Oil Palm Fibers: Morphology, Chemical Composition, Surface Modification, and Mechanical Properties

M. S. SREEKALA,¹ M. G. KUMARAN,¹ SABU THOMAS²

¹ Rubber Research Institute of India, Kottayam-686 009, Kerala, India

² School of Chemical Sciences, Mahatma Gandhi University, Priyadarshini Hills P.O., Kottayam-686 560, Kerala, India

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ABSTRACT: Oil palm fiber is an important lignocellulosic raw material for the preparation of cost-effective and environment-friendly composite materials. The morphology and properties of these fibers have been analyzed. The properties of two important types of fibers, the oil palm empty fruit bunch fiber and the oil palm mesocarp fiber (fruit fiber) have been described. The surface topology of the fibers has been studied by scanning electron microscopy. Thermogravimetry and differential thermal analysis were used to determine the thermal stability of the fibers. Fiber surface modifications by alkali treatment, acetylation, and silane treatment were tried. The modified surfaces were characterized by infrared spectroscopy and scanning electron microscopy. The chemical constituents of the fibers were estimated according to ASTM standards. Mechanical performance of the fibers was also investigated. Microfibrillar angle of the fibers was theoretically predicted. The theoretical strength of the fibers was also calculated and compared with the experimental results. © 1997 John Wiley & Sons, Inc. J Appl Polym Sci **66**: 821–835, 1997

Key words: oil palm fibers; surface modification; morphology; IR spectroscopy; thermal analysis; mechanical property

INTRODUCTION

Oil palm is one of the most economical and veryhigh-potential perennial oil crops. It belongs to the species *Elaeis guineensis* under the family *Palmacea*, and originated in the tropical forests of West Africa. Major industrial cultivation is in Southeast Asian countries such as Malaysia and Indonesia. Large-scale cultivation has come up in Latin America. In India, oil palm cultivation is coming up on a large-scale basis with a view to attaining self sufficiency in oil production.

Oil palm empty fruit bunch (OPEFB) fiber and oil palm mesocarp fiber are two important types of fibrous materials left in the palm-oil mill. Figure 1 shows the photographs of fruit bunch fiber and oil palm mesocarp fiber. OPEFB is obtained after the removal of oil seeds from fruit bunch for oil extraction. Photograph of an empty fruit bunch is shown in Figure 2. OPEFB fiber is extracted by the retting process of the empty fruit bunch. Average yield of OPEFB fiber is about 400 g per bunch. Mesocarp fibers are left as a waste material after the oil extraction. These fibers must be cleaned of oily and dirty materials. The only current uses of this highly cellulosic material are as boiler fuel and in the preparation of potassium fertilizers. When left on the plantation floor, these waste materials create great environmental problems. Therefore, economic utilization of these fibers will be beneficial. This requires

Correspondence to: S. Thomas.

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Figure 1 Photograph of (a) oil palm empty fruit bunch fiber and (b) oil palm mesocarp fiber.

extensive study of the chemical and physical characteristics of these fibers.

We have reported earlier about the possibilities of using OPEFB fiber as a potential reinforcement in phenol-formaldehyde resin.¹ The mechanical performance of phenol-formaldehyde resin is greatly improved by the incorporation of these fibers. The resultant composite product will be a cost-effective and value-added substitute for conventional building materials which can act as a better substitute for wood in building industry.

Many of the natural fibers—such as coir, banana, sisal, talipot, palmyrah, jute, pineapple leaf fiber, etc.-find applications as a resource for industrial materials.^{2,3} Properties of the natural fibers depend mainly on the nature of the plant, locality in which it is grown, age of the plant, and the extraction method used. For example, coir is a hard and tough multicellular fiber with a central portion called "lacuna." On the other hand, banana fiber is weak and cylindrical in shape. Sisal is an important leaf fiber and is strong. Pineapple leaf fiber is soft and has high cellulose content. Investigations based on these fibers are still ongoing. Many studies have reported on the natural fiber based composite products.⁴⁻⁶ Oil palm fibers are hard and tough, and show similarity to coir fibers in cellular structure. To date, no systematic work has been undertaken to evaluate the morphology and physical properties of oil palm fibers.

The physical properties of other natural fibers have already been reported.⁷⁻¹⁵ Barkakaty⁷ reported on the structural aspects of sisal fibers. Martinez and colleagues⁸ studied the physical and mechanical properties of the lignocellulosic henequen fibers. Thermal stability and moisture regain of wood fibers were studied by Rao and Gupta.⁹ They utilized a scanning electron microscope to study the morphological characteristics.

Chemical treatments of cellulosic materials usually change the physical and chemical structure of the fiber surface.¹⁰ Mukherjee and associates¹¹ reported the effect of ethylene diamine on the physicochemical properties of jute fibers. Xray and infrared (IR) studies can be used to investigate the changes in the fine structure of fiber surface.¹² Effects of alkali, silane coupling agent, and acetylation have been tried on the oil palm fibers. It is reported that the alkali treatment on coir fiber enhances the thermal stability and maximum moisture retention.13 Prasad and coworkers¹⁴ reported that the use of alkali-treated coir fibers greatly improves the mechanical properties of coir-polyester composites. Chemical analysis of the oil palm fibers shows that the principal component is cellulose. The cellulose content plays an important role in the fiber's performance. The properties of the particle boards prepared from OPEFB fiber and urea formaldehyde resin have been reported earlier.¹⁵ Many studies have reported on the determination of fiber strength using various techniques.^{16–18}

In this article, we report on the chemical, physical, and morphological characteristics of the oil palm fibers. Surface modifications of the fibers by alkali treatment, silane treatment, and acetylation have been tried. Morphological analysis has been carried out with the help of IR and scanning electron microscopy (SEM) studies. Chemical constituents of the fibers were determined. Mechanical properties such as tensile strength, Young's modulus, and elongation at break were evaluated.



Figure 2 Photograph of an oil palm empty fruit bunch.

Fiber	Lignin (%)	Cellulose (%)	Hemicellulose (%)	Ash Content (%)
OPEFB fiber	19	65	_	2
Oil palm mesocarp fiber	11	60	_	3
Coir	40 - 45	32 - 43	0.15 - 0.25	_
Banana	5	63 - 64	19	_
Sisal	10 - 14	66 - 72	12	_
Pineapple leaf fiber	12.7	81.5	—	—

Source: ref. 1.

The deformation characteristics of the fibers were studied with the help of stress-strain curves. Thermogravimetric (TGA) and differential thermal analyses (DTA) were carried out to study the thermal stability of the fibers. Microfibrillar angle and strength of the fibers were theoretically calculated.

MATERIALS AND EXPERIMENTAL

The fibers were collected from Oil Palm India Ltd., Kottayam, India. The empty fruit bunch was subjected to retting. Fibers were then cleaned, washed, and dried.

Fiber Surface Modification

Alkali Treatment

Fibers were dipped in 5% sodium hydroxide solution for about 48 h. These were further washed with water containing a few drops of acetic acid. Finally, the fibers were washed again with fresh water and dried.

Silane Treatment

Fibers were dipped in 1% silane solution (triethoxy vinylsilane) in water-ethanol mixture (40:60) for about 3 h. The pH of the solution was maintained to 3.5-4. Fibers were washed and then dried.

Acetylation

Fibers were treated in glacial acetic acid for 1 h. This was further treated with acetic anhydride containing concentrated H_2SO_4 as catalyst for 5 min. Fibers were then washed with water and dried.

SEM Examination

The SEM photographs of fiber surfaces and cross sections of untreated and treated fibers were taken using a scanning electron microscope, Philips model PSEM-500.

IR Spectra

KBr disk method was followed in taking IR spectra. The instrument used was a Shimadzu IR-470 infrared spectrophotometer.

Chemical Constituent	Oil Palm Empty Fruit Bunch Fiber (%)	Oil Palm Mesocarp Fiber (Fruit Fiber) (%)
Alcohol–benzene solubility	12	12
Ether solubility	12	20
1% caustic soda solubility	20	27
Cold-water solubility	8	12
Hot-water solubility	10	12

Table II Solubility of Oil Palm Fibers in Different Solvents



Figure 3 Distribution curve of fiber diameter of untreated and treated OPEFB fibers.

Thermal Analysis

Thermograms of untreated and treated fibers were taken in an inert atmosphere at a heating rate of 10° C/min. A Shimadzu DT-40 thermal analyzer was used for the study.



Figure 4 Distribution curve of fiber diameter of untreated and treated oil palm mesocarp fibers.





Figure 5 Scanning electron micrographs of untreated OPEFB fiber: (a) fiber surface ($\times 400$) and (b) cross section ($\times 200$).

Chemical Estimation

Chemical compositions of fibers were estimated according to the following ASTM procedures: lig-



Figure 6 Scanning electron micrograph of alkalitreated OPEFB fiber surface $(\times 400)$.



Figure 7 Distribution curve of pore size of the untreated and alkali-treated OPEFB fiber surface.

nin—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.

Mechanical Property Tests

Strength of the oil palm fibers was determined using a FIE TNE-500 electronic tensile testing machine. The fibers were mounted in a fixture made of paperboard with a central window and



Figure 8 Scanning electron micrograph of silane-treated OPEFB fiber surface $(\times 400)$.



Figure 9 Scanning electron micrograph of acetylated OPEFB fiber surface $(\times 400)$.

pulled at a strain rate of 20 mm/min. The gauge length was 50 mm and 20 mm in the case of OPEFB fiber and oil palm mesocarp fiber, respectively. Strength, Young's modulus, and elongation at break were evaluated.

RESULTS AND DISCUSSION

Chemical Analysis

Table I shows the various chemical components present in the OPEFB fiber and oil palm mesocarp fiber. The OPEFB fiber contains a higher percentage of cellulose. Lignin content is comparatively low. The total cellulose content (holocellulose) of the fiber was found to be 65%. The fiber was found to have a very low ash content. All these factors contribute to better performance of the fiber as a reinforcement in polymers. The fiber is hygroscopic and its moisture content was found to be 12%. Cellulose and lignin content of mesocarp fiber is less than that of OPEFB fiber.

Table I compares the results with those of some other important natural fibers. Compared with coir fibers, OPEFB fiber is highly cellulosic. Coir has a higher percentage of lignin than OPEFB fiber. However, the cellulose content of OPEFB fiber is slightly less than that of banana and sisal fibers, and much less than that of pineapple leaf fiber. The lignin contents of banana, sisal, and pineapple leaf fibers are less than that of OPEFB fiber.

Solubility of the fibers in different solvents is given in Table II. Caustic soda solubility is higher when compared with other solvent solubility. The OPEFB fiber contains 10% water-soluble matter.







Figure 10 Scanning electron micrographs of untreated oil palm mesocarp fiber: (a) fiber surface $(\times 400)$ and (b) cross section $(\times 800)$.

Mesocarp fiber surface contains traces of oils, dissolves on treatment with NaOH solution, and shows a higher-percentage solubility in ether, caustic soda, cold water, and hot water than does OPEFB fiber. Moisture content of the mesocarp fiber was found to be 11%.



Figure 11 Scanning electron micrograph of alkalitreated oil palm mesocarp fiber surface $(\times 400)$.



Figure 12 Scanning electron micrograph of silanetreated oil palm mesocarp fiber surface $(\times 400)$.

Chemical Modifications

Fiber treatments such as alkali treatment, acetylation, and silane treatment were tried for OPEFB fiber. Mesocarp fiber was subjected to alkali and silane treatments. Possible mechanisms of the chemical modifications are given as follows.¹⁹

Alkali Treatment

Fiber
$$- OH + NaOH \rightarrow Fiber - O^{-}Na^{+} + H_2O$$

NaOH treatment leads to the irreversible mercerization effect which increases the amount of amorphous cellulose at the expense of crystalline cellulose. Mercerization treatment improves the fiber surface adhesive characteristics by removing natural and artificial impurities, thereby producing a rough surface topography. The weight of the OPEFB fiber was decreased by 22% after the alkali treatment. For mesocarp fiber, the weight reduction observed was 25%.

Acetylation

Fiber – OH + CH₃COOH
$$\xrightarrow{(CH_3CO)_2O}_{Conc. H_2SO_4}$$

O
Fiber – O – C – CH₃ + H₂O

The extent of acetylation is estimated by the titrimetric method. The number of O—acetyl groups in a certain amount of acetylated fiber is estimated by hydrolyzing with excess normal caustic soda; the unreacted alkali is then determined by titrating



Figure 13 IR spectra of OPEFB fiber before and after treatments.

against normal oxalic acid. The number of O—acetyl groups in 0.5 g of acetylated OPEFB fiber was found to be 0.006. The reaction is expected to take place at the free OH groups available on cellulose molecules. There was no significant weight change of the fiber on acetylation. The fiber became more hydrophobic after the treatment.

Silane Treatment

The silane having the following chemical structure reacts with water to form a silanol and an alcohol.



 $\rm CH_2\rm CH~Si(\rm OC_2\rm H_5)_3 + 3\rm H_2\rm O \rightarrow$

$$\begin{array}{c} CH_2CH \,\,Si(OH)_3 \,+\, 3C_2H_5OH \\ \scriptstyle (Silanol) \end{array}$$

Silane molecules are chemisorbed onto the fiber. The extent of silane chemisorption depends on the availability of free OH groups in the fiber. In presence of moisture, silanol reacts with cellulosic hydroxyl groups in the fiber, forming stable covalent bonds to the cell wall.

$$CH_{2}CHSi(OH)_{3} + H_{2}O + Fiber - OH \rightarrow$$
$$CH_{2}CHSi(OH)_{2}O - Fiber + 2H_{2}O$$

The weight of the OPEFB fiber decreased by 6% on silynylation. A 7% decrease was observed for mesocarp fibers. Hydrophobicity of the fibers increased on silynylation. The hydrophobic coupling agent forms a protective monolayer on the proton-bearing surfaces and thus removes the sites for moisture absorption.

These modifications are most effective in the surface regions. As the concentration and time of treatment increases, the treatment effect may



Figure 14 IR spectra of treated and untreated oil palm mesocarp fibers.



Figure 15 TGA and DTA curves of OPEFB fibers: (a) untreated, (b) alkali-treated, (c) silane-treated, and (d) acetylated.

penetrate into the fiber. However, there will be a saturation point beyond which no further reaction takes place.

Dimensional Changes on Treatments

The dimensional changes of the fibers after treatments can be seen from the distribution curves (Figs. 3 and 4). The diameters of about 100 fibers before and after treatments were measured and distribution curves plotted. Chemical treatment significantly reduces the fiber diameter in both the fibers.

SEM Studies

Figure 5 shows SEM photographs of the untreated OPEFB fiber surface and its cross section.

Untreated (°C)	Alkali-treated (°C)	Acetylated (°C)	Silane-treated (°C)	Weight Loss (%)
150	235	145	180	10
260	290	240	300	20
300	325	285	328	30
315	350	308	360	40
340	352	325	370	50
340	352	338	370	60
345	360	340	370	70
395	415	370	420	80
440	460	435	440	90
480	510	495	520	100

Table III Weight Losses of Untreated and Treated OPEFB Fibers atVarious Temperatures

The photographs show minute pores on the surface of the fiber. The pores were found to have an average diameter of 0.07 μ m. The cross section of the fiber shows a lacuna-like portion in the middle. The porous surface morphology is useful for better mechanical interlocking with the matrix resin for composite fabrication. The alkali-treated fiber surface is presented in Figure 6. The pores became more prominent upon alkali treatment. Average diameter of the pores was found to be 0.15 μ m. The distribution of the pores of different size before and after alkali treatment can be understood from the distribution curve (Fig. 7). Pore size of about 100 micropores (selected at random) on the fiber surface were measured from SEM photographs and the distribution curve drawn.

Figures 8 and 9 give the micrographs of silanetreated and acetylated fiber surfaces, respectively. Acetylation clearly eliminates the waxy cuticle layer on the surface. This is evident from the micrographs.

Surface characteristics of the untreated oil palm mesocarp fiber are clear from Figure 10(a). The fiber surface is rough, exhibiting protruding portions and groove-like structures on its surface. The cross section of the fiber is not uniform [Fig. 10(b)]. The morphological changes of fiber upon alkali treatment and silane treatment are evident from the respective SEM photographs given in Figures 11 and 12. Alkali-treated fiber surface shows some protrusions (Fig. 11). This may be associated with the removal of the cuticle layer from the fiber surface. The fiber surface became clear on silane treatment (Fig. 12). Large number of micropores could be seen on the surface, having an average diameter of 0.2 μ m.

IR Studies

IR spectra of the untreated and treated OPEFB fibers are given in Figure 13. It can be understood from the spectra that some chemical reactions occurred during the different treatments. Major changes are observed in the IR absorbance of alkali-treated, acetylated, and silane-treated samples. Peaks at 770 and 2850 cm⁻¹, corresponding to C—O stretching and C—H stretching vibrations, are present in the untreated fiber. On modification, these peaks diminish. Alkali treatment may reduce the hydrogen bonding in cellulosic hydroxyl groups, thereby increasing the OH concentration. This is evident from the increased intensity of the OH peak (3450 cm⁻¹) in alkali-treated fiber. The alkali-soluble matter in the fiber is 20%. A peak at 1730 cm⁻¹ in acetylated fiber indicates the presence of an ester group. The peak at 1525 $\rm cm^{-1}$ in the untreated fiber is shifted to 1600 $\rm cm^{-1}$ upon silane treatment. This may be due to the C = C stretching.

On comparing the IR spectra of untreated OPEFB fiber and oil palm mesocarp fiber, it is seen that there is structural similarity between these fibers. Both of the spectra show intense peaks at 3450 and 2850 cm⁻¹ (O—H stretching and C—H stretching, respectively).

The fine structural changes of oil palm mesocarp fibers upon chemical modification can be understood from the IR spectra given in Figure 14. Major changes in the absorbance occur in the case of alkali treatment. The C=O stretching frequency of the carboxylic group (1730 cm⁻¹) disappears upon alkali treatment. This may be due to the removal of the carboxylic group by alkali. Car-



Figure 16 TGA and DTA curves of oil palm mesocarp fibers: (a) untreated, (b) alkalitreated, and (c) silane-treated.

boxylic groups may be present on the fiber surface from traces of fatty acids present. The intensity of the peak at 2125 cm⁻¹ increases upon alkali and silane treatments. This may be due to the C—H stretching. Intensity of the hydroxy vibration absorption (3425 cm⁻¹) increased considerably upon alkali and silane treatments, as in the case of OPEFB fiber. Cellulosic hydroxyl groups may be involved in hydrogen bonding. There are chances for bonding with carboxylic groups of the fatty acids present on the fiber. Presence of a peak at 1730 cm⁻¹ in untreated fiber gives evidence for this. On treatments, these bonds may break. The peak at 1560 cm⁻¹ (C=C stretching) disappeared upon alkali and silane treatments. This may be due to the treatments' removal of unsaturation present in the traces of oils. From these studies it is clear that several chemical reactions took place during treatments.

Sao and Jain²⁰ studied the mercerization effects

Untreated (°C)	Alkali-treated (°C)	Silane-treated (°C)	Weight Loss (%)
138	180	165	10
218	285	285	20
275	310	315	30
290	340	330	40
315	350	350	50
328	355	360	60
340	410	370	70
373	450	425	80
430	470	480	90
455	540	520	100

Table IVWeight Losses of Untreated and Treated Oil Palm MesocarpFibers at Various Temperatures

of aqueous NaOH on jute. Effect of weak alkalis such as sodium carbonate on jute fiber was reported by Sikdar and associates.²¹ The treatment cleaned the fiber surface and led to higher yarn productivity. The surface treatment of the fibers affects its mechanical properties.²² The incorporation of the treated fibers into plastics and rubber increases the usual strength of the composites.^{23–28}

Thermal Studies

Figure 15 shows the thermal degradation pattern of untreated, alkali-treated, silane-treated, and acetylated OPEFB fibers. Below 100°C, a 5-8%weight loss was observed. This may be due to the dehydration of the fibers. Initial degradation temperature is higher for the alkali-treated fiber. This is evident from the DTA and TGA curves [Fig. 15(b)]. Major weight losses of the untreated and acetylated fibers take place at about 325°C. Alkali treatment raises this temperature to 350°C,

whereas silane treatment raises it to 365°C [Fig. 15(c)]. The DTA curve shows a major peak in this region, which may be due to the thermal depolymerization of hemicellulose and the cleavage of the glucosidic linkages of cellulose.²⁹ This is an exothermic process. At the first stage of degradation, the DTA curve shows an endothermic peak in all cases (Fig. 15). This peak may be due to the volatilization effect. Breakage of the decomposition products of the second stage (second peak) leads to the formation of charred residue. The third exothermic peak present in the DTA curve is due to this oxidation and burning of the highmolecular-weight residues. In acetvlated fiber, the second peak is not prominent. Complete decomposition of all the samples takes place around 500°C. The percentage weight losses of untreated and treated fibers at various temperatures are given in Table III. From the table it can be understood that both alkali and silane treatment improve the thermal stability of the fibers.

Table V Mechanical Properties of Oil Palm Fibers

Fiber	Tensile Strength (MPa)	Young's Modulus (MPa)	Elongation at Break (%)
OPEFB			
Untreated	248	2,000	14
Alkali-treated	224	5,000	16
Silane-treated	273	5,250	14
Oil palm mesocarp fiber (fruit fiber)			
Untreated	80	500	17
Alkali-treated	64	740	6.5
Silane-treated	111	1,120	13.5



Figure 17 Stress-strain characteristics of untreated and treated OPEFB fibers.

Thermal stability of OPEFB fiber is higher than that of oil palm mesocarp fiber. Silanetreated OPEFB fiber is stable up to 365° C, whereas stability of the alkali-treated oil palm mesocarp fiber reaches 340° C. TGA and DTA scans of the untreated and treated mesocarp fibers are presented in Figure 16. The untreated fiber is stable up to 310° C. Alkali and silane treatment raises the stability of fiber to 340° C. Alkali treatment raises the initial degradation temperature (weight loss of 10%) to 180° C from 138° C. The initial weight loss may be due to the vaporization of water present in the sample. The DTA curve shows a corresponding endothermic peak in this region. Major degradation of untreated fibers occurs at about 310°C; treatment raises this temperature to 340°C. Decomposition of cellulose may occur at this stage. A corresponding exothermic peak is observed in the DTA curve. The third exothermic peak in the DTA curve may be due to the formation of charred residue from the first degradation products. Broadening of the DTA peak is observed for the silane-treated sample. The gradual degradation and percentage weight losses of untreated and treated fibers can be understood from Table IV. From these results, it can be concluded that alkali treatment is more effective in improving the thermal stability.

It was reported by Mahato and colleagues¹³ that 5 to 15% of alkali-treated coir fibers showed maximum thermal stability. Varma and associates³⁰ also investigated the effect of alkali treatment of natural fibers on thermal stability. Shah and coworkers³¹ reported that sodium hydroxide treatment of lignocellulosic fibers leads to the formation of a lignin–cellulose complex which gives more stability to the fiber.

Mechanical Performance

The effects of various chemical treatments on mechanical properties of the OPEFB fiber were studied. The important mechanical properties of the fiber are given in Table V. The nature and texture of the fibers obtained from different plants may not be the same. The diameter of the fibers varies in the range from 0.015×10^4 to 0.05×10^4 µm. The density of these fibers lies in the range from 0.7 to 1.55 g/c³. All these factors will affect the properties of the fiber, therefore there is large variation in the observed properties. An average value of the properties is reported.

The untreated fiber shows 14% elongation. Elongation at break remains more or less same

Fiber	Tensile Strength (MPa)	Elongation (%)	Toughness (MPa)
Sisal	580	4.3	1,250
Pineapple	640	2.4	970
Banana	540	3.0	816
Coir	140	25.0	3,200
OPEFB fiber	248	14	2,000
Oil palm mesocarp fiber	80	17	500

 Table VI
 Mechanical Properties of Some Important Natural Fibers

Source: ref. 32.

even after fiber treatment. This may be due to the firmly bound chemical structure of the fiber. Lignin binds the three-dimensional cellulose network as well as the fibrils. Figure 17 shows the stress-strain characteristics of the treated and untreated OPEFB fibers. At the very beginning (<1% elongation) there is linearity, and thereafter curvature is observed. As the applied stress increases, the weak primary cell wall collapses and decohesion of cells occurs, resulting in the mechanical failure of the fiber. The difference in stress-strain behavior of untreated and treated fibers is evident from Figure 17. Fiber modification by alkali treatment and silane treatment improves the overall mechanical performance of the fiber. Maximum tensile strength is given by silane-treated OPEFB fiber. The stiffness of the fiber is greatly improved upon modification. The properties of the fiber were compared with those of some important natural fibers (Table VI).³² The strength and stiffness of the OPEFB fiber is much higher than that of coir. Coir shows highest elongation among commonly used natural fibers. OPEFB fiber shows higher elongation than sisal, pineapple, and banana. The fiber is highly tough. However, the tensile strength of the fiber is less than that of sisal, pineapple, and banana fibers.

The strength and Young's modulus of the OPEFB fiber are greater than those of oil palm mesocarp fiber. But the mesocarp fiber shows a higher percentage of elongation. The mechanical performance of the mesocarp fiber is comparatively low with respect to other natural fibers (Table VI).

Properties of lignocellulosic fibers depend mainly on the cellulose content and microfibrillar angle. Various mechanical properties of oil palm mesocarp fibers are given in Table V. The density of the fiber was found to vary within the range from 0.6 to 1.18 g/c^3 . An average diameter of 0.02 \times 10⁴ µm was observed for these fibers. In fact, the diameter even varied within a single fiber. The stress-strain characteristics of treated and untreated fibers are given in Figure 18. The modulus of the fiber increased upon modification by alkali and silane coupling agent. Silane treatment was found to be more effective. Silane-treated fiber showed maximum tensile strength. However, alkali treatment slightly decreased the tensile strength. The elongation at break was maximum for untreated fibers. The value showed decrease upon treatment. The firmly bound three-dimensional network of cellular arrangement may be partly destroyed upon treatment. Alkali treat-



Figure 18 Stress-strain characteristics of untreated and treated oil palm mesocarp fibers.

ment reduced the tensile strength of the mesocarp fiber. This may be due to the bleaching of the oily and waxy materials from the fiber surface. Mesocarp fibers may contain traces of oil even after processing, since the oil is present in the flesh of the fruit. Silane treatment was found to be more effective in improving mechanical properties.

Theoretical Prediction of Microfibrillar Angle and Strength of the Fibers

Strength properties of the fibers are dependent mainly on the fibrillar structure, microfibrillar angle, and cellulose content. There is a correlation between percentage elongation ϵ and the microfibrillar angle θ as³³:

$$\epsilon = -2.78 + 7.28 \times 10^{-2}\theta + 7.7 \times 10^{-3}\theta^2 \quad (1)$$

Using this equation, the microfibrillar angle of OPEFB fiber is found to be 42°. There exists a relationship between the strength properties with microfibrillar angle and cellulose content.³³ This is given by

$$\sigma = -334.005 - 2.830\theta + 12.22W \tag{2}$$

where σ is the fiber strength, θ is the microfibrillar

angle, and *W* is the cellulose content. The strength of the fiber was calculated as 341 MPa; however, the experimental value was found to be 248 MPa.

The oil palm mesocarp fiber shows about 17% elongation at break. Using eq. (1), the microfibrillar angle of the fiber is calculated and found to be 46°. Using eq. (2), the strength of the fiber is predicted. The calculated strength of the fiber was 269 MPa, but the experimental value was very much lower than this. This may be due to the nature of cellular arrangement of the fiber and the effect of traces of oil present on the fiber surface.

CONCLUSIONS

Structure and properties of the two important oil palm fibers, OPEFB fiber and mesocarp fiber, were analyzed. Chemical compositions of the fibers were determined. The major constituents of these fibers were found to be cellulose. Lignin content is comparatively less. OPEFB fiber is more cellulosic than the mesocarp fiber. The oil palm mesocarp fiber contains a higher percentage of ether-soluble and caustic soda-soluble matter. Chemical modification of fibers by alkali treatment, acetylation, and silane treatment was carried out. This is to improve the strength and therefore the reinforcing ability of these fibers. Morphological studies revealed that treatment modified the fiber surface. The fine structural changes of the fibers can be seen from the respective scanning electron micrographs. IR studies give evidence for the chemical modifications that occurred during treatments. Thermal stability and degradation characteristics of the fibers were investigated by TGA and DTA. It was found that alkali and silane treatment increase the thermal stability of the fibers. Fibers are stable up to 300°C without any considerable weight loss.

The silane-treated OPEFB fiber showed maximum tensile strength. Alkali treatment slightly decreased the tensile strength. The Young's modulus of the fiber showed enhancement upon silane and alkali treatments. The strength of the mesocarp fiber is less than that of OPEFB fiber, because of the high cellulose content of OPEFB fiber. Silane treatment increased the strength of the fiber while alkali treatment decreased it. However, the stiffness of the fiber was increased by both alkali and silane treatments. The untreated mesocarp fiber showed very good elongation. Treatment reduced the elongation of the fiber.

Microfibrillar angle and strength of the fibers were theoretically predicted. The theoretical strength of the OPEFB fiber was found to be closer to the experimental value. However, in the case of mesocarp fiber, there was great deviation. Finally, it is important to mention that the properties of oil palm fibers are comparable to other natural fibers and therefore they could be successfully used as a potential reinforcing material for polymer matrices. Several studies are progressing in this direction at this laboratory.

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REFERENCES

- M. S. Sreekala, S. Thomas, and N. R. Neelakantan, J. Polym. Eng., 16, 265 (1977).
- K. G. Satyanarayana, A. G. Kulkarni, and P. K. Rohatgi, J. Sci. Ind. Res., 40, 222 (1981).
- K. G. Satyanarayana, A. G. Kulkarni, and P. K. Rohatgi, J. Sci. Ind. Res., 42, 425 (1983).
- A. N. Shah and S. C. Lakkad, *Fiber Sci. Technol.*, 15, 41 (1981).
- C. Pavithran, P. S. Mukherjee, M. Brahmakumar, and A. D. Damodaran, J. Mater. Sci. Lett., 6, 882 (1987).
- D. Maldas and B. V. Kokta, Polym. Plast. Technol. Eng., 29, 419 (1990).
- 7. B. C. Barkakaty, J. Appl. Polym. Sci., 20, 2921 (1976).
- M. N. C. Martinez, P. J. Herrera-Franco, P. I. Gonzalez-Chi, and M. Aguilar-Vega, J. Appl. Polym. Sci., 43, 749 (1991).
- D. R. Rao and V. B. Gupta, Ind. J. Fiber Tex. Res., 17, 1 (1992).
- C. David, R. Fornasier, W. Lejong, and N. Vanlautem, J. Appl. Polym. Sci., 36, 29 (1988).
- A. C. Mukherjee, S. K. Bandyopadhyay, A. K. Mukhopadhyay, and U. Mukhopadhyay, *Ind. J. Fiber Tex. Res.*, **17**, 80 (1992).
- D. M. Brewis, J. Comyn, J. R. Fowler, D. Briggs, and V. A. Gibson, *Fiber Sci. Technol.*, **12**, 41 (1979).
- D. N. Mahato, B. K. Mathur, and S. Bhattacherjee, Ind. J. Fiber Tex. Res., 20, 202 (1995).
- S. V. Prasad, C. Pavithran, and P. K. Rohatgi, J. Mater. Sci., 18, 1443 (1983).

- S. A. K. Yamini, A. J. Ahmad, J. Kasim, N. M. Nasir, and J. Harun, Proc. Int. Symp. Biocomposites and Blends Based on Jute and Allied Fibers, New Delhi, 1994, p. 135.
- 16. W. A. Curtin, Polym. Comp., 15, 474 (1994).
- M. R. Nedele and M. R. Wisnom, Comp. Sci. Technol., 51, 517 (1994).
- 18. P. K. Jarvela, Fiber Sci. Technol., 20, 83 (1984).
- E. T. N. Bisanda and M. P. Ansell, Comp. Sci. Technol., 41, 165 (1991).
- K. P. Sao and A. K. Jain, Ind. J. Fiber Tex. Res., 20, 185 (1995).
- B. Sikdar, A. K. Mukhopadhyay, and B. C. Mitra, Ind. J. Fiber Tex. Res., 18, 139 (1993).
- 22. O. P. Bahl, R. B. Mathur, and J. L. Dhami, *Polym. Eng. Sci.*, **24**, 455 (1984).
- 23. N. Chand, J. Mater. Sci. Lett., 11, 1051 (1992).
- 24. R. G. Raj, B. V. Kokta, G. Grouleau, and C. Daneault, *Polym. Plast. Technol. Eng.*, **29**, 339 (1990).

- J. George, S. S. Bhagawan, N. Prabhakaran, and S. Thomas, J. Appl. Polym. Sci., 57, 843 (1995).
- 26. K. Joseph, S. Thomas, and C. Pavithran, *Comp. Sci. Technol.*, **53**, 99 (1995).
- 27. V. G. Geethamma, R. Joseph, and S. Thomas, J. Appl. Polym. Sci., 55, 583 (1995).
- S. Varghese, B. Kuriakose, and S. Thomas, J. Appl. Polym. Sci., 53, 1051 (1994).
- K. C. Manikandan Nair, S. M. Diwan, and S. Thomas, J. Appl. Polym. Sci., 60, 1483 (1996).
- D. S. Varma, M. Varma, and I. K. Varma, *Thermochim. Acta*, **108**, 199 (1986).
- S. C. Shah, P. K. Ray, S. N. Pandey, and K. Goswami, J. Polym. Sci., 42, 2767 (1991).
- 32. P. S. Mukherjee and K. G. Satyanarayana, J. Mater. Sci., 21, 4162 (1986).
- K. G. Satyanarayana, C. K. S. Pillai, K. Sukumaran, S. G. K. Pillai, P. K. Rohatgi, and K. Vijayan, J. Mater. Sci., 17, 2453 (1982).