit fibers were immersed in the liquid. The one hour reflux time was provided and the fiber sample was dried. After drying, the fibers were weighed and the weight difference confirmed the wax content.

3.3.8 Moisture Content Test

The weighed quantity of Borassus fruit fibers were placed in an oven at the temperature range of 105 ±2°C for 4 hours. The weight of the fibers taken from the oven was measured and the difference in weight accounts for the moisture content present in the fiber.

3.3.9 Tensile Test

The tensile properties of Borassus fruit fibers were determined by INSTRAN 5500 R-60211 machine at 50 ± 5% RH and 26 ± 1°C temperature with a gauge length of 50 mm and cross head speed of 5 mm/min. Thirty samples of raw, 5%, 10% and 15% NaOH alkali treated Borassus fruit fibers were subjected to tensile test and the average values were noted.

3.4 PREPARATION OF MATRIX

The matrix employed to fabricate the composite was epoxy LY556 of density 1.15 g/cm³ and hardener HY951 of density 0.98 g/cm³. The weight ratio for the mixture of epoxy and hardener was 10:1. Epoxy is a thermosetting polymer that cures (polymerizes and cross links) when mixed with a hardener. It can be made flexible or rigid, transparent or coloured, fast setting or extremely slow. A precise electronic weighing machine was employed to weigh the resin and hardener (Figures 3.4 and 3.5).
3.5 PREPARATION OF MOULD

The rectangular mild steel plate was made to the size 180 mm x 140 mm x 10 mm. The mould was split into three parts (Upper portion, Rectangular side plate and lower base plate) (Figure 3.6).
3.6 PREPARATION OF SPECIMEN

Raw and alkali treated Borassus fruit fibers were reinforced to the epoxy and the composites were prepared using hand layup and compression mould technique. The dry raw and alkali treated Borassus fruit fibers were cut into the required lengths of 1 mm, 3 mm, 5 mm, 7 mm and 10 mm respectively. The epoxy resin with hardener was thoroughly mixed with the required fibers in the ratio of 65:35. The mould was made to 180 mm x 140 mm x 10 mm size. The fiber resin mixture was spread uniformly in the mould up to the required thickness. The mould was kept in an oven at a temperature of 60 °C to remove void contents and make sure uniform wetting. The top plate was placed over the mould and a compression load was applied for 24 hours. The sample was removed from the mould and the specimens were cut into the standard size. The fiber volume fraction was determined by Equation (3.4).

\[ v_f = \frac{w_f / \rho_f}{(w_f / \rho_f) + (w_m / \rho_m)} \]  

(3.4)

where,  
\( w_f = \) Fiber weight fraction  
\( w_m = \) Matrix weight fraction and is equal to \((1-w_f)\)  
\( \rho_f = \) Fiber density  
\( \rho_m = \) Matrix density

The density of Borassus fruit fiber reinforced epoxy composites were measured using the Equation (3.5).

\[ \rho_c = \frac{1}{(w_f / \rho_f) + (w_m / \rho_m)} \]  

(3.5)

where,  
\( \rho_c = \) Density of composite materials
Figure 3.7 Weighing of Borassus fruit fibers

Figure 3.8 Mixing of epoxy resin and Borassus fruit fibers

Figure 3.9 Preparation of Borassus fruit fiber epoxy composites

Figure 3.10 Mould covered with the top plate
3.7 TENSILE TEST SPECIMEN

The tensile test specimens were made from the composite plate as per the ASTM D 638-03 -Type I standard. Tensile test specimens were tested in Electronic Tensometer – Model PC - 2000 operated with a 20 KN load cell with digital load controller and extension microprocessor based elongation measurement set up. The cross head speed was 5 mm/min and the gauge length maintained was 50 mm. The tensile test was conducted at 28 °C and at a relative humidity of 50 ± 2%. The grippers held the specimen in the longitudinal axis and the load was applied over the specimen. The loads and the corresponding strains were noted. The tensile modulus and elongation at the break of the composites were calculated from the load-displacement curve. Five samples were tested for each composition and the mean value was reported.

Figure 3.11 Tensile test specimens (With grip lines)

Figure 3.12 Electronic tensometer with accessories
3.8 COMPRESSION TEST SPECIMEN

The compressive strength test specimens were made from the composite plates as per the ASTM D 695-02a standard. The size of the specimen was 12.7 mm x 12.7 mm x 50.8 mm. The compressive strength specimens were tested in compressometer. The test was conducted at 28 °C, at relative humidity of 50 ± 2% and at a cross head speed of 2 mm/min. The specimen was placed over the round disc and the load was applied over the specimen. The load cells and the strain gauge values for the corresponding loads and strains were noted. Five samples were tested for each composition and the mean value was reported.
3.9 IMPACT TEST OF SPECIMEN

The Impact test specimens were made from the composite plates as per the ASTM D 256-05 standard. The size of the specimen is 64 mm x 13 mm x 5 mm and the specimens were notched. The Izod digital impact tester, Frank–53568 was employed for conducting the impact test at room temperature. The test specimen was supported by a vertical cantilever beam and was broken by a single swing of a pendulum and the corresponding impact strength was recorded. Five samples were tested for each composition and the mean value was reported.
3.10 FLEXURAL TEST SPECIMEN

The flexural test specimens were made from the composite plate as per the ASTM D 790-03 standard. The size of the specimen was 127 mm × 12.7 mm × 5 mm. The Lloyd instrument LR 100 kN was used for conducting the flexural test at 28 °C and at a relative humidity of 50 ± 2%. The cross head speed was 2 mm/min. The sample was loaded till the core broke and the flexural strength, flexural loads were recorded. The flexural modulus values were calculated. Five samples were tested for each composition and the mean value was reported.
3.11 WATER ABSORPTION TEST SPECIMEN

Water uptake in Borassus fruit fiber reinforced epoxy composites was measured according to ASTM 570-98. The size of the specimen was 76.2 mm X 25.4 mm X 5 mm. The specimens were pre-conditioned by drying in open air until constant weight was obtained. Water absorption properties of raw and alkali treated specimens were determined by placing the weighed quantity of sample in a distilled water bath at room temperature and 100 °C boiled water. The water uptakes at 2 hours time intervals were measured. The percentage of water uptake was estimated.

Figure 3.20 Specimen immersed in cold water (29 °C)
3.12 MACHINABILITY

The composite specimen was cut into the rectangular shape of 40 mm × 40 mm × 10 mm size and it was fitted in a drill tool dynamometer hub. The machining properties of the Borassus fruit fiber reinforced epoxy composites were studied using drilling dynamometer analysis. In this work, the drill hole was made in the composite material by using 4 mm, 6 mm, 8 mm and 10 mm HSS drill bit and the thrust force was measured for a speed of 260 rpm (Figure 3.22).
3.13 FOURIER TRANSFORM INFRARED SPECTROMETRY (FTIR)

The Fourier Transform Infrared Spectrometry was performed using the thermo scientific NICOLET IS10 spectrometer at room temperature (29 °C). The infrared light was passed through the Borassus fruit fiber sample. When the infrared frequency was the same as the vibrational frequency of the bond, the absorption took place. The FTIR spectrometer recorded the interferogram and performed Fourier Transform on this interferogram to get the spectrum. The absorption spectrum was produced and based on the analysis of this absorption spectrum, the functional compounds of the sample were assigned. The spectrometer was used in the transmission mode with a resolution of 4 cm⁻¹ in the range of 4000-400 cm⁻¹. The FTIR spectrum was made for raw, 5%, 10% and 15% NaOH alkali treated Borassus fruit fibers and for raw, 5% alkali treated 1 mm, 5 mm length Borassus fruit fiber reinforced composites.

![Figure 3.23 Fourier Transform Infrared Spectroscopy (FTIR)](image)

3.14 SCANNING ELECTRON MICROSCOPY (SEM)

Scanning Electron Microscope images were taken by machine model JEOL-JSM-6390. The instrument scanned the surface by raster fashion with a high-energy electron beam. The surface topography information was
produced because the electron beam interacts with the atoms on the surface. The SEM produced very high-resolution images. The very thin electron beam in SEM micrographs made a characteristic three-dimensional manifestation which was useful for understanding the surface structure of the sample. The Scanning Electron Microscopy was made on raw, 5%, 10% and 15% NaOH treated Borassus fruit fibers. Also the surface morphology for the tensile, flexural and worn raw and alkali treated specimens were made.

3.15 THERMOGRAVIMETRIC ANALYSIS

The samples in the form of pellets were cut from the raw and alkali treated 1 mm, 3 mm, 5 mm, 7 mm and 10 mm length Borassus fruit fiber reinforced composite specimens for thermogravimetric analysis. The weight per sample taken was 10 mg. The samples were kept in an oven at 100 °C for 1 hour. The thermogravimetric analysis was conducted in the machine Netzsch STA 409 at a heating rate of 10 °C/min and in Nitrogen environment with a flow rate of 50 ml/min.

3.16 WEAR TEST

3.16.1 Specimen for Wear Test

The specimens of Neat epoxy (NE), raw and alkali treated Borassus fruit fiber reinforced epoxy composites with three different fiber lengths such as 3 mm, 5 mm and 7 mm were prepared using hand layup and compression mould technique. The rectangular shaped specimens were prepared with 10 mm sides and 75 mm length. The specimens were prepared by mixing chopped fibers of each length and resin at a ratio of 65:35 by manual stirring for a sufficient time to scatter the fiber in the matrix (Figure 3.24. a & b). This sample was cured for 24 hours at room temperature (29 ºC) and then taken out of the mould.


3.16.2 Wear Test Procedure

The wear tests were performed using a ‘pin on disc’ machine shown in Figure 3.25. The wear test specimens were slid against stainless steel disc. The loads applied were 15, 20, 25 and 30 N with varying sliding velocities of 1.413, 1.884 and 2.355 m/s corresponding to the speeds of 300, 400 and 500 rpm. The test duration of 300 seconds for a sliding distance of 90 mm was considered. The weight losses in the pin were determined using 0.0001 gram balance. The wear rate (W) of materials were calculated using the Equation (3.6).

\[ W = \frac{w}{\rho D} \quad (3.6) \]

where, \( w \) = Weight loss in kg
\( \rho \) = Density of materials in kg/mm\(^3\)
\( D \) = Sliding distance in m

The specific wear rate (P) was also calculated using the Equation (3.7).

\[ P = \frac{\text{Wear rate (W)}}{\text{Normal load (L)}} \quad (3.7) \]

The wear resistance of the material is reciprocal to the specific wear rate.

Figure 3.24. a - Neat epoxy wear test specimens
Figure 3.24. b- Borassus fruit fiber-epoxy composite wear test specimens

Figure 3.25 Pin on disc wear test apparatus with specimen

3.17 Experimental flow chart

The various experiments conducted on Borassus fruit fiber and the Borassus fruit fiber reinforced composites are illustrated in flow chart Figure 3.26. a & b follows.
Figure 3.26a Experimental flow chart- Borassus fruit fiber

Figure 3.26b Experimental flow chart- Borassus fruit fiber reinforced epoxy composites
Summary: The Borassus fruit fibers were carefully extracted through retting process. The fibers were alkali treated with 5%, 10% & 15% NaOH solution and dried. The physical, chemical and mechanical properties were evaluated as per the standard tests. Raw and 5% alkali treated Borassus fruit fibers of 1 mm, 3 mm, 5 mm, 7 mm and 10 mm lengths were reinforced in the epoxy matrix and the composites were made. The various test specimens according to the ASTM standards were tested to evaluate physical, chemical, mechanical and tribological properties. The surface morphology of the failure specimens was also made.