

CHAPTER 3

MATERIALS AND METHODS

3.1 INTRODUCTION

3.1.1 Regular and Compact Cotton Yarns

The Regular and compact yarns of the following counts were obtained from a well known textile mill in South India.

Table 3.1 Yarn samples

Sample No	Experimental yarns	Count (Ne)	Tex
1	Regular	60	9.84
2	Compact	60	9.84
3	Regular	80	7.38
4	Compact	80	7.38

3.1.2 Sisal Fibres

Sisal fibres of density of 1.45g/cc and diameter of 180-300(μ m) were obtained from an Industrial source. After obtaining the fibres, they were cut into size of 5mm and they were first washed thoroughly with 2% detergent water and air-dried. The dried fibres were designated as untreated fibres.

3.1.3 Chemicals

Chemicals used for yarn and fibre surface treatments were of AR grade and were used without purification. For enzyme treatment, cellulase enzyme (acidic pH) was obtained from reputed suppliers.

3.1.4 Polypropylene

Isotactic polypropylene (H110MA), density 0.91g/cc and melt flow index 11g/10 min, obtained from a well known supplier were used as the base polymer matrix.

3.1.5 Nano Clay (Cloisite 25A)

Cloisite 25A was obtained from a well known reputed supplier. Cloisite 25A is a natural montmorillonite modified with a quaternary ammonium salt. Cloisite 25A is an additive for plastics to improve various plastic physical properties, such as reinforcement, HDT and barrier.

- Specific Gravity 1.87
- Bulk Density 0.1935 g/cc (loose)
 0.3281 g/cc (packed)
- Loss On Ignition 34%
- Moisture Content <= 2.0 %
- XRD 18.6 A

3.1.6 PP-g-MA

PP-g-MA (Epolene G-3015), having <1.0 wt% maleic anhydride, with M_w 47,000 and acid number 15, obtained from M/s Eastman Chemical, Germany was used as compatilizer.

3.2 Conditioning of Yarn Sample

Cotton, being a hygroscopic fibre, changes its weight with respect to the relative humidity of its atmosphere. Thus the cotton yarn is to be conditioned prior to experimentation. Conditioning can be done by many methods like keeping the yarn in the air conditioned room or keeping the yarn in mini atmosphere of saturated salt solution. The yarn samples were kept in the desiccator and salt is added in the desiccator to maintain a RH. The yarn samples were kept for three days to achieve the correct amount of weight.

3.3 Yarn Pretreatments

3.3.1 Scouring

Batches of cotton yarns (144 samples) were scoured with caustic boil (3-4% Sodium Hydroxide, 3-4% Sodium Carbonate and soap) at 100° C for 60 minutes. The yarns were washed thoroughly with distilled water.

3.3.2 Bleaching

The wetted scoured leas were bleached with 12ml/L of 100% hydrogen peroxide at 80–85°C in stock solution of 2gpl (5%)sodium hydroxide, 2gpl (5%) sodium carbonate and 2gpl(5%) sodium silicate for 60 minutes followed by thorough washing with distilled water.

3.3.3 Mercerization

The scoured and bleached leas were mercerized at room temperature with 20% NaOH for 45 minutes. Then they were washed to remove the traces of alkali and dried.

3.4 ENZYMATIC TREATMENT

Enzymatic treatment of the original and the pretreated cotton yarns was performed with acidic cellulase enzyme. The treatments were conducted in 50mM Sodium acetate buffer (pH 5) at 40° and 50°C with various concentrations like 0.5, 1, 2 % solutions. The MLR ratio was 1:50 taken in high temperature, high pressure dyeing machine with gentle mechanical agitation for cotton yarns (9.84 and 7.38 tex). The treatments were performed at 50°C for 1.5 hour and carried out in triplicates. After the incubation period, enzyme activity was ceased by raising the temperature of the liquid to 80°C for 10 minutes. The yarns were washed three times with distilled water and dried at 40°C overnight prior to further analysis.

3.5 DESIGN OF THE EXPERIMENTS

The experiments were designed to study the (i) Weight loss, shrinkage loss, wicking, tensile and bending properties. (ii) FTIR & SEM images.

3.6 TESTING OF YARNS

Yarn number was defined according to the standard SFS 2703. Weight loss, shrinkage loss, wicking and tensile, bending properties were studied.

3.6.1 Weight Loss

The masses of the yarn samples before (w_0) and after the enzyme treatment (w) were determined and the weight loss was calculated by equation

$$\text{Weight loss} = \{w_0 - w/w_0\} * 100 \quad (3.1)$$

3.6.2 Wickability

When a textile yarn comes in contact with liquid water, the water rise due to the capillary phenomena. Vertical wicking test was carried out on treated yarns following DIN 53924.

3.6.3 Tensile Properties

Elongation and tenacity of all the yarns were measured on an Instron using 500mm test specimen and 200mm/min cross head speed. The mean yarn tenacity and breaking extension were averaged from 50 observations for each yarn sample.

Instron tensile tester was also used for the measurement of strength of cotton yarns at different gauge lengths. The traverse speed of 5mm/min and 500mm/min were selected. The tensile tests were performed at five different gauge lengths viz 254mm, 127mm, 76.2mm, 25.4mm and 12.7mm. The mean tenacity variability and elongation were estimated. Table 3.2 gives the details of particulars of Instron Tensile Tester. Figure 3.1 shows the Instron Tensile Tester used in this study.

Table 3.2 Details of the particulars of Instron tensile tester

Instrument	Model	Cross head speed (mm/min)	Gauge Length (mm)
Instron	5500R	5	254
		500	127
			76.2
			25.4
			12.7



Figure 3.1 a) Instron Tester 5500R, b) Instron measuring tensile strength at 20mm gauge length

3.6.4 Work of Rupture

Work of rupture is defined as the amount of energy needed to break a material and is represented by the area under the stress-strain curve. It is obtained from the formula,

$$\text{Work of rupture} = \frac{1}{2}(\text{Breaking load} \times \text{Breaking elongation}) \quad (3.2)$$

3.6.5 Initial Modulus

It is defined as the ratio between the stress and strain.

$$\text{Initial modulus} = \text{Stress} / \text{Strain\%} \quad (3.3)$$

3.6.6 Shrinkage

Shrinkage is mainly due to yarn swelling and the resulting crimp increase. Residual shrinkage of yarns was estimated by the method mentioned in BSI handbook.

3.7 SCANNING ELECTRON MICROSCOPY

The morphology of the different yarns was studied using a scanning electron microscope, ZEISS EVO 60, Germany at an acceleration voltage of 5-10 kV.

3.7.1 Bending Test

Peirce suggested a method of measuring yarn flexural rigidity in which the specimen is formed into ring of diameter 2cm. The ring is hooked and the deflection caused by a weight hung on the load at its lowest point is observed. The ring in all cases is small enough for it not to be appreciably distorted by its own weight: passing the yarn round a suitable test tube and

tying in with a reef knot may form it. The ends must be cut off closed to the knot, and the ring hung up the knot is at one end of the position of the tangent to the ring makes an angle of 45 degree with the vertical, the reason for this is that little bending occurs in this regions when the load is applied. A rider is loaded onto the loop such that θ lies between 40- 50 degree and the deflection d is measured Flexural rigidity is calculated using the formula:

$$\text{Flexural rigidity (g.cm}^2\text{)} = 0.0047 * W * L^2 * \cos\theta / \tan\theta \quad (3.4)$$

Where,

W : Weight in grams

L : Circumference of the loop (cm)

θ : $493 * d / L$

d : Deflection(cm)

Specific flexural rigidity is obtained from the following formula

$$\text{Specific flexural rigidity} = \text{Flexural rigidity [mN.mm}^2\text{]} / \text{Tex}^2$$

Flexural rigidity was measured by ring loop method proposed by Carlene (1950). In this method, a length of yarn or filament is bent through 360° to form a ring. The ends are gripped and the distortion produced in the ring shape by some externally applied weight is observed. In these circumstances, the flexural rigidity G is given by the equation

$$G = KWL^2 \left(\frac{\cos\theta}{\tan\theta} \right) \quad (3.5)$$

where K is a constant, the value of which is around 0.0047

W= applied load

Diameter of the loop =2cm

3.7.2 Minimum Twist of Cohesion

This parameter was measured by the method developed by Barella et al (1996). A twist tester was used and the yarn of 25mm length was fixed under a tension of 0.1g/tex. The method described by Gokarneshan et al (2005) was followed.

$$\text{Twist/inch} = \text{Twist multiplier} * \text{Count}^{1/2} \quad \text{ie } TM * C^{1/2} \quad (3.6)$$

3.7.3 Design and Development of Weibull Distribution

Weibull modeling is used to model extreme values such as failure times and fracture strength. The variability in bleached, bleached and enzyme treated cotton yarns is studied by Weibull modeling. The scale and shape parameters of the distribution function $F(x,b,c)$ are estimated from the values obtained. The methods which are employed in the estimation of these parameters are method of linear regression using MS Excel. This method involves transforming equation,

$$F(x,b,c) = 1 - \exp(-(x/b)^c) \quad \text{into} \quad 1 - F(x,b,c) = \exp(-(x/b)^c) \quad (3.7)$$

and taking double logarithms on both sides. Hence, a linear regression model in the form $Y = mX + c$ is obtained.

3.8 PREPARATION OF COMPOSITES

Various composites prepared are presented in 3.3 to 3.6.

Table 3.3 Various compositions of sisal/ Cotton fibres reinforced PP(mer-CSFPP)

PP (wt.%)	PPGMA (wt.%)	Sisal fibre (wt.%)	Cotton fibre (wt.%) Mercerised	Corresponding code Mer - CSFPP
100	-	-	-	0%
88	2	5	5	10%
78	2	10	10	20%
68	2	15	15	30%
58	2	20	20	40%

Table 3.4 Various compositions of sisal/ Cotton fibres reinforced PP (cell-CSFPP)

PP (wt.%)	PPGMA (wt.%)	Sisal fibre (wt.%)	Cotton fibre (wt.%) Cellulase treated	Corresponding code Cell-CSFPP
100	-	-	-	0%
88	2	5	5	10%
78	2	10	10	20%
68	2	15	15	30%
58	2	20	20	40%

Table 3.5 Various compositions of cotton fibres reinforced PP composite

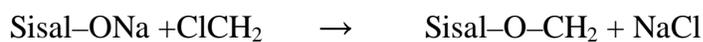
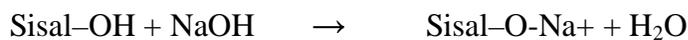
PP (wt.%)	PPGMA (wt.%)	Cotton fibre (wt.%) Mercerised	Corresponding code CFP
100	-	-	0%
88	2	10	10%
78	2	20	20%
68	2	30	30%
58	2	40	40%

Table 3.6 Various compositions of sisal/ Cotton fibres reinforced PP nano composite

PP (wt.%)	PPGMA (wt.%)	Sisal fibre (wt.%)	Cotton fibre (wt.%) mercerised	Nanoclay	Corresponding code CSFPP nano composite
68	2	15	15	0	30% CSFP
67	2	15	15	1	1%
65	2	15	15	3	3%
63	2	15	15	5	5%
61	2	15	15	7	7%

3.9 EXPERIMENTAL

Prior to benzylation, the fibres were chopped and cleaned using a benzene-ethanol mixture in a 2:1 liquor ratio by volume (v/v=2:1). Sisal fibres were soaked in NaOH solution of desired concentration while heated at 80°C and stirred for 1 hour and 30 minutes. Pre-treatment of sisal with NaOH can partially remove lignin and hemicellulose, and certainly results in weight loss of the fibres. To quantify the the amount of material removed, the alkali treated products were filtered and washed with distilled water to pH =6~7, and then dried under vacuum at 60°C until constant weight was reached. Benzlyation of lignocellulosic materials follows the typical Williamson synthesis reaction, which involves the nucleophilic substitution of an alkoxide or a phenoxide ion for a halide ion. The chemical modification of sisal fibre bundles using NaOH followed by benzoyl chloride treatment is represented in the following reaction scheme:



where Sisal-OH represents the hydroxyl groups present on the polymeric components of sisal fibres. Since cellulose constitutes the majority of sisal, and lignin contains little hydroxyl, benzyl chloride has to react mainly with the hydroxyl groups of cellulose.

3.9.1 Physical Properties

3.9.1.1 Water absorption (ASTM D570)

Mechanical properties of plastics are affected by the absorption of water and it depends on its exposure condition. Water absorption was measured according to ASTM D 570 by immersing the test specimen in the form of 50 mm disc and 3.2 mm thickness in water at room temperature for 24 hours. The increase in weight due to absorption of water was reported in percentage.

The percentage gain at any time “t” as a result of moisture absorption was determined by

$$\text{Water absorption} = \frac{W_w - W_d}{W_d} \times 100 \quad (3.8)$$

where W_d and W_w denote the weight of dry material (the initial weight of material prior to exposure to the water absorption) and weight of material after exposure to water absorption, respectively.

3.9.2 Mechanical Properties

The mechanical properties are often the most important sources to make a decision about product specifications.

3.9.2.1 Tensile Strength Test (ASTM D638)

Tensile test is a measurement of the ability of a material to applied forces tending to pull it apart and observe the extent of material stretches

before breaking. Different types of plastic materials are often compared based on tensile property data (i.e. strength, modulus, and elongation data). A testing machine of a constant-rate-of-crosshead movement, containing a stationary member carrying one grip, and a movable member carrying the second grip, is used. The test specimens are mostly either injection or compression moulded. Test specimen dimensions vary considerably depending on the requirements and are described in related section in the ASTM book of standards. The specimens are conditioned using standards of procedures. The recommended test conditions are $23 \pm 2^\circ\text{C}$ as a standard laboratory atmosphere and 50 ± 5 percent relative humidity.

There are basically five different testing speeds mentioned in the ASTM D638 Standard. As the specimen elongates, the resistance to the tension increases, and it is detected by a load cell. The tensile strength can be calculated by dividing the maximum load in newtons by the original minimum cross sectional area of the specimen in square millimeters, and the result can be explained in the term of Megapascal (MPa). The tensile strength at yield and at break (ultimate value) are calculated.

$$\text{Tensile strength} = \frac{\text{Force(Load)}(N)}{\text{Cross sectionalArea}(mm^2)} \quad (3.9)$$

$$\text{Tensile strength yield (MPa)} = \frac{\text{MaximumLoadrecorded}(N)}{\text{Cross sectionalarea}(mm^2)} \quad (3.10)$$

$$\text{Tensile Strength at Break (MPa)} = \frac{\text{LoadrecordedatBreak}(N)}{\text{CrossSectionalArea}(mm^2)} \quad (3.11)$$

Tensile modulus and elongation values are derived from the stress-strain curve. If the specimen gives a yield load larger than the load at break, percent elongation at yield is calculated; if not, percent elongation at break is calculated.

$$\text{Strain (E)} = \frac{\text{ChangeinLength}(elongation)}{\text{OriginalLength}(gauge\text{length})} = \frac{\Delta L}{L} \quad (3.12)$$

Elongation at yield : $\Delta L = \epsilon$ (the value at the yield point on the x - axis) $\times L$

$$\text{Percent elongation at yield} = \Delta L \times 100 \quad (3.13)$$

Tensile modulus (the modulus of elasticity) can be determined by extending the initial linear portion of the load-extension curve and dividing the difference in stress obtained from any segment of section on this straight line by the corresponding difference in strain, expressing the result in the unit of megapascal (MPa).

$$\text{Tensile Modulus} = \frac{\text{Difference in Stress}}{\text{Difference in corresponding Strain}} \quad (3.14)$$

In present study, the test was performed by using the specimen, with dimension of 165×12.7×3 mm. The gauge length was fixed at 50 mm and the test was carried out at a cross head speed of 50 mm/min.

3.9.3 Flexural Properties (ASTM D790)

3.9.3.1 Introduction

The flexural test measures the force required to bend a beam under 3 point loading conditions (Figure 3.2). The data is often used to select materials for parts that will support loads without flexing. Flexural modulus is used as an indication of a material's stiffness when flexed. Since the physical properties of many materials (especially thermoplastics) can vary depending on ambient temperature, it is sometimes appropriate to test materials at temperatures that simulate the intended end use environment. Flexural strength is the ability of the material to applied bending forces perpendicular to the longitudinal axis of the specimen. The stresses induced by flexural load are a combination of compressive and tensile stresses, and properties are calculated in terms of the maximum stress and strain occurring at the outside

surface of the test bar. These test methods are generally applicable to rigid or semi-rigid materials.

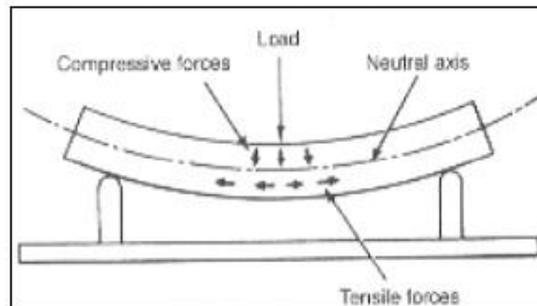


Figure 3.2 Representation of Flexural Test

3.9.3.2 Test Procedure and measurement

Most commonly the specimen lies on a support span and the load is applied to the center by the loading nose producing three points bending at a specified rate. The parameters for this test are the support span; the speed of the loading; and the maximum deflection for the test. These parameters are based on the test specimen thickness, and are defined differently by ASTM-D790 and ISO. Specimens of virgin PP, PP/sisal/cotton composites having dimensions 80x12.7x3 mm were taken for flexural test, under three point bending, using the same UTM in accordance with ASTM-D 790 at cross head speed of 1.5 mm/min and span length 50 mm [Figure 3.3]. Flexural strength, flexural stress at specified strain levels, and flexural modulus are calculated.



Figure 3.3 Flexural specimens

Two basic methods, including a three-point loading system utilizing center loading on a sample supported beam, and a four-point loading system utilizing two load points, are employed to determine the flexural properties. The former is designed particularly for materials undergoing small deflections, whereas the latter particularly for materials with large deflections during testing. The test specimens used for flexural testing are obtained from sheets, plates or moulded shapes by cutting as bars with rectangular cross section.

Flexural strength is equal to the maximum stress in the outer fibres at the moment of break, and calculated using the following equation,

$$S = \frac{3PL}{2bd^2} \quad (3.15)$$

Where S is the stress in the outer fibres at midspan (MPa), P is the load at a given point on the load-deflection curve (N), L is the support span (mm), b and d are the width and the depth of beam tested respectively (mm).

The maximum Strain in the outer fibres occurs at midspan which is calculated as follows,

$$r = \frac{6Dd}{L^2} \quad (3.16)$$

Where r is the maximum strain in the outer fibres (mm/mm), D is the maximum deflection of the center of the beam (mm), L is the support span (mm), and d is the depth of the sample (mm).

$$E_n = \frac{L^3}{4Bd^3} \quad (3.17)$$

The modulus of elasticity is the ratio, within the elastic limit of stress to corresponding strain, and can be represented by the slope of the initial straight-line portion of the stress-strain curve, calculating as follows.

Where, E_b shows the modulus of elasticity in bending (MPa), L is the support span (mm), b and d are the width and the depth of beam tested, respectively (mm), and m is the slope of the tangent to the initial straight line portion of the load-deflection curve (N/mm).

3.9.4 Impact Properties (ASTM D256)

3.9.4.1 Introduction

Izod impact strength testing is an ASTM standard method (ASTM-D256), of determining impact strength. A notched sample is generally used to determine impact strength. The impact properties of the polymeric materials depend mainly on the toughness of the material. Toughness can be described as the ability of the polymer to absorb applied energy. The molecular flexibility has a great significance in determining the relative brittleness of the material. Impact energy is a measure of toughness, and the impact resistance is the ability of a material to resist breaking (fracture) under a shock loading. An arm held at a specific height (constant potential energy) is released. The arm hits the sample and breaks it. From the energy absorbed by the sample, its impact strength is determined. Impact strength is so sensitive to molecular orientation, crystallinity and crystal texture that some variations in the strength point to point are to be expected in injection mouldings.

3.9.4.2 Test Procedures and measurements

Specimens of dimension 63.5 x 12.7 x 3 mm. were taken for measurement of impact test in an Impactometer (M/s Ceast, Italy) as per ASTM D 256. The specimens were notched at angle of 45° and depth of 2.54mm using notch cutter (M/s Ceast, Italy) prior to test.

Two basically different test methods, namely Izod type and Charpy type, are used generally. Present study utilizes the Izod type impact testing. In Izod type testing, the specimen is clamped vertically to a cantilever beam and the pendulum arm swung from certain height is made to impact on a notched cantilevered specimen. The loss of energy is measured in ft-lb/in or J/m or kg-mm/c m of specimen thickness, is known as Izod impact strength. The Izod impact test differs from the Charpy impact Test in that the sample is held in a cantilevered beam configuration as opposed to a three point bending configuration.(Figure 3.4).

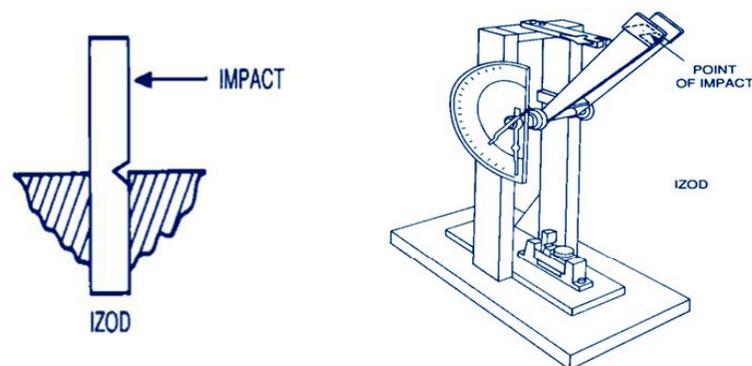


Figure 3.4 (a) specimen in hold position (b)Izod Impact meter

3.10 ABRASION RESISTANCE (ASTM D1044)

Abrasion resistance is defined as the ability of a material to withstand mechanical action of rubbing, scrapping or erosive that tends to progressive removal of the material from its surface. The resistance of abrasion is most often measured by loss of weight when abraded by abrasive. The loss of material due to mechanical action causes damage to the product such as weakening of the product, loss of surface gloss, transparency and degradation etc. Therefore abrasion resistance is the determining factor for the performance of a product. ASTM D-1044 Standard test method for resistance of transparent plastics to surface abrasion.

Taber Abrasion: The basic principle of tabor abrader is the rotation of test disc at a prescribed speed under the abrading wheels freely resting there on with load of 250, 500 or 1000g acting of them. Material to be tested is subjected to wear action of two abrasive wheels at known pressure against surface of test samples. Results are evaluated in weight loss of the test disc induced by abrading wheels.

Test specimen and conditioning: Test sample of 100 mm diameter disc and thickness of approximately 2.5 mm is used. Surface shall be plane and parallel. Sample is a drilled with a hole of 6.3mm at the centre and fixed on turntable. Test specimen was conditioned at standard test temperature and humidity of $23\pm 2^{\circ}\text{C}$ and $50\pm 5\% \text{RH}$ for a specified period of time.

Equipment: Taber abrader consisting of abrasive wheel with standard load 250g was attached to it and connected to a vacuum pump to remove abraded material. CS-10 was used for rigid material and analytical balance of 0.0001g accuracy was used for weighing the sample by loss of

weight method. The initial weight of the test sample was taken in analytical balance of accuracy of 0.1mg. The specimen was placed on revolving turn table on taber abrader. A pair of abrasive wheels was mounted on respective flange holder and calibrated using abrasive wheel CS-10. The test was carried out for 500 revolutions. The vacuum device was positioned over specimen sample so as to take away the abraded particles. The loss in weight was measured by analytical balance for weight loss in milligram per 500 cycles. It is expressed in % Loss of weight.



Figure 3.5 Taber abrader machine

3.11 THERMAL ANALYSIS

3.11.1 Differential Scanning Calorimetry (DSC):

Using DSC (Diamond DSC Perkin Elmer, USA, melting and crystallization behaviour of virgin PP, composite and hybrid composite samples were studied under nitrogen atmosphere. Samples of 5 – 10 mg weight were heated from 40 to 200°C at the heating rate of 10°C/min to detect the melting characteristics of the virgin matrix, composites and hybrid composite materials for isothermal crystallisation, the samples were heated from 40 – 20°C, followed by cooling to 40°C at the cooling rate of 10°C/min. Figure 3.6 shows details of DSC sample cell.

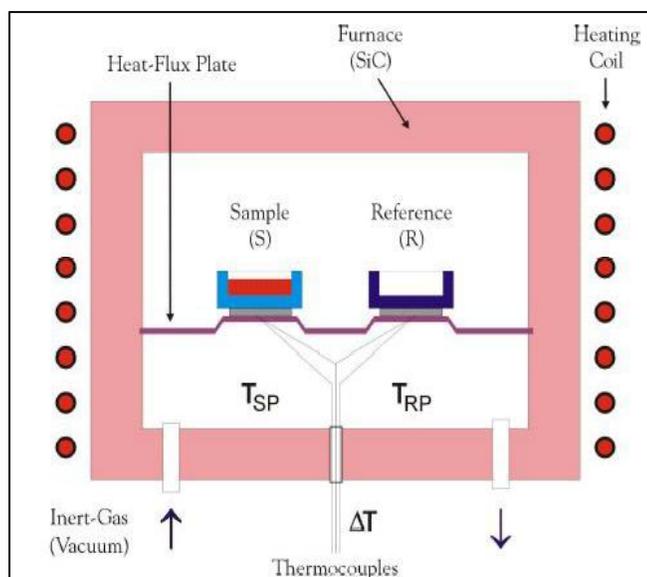


Figure 3.6 Schematic representation of typical DSC sample cell

Make	Perkin Elmer, SA
Model	Pyris Diamond DSC
Maximum heating /Cooling rates	500 °C/min.
Temperature range	-170 to 800 °C
Sensitivity	0.2 μw
Dynamic range	02 μw to 800 mw

3.11.2 Thermo-Gravimetric Analysis (TGA)

TGA is a technique that determines the weight change of a sample (TG) and measures the change in temperature between a sample and the reference as a function of temperature. Thermo gravimetric analysis (TGA) measures the mass change in a sample as a function of temperature or time. The technique provides the following valuable information on materials:

The rate of decomposition and thermal and oxidative stability of the virgin polymer and composites were investigated under Nitrogen atmosphere using thermogravimetric analysis (TGA) (Pyris 7, Perkin Elmer, USA). Sample ≤ 10 mg weight were heated from 50 to 600 °C at the heating rate of 20 °C/min to investigate the thermal stability of the materials.

Make	Perkin Elmer, USA
Model	Pyris 7 – TGA
Balance capacity	500 °C/min
Temperature range	5 to 1000 °C
Scanning rate	0.1 to 200 °C/min



Figure 3.7 TGA Instrument (Perkin Elmer Inc.)

3.11.3 Heat Deflection Temperature (HDT)(ASTM D648)

As per the standard, ASTM D648, the distortion temperature under a 0.455 MPa load was measured with a heat deflection temperature tester, GEOTECH Testing Machine. Test specimens of size 127 x 12.7 x 3.2 mm³ obtained at rate of 2°C/min in oil bath were used. The temperature was measured when the test pieces had deflected to 0.25 mm.

The heat deflection temperature or heat distortion temperature (HDT, HDTUL or DTUL) is the temperature at which a polymer or plastic sample deforms under a specific load. This property of a given plastic material is applied in many aspects of product design, engineering, and manufacture of products using thermoplastic components. The heat deflection is determined by the following test procedure outlined in ASTM D638. The test specimen is loaded in three-point bending in the edging direction. The outer fibre stress used for testing is either 0.455 MPa, and the temperature is increased at 2°C/min until the specimen deflects 0.25 mm, this is similar to the test procedure defined in the ISO limitations that are associated with the determination of HDT in the sample is not thermally isotropic and, thick samples in particular, will contain a temperature gradient. The HDT of a particular material can also be very sensitive to stress experienced by the component which is dependent on the component's dimensions. The selected deflection of 0.25mm (which is 0.2% additional strain) is selected arbitrarily and has no physical meaning.

3.12 SCANNING ELECTRON MICROSCOPY

The morphology of the impact fractured surface of the virgin PP and the composites are examined employing SEM, JOEL JSM-5800, Japan. The samples were gold sputtered (50nm) and dried for 30 min in vacuum prior to study.

3.13 X-RAY DIFFRACTION

The interlayer distance of nanoclay in the nanocomposites was studied by wide angle X ray diffraction. Both for the clays and nanocomposites, XRD was recorded using Philips X'pert MPD (Japan), which had a graphite monochromator and a Cu K α radiation source operated at 40kv and 30 mA. The basal spacing of d_{001} reflection of the samples was calculated from Bragg's equation by monitoring the diffraction angle 2θ from $2-10^\circ$ at a scanning rate of $0.5^\circ/\text{min}$.

3.14 COMPOUNDING AND PROCESSING

3.14.1 Haake Rheocord

Instrumentation

The HAAKE Rheocord base unit contains all elements of a torque rheometer that either drive the measuring sensors or detect the resulting torque. Built-in interfaces enable communication with:

- A control and analysis module
- A remote control unit
- Measuring sensors
- External devices
- A local area network



Figure 3.8 HAAKE Rheocord

Due the flexibility of the measuring bus technology, a single unit satisfies all needs – both of current and future applications. Measuring sensors are driven by a state-of-the art power unit, enabling operation at the optimum bias point of the drive unit at all times. This leads to load characteristics that can compensate even peak demands under extreme operating conditions, e.g. in high torque/high speed conditions. Torque measurement is carried out via a measuring cell with integrated amplifier that has been custom designed for the HAAKE Rheocord thus providing the highest level of measuring accuracy and tolerance to interference for your application.

The measuring accuracy of the unit is sufficient for all applications. Additionally, a number of easy-to install, interchangeable, coded torque sensors provide extra high resolution for application threshold ranges, should this be required. A real-time operating system with the look and feel of a standard Microsoft Windows GUI provides functions for drive unit control, measuring data bus control and data transfer in.

Applications

Thermo can supply a range of measuring systems for the rheological testing of your polymers:

- A HAAKE polylab system in combination with a HAAKE Rheomex measuring extruder and capillary dies as an extrusion capillary rheometer
- HAAKE polylab system in combination with a HAAKE Rheomix mixer torque rheometer for relative measurements of rheological properties

- A HAAKE rheostress unit equipped with a HAAKE roto visco rotation rheometer for plate-plate or plate-cone measuring geometries

3.14.2 Compression Moulding

Compression moulding is a method of moulding in which the moulding material, generally preheated, is first placed in an open, heated mould cavity. The mould is closed with a top force or plug member, pressure is applied to force the material into contact with all mould areas, while heat and pressure are maintained until the moulding material has cured. The process employs thermosetting resins in a partially cured stage, either in the form of granules, putty-like masses, or preforms. Compression moulding is a high-volume, high-pressure method suitable for moulding complex, high-strength fibre glass reinforcements. Advanced composite thermoplastics can also be compression moulded with unidirectional tapes, woven fabrics, randomly oriented fibre mat or chopped strand. The advantage of compression moulding is its ability to mould large, fairly intricate parts. Also, it is one of the lowest cost moulding methods compared with other methods such as transfer moulding and injection moulding; moreover it wastes relatively little material, giving it an advantage when working with expensive compounds. However, compression moulding often provides poor product consistency and difficulty in controlling flashing, and it is not suitable for some types of parts. Fewer knit lines are produced and a smaller amount of fibre length degradation is noticeable when compared to injection moulding. Compression-moulding is also suitable for ultra-large basic shape production in sizes beyond the capacity of extrusion techniques. Materials that are typically manufactured through compression moulding include: Polyester fibreglass resin systems (SMC/BMC), Torlon, Vespel, Poly(p-phenylene sulfide) (PPS), and many grades of PEEK.

Materials may be loaded into the mould either in the form of pellets or sheet, or the mould may be loaded from a plasticating extruder. Materials are heated above their melting points, formed and cooled. The more evenly the feed material is distributed over the mould surface, the less flow orientation occurs during the compression stage.

Thermoplastic matrices are commonplace in mass production industries. Significant examples are automotive applications where the leading technologies are long fibre reinforced thermoplastics (LFT) and glass fibre mat reinforced thermoplastics (GMT).



Figure 3.9 Compression moulding machine

3.14.3 Fibre Surface Treatment

The physical properties of fibre reinforced composite materials depend on the ability of the polymer matrix both to transmit stresses to the fibre reinforcement and to protect the fibres from damage. To promote these characteristics, fibres are treated during manufacture with mixtures of sizing and coupling agents. The optimization of a given coating is complicated by the fact that individual components often perform more than one function. An experimental technique and a theoretical analysis have been developed which

allow evaluation of both the stress transfer capability and the fibre protection performance of a given surface treatment. The analysis should be of particular value in the optimization of commercial fibre treatments.

3.14.3.1 Alkali treatment

The sisal/cotton fibre was soaked in 5% concentration of NaOH solution in the water bath for 1 hour at room temperature. The ratio of the fibres and the solution was 1:20 (w/v). Subsequently, the fibres were washed several times with distilled water and pH of the solution was maintained at 7 to bring about complete removal of alkali. After that, the fibres were then dried at room temperature for 48 h, followed by oven drying at 70°C for 3 h to remove the moisture content.

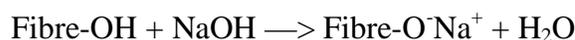
3.14.3.2 Benzoylation

As discussed earlier, benzoylation is an important transformation in organic synthesis. Benzoyl chloride is often used in fibre treatment. The pre-treated fibres were suspended in 10% NaOH solution and agitated with benzoyl chloride. The mixture was kept for 15 min, filtered, washed thoroughly with water and dried between filter papers. The isolated fibres were then soaked in ethanol for 1 h to remove the benzoyl chloride and finally was washed with water. After that the fibres were then dried at room temperature for 48 h, followed by oven drying at 60°C for 3 h to remove the moisture content.

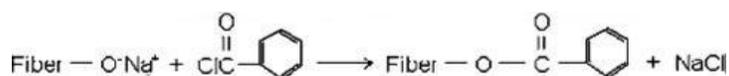
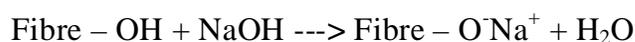
3.14.3.3 Effect of Fibre Surface Treatment

The alkaline treatment has two effects on the fibre: (1) it increases the surface roughness that results in a better mechanical interlocking; and (2) it increments the amount of cellulose exposed on the fibre surface, thus

increasing the number of possible reaction sites. The fibre pre-impregnation allows a better fibre wetting which in a normal fibre-polymer mixing procedure would not be possible because of the high polymer viscosity. Thus, the pre-impregnation enhances the mechanical interlocking between fibre and matrix. Addition of aqueous sodium hydroxide (NaOH) to natural fibre promotes the ionization of the hydroxyl group to the alkoxide.



Benzoylation of fibre improves fibre matrix adhesion, thereby considerably increasing the strength of composite, decreasing its water absorption and improving its thermal stability. The reaction between the cellulosic hydroxyl group of the fibre and benzoyl chloride is shown in the following equations :



3.14.4 Compounding

Compounding was carried out using a melt mixer (Haake Rheocord 9000, M/s Haake, Germany) having two ban bury rotors and a mixing chamber with volu-metric capacity of 69 mm³. The compounding was done in two steps. In the first stage, PP nano composites containing different weight% values of Cloisite 25A nanoclay (3, 5, and 7 wt.%) with maleic anhydride polypropylene copolymer (MAPP) at 5 wt.%, were prepared at a temperature of 180°C with a mixing speed of 40 rpm for a duration of 15 min. In the second stage, optimized PP (OPP) nano composites was melt blended with 15 wt. % of cotton and sisal fibres with a mixing speed of 45 rpm for a duration

of 15 min at 180°C. The melt mixers were brought down to room temperature and stored in airtight containers prior to use.

3.14.5 Specimen Preparation

Sheets were prepared by compression moulding at 170°C temperature employing a cycle time of 10–15 min to produce sheets of 3 mm thickness. Specimens were prepared by saw cutting for testing of flexural strength, impact strength and DMA. Specimens for tensile strength were prepared by contour cutting.