

# Chapter 11

## Isora Fibre: A Natural Reinforcement for the Development of High Performance Engineering Materials

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**Abstract** In this chapter, the technical potential of a natural fibre namely “ISORA” has been examined as an effective reinforcing material to design and manufacture high performance eco friendly composites in various polymers like natural rubber, polyester, epoxy resin, etc.; “Isora” a bast fibre separated from the bark of *Helicteres isora* plant is an important raw material can be used for the preparation of cost-effective and eco friendly composites. Morphology and physical properties of these fibres have been studied. Density and microscopic methods are used to determine the cross-sectional area and diameter of fibre bundles. Surface modification by alkali treatment and silane treatment were tried. Tensile properties of the treated and untreated fibres were determined by density method. The thermal characteristics, crystallinity index, reactivity, and surface morphology of the untreated and treated fibres have been studied by TGA, DSC, DTA, WAXRD, FTIR, and SEM. Average tensile strength of the fibre decreased and density increased to some extent on treatment with alkali and silane. Chemical constituents of the fibre were determined according to ASTM standards. SEM studies showed that as a result of chemical treatment fibre surface becomes rough promoting the fibre matrix adhesion which in turn improves the mechanical performance of the composites. Thermal analysis showed that chemical modification improves the thermal stability of the fibre. The strength of the fibre was theoretically calculated. For the successful design of a composite material using isora fibre and various polymers like natural rubber and thermosets (polyester and epoxy resin) several parameters like fibre aspect ratio, fibre orientation, fibre loading, chemical modification of fibre surface, fibre matrix adhesion that influences the performance of a short fibre composite were studied and optimised.

**Keywords** Adhesion · Bonding agent · Interface · Isora fibre · Natural fibre · Polymers · Reinforcement

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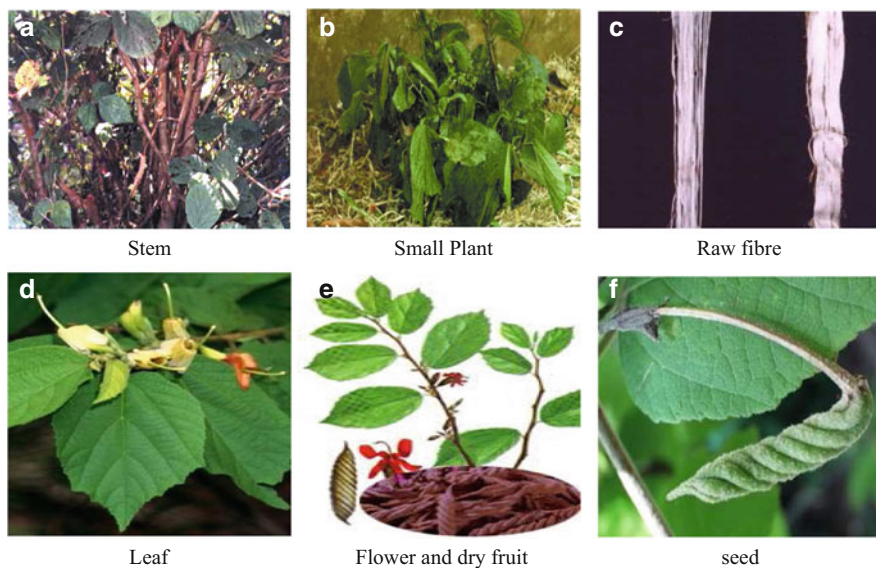
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## 11.1 Introduction

Nature is endowed with abundant quantity of natural fibres and now industrialists have focused on the development of natural fibre composites primarily to explore value added application avenues. Natural fibres do have a number of techno-economical and ecological advantages over synthetic fibres. The combination of interesting mechanical and physical properties together with environment friendly character has triggered a number of industrial sectors to consider these fibres as potential candidates to replace synthetic fibres in environmentally safe products. Composites fabricated with these natural fibres have the potential to be an attractive alternative to synthetic fibre composites. Interest in using natural fibres as reinforcement in polymer matrices and also in certain application as partial replacement of glass fibres has grown up significantly in recent years for making low-cost building materials and automobile components. Composites, the wonder material with light weight, high strength to weight ratio, and stiffness properties have come a long way in replacing the conventional materials like metals and woods. In this context, we have made efforts in search for some natural materials that can be effectively used as reinforcement for composites. There have been several attempts to utilise the abundant and renewable resources of plant fibres in composite materials with a view to replace the use of expensive synthetic fibres. Their application is diversified into engineering end uses such as building materials and structural part of motor vehicles where lightweight is required. The attractive features of natural fibres are their low cost, lightweight, high specific modulus, renewability and biodegradability. Low cost and less tool wear during processing are among the known advantages of plant fibres and ease of recycling makes them eco friendly. There are at least 1,000 types of plant that bear useful fibres. However

factors like lack of wettability and interfacial bonding between natural fibres and the well-known commercial polymers are the major problems that usually encountered with many of the applications. Experimental studies have shown that control of the fibre matrix interfacial bond strength is a critical factor in obtaining the best mechanical properties for the composites [1]. Hence studies have been focused on the treatment of fibres to improve bonding with the polymer matrix [2–4]. A wide variety of natural fibres like coir, sisal, oil palm, flax, banana, jute, pineapple, bamboo, etc. have been studied by several researchers [5–11]. However the use of “isora” has not yet been investigated by other researchers. Isora is a bast fibre separated from the bark of *Helicteres isora* plant by retting process. Two varieties of the plant are distinguished: *tomentosa* and *glabrescens* in which in the former the under side of the leaves is glabrous and in the latter both sides of the leaves are glabrous. The plant occurs as undergrowth especially as a secondary growth in forests. It coppices well, shooting up rapidly when cut or burnt. Seed sown during the rainy season easily propagates it. Roots, leaves and fruits of the plant are used for medicinal applications (Fig. 11.1). The stem bark is exploited for the fibre. Retting the stem in water and removing the fibre, which is pale yellow to light brown in colour, extract the fibre. The best type of fibre is obtained when the plants are 1–1.5 years old; plants older than 2 years yield coarse and brittle fibre. Stalks can be harvested annually for fibre extraction from regenerated shoots [12]. Fibre of good quality and colour is obtained when retting is effected in running water. Properties of natural fibres depend mainly on the nature and age of the plant, and the extraction method used. Isora fibre resembles jute in appearance, with comparable strength and better durability [13]. The fibre is used for making cordages,



**Fig. 11.1** *Helicteres isora* plant and its various parts

bags, fancy items, canvas, etc. Ropes and cordages made of the fibre are better and smoother than coir products. Recently we have reported about the possibilities of using isora fibre as a potential reinforcement in natural rubber and polyesters [14, 15]. The mechanical properties of these matrices are greatly improved by incorporation of this fibre. The resultant composite product will be a cost-effective and value added material for various components of machines like conveyor belts, v belts, and automobile parts. Use of biodegradable matrix and natural fibres as reinforcement has open new potential applications to these composites as they are eco friendly materials. To date no systematic work has been taken undertaken to study the properties of isora fibre from other researchers. This chapter reports on the physical, chemical, mechanical, thermal, and morphological characteristics of isora fibre and its utilisation as reinforcement in the development of polymer composites.

Short fibre reinforced rubber composites is a rapidly growing class of materials because of their improved physical and mechanical properties, easy process ability, and economic advantages. These materials bridge the gap between elastomers and fibres by combining the strength and stiffness of short fibres with the elasticity of rubber [1, 16]. Complex shaped engineering components may be developed using short fibre reinforced elastomers. The most important parameters that affect the short fibre reinforcement are fibre aspect ratio, fibre dispersion and orientation, fibre loading, fibre length, and adhesion between the fibre and the matrix [2, 17]. Use of biodegradable matrix and natural fibres as reinforcement has open new potential applications to these composites as they are eco friendly materials. Nowadays natural fibre is getting more attention from researchers. Natural cellulosic fibres when used as reinforcement imparts mechanical properties comparable to those of synthetic fibres like glass. Composites fabricated using these natural fibres have the potential to be an attractive alternative to synthetic fibre composites and are currently being explored in sectors like automobiles and buildings. In addition, these fibres offer an excellent opportunity to utilise an abundant source of such materials. These fibres, however, exhibit large variation in quality and are sensitive to moisture. The main problems that are usually encountered while using natural fibres as reinforcement are that the incorporation of a hydrophilic fibre into a hydrophobic polymer leads to a heterogeneous system with the result that they exhibit properties inferior to those of the unfilled system due to the poor adhesion at the fibre matrix interface. The surface characteristics of the reinforcing fibres are therefore important in transferring stresses from the matrix to the fibre. In the past, many attempts have been made to modify the surface properties of natural fibres in order to enhance adhesion with the polymer. Various methods such as corona treatment, plasma treatment, mercerisation, heat treatment, graft co polymerisation, and silane treatment have been reported to enhance the compatibility in natural fibre composites [18–24]. Unsaturated polyester is one of the thermoset matrices most frequently employed in the production of fibre reinforced composites, as it is available in liquid form, easily processed and cured, low in cost, easily available and has good mechanical properties when reinforced. Fibres are known to confer strength and rigidity to the weak and brittle matrix [25, 26].

## 11.2 Isora Fibre: Characterisation

### 11.2.1 Materials and Experimental Techniques

Isora fibre was separated from the bark of *H. isora* plant by retting process. Fibre presented in the inner part of the bark was peeled off, washed, and dried. The photographs of the stem, small plant, and the raw fibre are given in Fig. 11.1. Fibre surface was modified using alkali and silane treatments. For alkali treatment, fibres were dipped in 5% NaOH for about 4 h, then washed with water containing little acetic acid, washed well with water, and dried in an air oven. For silane treatment (Silane A 172), the alkali treated fibres were dipped in 1% silane solution in alcohol water mixture (60:40) for 2 h. The pH of the solution was maintained between 3.5 and 4. The fibres were washed with water and dried.

### 11.2.2 Chemical Composition and Mechanical Property Studies

Chemical composition of the fibre was estimated according to the following ASTM procedures: lignin-ASTM D1106, holocellulose-ASTM D1104, ash content-ASTM D1102, alcohol benzene solubility-ASTM D1107, ether solubility-ASTM D1108, 1% caustic soda solubility-ASTM D1109, and water solubility-ASTM D1110. Metal ions present were estimated using ICP AES analyser model IRIS INTREPID II XSP. For mechanical property studies, the cleaned isora fibres were cut into 70 mm, weighed, and finally mounted on a manila paper card mounts with a central cut out using a strong adhesive and the tensile test was then conducted according to ASTM 2256. Tensile strength was then calculated using cross-sectional area obtained from the weight and density of the fibre as shown in the equation.  $\sigma_T = F_{\max}/A$ ,  $\sigma_T = (\rho \cdot F_{\max} \cdot l_f)/m_f$ , where  $F$  is the maximum force,  $\rho$  the density of the fibre, and  $l_f$  the length of the fibre. The bulk density of the fibre was determined using a liquid of lower density (benzene) than the fibre.

From Table 11.1 it is clear that isora is a cellulose rich fibre with comparatively low lignin content which contributes to better performance of the fibre as reinforcement in polymers. The fine structure of cellulose materials is composed of crystalline and amorphous regions. The amorphous regions easily absorb chemicals such as dyes and resins, whereas the compactness of the crystalline regions make it difficult for chemical penetration. The physical characteristics and chemical constituents of the fibre are given in Table 11.1. The bulk density includes all the solid material and pores within the fibre which is always less than the absolute density. The absolute density of most plant fibres is between 1.4 and 1.5 g/cm<sup>3</sup>. The fibre density is assumed to be closely related to the mechanical properties, moisture absorption, homogeneity, and degree of order of the fibre. After chemical treatment there is a slight positive change in the bulk density which normally signifies cell wall densification. The nature and texture of the fibres obtained from different

**Table 11.1** (a) Chemical constituents of isora fibre, (b) Physical and mechanical properties of the fibre

(a)			
Chemical constituents (%)		Solubility in various solvents (%)	
Cellulose	74.8	Alcohol–benzene	6.3
Lignin	14.0	Ether	5.7
Ash	0.92	1% Caustic soda	14
Fat	1.09	Cold water	10
Moisture content	6–7	Hot water	12
Ca (ppm)	713.45	Acetone	5.6
Mg (ppm)	37.93		
Fe (ppm)	16.10		
K (ppm)	10.54		
Na (ppm)	10.42		
(b)			
Properties	U	A	Si
Tensile strength (MPa)	561	500	474
Young's modulus (GPa)	19	22	20
Elongation break (%)	5	4	4
Diameter ( $\mu\text{m}$ )	10	–	–
Density ( $\text{gm cm}^{-3}$ )	1.39	1.42	1.51
Crystallinity index (%)	70.9	80.6	77.8
Micro fibrillar angle ( $^\circ$ )	20–26	–	–
Intrinsic strength (g/denier)	0.987	–	–
Length to diameter ratio	99	–	–

U Untreated, A Alkali treated, Si Silane treated

plants may not be the same. Hence this will affect the properties of the fibre; therefore, there is a large variation in the observed properties. An average value of the properties is reported. The tensile properties like tensile strength and elongation at break of the fibre change slightly after chemical treatment. Tensile strength of the treated fibre is less than that of the untreated fibre. Lignin, the binding material that binds the three-dimensional cellulose structures as well as the fibrils, get partially removed on chemical treatment and hence tensile strength decreases. Elongation at break remains more or less same even after chemical treatment. The modification of plant fibres may involve the removal of the surface impurities, the swelling of the crystalline region, and the removal of the hydrophilic hydroxyl group of the cellulose. The effective reinforcement of composites with plant fibres is dependent on the moisture content, fibre matrix adhesion, crystalline, and cellulose content.

### 11.2.2.1 Theoretical Prediction of Microfibrillar Angle and Strength of the Fibre

Strength properties of the fibre are dependent mainly on the fibrillar structure, microfibrillar angle, and the cellulose content. There is a correlation between percentage elongation  $\varepsilon$  and microfibrillar angle  $\theta$  as follows:

$$\epsilon = -2.78 + 7.28 \times 10^{-2}\theta + 7.7 \times 10^{-3}\theta^2, \tag{11.1}$$

$$\sigma = -334.005 - 2.830\theta + 12.22W, \tag{11.2}$$

where  $W$  is the cellulose content of the fibre and  $\sigma$  the fibre strength [27].

The theoretical strength of the fibre was found to be 520 MPa which is in close agreement to the experimental value (568 MPa).

### 11.2.3 Surface Morphology Studies

The surface morphology of the fibre was studied using scanning electron microscopic studies (SEM). The SEM photographs of the fibre surface and cross section of the untreated and treated fibres were taken using a JEOL JSM 35 C model SEM. To avoid electron charging effects, the samples were gold coated in a Polaron SEM coating unit SC515. Figure 11.2 is the SEM photographs revealing the morphology of untreated isora fibres. The scanning electron micrograph of a single fibril and the cross section of isora fibre are given in Fig. 11.2a, b. The diameter of the fibre was found to be 10  $\mu\text{m}$ . The cross section is polygonal with a circular or oval lumen. The fibres are arranged in a reticulate pattern in a series of zones alternating with zones of soft tissue in the phloem region. The fibre consists of cells embedded in a matrix; the cells are the crystalline cellulose arranged in a matrix consisting of noncrystalline cellulose–lignin complex. The cell wall of the fibre elements is thick and lignified. The central core is referred to as “lacuna” can also be seen in Fig. 11.2b. For the alkali treated fibre (Fig. 11.2d), it was observed that the size of the central lacuna

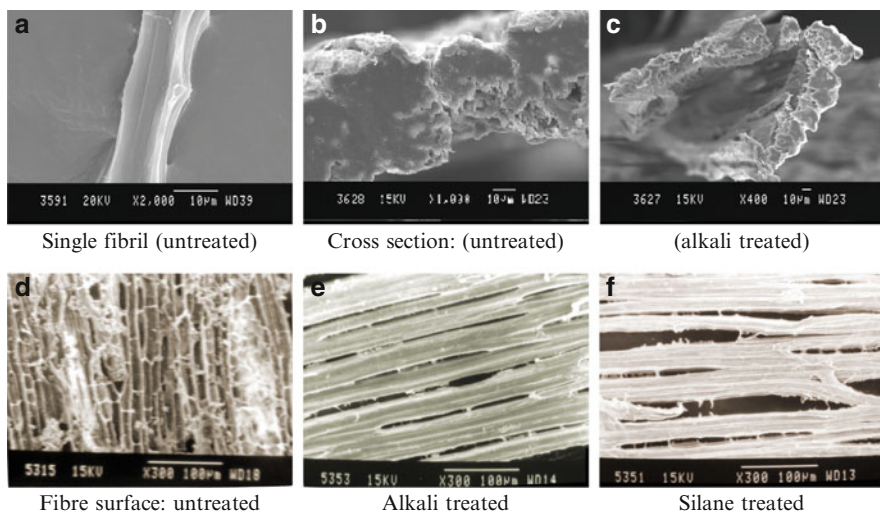
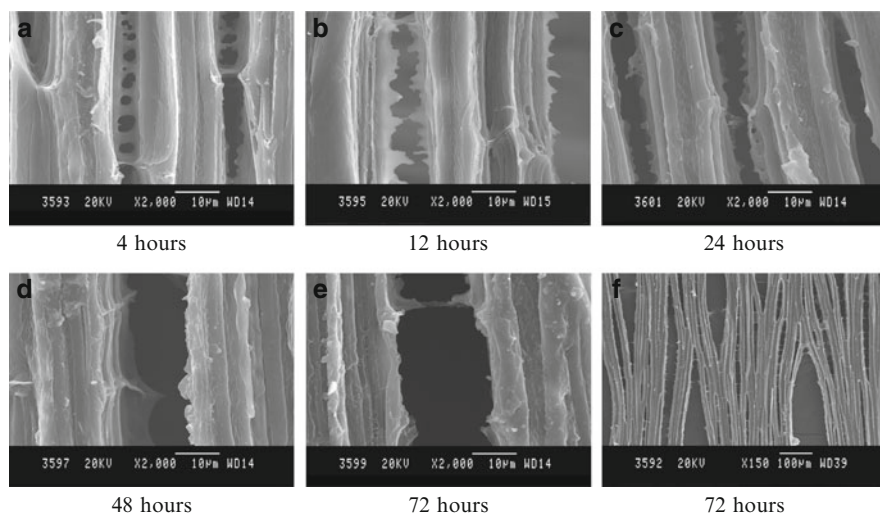


Fig. 11.2 SEM photographs of untreated and chemically treated fibres

is less than that of the untreated fibre indicating cell wall thickening and shrinkage of fibres during alkali treatment. From these pictures, it was found that isora fibres consisted of aligned fibrils with materials cementing the fibres together. Based on the results of FTIR and the structure of the natural fibre, the cementing material would be expected to be hemicellulose and lignin. It is well known that hemicellulose is a branched amorphous polymer with a low degree of polymerisation, which always associated with cellulose by hydrogen bonding. Therefore isora fibres should thought of as a composite material with fibrous reinforcement and a mixture of hemicellulose and lignin as matrix. SEM photographs of the alkali treated fibre were given in Fig. 11.3a–f. When the soaking time of the fibre in alkali was increased, it can be seen that the materials in the interfibrillar region were obviously etched away and the fibrous region becomes more pronounced as interfibrillar region is removed. SEM picture of the untreated isora fibre (Fig. 11.2d) shows that the fibre surface is very smooth. A series of globular particles can be seen to be embedded in the fibre surface at regular intervals. They are identified as tyloses which covers the pits on the cell walls. When the fibres are soaked for 4–12 h, most of the tyloses are intact, but at a few isolated places, it was removed creating holes as shown in Fig. 11.3b. When the soaking time was increased to 24–48 h, a much greater proportion of the tyloses appeared to be removed. At still higher soaking time, the SEM pictures show complete exposure of the fibrils indicating the leaching of the intercellular binding material and the cuticle layer (Fig. 11.3c–f). Fibre modification by alkali and silane treatment renders roughness to the fibre surface as evident from the SEM photographs (Fig. 11.2e, f). This will enhance the interlocking with the matrix, thereby the property of the composite increases [14, 15] by the incorporation of treated fibres.

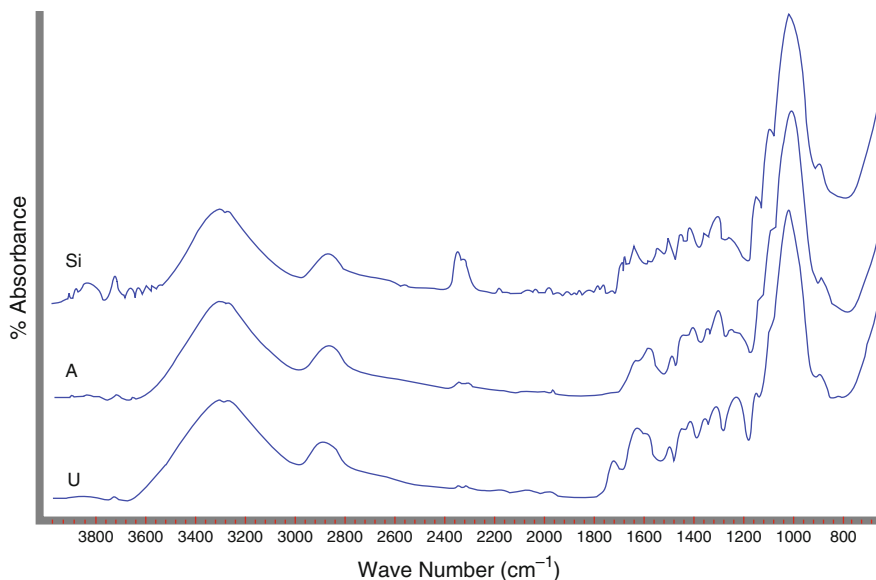


**Fig. 11.3** SEM photographs of alkali treated fibres (at different intervals of soaking in NaOH) at 2,000× magnification



### 11.2.4 Chemical Reactivity of the Fibre

The chemical reactivity of the fibre was clearly evident from the IR spectra. The IR spectra of the raw and chemically modified isora fibres were taken using a Shimadzu IR 470 Infra red spectrophotometer, by the KBr pellet technique. The IR spectra of untreated and treated fibres are given in Fig. 11.4. It can be understood from the spectra that some chemical reactions are occurred during different treatments. Major changes are observed in the IR absorbance of alkali and silane treated fibres. By alkali treatment, a peak at  $1,730\text{ cm}^{-1}$  due to the C=O stretching frequency of the carboxylic acid or ester present at the surface of the untreated fibre was disappeared due to the partial removal of lignin and hemicellulose. Also alkali treatment reduces the hydrogen bonding in the cellulosic hydroxyl groups, which is evident from the increased intensity of the -OH peak ( $3,350\text{ cm}^{-1}$ ) in the alkali treated fibre. In the case of silane treated fibres, an additional peak at  $3,400\text{ cm}^{-1}$  indicated intermolecular hydrogen bonding between the silanol -OH and that of cellulosic -OH of the fibres. The peak at  $1,525\text{ cm}^{-1}$  in the untreated fibre is shifted to  $1,600\text{ cm}^{-1}$  upon silane treatment. This may be due to the C=C stretching of the vinyl group. Additionally a peak at  $2,900\text{ cm}^{-1}$  belonging to the CH stretching vibration in the cellulose and hemicellulose decreased after alkali treatment indicating the partial removal of lignin and hemicellulose.



**Fig. 11.4** IR spectra of various chemically treated fibres (*U* untreated; *A* alkali treated; *Si* silane treated)

### 11.2.5 Thermal Analysis

TGA and DSC studies of the fibres were carried out using a TGA Q-50 (TA Instruments) and a Mettler Toledo model DSC 822 thermal analyser with a heating scheme of 30–550°C and at a heating rate of 10°C min<sup>-1</sup> in nitrogen atmosphere purged at 25 ml min<sup>-1</sup>. DTA and TGA studies were also carried in an oxygen atmosphere using a Perkin Elmer model diamond TGA/DTA thermal analyser.

Thermal degradation pattern of untreated, alkali treated, and silane treated fibres are shown in Figs. 11.5–11.7 (DTG, DSC, and DTA). Figure 11.5 shows the DTG curves for the fibre with different treatments. Around 100°C an initial weight loss was observed due to the removal of sorbed moisture from the fibres. The initial degradation temperature is higher for the alkali treated fibre. Major weight losses of the untreated and silane treated fibres take place at between 360 and 370°C. Alkali treatment raises this temperature to 380°C. The DTA curve (Fig. 11.6) in oxygen shows a major peak between 300 and 350°C that may be due to the thermal depolymerisation of hemicelluloses and the cleavage of the glycosidic linkages of cellulose. This is an exothermic process. At the first stage of the degradation, the DTA curve shows an endothermic peak in all cases around 100°C (Fig. 11.6). This peak may be due to the removal of moisture. Breakage of the decomposition products of the second stage (second peak) leads to the formation of charred residue. The third exothermic peak present in DTA curve is due to this oxidation and burning of the high molecular weight residues. In alkali treated fibre, the second peak is not prominent and broadening of the DTA peak was observed. In DTA (oxygen), the initial degradation temperature is higher for the untreated fibre. The DSC studies in nitrogen also show a similar trend (Fig. 11.7). A broad endotherm in the temperature range of 75–120°C was observed. The peak of maximum weight

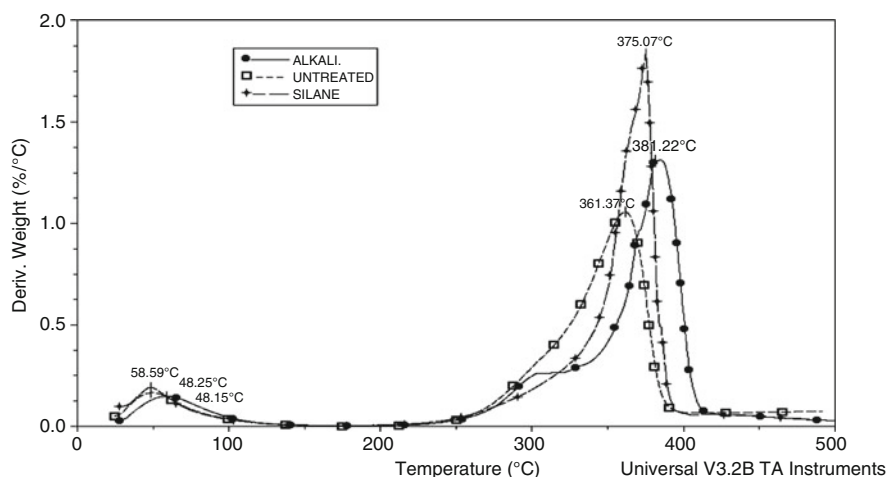
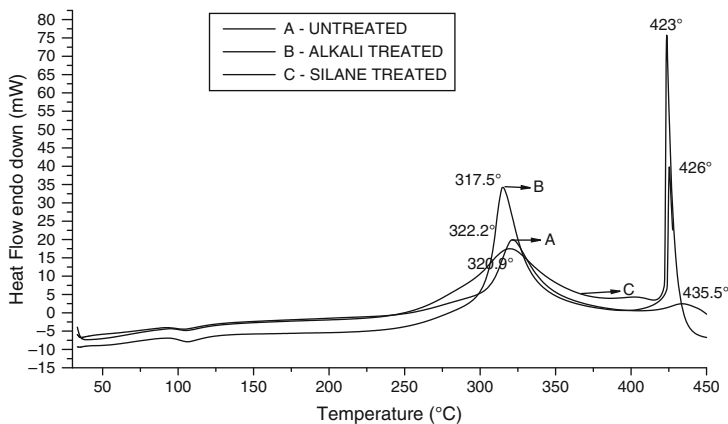
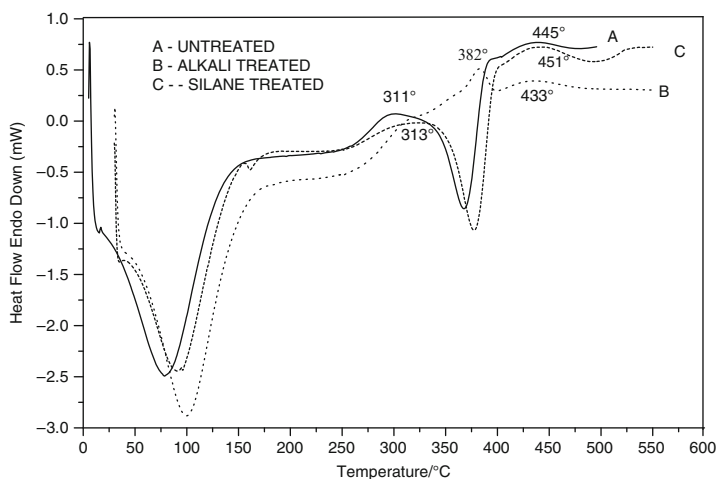


Fig. 11.5 DTG curves of untreated, alkali, and silane treated fibres in nitrogen atmosphere



**Fig. 11.6** DTA curves of the untreated, alkali, and silane treated fibres in oxygen atmosphere



**Fig. 11.7** DSC curves of untreated, alkali treated, and silane treated fibres in nitrogen atmosphere

loss was observed for untreated and treated fibres between 300 and 350°C. Fibre treatment slightly increased the thermal stability of the fibres in nitrogen atmosphere. This is evident from the DSC curves (Fig. 11.7) which is an indication of the increase in the crystalline cellulose, which is known to have good thermal resistance. Two and three exothermic peaks are observed for the untreated and treated fibres, respectively. In cellulose fibres, lignin degrades at a temperature around 200°C [28]. While the other polysaccharides such as cellulose degrade at higher temperatures. Therefore these exothermic peaks which were higher than 200°C indicate the decomposition temperature of the cellulose in the fibres. The region between 150 and 270°C shows no exothermic or endothermic reactions which suggest that the fibres are stable between these temperatures. From the first

exothermic peak, it can be deduced that the alkali treated fibre is more thermally stable compared to the untreated and silane treated fibre. Similar observations have also been reported in the case of other natural fibres [29]. It was reported that alkali and silane treatment increase the thermal stability of the fibres [6].

### 11.2.6 Wide Angle X-Ray Diffraction Studies

The crystallinity index of the fibres was studied using a wide angle X-ray diffractometer Bruker model D8 Advance, equipped with a scintillation counter and a linear amplifier was used. The diffraction intensities were recorded between 5 and 60° ( $2\theta$  angle range). The crystallinity index was determined using the equation:

$$I_c = \frac{[(I_{002} - I_{am}) \times 100]}{I_{002}},$$

where  $I_c$  is the crystallinity index,  $I_{002}$  is the counter reading at peak intensity at a  $2\theta$  angle close to 22° representing crystalline material, and  $I_{am}$  is the counter reading at peak intensity at a  $2\theta$  angle close to 18° representing amorphous material in the fibre.

From Table 11.1 and Fig. 11.6, it is clear that the treated fibres show an overall initial increase in the crystallinity index, with maximum for alkali treated fibres which is an indication of the improvement in the order of crystallites as the cell wall thickens upon chemical treatment. The use of wide angle X-ray diffraction studies (WAXRD) counts offers a simple and quick method of determining the crystallinity index, and the minimum between 101 and 002 peaks (Fig. 11.8) is an indication of the reflection intensity of the amorphous material. Alkalisiation and silane treatment increases the crystallite packing order. Crystallinity index is a measure of the order of the crystallites rather than the crystallinity of the crystallites.

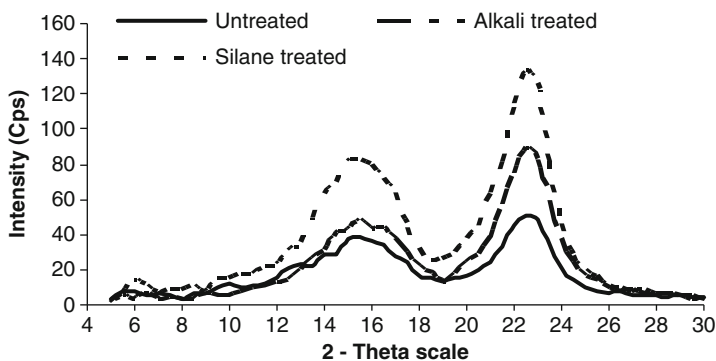


Fig. 11.8 WAXRD spectra of untreated, alkali treated, and silane treated fibres

## 11.3 Isora: Polymer Composites

### 11.3.1 Isora: Natural Rubber Composites

#### 11.3.1.1 Preparation and Characterisation of Isora/Natural Rubber Composites

Fibre, separated from the bark of *H. isora* plant by retting process, was chopped almost accurately to different lengths of 6, 10, and 14 mm using a fibre chopper on which the length of fibre cuttings can be adjusted and washed with water to remove the undesirable impurities and dried. Treated fibre was prepared from raw fibre by immersing it in 5% aqueous NaOH for 4 h. Washing with water was done several times followed by drying in an air oven.

The composites were prepared by incorporating short isora fibres of different lengths 6, 10, and 14 mm (15 phr) and different loadings 10, 20, 30, 40 phr (10 mm) both for treated and untreated fibres into natural rubber matrix as per the formulation given in Tables 11.2 and 11.3. Mixes were prepared in a laboratory (150 × 300 mm) two roll mixing mill as per ASTM standards D 3184-80 at a nip gap of 1.3 mm. The samples were milled for sufficient time to disperse the fibres in the matrix. The final sheeting was done by passing the compound through a tight nip gap of 0.8 mm. The bonding agent (resorcinol, hexa, and silica) was incorporated

**Table 11.2** Mixes with varying fibre length (phr<sup>a</sup>)

Ingredients	Gum	L <sub>1</sub>	L <sub>2</sub>	L <sub>3</sub>
Natural rubber	100	100	100	100
Zinc oxide	5.0	5.0	5.0	5.0
Stearic acid	2.0	2.0	2.0	2.0
TDQ	1.0	1.0	1.0	1.0
CBS	0.6	0.6	0.6	0.6
TMTD	0.1	0.1	0.1	0.1
S	2.5	2.5	2.5	2.5
Untreated isora fibre	0	15	15	15
Fibre length (mm)	0	6	10	14

<sup>a</sup>parts per hundred rubber

**Table 11.3** Mixes (phr)<sup>a</sup> with varying fibre loading and bonding agent

Mixes	Gum	X <sub>10</sub>	X <sub>20</sub>	X <sub>30</sub>	X <sub>40</sub>	Y <sub>10</sub>	Y <sub>20</sub>	Y <sub>30</sub>	Y <sub>40</sub>
Fibre (untreated)	0	10	20	30	40	–	–	–	–
Fibre (treated)	0	–	–	–	–	10	20	30	40
Mixes	–	X <sub>10b</sub>	X <sub>20b</sub>	X <sub>30b</sub>	X <sub>40b</sub>	Y <sub>10b</sub>	Y <sub>20b</sub>	Y <sub>30b</sub>	Y <sub>40b</sub>
Resorcinol	0	2.5	5.0	7.5	10	2.5	5.0	7.5	10
Hexa	0	1.6	3.2	4.8	6.4	1.6	3.2	4.8	6.4
Silica	0	1	2	3	4	1	2	3	4
Fibre (untreated)	0	10	20	30	40	–	–	–	–
Fibre (treated)	0	–	–	–	–	10	20	30	40

<sup>a</sup>Basic recipe: NR-100, ZnO-5, Stearic acid-2, TDQ-1, CBS-0.6, TMTD-0.1, S-2.5

along with the other ingredients. The fibre was incorporated at the end of mixing process taking care to maintain the direction of compound flow, so that majority of fibres followed the direction of flow. In order to study the extent of fibre breakage, which occurs during milling operations, the fibres were extracted from the green compound by dissolving it in toluene, and their length and diameter were measured by using a travelling microscope. The cure characteristics were studied by an oscillating disc rheometer (Goettfert Elastograph). The samples were vulcanised at 150°C in a hydraulic press up to their respective optimum cure time  $t_{90}$  as measured on a Goettfert Elastograph.

### 11.3.1.2 Evaluation of Fibre Breakage

Mixes  $L_1$ ,  $L_2$ , and  $L_3$  are the NR mixes containing fibres of lengths 6, 10, and 14 mm, respectively (Table 11.2). The control of fibre length and aspect ratio of fibres in rubber matrix is difficult because of fibre breakage during processing. The severity of fibre breakage depends mainly on the type of fibre, the initial aspect ratio and the magnitude of stress and strain experienced by the fibres during processing [20]. Shear force during mixing orients most of the fibres along the mill grain direction. It may cause breakage of fibres also. The average diameter (10  $\mu\text{m}$ ) of the fibre remained unchanged after mixing. The results of the fibre breakage analysis are given in Table 11.4. From the data it is seen that the compound  $L_2$  contained a total of 52.3% of the fibre in the range of 2–6 mm length and 63.3% of fibre in the range of 4–8 mm length after mixing and the aspect ratio remains higher than 200 which is generally required for effective stress transfer in short fibre elastomer composites [30]. From Table 11.7 it is clear that the reinforcement is high for the compound  $L_2$  which contained fibres having an original length of 10 mm before mixing as evident from the high tensile strength, modulus, and tear strength of the compound  $L_2$  compared to that of  $L_1$ . The tensile strength and modulus of the compound  $L_3$  are comparable to those of  $L_2$ , even though it contained almost the same level of fibres (48.9%) having a length in the range 2–6 mm as that of the compound  $L_1$  (48.3%). This is likely to be due to the presence of 39.7% of fibres having a final length in the range of 6–10 mm in  $L_3$ , which is almost comparable to that present in  $L_2$  (38.7%). These observations indicate that an original fibre length

**Table 11.4** Distribution of fibre length after mixing

Length of fibre after mixing (mm)	Percentage available in the mix after mixing		
	$L_1$	$L_2$	$L_3$
0–2	51.7	9.0	11.4
2–4	27.9	22.0	23.3
4–6	20.4	30.3	25.6
6–8	–	33.0	32.7
8–10	–	5.7	7.0

Mixes  $L_1$ ,  $L_2$ , and  $L_3$  contain fibres of length 6, 10, and 14 mm before mixing

of 10 mm is the critical fibre length which is essential for getting better reinforcement in short isora fibre natural rubber composites.

### 11.3.1.3 Cure Characteristics

Tables 11.5 and 11.6 show the variation of cure characteristics of composites with varying fibre length and loading, respectively. The maximum torque is a measure of cross link density and stiffness in the rubber. In general, for all the mixes, the torque initially decreases, then increases, and finally levels off. The initial decrease in torque to a minimum value is due to the softening of the rubber matrix while the increase in torque is due to the cross linking of rubber. The levelling off is an indication of the completion of curing. It is found that the addition of fibres into the mix generally increases the torque values. It also shows that the torque increases with increase in fibre length and reaches a maximum at 10 mm fibre length. This increase is due to the presence of longer fibres, which imparts more restriction to deformation. However, the maximum torque was slightly higher for 10 mm than 14 mm. This may be due to the fact that longer fibres undergo fibre entanglement and breakage during mixing. The maximum torque also increases with increase in fibre loading. This is due to the increase in the stiffness and hardness of the composite. These torque values are also increased by alkali treatment as the treated fibre may provide a better surface for reinforcement. It is observed that the curing time is not very much affected by the modification of fibre surface. The optimum cure time is found to increase with addition of bonding agent. The longer curing time is due to the better bonding between the fibre and the matrix when bonding agent is used. Maximum and minimum torque values also increase in the presence of bonding agent. This is due to the strong bonding at the fibre/rubber interface and consequently the composite becomes stronger, harder, and stiffer.

**Table 11.5** Vulcanisation parameters for mixes with varying fibre length

Mixes	Gum	L <sub>1</sub>	L <sub>2</sub>	L <sub>3</sub>
Min. torq. (Nm)	0.01	0.03	0.02	0.02
Max. torq. (Nm)	0.21	0.24	0.30	0.28
Scorch time	2.36	1.8	1.76	1.76
Cure time $t_{90}$	4.32	4.62	5.42	5.46

**Table 11.6** Vulcanisation parameters for mixes with varying fibre loading and bonding agent

Mixes	Gum	X <sub>10</sub>	X <sub>20</sub>	X <sub>30</sub>	X <sub>40</sub>	Y <sub>10</sub>	Y <sub>20</sub>	Y <sub>30</sub>	Y <sub>40</sub>
Curetime $t_{90}$ (min)	4.32	5.4	5.5	5.7	5.85	4.61	4.91	5.22	5.64
Max. torq. (Nm)	0.22	0.29	0.32	0.35	0.40	0.32	0.34	0.40	0.51
Mixes		X <sub>10b</sub>	X <sub>20b</sub>	X <sub>30b</sub>	X <sub>40b</sub>	Y <sub>10b</sub>	Y <sub>20b</sub>	Y <sub>30b</sub>	Y <sub>40b</sub>
Curetime $t_{90}$ (min)	4.32	7.24	7.76	8.12	8.36	6.9	7.0	7.32	7.68
Max. torq (Nm)	0.22	0.33	0.35	0.52	0.64	0.35	0.47	0.58	0.69

### 11.3.1.4 Extent of Fibre Orientation from Green Strength Measurements

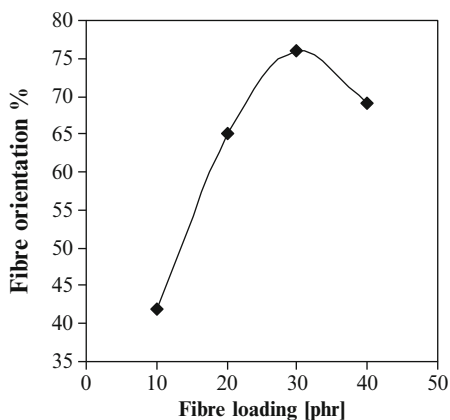
The green strength of short fibre-reinforced composites depends on the degree of fibre orientation. The extent of fibre orientation can be calculated by using the following equation:

$$\text{Orientation \%} = \frac{S_L/S_{G,L}}{S_L/S_{G,L} + S_T/S_{G,T}},$$

where  $S$  is the green strength and subscripts G, L, and T denote gum, longitudinal, and transverse, respectively. The effect of fibre loading on the percentage orientation is shown in Fig. 11.9. At low fibre loading, the percentage orientation is the lowest as the fibres can randomly move around leading to increased chaoticity and decreased levels of orientation. As fibre loading increases, percentage orientation increases with the maximum value for composite containing 30 phr fibre. At 40 phr fibre loading, the percentage orientation decreases indicating that the fibres cannot orient themselves due to the entanglement caused by the increased content of fibres.

### 11.3.1.5 Mechanical Properties

Dumb bell and crescent-shaped tensile and tear specimens with longitudinal and transverse fibre orientations were punched from the vulcanised sheets. Stress–strain measurements were carried out at a crosshead speed  $500 \text{ mm min}^{-1}$  on a Schimadzu Model AG1 universal testing machine. Tensile and tear strengths were measured according to ASTM D 412-68 and D 624-54, respectively. Compression set of the specimens was measured in accordance with ASTM D 395-86 (method B). Abrasion resistance of the samples was tested using a DIN 53516. The hardness was measured using the Shore A type Durometer according to ASTM 2240-81. The SEM photographs of the fibres and the fractured surfaces were taken using a JEOL



**Fig. 11.9** Effect of fibre loading on percentage of fibre orientation of the composites



JSM 35 C model SEM. The fibre and the fracture surfaces were sputter coated with gold within 24 h of testing by using a fine coat JFC-1100.

### Effect of Fibre Length

The effect of fibre length and orientation on the properties of the composites is given in Table 11.7. The properties are found to increase with increase in fibre length. The increase in the strength with increase in fibre length is attributed to the fact that the extent of load transmittance is a function of fibre length and magnitude of fibre matrix interfacial bond [31]. In fibre reinforced composites, there exists a critical fibre length at which the load transmittance from the matrix to the fibre is maximum. Critical fibre length is that length which is required by the fibre to develop its fully stressed condition in the matrix. Longer fibres impart more restriction to deformation while shorter fibres create friction and heat generation due to the increased number of fibre ends. Tensile and tear strengths show a maximum value for the composite with fibres having an original length of 10 mm before mixing. The interaction between the fibre and the matrix reaches its maximum at this fibre length and the effect of fibre length decreases with longer fibres because of fibre entanglement and breakage. At higher fibre length, the dispersion of fibres in rubber matrix is difficult. Hence a further increase in fibre length beyond 10 mm decreases the mechanical properties like tensile strength and tear strength. These observations indicate that an original fibre length of 10 mm is the critical fibre length which is essential for getting better reinforcement in short isora fibre natural rubber composites.

### Effect of Fibre Orientation

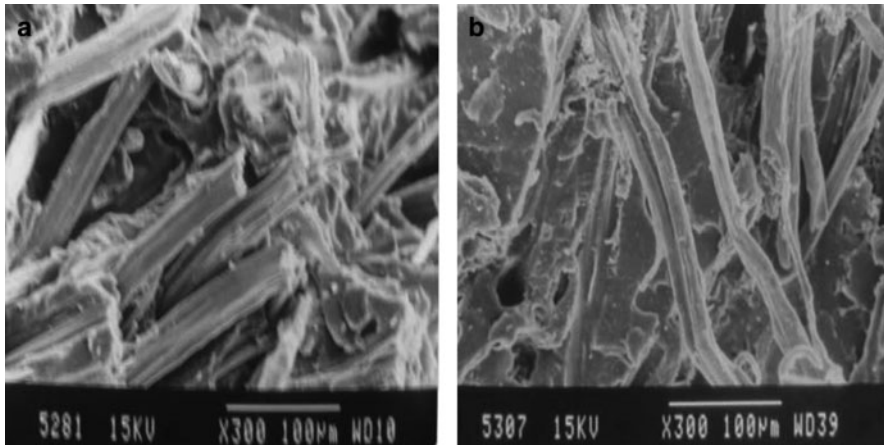
Fibre orientation affects composite properties. During milling of rubber composites, majority of the fibres tend to orient along the flow direction, causing the mechanical properties to vary in different directions. In case of short fibre reinforced composites, longitudinal and transverse orientations are possible. In longitudinal orientation, the fibres are aligned along the mill grain direction and in transverse one the fibres are aligned across the grain direction. Properties like tensile modulus, tensile strength,

**Table 11.7** Mechanical properties of vulcanisates with varying fibre length

Properties	O	Gum	L <sub>1</sub>	L <sub>2</sub>	L <sub>3</sub>
Tensile modulus (300% E) MPa	L	2.3 (0.28)	2.71 (1.18)	2.95 (0.55)	2.80 (1.05)
	T	2.3 (0.22)	2.30 (0.95)	2.40 (0.73)	2.30 (0.91)
Tensile strength (MPa)	L	25.9 (0.48)	14.0 (1.02)	16.2 (0.87)	15.9 (0.90)
	T	25.0 (0.39)	12.1 (0.98)	13.8 (0.97)	14.0 (0.95)
Elongation break (%)	L	1,050 (0.29)	700 (1.15)	625 (0.45)	695 (0.77)
	T	1,045 (0.31)	725 (1.06)	650 (0.88)	715 (1.11)
Tear strength (kN m <sup>-1</sup> )	L	35.1 (0.42)	37.5 (0.92)	42.0 (0.65)	42.5 (0.61)

L<sub>1</sub>, L<sub>2</sub>, and L<sub>3</sub> are mixes with fibre length 6, 10, and 14 mm, respectively. Figures given in the parentheses are standard deviations

O Orientation, L longitudinal, T transverse



**Fig. 11.10** SEM photographs showing (a) longitudinal and (b) transverse orientation of fibres in the composite  $X_{30}$

and tear strength of the composites with longitudinal orientation are always higher than the composites with transverse fibre orientation (Table 11.7). The extent of fibre orientation can also be understood qualitatively from the examination of the SEM photographs. Figure 11.10a, b are tensile fracture surfaces of the longitudinally and transversely oriented composite ( $X_{30}$ ). The broken fibre ends protruding from the fracture surface (Fig. 11.10a) indicate that the fibres are well aligned longitudinally in the direction of applied force. While in transverse orientation, the fibres are aligned across the direction of applied force (Fig. 11.10b). The tensile strength of the composites depends on the fibres, which obstruct the progress of the fracture front. In longitudinal orientation, the crack progresses in the direction perpendicular to the fibre alignment causing greater obstruction by the fibres and hence tensile strength increases. Breakage and pulling out of fibres take place when fibres are oriented longitudinally, whereas in transverse orientation crack progresses in the direction of fibre alignment experiencing a lower resistance by the fibres. The increase in tear strength in longitudinal orientation is due to the obstruction caused to the tear path by the short fibres.

#### Effect of Chemical Treatment and Fibre Loading

Good interfacial strength between the fibre and rubber is the essential factor to achieve good fibre reinforcement. The interfacial strength depends on the surface topology of the fibre. The cellulose fibres even though possess hydroxyl groups on its surface, the lignin and other waxy contents make it a less effective reinforcement. Hence to improve adhesion between isora fibre and rubber, it should be subjected to some chemical treatment to remove the lignin and other waxy impurities. So the mixes  $Y_{10}$ ,  $Y_{20}$ ,  $Y_{30}$ , and  $Y_{40}$  are prepared using alkali treated fibres. The surface

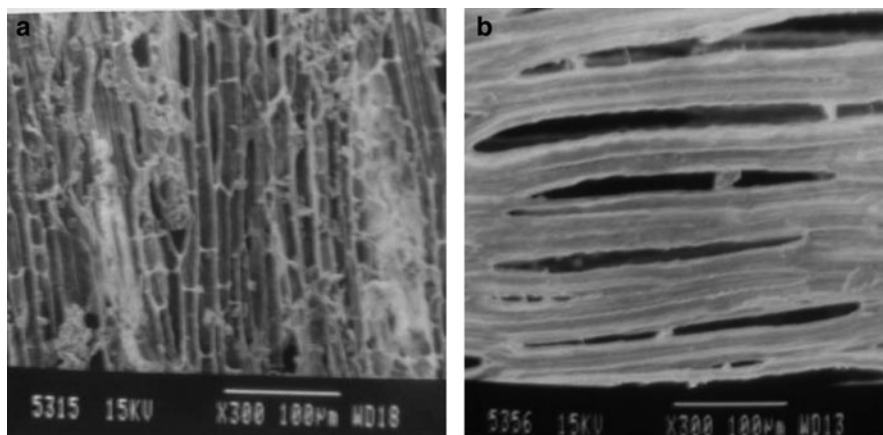


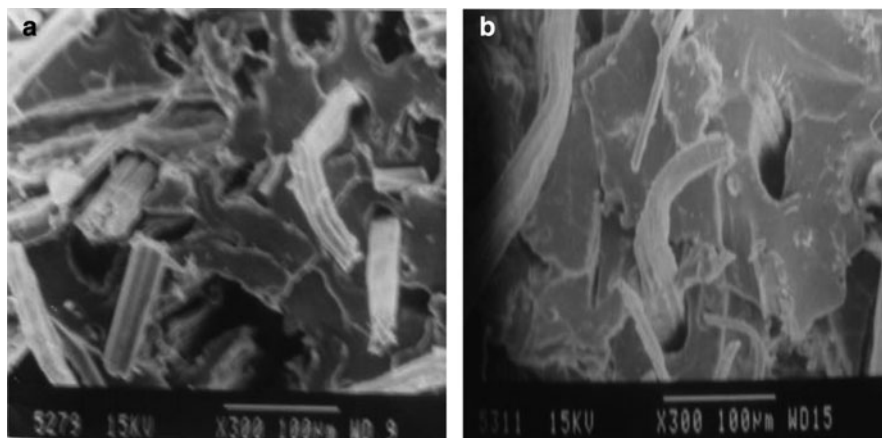
Fig. 11.11 SEM photographs of (a) raw fibre (b) alkali treated fibre

topology of isora fibre was studied by SEM, as shown in Fig. 11.11a, b. In Fig. 11.11a, the multicellular nature of the raw fibre is seen. The fibrillar nature and porosity of the fibre is revealed from the fibre topography. The porous surface morphology is useful to have better mechanical interlocking with the matrix for composite fabrication. The SEM of alkali treated fibre (Fig. 11.11b) gives strong evidence for the physical microcellular structural changes occurred to the fibre surface on mercerisation. Here pores become clearer and fibre becomes thinner, also fibres undergo fibrillation as shown in the figure. This may be due to the dissolution and leaching out of fatty acids and lignin components of the fibre. This renders roughness to the fibre, thereby enhancing the mechanical interlocking at the interface. The development of a rough surface topography offers better fibre rubber interface adhesion and increase in mechanical properties. Table 11.8 shows the mechanical properties of the composites containing treated and untreated fibres for varying fibre loadings. The tensile properties (tensile modulus, tensile strength, tear strength, etc.) of the composites filled with treated fibres are higher than those with untreated fibres at similar loadings. To obtain good fibre reinforcement in rubber composites, the adhesion between the rubber and the fibre is very important. From these results it is clear that the aqueous alkali treatment of isora fibre has improved the fibre adhesion to rubber matrix. It is reported that the surface of fibres can be modified by aqueous alkali treatment at elevated temperatures, and this was found to improve the adhesion properties significantly [32]. Fibre treatment can be used to prevent the debonding at the fibre interface since it can form covalent bonds between the rubber matrix and fibre. Strong adhesion between treated fibre and rubber leads to higher shear strength at fibre rubber interface. Stronger force must be used to overcome the shear strength, which resulted in higher tensile strength. The elongation at break for composites with treated fibres is lower than composites with untreated fibre at similar loading. This is due to the better strength and stiffness achieved from strong adhesion between fibre and rubber. Consequently,

**Table 11.8** Mechanical properties of vulcanisates with varying fibre loading

Properties	O	Gum	X <sub>10</sub>	X <sub>20</sub>	X <sub>30</sub>	X <sub>40</sub>	Y <sub>10</sub>	Y <sub>20</sub>	Y <sub>30</sub>	Y <sub>40</sub>
Modulus (300% elongation) (MPa)	L	2.3 (0.32)	2.8 (0.19)	2.9 (0.41)	3.2 (0.92)	3.0 (0.65)	3.1 (1.12)	3.3 (0.84)	3.8 (0.64)	3.5 (1.05)
	T	2.3 (0.30)	2.0 (0.89)	2.1 (0.16)	2.2 (1.25)	2.4 (2.18)	2.5 (0.14)	2.6 (0.78)	2.8 (0.55)	2.9 (1.12)
Tensile strength (MPa)	L	25.9 (0.96)	20.9 (0.88)	14.0 (1.01)	9.8 (1.28)	9.0 (0.77)	21.1 (0.32)	15.2 (0.28)	10.7 (1.31)	9.8 (0.68)
	T	25.0 (0.93)	13.2 (0.82)	10.1 (1.44)	6.8 (1.05)	6.2 (0.79)	15.2 (0.59)	11.5 (1.34)	8.2 (1.28)	7.8 (0.37)
Elongation at break (%)	L	1050 (0.88)	860 (0.96)	615 (0.16)	437 (0.83)	380 (0.75)	790 (2.04)	605 (1.16)	400 (0.81)	310 (0.95)
	T	1045 (0.86)	900 (1.08)	690 (0.28)	480 (0.84)	425 (1.17)	612 (0.93)	625 (0.48)	412 (2.03)	395 (0.19)
Tear strength (kN m <sup>-1</sup> )	L	35.1 (0.68)	36.5 (0.96)	42.8 (0.77)	46.8 (0.18)	40.9 (0.99)	38.1 (0.83)	44.6 (1.52)	47.9 (1.17)	43.1 (0.31)
	T	34.5 (0.74)	35.5 (1.11)	40.1 (1.41)	43.9 (0.32)	39.5 (0.53)	37.5 (1.00)	41.9 (0.86)	44.2 (0.56)	40.8 (0.57)
Hardness (shore A)	-	45	50	63	69	72	55	65	73	76

Figures given in parentheses are standard deviations



**Fig. 11.12** SEM of the fractured surface containing (a) untreated (b) alkali treated fibres of the composites  $X_{30}$  and  $Y_{30}$

the toughness of the composites is reduced resulting in lower elongation at break. Higher toughness is obtained from weak interfacial adhesion as shown by higher elongation at break for composites filled with untreated fibres. Figure 11.12a, b are the SEM of the tensile fracture surfaces of composites with untreated and treated fibres ( $X_{30}$  and  $Y_{30}$ ). In the case of untreated fibre composites, due to the weak interfacial adhesion between the fibre and rubber, fibre pull out may take place leaving holes on the surface when stress is applied (Fig. 11.12a). Figure 11.12b shows the presence of broken fibres on the fracture surface which is due to the strong adhesion between the fibre and rubber matrix for composites with treated fibres. Composites containing treated fibres show enhancement in modulus compared to the untreated ones for similar fibre loading.

Natural rubber inherently possesses high tensile strength due to strain-induced crystallisation. When fibres are incorporated into NR, the regular arrangement of rubber molecules is disrupted and hence the ability of crystallisation is lost. Hence the fibre reinforced natural rubber composites possess lower tensile strength than gum compounds. When fibre reinforced rubber composites are subjected to a load, the fibres act as carriers of load and stress is transferred from matrix along the fibres which results in a composite having good mechanical properties. The uniform distribution of stress is dependant on the population and orientation of fibres. At low levels of fibre loading, the orientation of fibres is poor and the fibres are not capable of transferring load to one another and stress get accumulated at certain points of composite, leading to low modulus. From Table 11.8, it is clear that modulus shows a continuous increase up to 30 phr fibre loading in the case of longitudinal orientation. At higher levels of fibre loading, the increased population of fibres leads to agglomeration, and stress transfer gets partially blocked. On transverse orientation, the modulus increases gradually but with a lower value than longitudinal orientation. Also with the increase of fibre loadings, tensile strength of the

composites shows an abrupt decrease up to a loading of 30 phr and there afterwards a gradual decrease both in longitudinal and in transverse orientations. At intermediate levels of loading (30 phr), the population of fibres is just sufficient for maximum orientation and fibres actively participate in stress transfer. As fibre loading increases tear strength gradually increases and reaches a maximum at 30 phr fibre loading. When the fibre loading increased further, tear strength again decreases as the increased strain in the matrix between closely packed fibres increases tearing and reduces the tear strength. Maya and Thomas [33] also have observed similar results. There is a reduction for elongation at break with increasing fibre loading. Increased fibre loading in the rubber matrix resulted in composites becoming stiffer and harder. This will reduce the composite's resilience and lead to lower elongation at break. The elongation at break for composites with treated fibres is lower than composites with untreated fibre at similar loading. This is due to the better strength and stiffness achieved from strong adhesion between fibre and rubber. Consequently, the toughness of the composites is reduced resulting in a still lower elongation at break. Higher toughness is obtained from weak interfacial adhesion as shown by higher elongation at break for composites filled with untreated fibres.

### Effect of Bonding Agent

Further increase in properties is seen by the incorporation of bonding agent to the system. It has already been established that a tricomponent system consisting of hexamethylene tetramine, resorcinol, and fine particle of silica can be used as a bonding agent for most rubber and fibre combinations [34]. Presence of bonding agent in the mixes improved the mechanical properties like modulus, tensile strength, and tear strength. Alkali treatment of fibres further enhances the effect of bonding agent as seen from the higher modulus, tensile strength, tear strength, etc. from Figs. 11.13–11.15. The treated fibre provides a better surface for strong adhesion between fibre and matrix, and the stress transfer becomes more efficient and consequently better enhancement in the properties. Elongation at break for composites with bonding agent has a lower value than composites without bonding agent. Again treated fibres show a lower elongation at break than untreated fibres (Fig. 11.16). The variation of compression set, abrasion loss, and hardness with fibre loading for the treated and untreated fibre composites with bonding agent are given in Figs. 11.17–11.19. Compression set increased steadily with increase in fibre loading. The rate of increase in set, however, decreased as loading was increased. But the set was lower for the composites having alkali treated fibres (Fig. 11.17). It has been reported that this behaviour is due to the buckling of the fibre, taking place invariably when the closely packed fibres are compressed in the direction of their alignment [35]. Due to the strong adhesion between the treated fibres and rubber, the extent of buckling is reduced in treated fibre composites resulting in a low value for the set. Abrasion loss decreased with increase in fibre concentration in the composite (Fig. 11.18). Here also the treated fibre composites

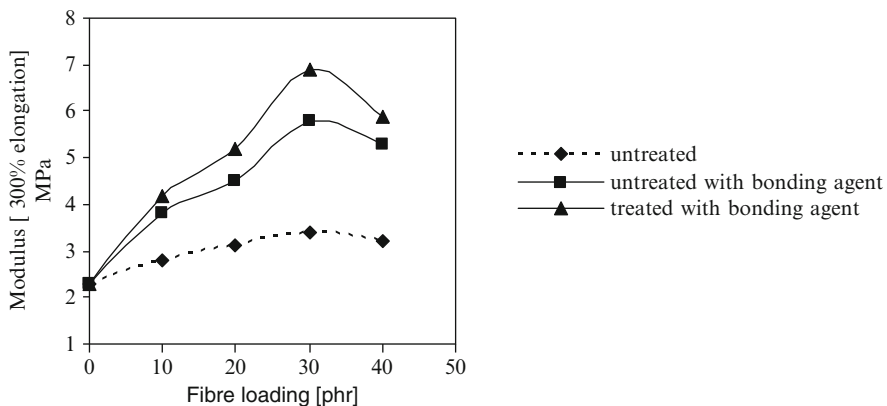


Fig. 11.13 Effect of fibre loading on modulus of the composites

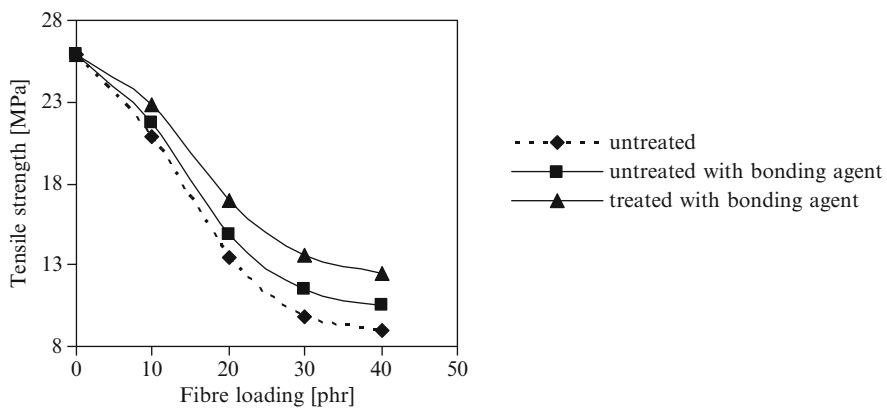


Fig. 11.14 Effect of fibre loading on tensile strength of the composites

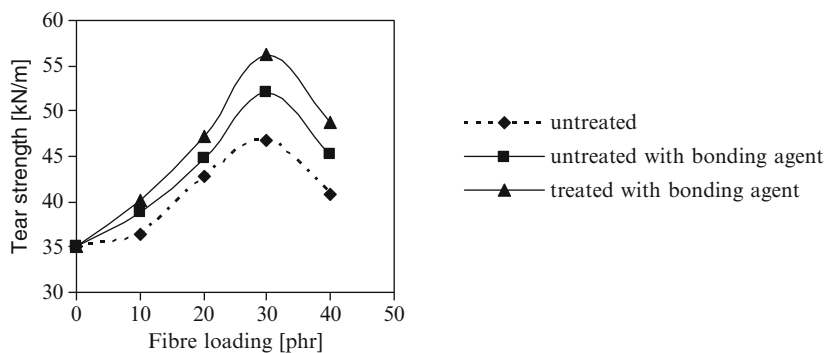
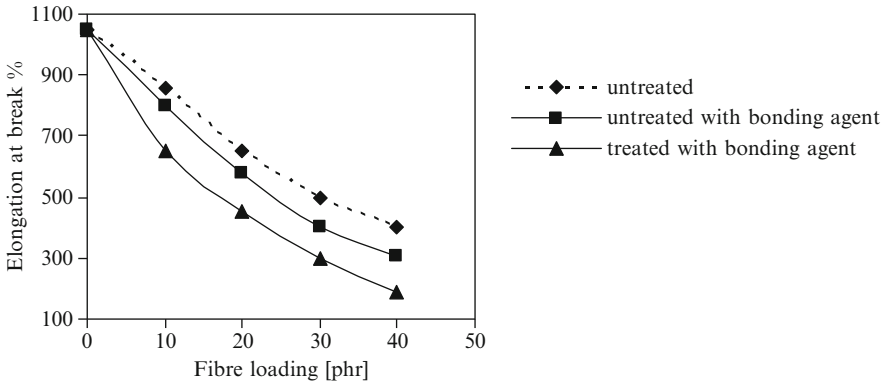
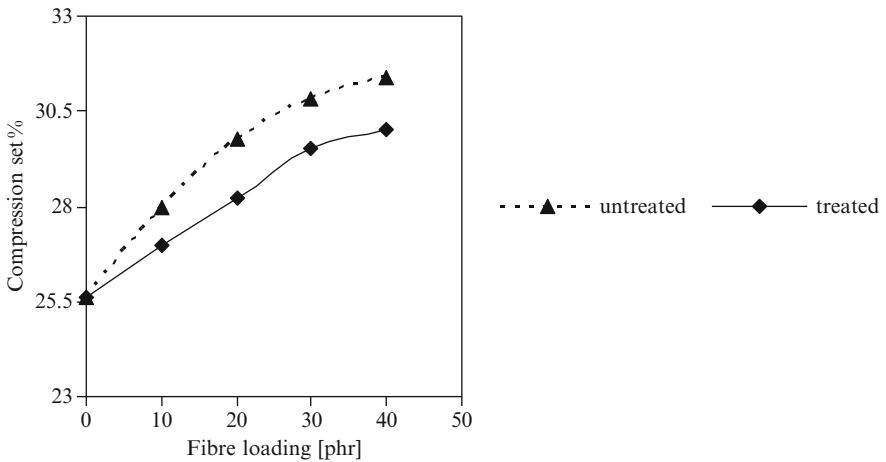


Fig. 11.15 Effect of fibre loading on tear strength of the composites



**Fig. 11.16** Effect of fibre loading on elongation at break of the composites



**Fig. 11.17** Variation of compression set with fibre loading for the composites with bonding agent

showed better resistance to abrasion compared to those containing untreated fibres. The better abrasion resistance of the treated fibre composites may be resulting from the combination of higher tear strength, tensile strength, and modulus achieved through better bonding with the rubber matrix. The hardness of the composites also increases in the presence of bonding agent (Fig. 11.19). The SEM studies revealed the indications of high interfacial adhesion. Figures 11.20a, b and 11.21a, b are the SEM of the tensile and tear fracture surfaces of composites ( $X_{30}$  and  $Y_{30b}$ ) with and without bonding agent. SEM studies also revealed that for composites without bonding agent, failure occurred at the weak fibre/rubber interface while for composites containing treated fibre and bonding agent failure occurred at the fibre due to strong adhesion between fibres and matrix.



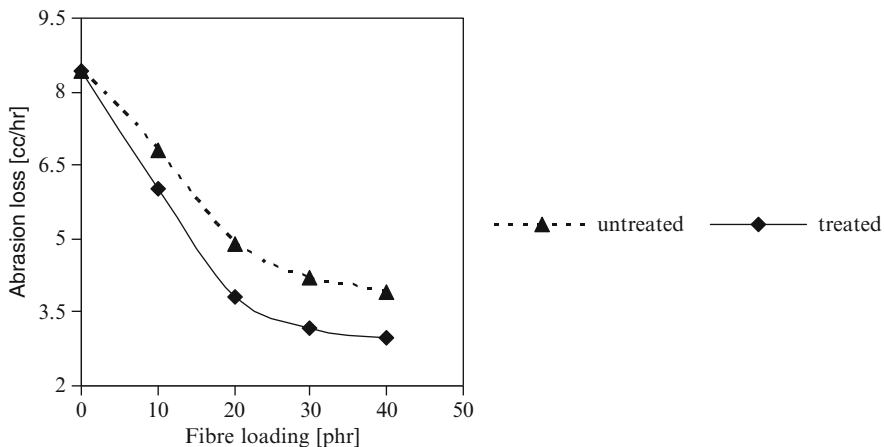


Fig. 11.18 Variation of abrasion loss with fibre loading for the composites with bonding agent

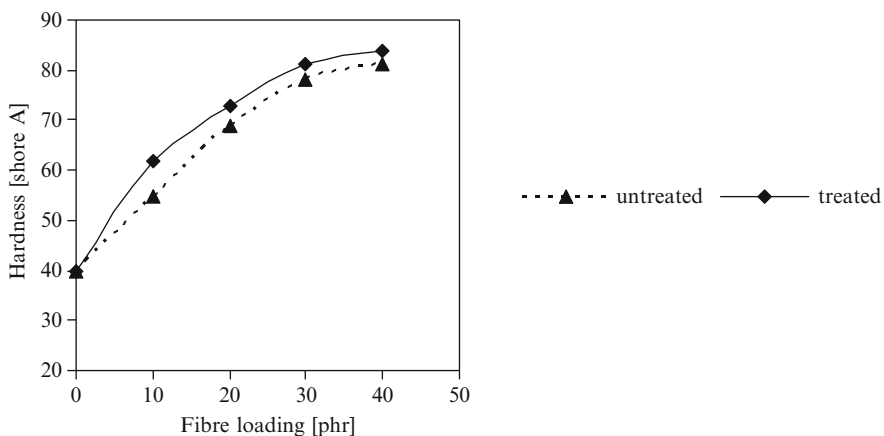
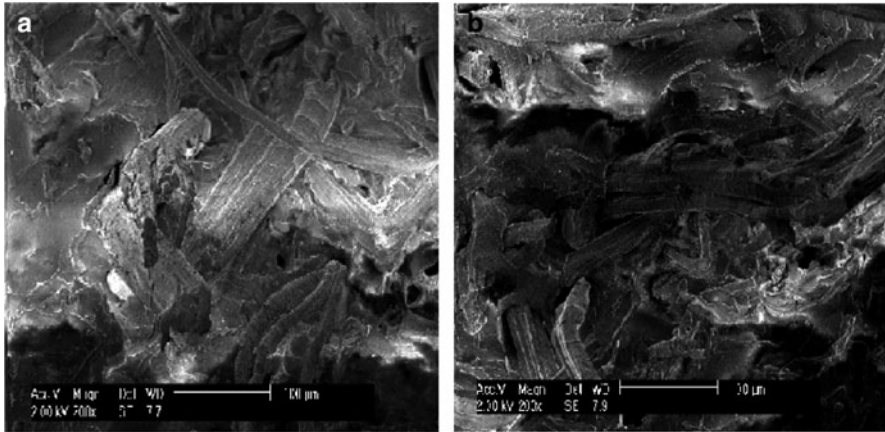


Fig. 11.19 Variation of hardness with fibre loading for the composites with bonding agent

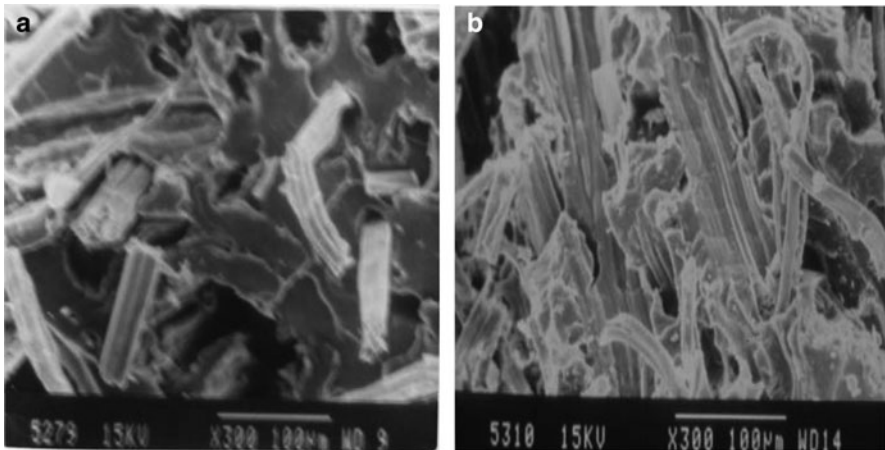
### 11.3.2 Isora: Polyester Composites

#### 11.3.2.1 Composite Preparation

Unidirectional isora fibre–polyester composites containing varying fibre volume fractions were prepared by hand lay up technique using a three-piece stainless steel mould having dimensions  $150 \times 70 \times 2 \text{ mm}^3$ . 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



**Fig. 11.20** SEM of the tensile fractured surfaces containing (a) untreated fibre without bonding agent and (b) treated fibre with bonding agent of the composites X<sub>30</sub> and Y<sub>30b</sub>



**Fig. 11.21** SEM of the tear fractured surfaces containing (a) untreated fibre without bonding agent and (b) treated fibre with bonding agent of the composites X<sub>30</sub> and Y<sub>30b</sub>

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 room temperature and the excess resin was allowed to flow out as “flash.” The pressure was increased very slowly taking care to avoid the slippage of the fibre sheets, so that the unidirectional nature of the fibre is not lost. 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 h. From the sheet, samples were cut for measuring tensile and flexural properties according to

ASTM standards. Composites containing varying fibre loading were prepared using untreated and alkali treated fibres.

The mould with dimensions  $120 \times 60 \times 4 \text{ mm}^3$  was used for preparing samples to determine the impact strength. Composites were prepared in a similar manner described above, using untreated and alkali treated fibres.

### 11.3.2.2 Characterisation of the Composites

The SEM photographs of the fractured surfaces of the composites were taken using a JEOL JSM 840 SEM. Dynamic mechanical analysis of the composites was carried out using rectangular specimens of size  $60 \times 10 \times 3 \text{ mm}^3$ . The dynamic moduli and mechanical damping ( $\tan \delta$ ) were measured using a dynamic mechanical thermal analyser DMA Q80, TA instruments. The temperature range over which properties were measured ranged from 30 to  $130^\circ\text{C}$  at a heating rate of  $3^\circ\text{C min}^{-1}$ . The test was carried out at a frequency of 1 Hz.

Tensile testing of the composite specimens was carried out according to ASTM D5083 on a Shimadzu Model AG1 50 kN universal testing machine at a cross head speed of  $20 \text{ mm min}^{-1}$  and a gauge length of 50 mm. Rectangular specimens of dimensions  $150 \times 10 \times 2 \text{ mm}^3$  were used for testing. The tensile strength and Young's modulus were determined from the stress–strain curves.

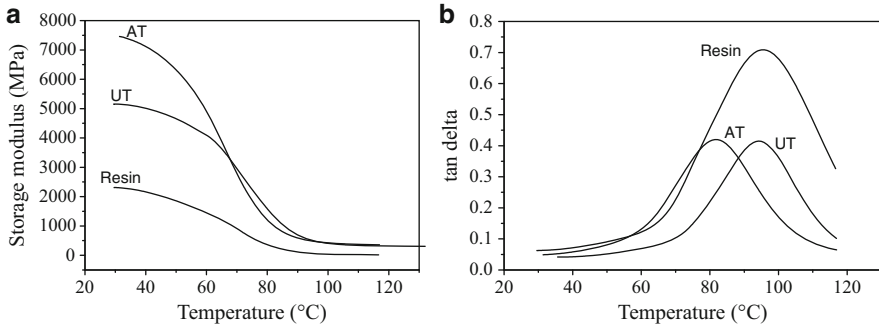
Flexural tests were performed according to ASTM D790 with rectangular strips of size  $75 \times 20 \times 2 \text{ mm}^3$  at a cross head speed of  $2 \text{ mm min}^{-1}$  and lower support 32 mm. Flexural strength and flexural modulus were determined from the stress–strain curves.

Izod impact strength of unnotched sample of the composite was determined according to ASTM D256 using Tinius Olsen Model 503.

### 11.3.2.3 Dynamic Mechanical Analysis of the Composite

Dynamic mechanical thermal analysis was carried out on the cured neat resin and composite samples. The DMA curves of storage modulus ( $E'$ ) versus temperature of neat resin (cured) and composites containing raw (UT) and alkali treated (AT) fibres are given in Fig. 11.22a. It is evident from the figure that the  $E'$  values of the composites are higher than that of neat resin. Also the alkali treated fibre composite has higher  $E'$  value compared to untreated one. The higher  $E'$  value of the treated long isora fibre reinforced polyester composite is due to greater interfacial adhesion and bond strength between matrix resin and fibre as reported by several authors [36, 37].

Figure 11.22b shows  $\tan \delta$  versus temperature plots of neat resin (cured) and composites containing UT and AT fibres. It has been reported by other researchers that incorporation of stiff fibres reduced the  $\tan \delta$  peak by restricting the movement of polymer molecules [36, 38]. From Fig. 11.22b it can be observed that the height of the  $\tan \delta$  peaks of the composites lie far below that of the neat resin. It can also be noted that the peak height of the UT and AT fibre composites are almost same, indicating the same damping capabilities of the composites. Similar results have been reported



**Fig. 11.22** (a) Variation of storage modulus with temperature of neat resin (cured) and composites containing untreated and alkali treated fibres. (b) Variation of  $\tan \delta$  with temperature of neat resin (cured) and composites containing untreated and alkali treated fibres

for hemp fibres by Aziz and Ansell [37]. This is also supported by the impact strength of the UT and AT fibre composites.

#### 11.3.2.4 Mechanical Properties of the Composite

(a) *Tensile properties.* The effect of alkali treatment of the fibre on tensile strength of the composite with fibre loading is given in Fig. 11.23. Tensile strength for the UT and AT fibre composite increased regularly with fibre loading. For UT fibre composite the tensile strength showed maximum value at 45% fibre loading. AT fibre composites showed higher tensile strength values at all fibre loadings indicating enhanced adhesion in the composite. The optimum fibre loading is increased from 45 to 66%, which is again an indication of enhancement in fibre/matrix adhesion in the composite. At higher fibre loadings, the tensile strength values showed a gradual decrease. Young's modulus values for the treated and untreated fibre composites varied in a similar way (Fig. 11.24).

At the optimum fibre loading, there is maximum wetting of the fibre and effective stress transfer at the fibre/matrix interface occurred. The decrease in tensile properties at higher loadings may be due to improper wetting and adhesion between fibre and matrix resulting in inefficient stress transfer.

(b) *Flexural properties.* The effect of alkali treatment on the flexural strength with fibre loading of UT and AT fibre composites is given in Fig. 11.25. The flexural strength of the UT and AT fibre composite increased regularly with fibre loading, the latter values being always higher than the former at all loadings. UT fibre composite showed maximum flexural strength at 56% loading and AT fibre composite at 66% loading, after which the values decreased. So the optimum fibre loading for flexural properties of the untreated fibre composite is 56%. Increase in flexural strength with fibre loading is due to better interaction between fibre and matrix, whereas the decrease of flexural strength at higher fibre loading is due to increased fibre to fibre interaction and also due to dispersion problems. Alkali treatment enhances the

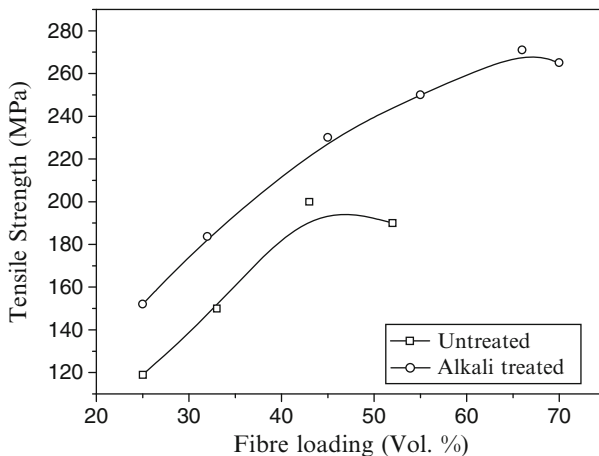


Fig. 11.23 Effect of alkali treatment on the tensile strength of the composite

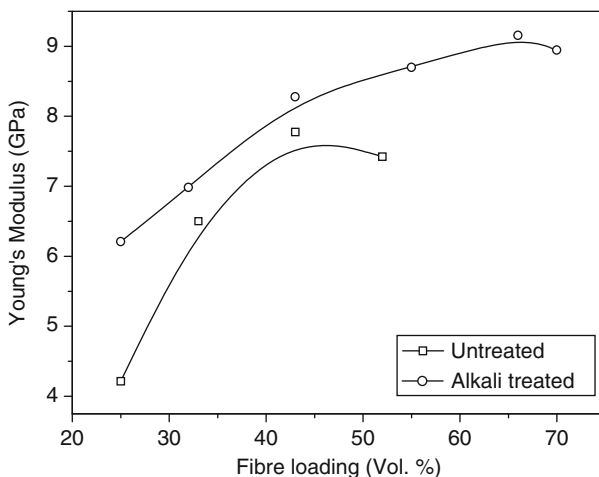
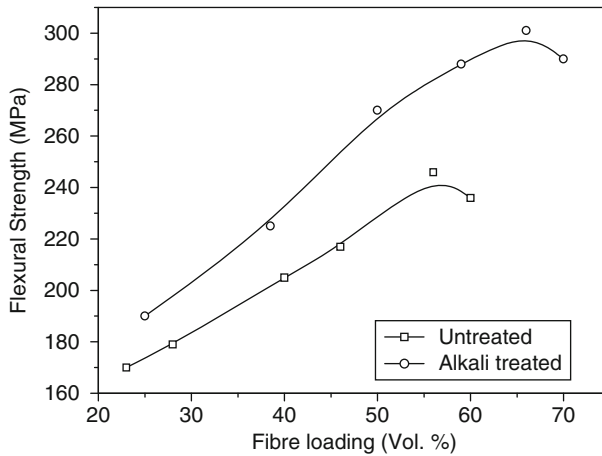


Fig. 11.24 Effect of alkali treatment on the Young's modulus of the composite

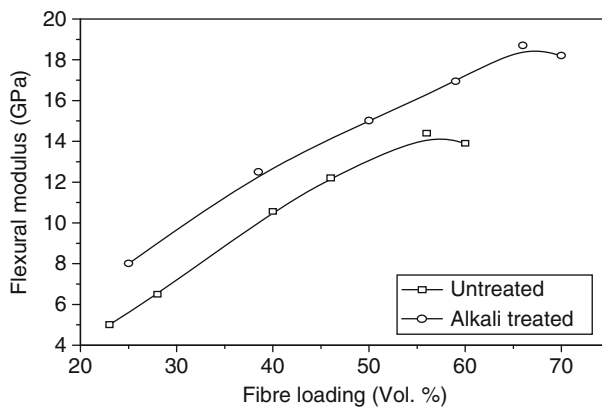
optimum fibre loading for flexural properties from 56 to 66% which indicates enhanced fibre/matrix adhesion in the composite. Flexural modulus of the treated and untreated fibre composites varied in the same manner (Fig. 11.26).

### 11.3.2.5 Impact Strength

Figure 11.27 shows the variation of impact strength of the untreated fibre composite as a function of fibre loading. Impact strength increases with fibre loading, reached



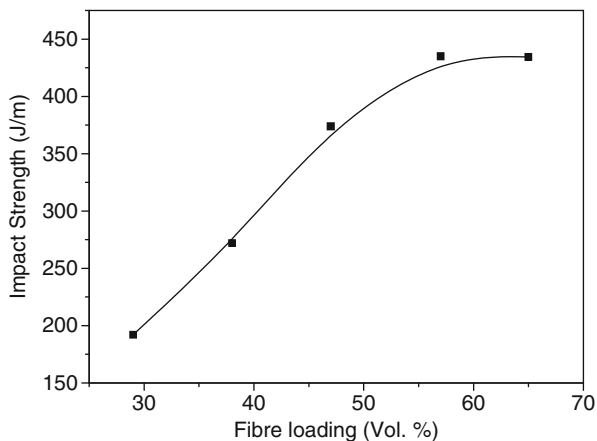
**Fig. 11.25** Effect of alkali treatment on the flexural strength of the composite



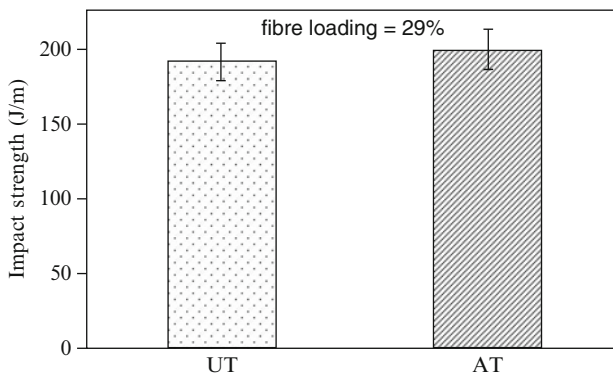
**Fig. 11.26** Effect of alkali treatment on the flexural modulus of the composite

a maximum at 57% by volume of fibre. With further increase in fibre loading, there is no significant increase in impact strength. There is only a nominal increase in impact strength on alkalisation of the fibre (Fig. 11.28). This is supported by the DMA analysis. The fibres play an important role in the impact resistance of the composites as they interact with the crack formation in the matrix and act as stress transferring medium.

On alkali treatment, lignin and hemicellulose, the cementing materials present in the fibre, get dissolved. This makes the interfibrillar region less dense and less rigid as a result of which the fibrils become more capable of orienting themselves along the tensile deformation [39]. Other authors have also reported on the change in crystallinity of alkali treated fibre because of the removal of the cementing



**Fig. 11.27** Variation of impact strength of the untreated fibre composite with fibre loading

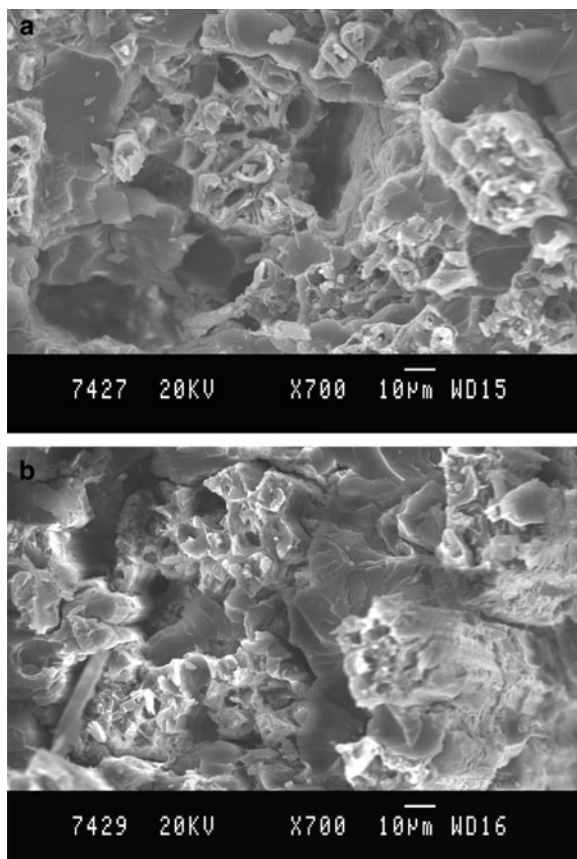


**Fig. 11.28** Effect of alkali treatment on the impact strength of the composite

materials which leads to a better packing of cellulose chains [40, 41]. More than that increase in surface area of the fibre occurs due to the dissolution of lignin, hemicellulose, and alien substances associated with the fibre. Alkali treatment cleans the surface debris and develops microporosity with many pits and holes on the fibre surface by the removal of globular protrusions of fibre surface present in the untreated fibre. This leads to a larger area of contact and greater mechanical interlocking between fibre and matrix, making the interfacial adhesion stronger and the mechanical properties higher.

SEM micrographs of the tensile fractured surfaces of the composites containing untreated and alkali treated fibres (Figs. 11.29a, b) were investigated to evaluate the fibre/matrix adhesion which again indicates a better fibre–matrix interfacial adhesion for the alkali treated fibre composite.

**Fig. 11.29** (a) SEM photograph of the tensile fractured surface of untreated fibre composite. (b) SEM photograph of the tensile fractured surface of alkali treated fibre composite



## 11.4 Conclusions

### 11.4.1 Fibre Characterisation

Isora is found to be a cellulosic rich fibre with comparatively less lignin content.

A small positive change in the fibre density was observed for the treated fibres indicating cell wall densification. Isora fibre contains a higher percentage of hot water soluble and caustic soda soluble matter.

Chemical treatment slightly reduced the tensile strength of the fibre as the binding component of the fibrils gets removed on alkali treatment. Elongation at break more or less same even after the treatment.

WAXRD studies indicate an increase in the crystallinity index after chemical treatment.

FTIR spectroscopy has provided additional information on the reactivity of the fibres following treatment with alkali and silane



SEM studies revealed that chemical treatment modified the fibre surface. Fine structural changes of the fibres can be seen from the respective SEM photographs.

Thermal stability and degradation characteristics of the fibre was investigated by TGA/DTG, DTA, and DSC which indicate that thermal stability of the fibres was increased upon treatment with alkali and silane. Fibres are stable up to 300°C without any considerable weight loss.

Finally, it is important to mention that properties of isora fibre are comparable to other natural fibres and therefore they could be successfully used as a potential reinforcing material for polymer matrices.

### ***11.4.2 Isora: Rubber Composites***

The mechanical properties of short isora fibre reinforced natural rubber composites are enhanced by chemical treatment on the fibre surface and by the use of bonding agent. Longitudinally oriented fibre composites have superior properties than transversely oriented ones.

The optimum length and loading of isora fibre in NR composite are found to be 10 mm and 30 phr, respectively, to achieve good reinforcement.

The surface morphology of isora fibre is modified by alkali treatment. SEM analysis revealed that better adhesion is observed between alkali treated isora fibre and NR in the composites.

Presence of bonding agents in the composites prolonged curing time.

SEM studies also revealed that for the control compound (untreated fibre without bonding agent), failure occurred at the weak fibre/rubber interface, while for composites containing treated fibre and bonding agent, failure occurred in the fibre due to the strong adhesion at the fibre/rubber interface.

### ***11.4.3 Isora: Polyester Composites***

The optimum fibre loading for untreated fibre composite for tensile and flexural properties were found to be 45 and 49 vol.%, respectively.

On alkali treatment, the optimum fibre loading for tensile and flexural properties was increased to 66 vol.%, indicating enhanced fibre–matrix adhesion.

Impact strength of the composite increased with increase in fibre loading and remained constant at a loading of 57 vol.%.

Alkali treatment has only a marginal effect on the impact strength.

DMA analysis revealed that treated fibre composite has higher storage modulus ( $E'$ ), equal  $\tan \delta$ , and impact strength indicating greater interfacial bond strength and adhesion between the matrix and the fibre compared to untreated fibre composites.

SEM studies of the tensile fractured surface of the composites gave evidence for the enhanced interfacial adhesion between the fibre and the matrix on alkalisation of the fibre.

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