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## Chemical modification of coir, empty fruit bunch and palm kernel fibres for polymer reinforcement

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### A B S T R A C T

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*Reinforcing polymers with natural fibres to form materials with improved strength has been globally accepted as one of the environmental friendly ways of harmonizing the present technology with future engineering material property required. The major limitation to reinforcement of hydrophobic polymer with hydrophilic natural fibre is that of poor interfacial bonding. Solution to the problem arising from this technology was examined by using mercerization, alkaline bleaching and acetylation surface modification technology, to incorporate water resistance to the hydrophilic coir (CCF), empty fruit bunch (EFB) and palm kernel fibres (PKF). The moisture content (MC) of EFB is higher than that of CCF but lower than that of PKF. Up to about 70 % reduction was observed in MC of the fibres as a result of chemical modification. Acetylation method of fibre modification was the one that gave the best hydrophobic nature to the fibres, followed by alkaline bleaching method of modification. The reduction in hydrophilic nature of these natural fibres might make them compatible with hydrophobic matrices and promotes interfacial adhesion between them.*

### 1. Introduction

Technology of converting waste to wealth is another global path way to economic growth. Abandoned natural fibres from post harvest are environment pollutants. Presently, these wastes have found a place in the reinforced polymer industry, where value has been added to them (Bei, 2004). Conventional polymer materials lack some of the desired properties for modern day technology and these properties are found in fibre reinforced polymer. Reinforcing polymer matrix with fibres is the only bridge for marrying the need of the present technology with material property required (Mathews and Rawlings, 1999). A lot of work have been done using different plant fibres but only few of these researchers have considered coconut coir (CCF) and empty fruit bunch fibres (EFB) and very few have considered palm kernel fibre (PKF) as reinforcement agents in polymer composite reinforcement.

The CCF, EFB and PKF fibres belong to the class of fruit fibres and have been proved to possess good compatibility with resin like polyisocyanate to form and reinforce polyurethane composite as a result of their constituents like cellulose and lignin with hydroxyl functional group that is highly reactive with

isocyanate group, especially when chemically modified (Mizanui and Mubarak, 2007; Myrtha et al., 2008).

The major problem encountered when lignocellulosic (natural fibres) materials are to be used to reinforce thermoplastic / thermosetting polymer is incompatibility between hydrophilic natural fibres and hydrophobic thermoplastic /thermosetting polymer. Quality interfacial adhesion between natural fibres and polymer matrix can be achieved by reducing hydrophilic nature of the natural fibres through chemical modification of the natural fibres, grafting of the polymer on to the natural fibres or the use of coupling agents (Khalil et al., 2001).

Chemical coupling agents are molecules possessing two functional groups of the same or different kinds. One is to react with hydroxyl functional group in the fibre and the other to react with functional group in the matrix (Bledzki et al., 2008). Grafting is formation of a more hydrophobic composite through compounding of the hydrophilic natural fibres and hydrophobic matrix. Chemical modification process uses chemical compounds that react with hydroxyl functional group in the amorphous constituents of the fibre, i.e lignin, hemicellulose and amorphous cellulose and converts hydrophilic to hydrophobic fibre (Sreekala and Thomas, 2003; Seena et al., 2005; Mishra and Luyt, 2008; Mishra et al., 2001).

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These treatments reduce hydrophilic nature of the natural fibres, stabilize the cell wall against moisture, improve dimensional stability, environmental degradation and compatibility with hydrophobic polymer (Bledzki *et al.*, 2008). Bledzki and Gassan (1999) investigated the role of chemical treatment on hydrophilic nature of jute and pine fibres and reported jute fibre having about 50 % hydrophilic reduction and pine fibre having up to 65 % hydrophilic reduction.

This research work was designed to study the effect of mercerization, alkaline bleaching and acetylation on the hydrophilic nature of CCF, EFB and PKF fibres.

## 2. Materials and Methods

### 2.1. Materials

The coir fibres used were obtained from Cassava Avenue, FUTA road Akure. The empty fruit bunch and palm kernel fibres were obtained from FUTA research farm, Akure, Nigeria. Reagents used were of AnalaR grade.

### 2.2. Methods

#### 2.2.1. Sample preparation and pretreatment

Coir (CCF), empty fruit bunch (EFB) and palm kernel fibres (PKF) (about 0.500 g each) were pretreated with 2 % detergent solution in liquor ratio 1:200 at 80°C for 1 h, washed with distilled water and finally oven dry at 105°C to a constant weight. The physically treated CCF, EFB and PKF fibers were divided into four parts each. The first portion was treated with NaOH (5 %) solution (Ray *et al.*, 2001), the second portion treated with alkaline peroxide solution (Sun *et al.*, 2004; Habibi *et al.*, 2008) and the third portion treated with 10 % acetic anhydride with a drop of conc. H<sub>2</sub>SO<sub>4</sub> as catalyst (Bledzki *et al.*, 2008). The procedures adopted for various surface modifications were described below.

#### 2.2.2. Mercerization

The natural fibres (about 0.100 g each) were treated with NaOH (5 %) solution for 2 h at room temperature, using liquor ratio 1:200. At the end of treatment time, the fibres were washed with distilled water to obtain alkaline free fibres. The fibres were then oven dried at 105°C for 2 h before finally dried to a constant weight at 15 min interval to obtain cellulose II fibres of a known mass (Maya and Rajesh, 2008).

#### 2.2.3. Alkaline bleaching

The natural fibres (about 0.100 g each) were soaked in a bleaching solution containing 2 % H<sub>2</sub>O<sub>2</sub> in NaOH (25 %) in liquor ratio 1:200 at room temperature for 2 h of continual stirring. At the end of treatment time the bleached fibres were washed with distilled water to free them from bleaching solution, oven dried at

and unmodified) were obtained by using ground fibers mixed with potassium bromide at ratio 1:100 in a mortar pestle. The mixture was then taken in a dice of specific dimension to form pellet by pressing with a hand press machine, placed on the sample holder of the IR spectrometer (Perkin Elmer) operated at spectral range 4000 - 350 cm<sup>-1</sup>.

#### 2.2.7. Scanning electron microscopy (SEM)

The scanning electron micrographs of the samples were taken in an XL 20 Philips SEM. The surface of the fibres was studied with the microscope operated at 10.0 kV. The samples were coated with a 10 nm thick layer of gold.

## 3. Results and Discussion

### 3.1. Moisture content and fibre hydrophilic nature

The moisture content (MC) and reduction in hydrophilic nature (J) of each of unmodified, mercerized, alkaline bleached and acetylated CCF, EFB and PKF fibres are summarized in Table 1. The moisture content of the fibre samples was found to be in order CCF < EFB < PKF and effect of the chemical surface modifications on the fibres hydrophilic nature was in order acetylation > alkaline bleaching > mercerization. These observations show that PKF has the highest inherent water retention property, followed by EFB and CCF has the least. Acetylated fibres have the lowest moisture content, suggesting acetylation reaction to have highest fibre conversion from hydrophilic nature to hydrophobic nature, followed by alkaline bleaching, while mercerization reaction gave the least conversion (Table 1). These observations are in agreement with the findings of Suradi *et al.*, (2009) and Bei, (2004).

Equally their compatibility and interfacial adhesion with hydrophobic matrices are expected to be in this order. These are in agreement with Khalil *et al.*, (2001), Suradi *et al.*, (2009) and Bhat *et al.*, (2011).

### 3.2. FT-IR spectroscopy

The IR spectra of the lignocellulosic CCF, EFB and PKF fibres that confirmed their chemical modification are similar to one another. Therefore, spectrum of CCF fibres (Figure 1A) before modification is similar to those of EFB and PKF fibres and has the same trend of absorption shift after mercerization (Figure 1B), alkaline bleaching (Figure 1C) and acetylation reaction (Figure 1D). The absorbance peaks at 3402 - 3435 cm<sup>-1</sup> were attributed to the stretching of -OH group in cellulose, lignin and hemicellulose and absorbed water molecule in all unmodified and chemically modified fibres. The major observation noticed in the spectrum in Figure 1A was medium intensity bands in unmodified CCF, EFB and PKF fibres due to C=O stretching vibration of lignin and hemicellulose that appeared at 1726.42 cm<sup>-1</sup>, 1733.91 cm<sup>-1</sup> and

1733.00  $\text{cm}^{-1}$  respectively. These bands disappeared in mercerized (Figure 1B) and alkaline bleached (Figure 1C) fibres.

This observation agreed with the findings made by Bhat *et al.*, (2011). However, strong and intense bands at 1731.57  $\text{cm}^{-1}$ , 1735.82  $\text{cm}^{-1}$  and 1729.00  $\text{cm}^{-1}$  were observed in acetylated CCF, EFB and PKF fibres respectively (Figure 1D). This can be assigned to the ester carbonyl functionality of acetic anhydride according to Khalil *et al.*, (2001). Other functional groups assignments are presented in Table 2.

### 3.3 Scanning electron microscopy

Morphological examinations of the fibre samples were done using scanning electron microscope (SEM) on the surface, before and after modifications (Plates 1 – 4) to further proof the chemical modifications of the fibre. Unmodified CCF, EFB and PKF fibres have rough surfaces as shown in Plate 1. The surface roughness of the untreated fibres was due to the presence of residual silica nodules, hemicellulose, lignin and other impurities, according to the findings of Suradi *et al.*, (2009).

The mercerized CCF, EFB and PKF fibres have most of their impurities and residual hemicellulose removed. They appear cleaner with wider pore sizes and clearly seen silica nodules (Plate 2). This is as a result of alkaline reaction with cementing materials of the fibres and splitting the fibers into finer filaments according to Bhat *et al.*, (2011)

In the case of alkaline bleached CCF, EFB and PKF fibres, the fibres have not only their lignin component degraded but also most of their hemicellulose removed with formation of widest pore sizes on the surfaces of the fibres (Plate 3). These observations agreed with the observation of Suradi *et al.*, (2009).

After treatment with acetic anhydride, the surfaces of the CCF, EFB and PKF fibres appear smoother with a little increase in surface pore sizes (Plate 4). This is as a result of conversion of hydroxyl group on the fibres surface to hydrophobic acetyl group (Khalil *et al.*, 2001).

## 4. Conclusion

The chemical modifications reduced the problem of incompatibility between hydrophilic fibres and hydrophobic matrices through conversion of the natural fibres to less hydrophilic materials. Acetylation reaction was the chemical modification that gave the best conversion of the natural fibres to less hydrophilic materials, followed by alkaline bleaching and mercerization reaction. The increase in porosity and pore sizes of the chemically modified natural fibres might improve physical and mechanical interlocking leading to better interfacial bonding between fibres and matrices.

A  
T  $\epsilon = \left(1 - \frac{\rho_b}{\rho_t}\right) \times 100$  : Bello Kamilu A. (Ph.D research scholar) and Ibrahim Razali of the Department of Manufacturing and Materials Engineering, the International Islamic University, Malaysia for using scanning electron microscope in their laboratory for the SEM the analysis. We thank all staff of the multidisciplinary laboratory, University of Ibadan for running FT-IR analysis of the fibre samples.

## References

- Adebayo, G.B., and Elelu, S.A. (2011). Physico-chemical characterization of adsorbents prepared from different parts of physic nut plant (*Jatropha curcas*). *Proceeding of Chemical Society of Nigeria Conference held between 20<sup>th</sup> and 23<sup>rd</sup> September 2011 at University of Ilorin, Ilorin Nigeria*, PHY 078-084.
- Alinnor, I.J., and Madu, P. (2007). Effect of urea and onion (*Allium cepa*) extract on the combustion characteristics of woods. *Journal of Chemical Society of Nigeria*, 32(2), 226-233.
- Bei, W. (2004). Pre-treatment of flex fibre for use in rotationally moulded Biocomposites. *Unpublished M.Sc. Thesis. University of Saskatchewan, Department of Agricultural and Bioresources Engineering*, Saskatchewan, Canada 92pp.
- Bhat, I., Abdul Khalil, H.P.S., Ismail. H., and Alshammari, T. (2011). Morphological, spectroscopic and thermal properties of alkali-treated and chemical modified oil palm empty fruit bunch and oil palm frond fibres: A comparative study. *Bioresources*, 6(4): 4673-4685.
- Bledzki, A.K. and Gassan, J. (1999). Composites reinforcement with cellulose based fibres. *Progress in Polymer Science*, 24: 221-274.
- Bledzki, A.K., Mamun, A.A., Lucka-Gabor, M. and Gutowski V.S. (2008). The effect of acetylation on properties of flax fibre and its polypropylene composites. *Express Polymer Letters*, 2(6): 413-422.
- Khalil H.P.S.A, Ismail B., Rozman H.D. and Ahmad M.N. (2001). The effect of acetylation on interfacial shear strength between plant fibres and various matrices. *European Polymer Journal*, 37: 1037-1045.
- Mishra, S.B. and Luyt, A.S. (2008). Effect of organic peroxides on the morphological, thermal and tensile properties of EVA-organoclay nanocomposites. *Express Polymer Letters*, 2: 256-264.

Mishra, S., Misra, M., Tripathy S.S., Nayak, S.K. and Mohanty, A.K. (2001). Graft copolymerization of acrylonitrile on chemically modified sisal fibres. *Macromolecular Materials and Engineering*, 286: 107–113.

Mizanui, M.R. and Mubarak, A.K. (2007). Surface treatment of coir fibre and its influence on the fibres physicochemical properties modification and characterization of natural fibre reinforced plastic composite. *Journal of Polymer Science*, 41: 1471-1485.

Myrtha, K., Holia, O., Abdullah, D.A.H. and Anung, S. (2008). Effect of oil palm empty fruit bunch fibre on the physical and mechanical properties of fibre glass reinforced polyester resin. *Journal of Biological Science*, 8(1): 101-106.

Rakesh, K., Sangeeta, O. and Aparna, S. (2011). Chemical modification of natural fibre for composite material. *Der Chemical Snica*, 2(4): 219-228.

Seena, J., Koshy, P. and Thomas, S. (2005). The role of interfacial interactions on the mechanical properties of banana fibre reinforced phenol formaldehyde composites. *Composite Interfaces*, 12: 581–600.

Sreekala, M.S. and Thomas, S. (2003). Effect of fibre surface modification on water-sorption characteristics of oil palm fibres. *Composites Science and Technology*, 63: 861–869.

Suradi, S.S., Yunus, R.M., Beg, M.D.H. and Yusof, Z.A.M. (2009). Influence of pre-treatment on the properties of lignocellulose based biocomposite. *National Conference on Postgraduate Research held on 1st October at UMP Conference Hall, Universiti Malaysia Pahang, Malaysia*, 67-78.

Table 1: Characterization of modified and unmodified CCF, EFB and PKF fibres

<b>Fibre</b>	<b>MC (%)</b>	<b>J (%)</b>	<b>Fibre mass(g)</b>
UCCF	9.66±0.24	-	0.1023
MCCF	6.02±0.08	37.68	0.1030
BCCF	3.98±0.08	58.80	0.1000
ACCF	3.15±0.08	67.39	0.1005
UEFB	12.09±0.40	-	0.1026
MEFB	7.43±0.08	38.54	0.1009
BEFB	4.66±0.03	61.46	0.1008
AEFB	3.77±0.08	68.82	0.1007
UPKF	15.44±0.32	-	0.1004
MPKF	8.93±0.08	42.16	0.1079
BPKF	5.90±0.03	61.79	0.1004
APKF	4.18±0.08	72.93	0.1016

Key: CCF = coconut coir fibre  
 EFB = empty fruit bunch fibre  
 PK = palm kernel fibre  
 MC = moisture content  
 J = hydrophilic nature

Table 2: The peak assignments of CCF, EFB and PKF natural fibres #  
for FT-IR analysis

Wave number (cm-1)				Groups
Unmodified	Mercerized	Alkaline bleached	Acetylated	
Below 1000	Below 1000	Below 1000	Below 1000	=C-H Strong bend
1050-1085	1050-1085	1050-1085	1050-1085	Alcoholic C-O stretch
1310-1340	1310-1340	1310-1340	1310-1340	-CH <sub>2</sub> from holocellulose
1370-1390	1370-1390	1370-1390	1370-1390	-CH from cellulose & hemicellulose
1410-1470	1410-1470	1410-1470	1410-1470	-CH <sub>2</sub> from cellulose
1500-1510	1500-1510	1500-1510	1500-1510	-C-C- Stretching vibration in ring from lignin
1600-1680	1600-1680	1600-1680	1600-1680	-C=C- stretching vibration in lignin
1720-1740	-	-	1720-1740	-C=O from holocellulose and lignin
2360-2370	2360-2370	2360-2370	2360-2370	
2900-3000 and conjugated bond	2900-3000	2900-3000	2900-3000	-C=C- Stretch in alkene, aromatic ring
3200-3500	3200-3500	3200-3500	3200-3500	Broad band of the -OH group

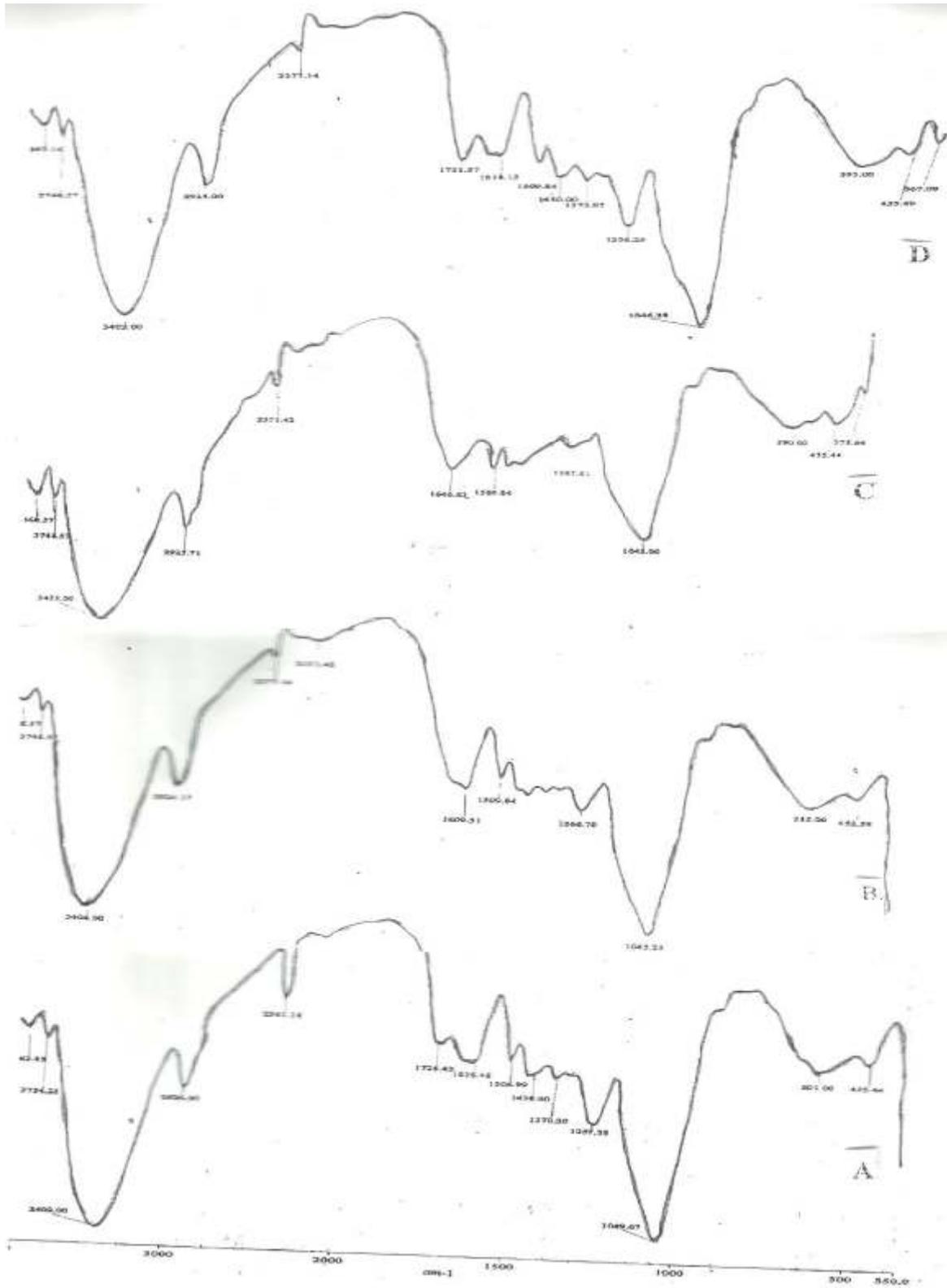


Figure1: IR spectra of unmodified (A), mercerized (B), alkaline bleached (C) and acetylated (D) coir fibres

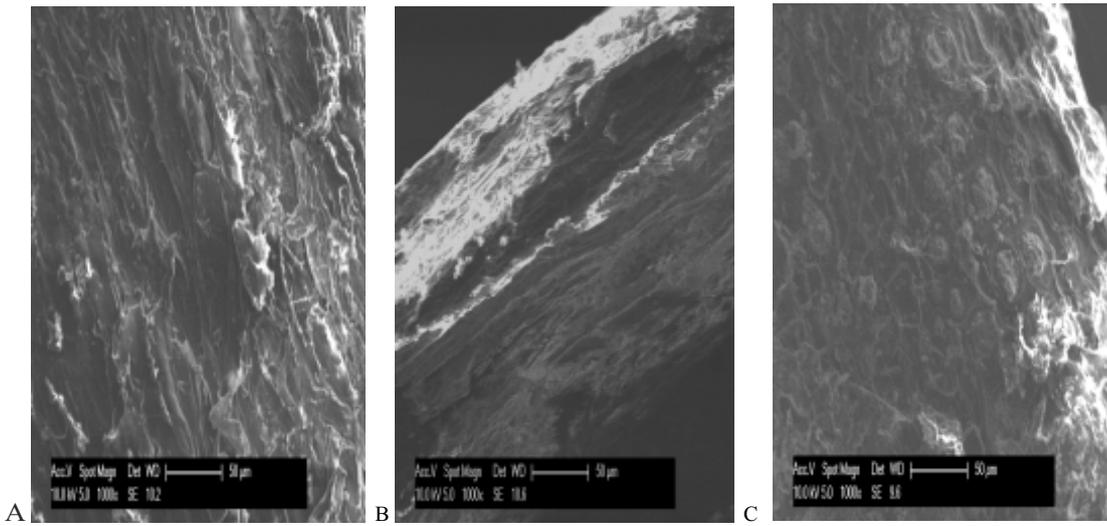


Plate 1: A, B and C are SEM images of UCCF, UEFB and UPKF fibres respectively

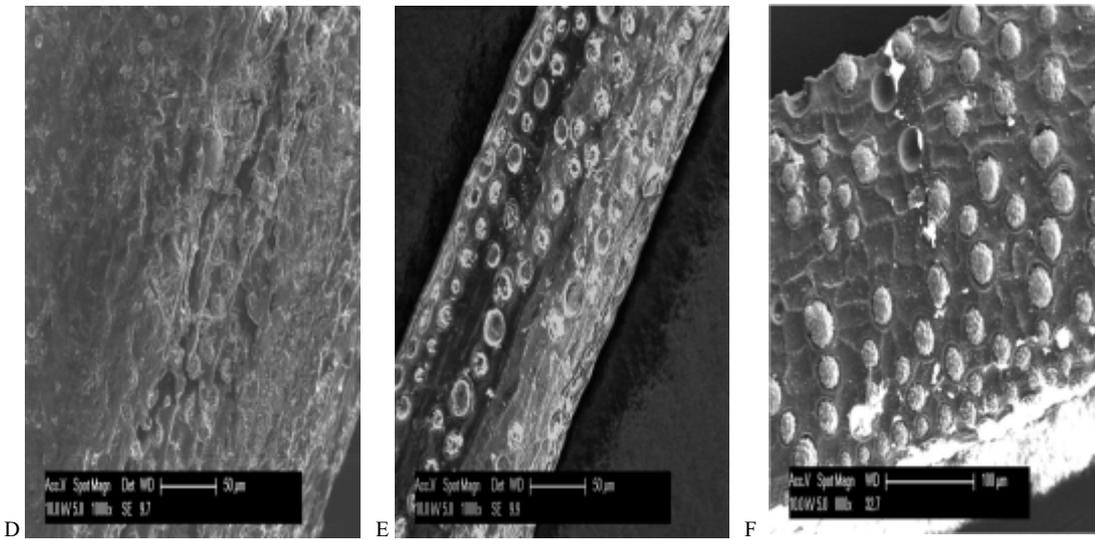
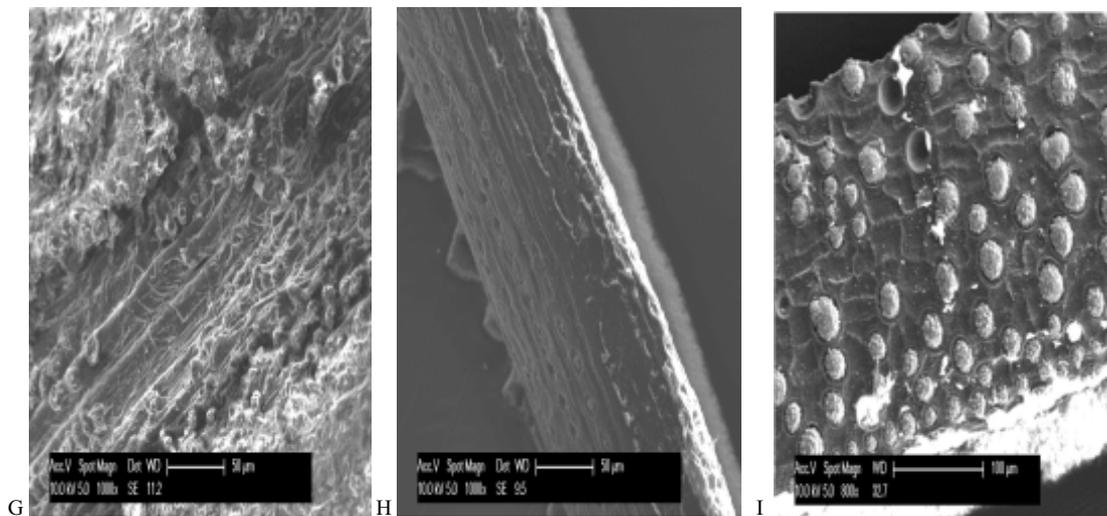


Plate 2: D, E and F are SEM images of MCCF, MEFB and MPKF fibres respectively



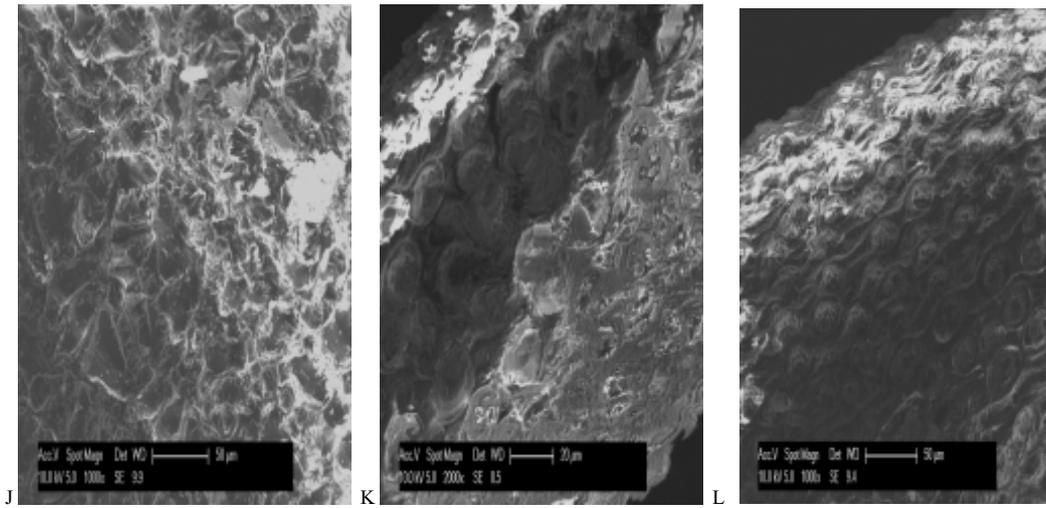


Plate 4: J, K and L are SEM images of ACCF, ABEFB and APKF fibres respectively

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