J. Indian Chem. Soc.,
Vol. 88, April 2011, pp. 583-585

NOTE

FTIR, XRD and SEM studies on sugar palm (*Borassus flabellifer* L.) seed fibre

L. K. Navak*. P. Moiumder and S. K. Bhaduri

National Institute of Research on Jute & Allied Fibre Technology, 12, Regent Park, Kolkata-700 040, India

E-mail : laxmikanta8495@rediffmail.com *Fax* : 91-33-24712583

Manuscript received 01 June 2010, revised 31 July 2010, accepted 16 August 2010

Abstract : Palm seed fibre, a non-conventional lignocellulosic fibre extracted from the seed of sugar palm (*Borassus flabellifer* L.) was characterized by spectral (FTIR), scanning electron microscopy (SEM) and X-ray diffraction analyses (XRD). The infra-red spectra of defatted palm seed fibre showed peaks in the region as observed for other lignocellulosic fibres like jute.

Keywords : Palm seed fibre, XRD, SEM, FTIR, bleaching.

Introduction

The Palmyra is a tropical palm tree that grows indigenously in India, Srilanka, Malaysia, Philippines, Indonesia and many parts of East Africa.

Palm tree yields about 200 fruits in a season, which are large and fibrous and usually contain three seeds in each fruit. When the fruit is tender, the seeds are present in a soft, sweet and gelatinous pulp and have delicious taste and after it ripens the pulpy tissue beneath the outer skin of the fruit become sweet and edible. The seeds after extraction of the pulpy juice are thrown away as agro-waste that contains about 15 g fibre attached to the surface of each seed. Considering the potential availability of the fibre in large quantity, the present work was carried out to explore into the characteristics of palm seed fibre. The work is of practical importance for comparison of characteristics with other natural fibres. It can be utilized for industrial purpose to produce different natural fibre based products.

Results and discussion

FTIR analysis :

The assignment of infra-red spectra of defatted, H_2O_2 bleached and 5% alkali treated palm seed fibre are recorded in Table 1. The infrared spectra of defatted fibre showed peaks characteristics of lignocellulosic fibres like jute[1]. The strong broad band in the region 3410-3414 cm^{-1} is due to hydrogen bonded O-H stretching vibration[2]. The medium density band in the region 2919-2921 cm^{-1} is assigned to C-H stretching frequency in methyl and methylene group. The sharp strong peak at 1738 cm^{-1} in defatted fibre is assigned to C=O stretching of carboxyl and acetyl groups in hemicellulose of the fibre

Table 1. FTIR spectra of defatted, alkali treated and bleached palm seed fibre

	Absorbance (cm^{-1})		
Functional group	Deatted	5% Alkali treated	H_2O_2 bleached
Hydrogen bonded O-H stretching	3414(s)	3413(s)	3410(s)
C-H stretching	2921(s)	2919(s)	2920(s)
C=O stretching	1738(s)	1738(sh)	1738(sh)
Adsorbed water vibration	1641(w)	1641(w)	1641(w)
Aromatic C=C	1598(m)	1598(m)	1598(m)
stretching	1502(w)	1502(w)	1502(w)
COO-/O-H	1462(w)	1462(w)	1462(w)
in plane blending			
CH_2 bending	1382(w)	1381(w)	1382(w)
O-H in plane	1325(w)	1332(w)	1332(w)
bending			
C-O stretching	1251(m)	1251(sh)	1251(sh)
C-O-C stretch	1164(w)	1164(w)	1165(w)
antisymmetric bridge			
C-O stretching/	1036(s)	1034(s)	1039(w)
C-C stretching			
β-Glucosidic bond	897(w)	896(w)	896(w)

Relative intensities . s = strong, m = medium, w = weak, sh = shoulder.

This peak is reduced to a shoulder in alkali treated and bleached fibre samples due to partial loss of hemicellulose containing carboxyl and acetyl group. The peak at 1641 cm^{-1} is attributed to adsorbed water molecules in the non-crystalline region of fibre. The adsorption bands at 1598 cm^{-1} and 1502 cm^{-1} indicate aromatic skeletal ring vibration of the lignin component of the fibre[3]. The O-H in plane bending is assigned at 1462 cm^{-1} and in the region 1325–1331 cm^{-1} in the spectra. The peak for CH_2 bending appears in the region 1426–1427 cm^{-1} and 1381–1382 cm^{-1}. The medium density peak of C-O stretching mode at 1251 cm^{-1} in the defatted fibre sample reduces to a shoulder in the alkali treated and bleached fibre samples. The C-O-C/C-O stretching vibration in the region 1164–1165 cm^{-1} in the spectra of defatted and treated fibre samples are characteristics of lignocellulosic material. The broad band in the region 1034–1039 cm^{-1} in the spectra of all the fibre samples is assigned to C-O/C-C stretching mode while the peak in the region 896–897 cm^{-1} indicates a glycosidic bond of cellulose and hemicellulose in the fibre. Due to very low fat and wax (0.78%) content of palm seed fibre, there is no significant change in spectral pattern of raw and defatted fibre.

SEM analysis :

The electron micrographs of raw, defatted, bleached and alkali treated palm seed fibre samples are shown in Figs. 1(a) and 1(b) and these show the deposits and debris on the raw fibre surface obscuring the individual cells. Defatting removes the amorphous deposits on the fibre cells and make them visible (Fig. 1(b)). Alkali treatment and bleaching further removes the carbohydrate and lignin fraction to some extent and the fine structure of the fibrils are distinctly visible.



Fig. 1a. SEM microphotograph of raw palm seed fibre.

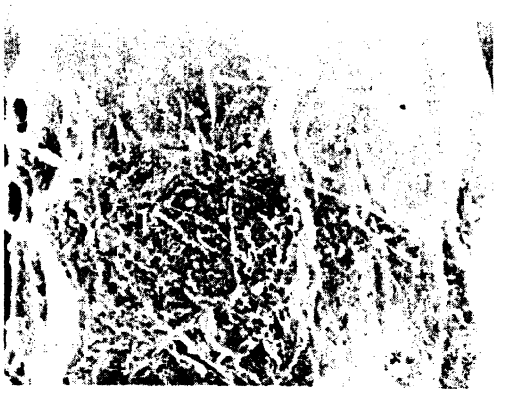

Fig. 1b. SEM microphotograph of defatted palm seed fibre.

The degree of crystallinity of defatted palm seed fibre is recorded and compared with that of other natural fibres[4]. In Table 2, it was observed that palm seed fibre is less crystalline (d.c. 50.9) than other lignocellulosic fibres. This is due to the fact that palm seed fibre contains less cellulose (50.1%) and a high amount of amorphous carbohydrate content (pentosan 31.3%) which contribute to less crystallinity as compared to other natural fibre[5]. The degree of crystallinity of raw pam seed fibre is less than that of the de-fatted palm seed fibre i.e. 50.9. The lower value for raw fibre is due to the fact that the amorphous fraction of fat and wax are removed during defatting. It can be explained from SEM analysis that, the photomicrograph (Fig. 1(a)) of raw fibre shows the deposits and debris on the raw fibre surface obscuring the individual cells which is removed by solvent extraction process for defatted fibre. Hence, its evident that palm seed fibre (both raw and defatted) has degree of crystallinity less than that of other natural fibres.

Table 2. Degree of crystallinity of defatted palm seed fibre and other natural fibres

Fibre	Degree of crystallinity
Palm seed fibre	50.9
Khimp	60.0
Jute	59.5
Sunhemp	57.7
Pineapple	52.3
Cotton	67.6

Experimental

Ripened fruits were collected from the local area. After removal of the pulpy tissue from the ripe fruits, the seeds were washed clean in cold water and sun dried. Fibre was collected through shearing the surface of the dried seed by a sharp blade keeping the blade as close as possible to the surface of the seed, so that fibres of maximum length were obtained.

Raw palm seed fibre contains only 0.78% fat and wax which was removed during defatting. Defatted fibre being taken for bleaching with hydrogen peroxide and treatment with sodium hydroxide solution to get bleached and alkali treated fibre. The fat content after bleaching and alkali treatment is insignificant. The methods followed for defatting, bleaching and alkali treatment of palm seed fibre are described below.

The raw fibre was defatted with alcohol-benzene (1 : 2 v/v) mixture for 6 h in soxhlet apparatus and then dried in air.

The defatted palm seed fibre sample was treated with 2 volume of hydrogen peroxide, 1.1% of sodium silicate and 0.5% sodium phosphate for 2 h at the temperature of 80–85 °C.

Defatted fibre was treated with 5% (w/v) sodium hydroxide solution in material to liquor ratio 1 : 10 for one hour at room temperature and then washed with distilled water. Then the sample was immersed in 10% acetic acid solution for 10 min (liquor ratio 1 : 10). Finally the sample was washed with water and dried in air.

FTIR study :

The infra-red spectra of defatted, bleached and alkali treated palm seed fibre samples were recorded in a Jasco 4200 FTIR spectrophotometer with dry KBr pellet of the fibre samples.

The surface topology of raw, defatted, bleached and alkali treated palm seed fibre samples were investigated by scanning electron microscopy. Individual filament samples of palm seed fibre were mounted on standard specimen holders about 1 cm in diameter, durafix being used as adhesive. The filaments were then coated *in vacuo* with gold coating of about 20 nm thickness in Edward Sputter Coater. The coated samples were examined in a scanning electron microscope (Hitachi, S-430) at an accelerating potential of 20 KV. Representative micrographs were taken at a magnification X 750 to study the changes in surface morphological features with different treatments in comparison to that of raw fibre.

The degree of crystallinity of defatted palm seed fibre was determined by X-ray diffractometry technique using a Philips X-ray Diffractometer (Model PW 1710) and compared with the crystallinity values of cotton, jute, sunhemp, khimp and pineapple leaf fibre. Nickel filtered Cu Kα radiation (1.54 A) from X-ray tube run at 40 KV and 30 mA was used for the experiment.

Acknowledgement

Authors are grateful to Director, NIRJAFT for his constant encouragement.

References

1. K. P. Sao, M. D. Mathew, A. K. Jain and P. K. Roy, *Cellulose Chemistry and Technology*, 1987, **21**, 17.
2. M. Tsuboi, *Journal of Polymer Science*, 1957, **25**, 159.
3. K. V. Sarkanen, H. H. Chang and G. G. Allan, *TAPPI*, 1967, **50**, 583.
4. S. K. Kundu, P. Mojumder, S. K. Bhaduri and B. K. Das, 2005, *Indian Journal of Fibre and Textile Research*, 2005, **30**, 153.
5. S. Ghosh and N. Geo Paul, *Indian Journal of Textile Research*, 1980, **5**, 103.