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Structural Characteristics of Batch Processed Agro-Waste Fibres

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***Abstract*—**The characterisation of agro-wastes fibres for composite applications from Nigeria using X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM) has been done. Fibres extracted from groundnut shell, coconut husk, rice husk, palm fruit bunch and palm fruit stalk are processed using two novel cellulose fibre production methods developed by the authors. Cellulose apparent crystallinity calculated using the deconvolution of the diffractometer trace shows that the amorphous portion of cellulose was permeable to hydrolysis yielding high crystallinity after treatment. All diffratograms show typical cellulose structure with well-defined 110, 200 and 040 peaks. Palm fruit fibres had the highest 200 crystalline cellulose peaks compared to others and it is an indication of rich cellulose content. Surface examination of the resulting fibres using SEM indicates the presence of regular cellulose network structure with some agglomerated laminated layer of thin leaves of cellulose microfibrils. The surfaces were relatively smooth indicating the removal of hemicellulose, lignin and pectin.

***Keywords*—**X-ray diffraction, SEM, cellulose, deconvolution, crystallinity.

I. Introduction

THE world needs an assortment of material of forest based products for an environmentally friendly and sustainable development. Their successful application will decrease dependence on non-renewable fossil (geologic deposits) resources and improve the economics of the forestry/wood-based industry. This area of research is of great importance now as there are global indications of steady depletion of fossil resources which serve as energy sources for most technologies. It is therefore imperative to develop alternative solutions with renewable resources.

Natural fibres have attractive technical, economic and environmental advantages. These include low cost, low abrasion, medium mechanical and thermal properties, high disposability, high renewability and biodegradability, low energy consumption and generation of neutral carbon dioxide [1]-[4].

Several studies carried out on fibres with high consumption rate include flax [4], [5], jute [6], [7] and sisal [8], [9]. Fibrous agricultural residues of annual plants such as pineapple [10] and banana [11], [12] have been identified as important source of natural fibres, especially in developing countries [13], [14]. These fibres have many attractive features for the composite industry. On a weight basis, the fibres have comparable strengths and in some cases even higher stiffness than E-glass fibres [15]–[17]. The hollow tubular or cellular nature of the fibres confers lightweight [17] and improved acoustic and thermal insulation performance [18]. In addition, natural organic fibres from renewable natural resources offer the potential to act as biodegradable reinforcing materials alternative to the use of glass or carbon fibre and inorganic fillers.

Over the years, there has been renewed interest in the use of naturalfibres from plants to replace glass fibre in composite applications. A great deal of work has been published on the mechanical responses of individual plant fibres [19], [20] and interaction of fibres with various thermosets and thermoplastics [19]–[24].

Earlier studies by some authors revealed that quality fibres suitable as reinforcement in composites can be produced from maize stems and hulls, rye straw, rice straw and husk, banana bundles, switch grass, wheat straw, sorghum stems and leaves, pineapple, coconut, nettle, velvet, empty palm fruit bunch, hemp, flax, kenaf, cotton, soy hulls and straw, milk weed stems, hop stems and bamboo. However, natural fibres have some major draw backs which include poor compatibility with non-polar polymers, poor moisture resistance, and inconsistent or variable properties. Plant fibres are usually 'pulped' and refined to liberate fibrils cellulose that are responsible for its inherent strength and performance [25] using different methods. The fibre is extracted from retted tissue by hand carding (passing the fibres through differential moving surface) or by various mechanical processes [26]–[28]. The fibre produced is in bundle form of individual cells with varying quality. These variations are often due to differences in the fibre bundle structure caused by the retting process (partial decay of stems in the field, carried out to make fibre extraction easier), [24]. Various techniques have been developed to assess and improve the quality of the fibres [29], [30]. The high quantity of non-cellulosic components in natural fibres (hemicelluloses, lignin, pectin and waxes) and impurities negatively influence further fibre processing and fibre properties. The predominant task in preparing natural fibres for further processing is to remove these non-cellulosic components in order to improve fibre properties without damage to the fibre cellulose [31].

Individual methods used for extracting fibres include steam

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explosion, alkaline treatment, thermal alkaline degradation, shear alkaline peroxidation, silane treatment, acid hydrolysis and high pressure homogenization. These methods yield improved fibre mechanical and chemical properties. The adhesion property is, however, only marginally improved. As mentioned in our previous study [32] the adhesion property of fibres depend basically on the amount of non-cellulosic materials present in the fibre structure. A well designed process that can eliminate/reduce the presence of non-cellulosic materials without damage to the fibre is thus necessary. Two new methods based on the chemistry of cellulose have been designed by the authors for studies on the removal of non-cellulosic materials from five agro-waste resources (i.e. Palm fruit stalk, palm fruit bunch, rice husk, groundnut shell, coconut shell) which are major agricultural waste in Nigeria. In the present investigation, the morphology and crystalline structure of fibres from these plant wastes after treatment using newly designed methods are studied.

Surface examination and crystallinity of fibres are of importance as they help to measure the level of non-cellulosic content that the treatment has been able to remove from the structure. It also helps to examine the internal structure of the material and gives insight into a new system of modification that will make the fibres useful in some applications such as polymer composites. The observations at different structural level have been generated from a combination of SEM and X-ray diffraction. By this analysis, the major factor(s) responsible for the structural properties are identified.

II. Experimental Methodology

A. Materials

Palm fruit stalk, palm fruit bunch, rice husk, groundnut shell and coconut shell, which are agricultural wastes, are used for the study. These wastes materials are collected from farm fields in Ekpoma, a city located in southern Nigeria. The unwanted parts are removed and the fibrous sheaths dried in air for about two weeks. Each agro-waste material is processed using two methods of treatment designated as Route A and Route B.

B. Route A

The plant wastes are collected, washed, dried in sunlight and then cut into small pieces. The cut stems or straws are ground to pass a 10 mm screen in a mechanical crusher. The resulting fibres are fed into an autoclave, saturated steam is admitted into its chamber, the valve is closed and the temperature rose to 175 at 1bar. When the material attains the reaction temperature the valve is released to cause rapid pressure reduction resulting in explosive decomposition of the fibre. The fibres are then soaked in 2% solution of NaOH overnight, washed in water and neutralized in ethanoic acid to remove the alkali. The alkaline treated fibres are further soaked in 8% solution of hydrogen peroxide overnight (this is the bleached pulp stage), and again rinsed with distilled water. Further treatment of the resulting fibres are done with equal amount of 10% (w/w) nitric acid and 10% (w/w) chromic acid by dipping in equal amount of solution and raising the temperature to 60 for 15 minutes to initiate the reaction and then allowed to stay for 24 hours. The treated fibres are washed in water after this stage, centrifuged in ethanol and dried.

C. Route B

The study plant wastes are collected, washed, dried in sunlight and then cut into small pieces. The cut stems or straws are ground to pass 10 mm screen in a mechanical crusher. These are de-waxed using benzene – ethanol treatment in a Soxhlet extractor, washed to neutrality and oven dried at 45. The resulting fibres are fed into an autoclave. Saturated steam is admitted into its chamber, the valve is closed and the temperature raised to 175 at 1bar. When the material attains the reaction temperature the valve is released to cause rapid pressure reduction giving rise to explosive decomposition of the fibre and the resulting fibres are oven dried. Fibres from the previous step are treated with crude cellulase (enzyme for the hydrolysis process) by suspending in 50 mL of 50 mM sodium (Na) acetate buffer pH 4.8 in a conical flask. The hydrolysis is performed at 50 in a shaking bath at 15 rpm for two hours. The hydrolyzed fibres are soaked in 2% solution of NaOH overnight to remove some cementing ingredients like hemicellulose and pectin through beta elimination. These alkaline treated fibres are neutralized by washing in distilled water and bleached by soaking in 8 % solution of hydrogen peroxide overnight. The bleached pulp is rinsed in distilled water. The bleached fibres are further treated with equal amount of 10% (w/w) nitric acid and 10% (w/w) chromic acid by dipping in equal amount of solution and raising the temperature to 60 for 15 minutes to initiate the reaction and then allowed to stay for 24 hours. The treated fibres are then washed in distilled water, centrifuged in ethanol and dried in still air.

D. X-Ray Diffraction

A Panalytical X'Pert Pro MPD model diffractometer equipped with an X'celerator detector and GAADS software is used to observe the diffraction pattern of the cellulose crystals in the fibre. The diffraction patterns are collected by using small mass of fibre placed on a rectangular flat glass 45cm by 3.6cm to ~ 1mm thick over a rectangular space of 1.5cm by 2cm at a scan range of 5o~600(2θ) in step size of 0.0334, using a Ni filter. The diffraction patterns are generated within 7 -8 min with the X-ray beam set to 40 kV and 40 mA at 290C (inner temperature), 250C (outside temperature).The glass spatula and the rectangular flat glass are cleaned with a tissue paper soaked in methanol before and after each sample test.

To calculate apparent crystallinity of the fibres the deconvolution method is used. The curve fitting is performed with PeakFit 4.12 (www.systat.com) assuming Gaussian (area) functions with free adjustment of intensity, position and width of both crystalline and amorphous peaks for most of the cases, except for three fibres where the Gaussuain-Lorentzian function is used to obtain a more accurate fitting. Linear backgrounds are fitted to a simulated profile based on the

convolution of four sharp peaks [1-10, 110, 200, 040] for crystalline reflections and one broad peak for amorphous reflection in groundnut shell, rice husk, palm fruit stalk and palm fruit bunch fibres. The coconut fibres had a conspicuous extra sharp peak between 002 and 040 peaks. This peak was used as part of the crystalline peaks during the peak fitting. In each case iterations were repeated until maximum F (>10000) value is attained with an R2> 0.99 and the crystallinity index calculated from the equation:

$$C = \frac{A_{1-10}+A_{110}+A_{200}+A_{040}}{A_{1-10}+A_{110}+A_{200}+A_{040}+A_{non-cr}} \times 100 \quad (1)$$

where: C is apparent crystallinity [%], $A_{1\text{-}10}$ represents the area under the crystalline peak in the diffraction pattern corresponding to the Miller index 1-10. A_{110} represents the area under the crystalline peak in the diffraction pattern corresponding to the Miller index 110. A_{200} represents the area under the crystalline peak in the diffraction pattern corresponding to the Miller index 200. A_{040} represents the area under the crystalline peak in the diffraction pattern corresponding to the Miller index 040. $A_{non\text{-}cr}$ represents the area under the non-crystalline peak in the diffraction pattern. In the case of coconut husk fibres an extra term corresponding to the extra peak is included to the numerator and the denominator.

E. Scanning Electron Microscopy

A Hitachi S-4700 model variable pressure Scanning Electron Microscope (SEM) fitted with an EDAX head is used to observe the longitudinal features of the fibres. The samples to be observed under the SEM are mounted on conductive adhesive tape prepared by placing the samples on circular disc lined with carbon and coated with Au for 5mins to enable it conduct electricity using an E-1010 Hitachi model machine.

III. Results/Discussion

A. X-Ray Diffraction

Figs. 1–6 show X-ray diffraction traces for the five fibres processed by two methods (Route A and Route B). Fibres from groundnut shell from route A show two distinct peaks at 2θ of 15.85 and 22.30 degrees and a third dull peak at 2θ =34.43 (see Fig. 1). The first peak (15.85) is attributed to 101 and 1-10 peaks combining to form a broad peak. The second peak (22.30) is attributed to the crystalline portion of cellulose at 200 while the third peak is attributed to the crystalline portion of the fibres at 040. Fibres processed with route B also possess these peaks at 15.55, 21.97 and 34.47.The fibre treated with route A tends to have a sharper peak at 200 than that of route B. Groundnut shell fibres processed using route A show an apparent crystallinity of 89.71 % while that processed via route B gave an apparent crystallinity of 96.11%. This indicates that treatment of groundnut shell fibres in the route B is able to remove more non-cellulosic materials than the route A treatment.

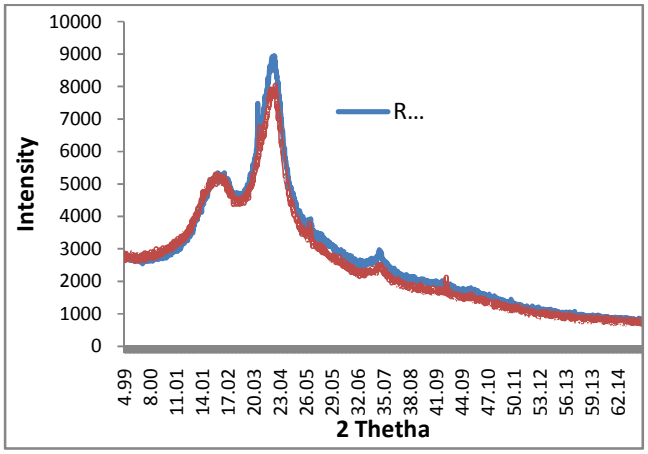


Fig. 1 XRD of processed Groundnut shell fibre

The X-ray traces of coconut fibres shown in Fig. 2, has an extra sharp peak between 200 and 040 that is different from the groundnut shell pattern. For fibre processed via route A the first peak appears at 2θ of 15.92, the second at 22.30 and third at 34.50, while the extra peak appeared at 26.55. For route B these peaks appear at 15.92, 22.37, 34.67 and 26.41 respectively. The 200 peak in route B appears to be sharper than that of route A. The apparent crystallinity of coconut husk fibres processed through routes A and B are 90.91% and 86.76% respectively. This indicates that treatment with route A is more effective compared to that of route B.

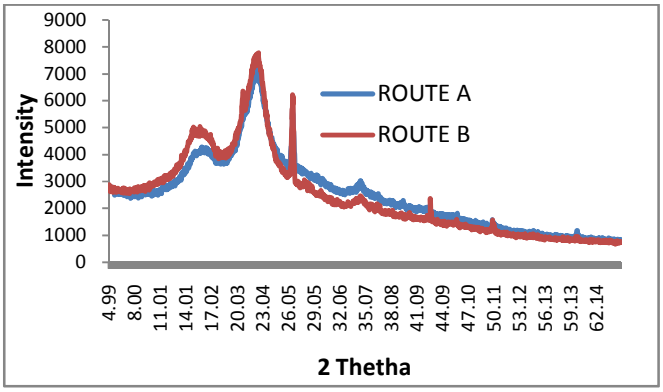


Fig. 2 XRD of processed Coconut husk fibre

Similar to the fibres from groundnut shell, fibres from rice husk (see Fig. 3) possess three distinct peaks at 2θ of 15.79, 22.27 and 34.70 for route A and 15.59, 22.14 and 34.80 for route B. Fibres processed with route B has a sharper 200 peak compared to that processed with route A. Processed rice husk fibres also show percentage crystallinity of 86.59% and 89.07% for route A and B respectively.

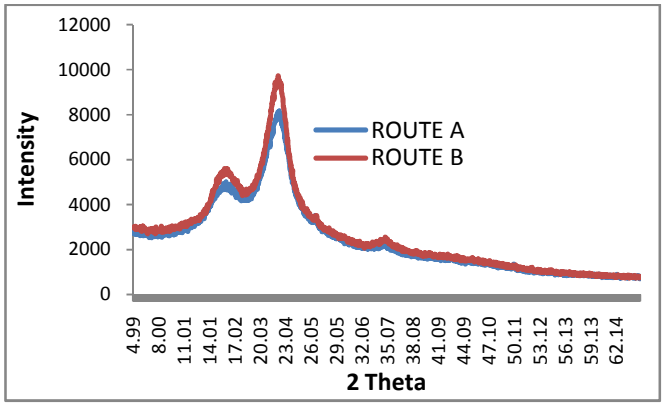


Fig. 3 XRD of processed Rice husk fibre

Fig. 4 shows the X-ray diffractogram of processed palm fruit bunch fibres. The X-ray trace is similar to that of groundnut shell and rice husk following the conventional cellulose XRD trace reported in most literatures. Three peaks appear at 2θ of 15.99, 22.24 and 34.47 for route A and 15.95, 22.30 and 34.43 for route B. There is no significant difference between the peaks in fibres processed via the two routes (A and B). This is evident as both fibres possess similar % crystallinity (88.55, A; 89.185, B).

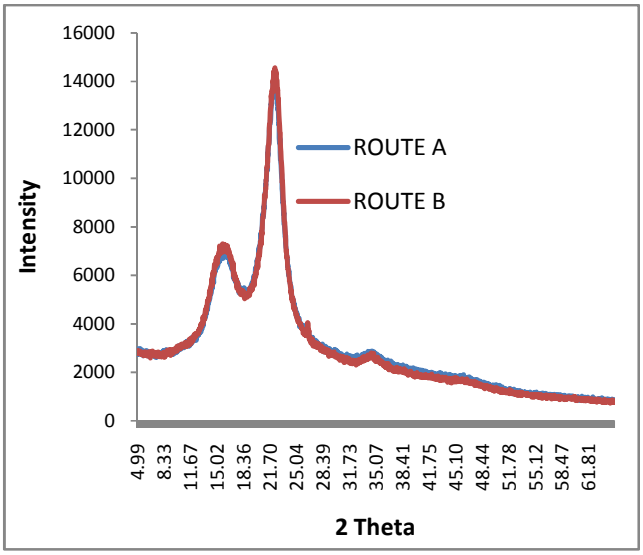


Fig. 4 XRD of processed Palm Fruit bunch fibre

Like the palm fruit bunch, palm fruit stalk shows a typical cellulose diffraction trace with three distinct peaks at 2θ of 15.82, 22.24 and 34.50 for route A; 15.82, 22.34 and 34.6 for route B. The traces coincide with apparent crystallinity of 89.74 and 89.56% for fibres processed via routes A and B respectively (see Fig. 5).

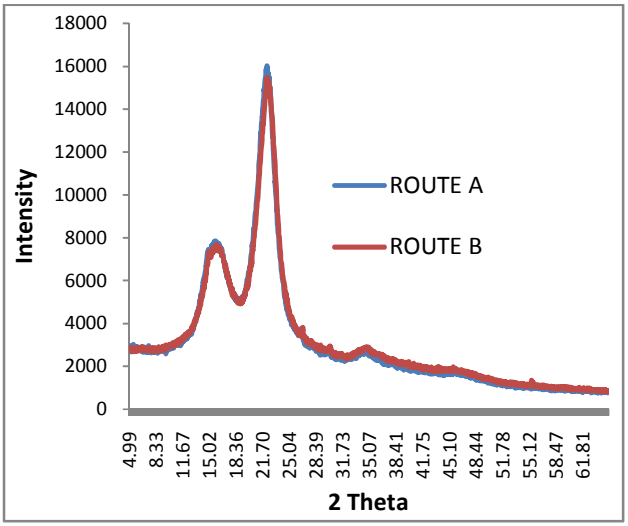


Fig. 5 XRD of processed Palm Fruit stalk fibre

B. Crystallinity

Crystallinity is an important feature in the physical analysis of cellulose fibres [33]–[36]. Pre-treatments can change cellulose crystal structures and degree of crystallinity by disrupting inter- and intra-chain hydrogen bonding of cellulose fibrils [37]. The features of fibres processed (groundnut shell, coconut husk rice husk, and palm fruit bunch and palm fruit stalk) using two methods are shown in Figs. 11 and 12. The crystallinity index (C) for all samples is calculated from the XRD data using the deconvolution method and the results are summarized in Table I.

TABLE I
DECONVOLUTION PARAMETERS

S/N	Fibre	Processing route	F value	R^2 value	% crystallinity
1.	Groundnut Shell	A	12825.6701	0.99017869	89.71
2.		B	21234.0320	0.99509864	96.11
3.	Coconut Husk	A	9902.44646	0.99111718	90.91
4.		B	13564.7121	0.99349982	86.76
5.	Rice Husk	A	12891.8194	0.99022859	86.59
6.		B	14562.3401	0.99133981	89.07
7.	Palm fruit bunch	A	20394.3692	0.99380095	88.55
8.		B	18501.6194	0.99317111	89.19
9.	Palm fruit stalk	A	12700.2318	0.99008265	89.74
10.		B	16259.4952	0.99223674	89.56

Fibres from groundnut shell processed through route B possess the highest crystallinity of 96.11%. The resulting crystallinity has shown that the treatment methods utilized in the study has the propensity to improve the yield of cellulose nano-crystals [38]. The increase in the number of crystalline regions increases the rigidity of cellulose. Higher crystallinity of these fibres is associated with higher tensile strength of the fibres. Therefore, there is a strong indication that mechanical properties of composite material will improve if these treated fibres are used [39]–[41].

The peak patterns for all diffractograms are typical crystalline structures of native cellulose with main diffraction signals at 2 theta in the ranges of 15.55 – 15.99, 21.97 – 22.37

and 34.43 – 34.80. This is consistent with findings of other authors [38]-[46]. The first peak (15.55 – 15.99) is a result of the combination of two crystalline peaks to form a broad peak and are normally assigned the diffraction planes 1-10 and 110. The second peak (21.97 – 22.37) is normally assigned to crystalline portion of cellulose on the 200 diffraction plane while the third peak (34.43 – 34.80) is assigned the diffraction plane 040. It is noted that palm fruit stalk show the highest peak among the 200 peaks in both treatment methods employed and followed by palm fruit bunch (see Figs. 11 and 12). The results show palm fruit fibres to be richer in cellulose than the other fibres.

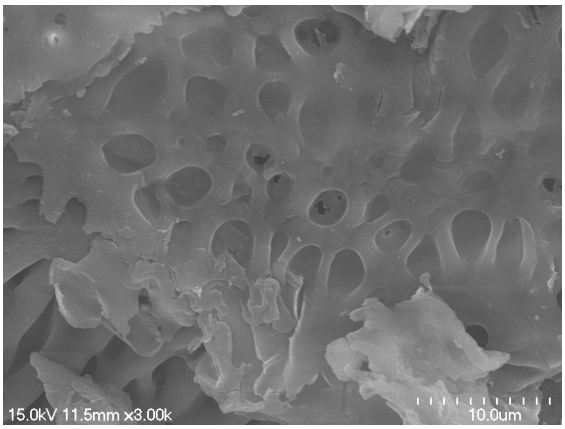
C. Morphology

Fig. 6 shows the SEM micrographs of groundnut shell fibres processed via routes A and B. In Fig. 6 (a) the fibre displays a network structure of randomly assembled fibrils. The fibre network in B consists of terraces, steps and kinks traceable to its hydrolysis. The surface of fibres through route A appears smooth while that of B is slightly rough owing to the residual effect of steam explosion of its fibres. The SEM images for coconut husk fibres (see Fig. 7) have fibrous network structure with distortion arising from the steam explosion, hydrolysis and mechanical treatments of its fibres. The fibre structure through route A appears agglomerated compared to that of B with the presence of irregularly-sized fibrils. The terraces, steps and kinks are more in B than A, which could have resulted due to removal of more reactive amorphous cellulose from its surface than those through route A. The peak obtained in fibre of route B is sharper than that of route A (see XRD chart in Fig. 2). Fig. 7 (b) clearly shows that each thin leaf of cellulose fibrillar lamellae exists separately though they are stuck to each other and produce torn-like laminate structure overlapping each other. This behaviour is caused by the ease of formation of hydrogen bonding among the hydroxyl groups present on the different surfaces [47].

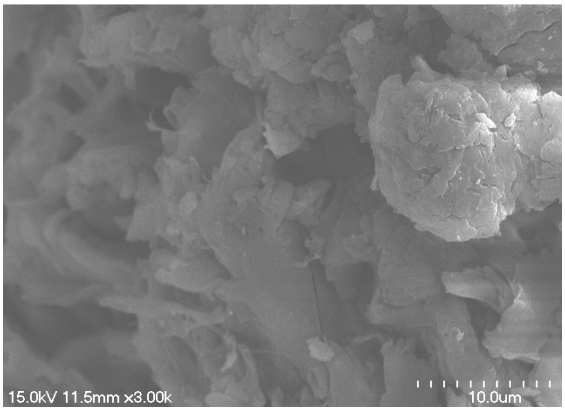
Figs. 8 (a), (b) show SEM images of rice husk fibres that are hydrolysed. Similar to coconut husk fibres processed via route B the fibrous network of both routes show structural distortion occasioned by steam explosion, hydrolysis and mechanical treatments given to the fibres. The severe aggregate structure of the fibrils is seen in both images with the surface regularly coagulated to form a laminate thin leaves layer. Each leaf lamella consists of cellulose fibrils. The fibre structure in B appears fines than that through process route A due to severity of treatment in route B. The coagulation of fibre here may equally be attributed to ease of hydrogen bonding formation among the hydroxyl groups present on the different surfaces [47].

Figs. 9 (a), (b) show the SEM images of palm fruit bunch which is similar to the rice husk fibrous network with structural distortion emanating from steam explosion, hydrolysis and mechanical treatments of its fibres. The aggregate structure of the fibrils laminates of thin leaves with each leaf appearing as lamellae of cellulose fibrils. Figs. 10 (a), (b) are SEM images of palm fruit stalk fibres. In Fig. 10 (a) the result point to strong fibre resistance to hydrolysis treatment. The distortion appears minimal with insignificant fibre-lamellae structure but longer fibre length than others. Fig. 10 (b) shows the presence of light lines which are attributed to steps formed in hydrolysis. The palm fruit fibre is stronger than its bunch and this may account for its resistance to mechanical, chemical and enzymatic hydrolysis treatments than its bunch. The palm fruit fibre network structure and surface roughness indicate that it is rich in crystalline cellulose and will be a suitable reinforcement in polymer composites.

Generally, the surfaces of the fibres are not smooth but covered with ridges. This is an indication of the organization of the fibrils within the fibres. Furthermore the effect of steam explosion pre-treatment on the softening of lignin is clearly seen. Steaming is believed to lower the softening point and reduce the molecular weight of the lignin for its removal via subsequent treatments. It is evident that steam explosion pre-treatment significantly destroyed lignocellulosic structure with more structural distortion. The combination of mechanical treatment, steam explosion and alkaline hydrolysis (route A) activates large reactive surface area on the fibre with improved cellulose accessibility [42]. The combined effect of enzymatic and alkaline hydrolysis (route B) is seen in the fibre surface roughness in all the SEM images (see Figs. 6 (b), 7 (b), 8 (b), 9 (b), 10 (b)) processed. This suggests the removal of hemicelluloses, lignin and pectin after the treatment [41].

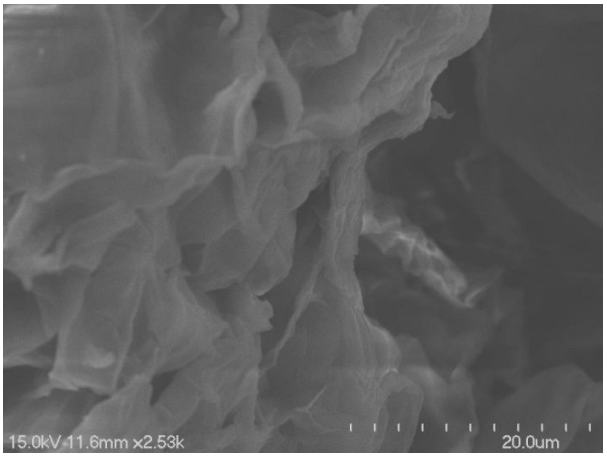


(a)



(b)

Fig. 6 SEM images of Groundnut Shell fibres (a) route A (b) route B



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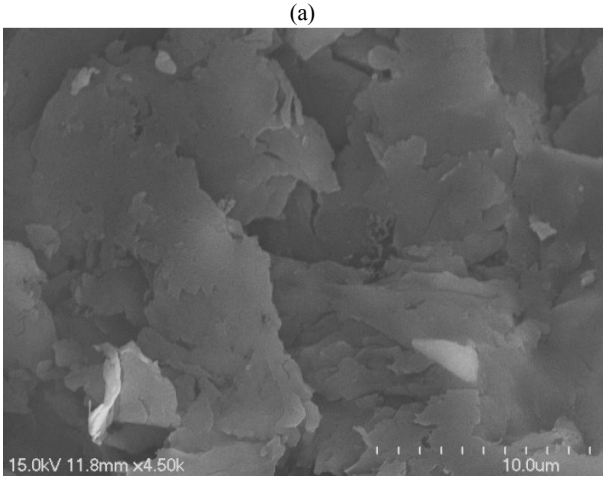
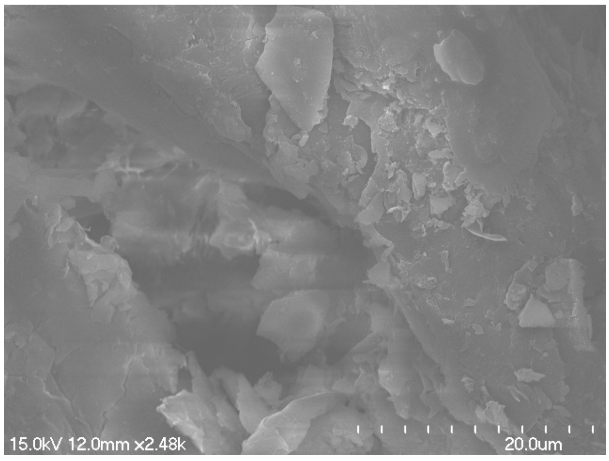


(b)

Fig. 7 SEM images of Coconut husk fibres (a) route A (b) route B



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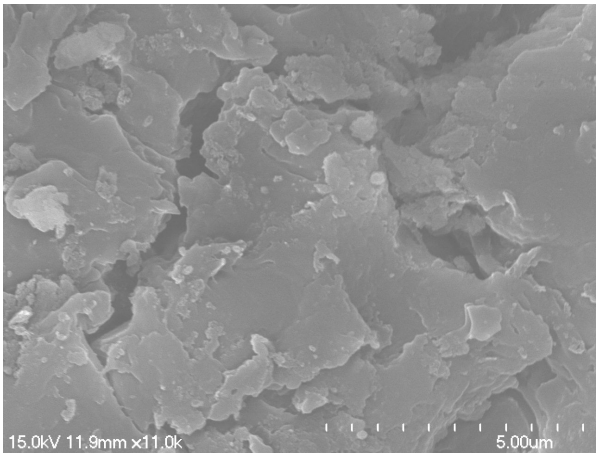
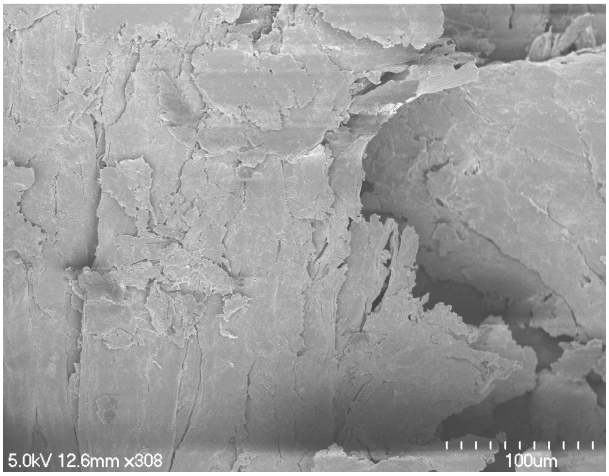
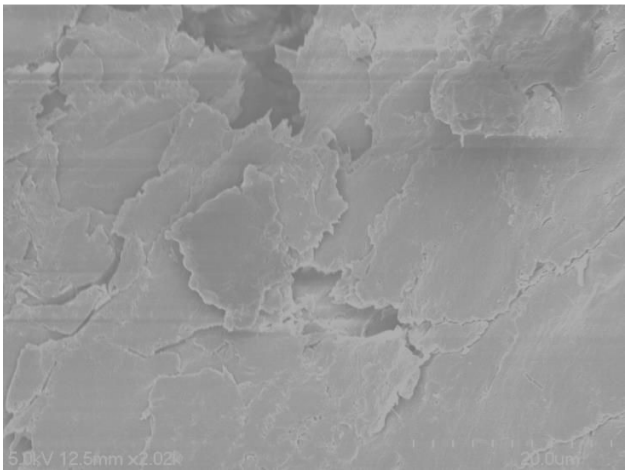


(b)

Fig. 8 SEM images of Rice husk fibres (a) route A (b) route B

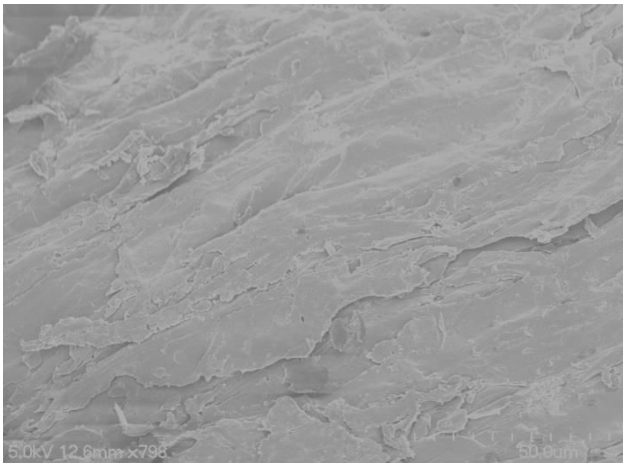


(a)

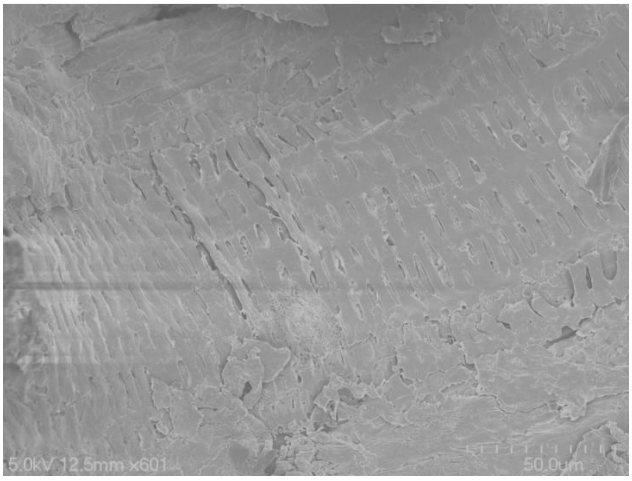


(b)

Fig. 9 SEM images of Palm fruit bunch fibres (a) route A (b) route B



(a)



(b)

Fig. 10 SEM images of Palm fruit stalk fibres (a) route A (b) route B

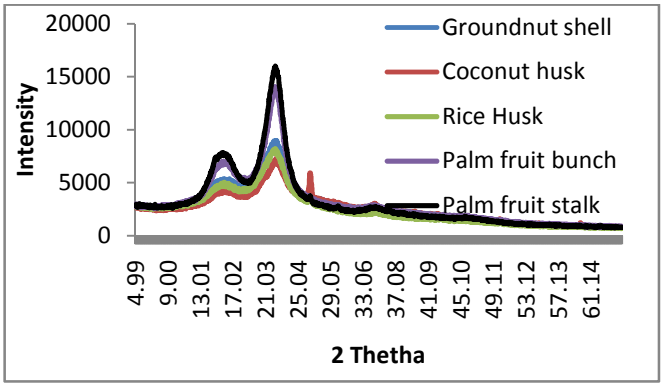


Fig. 11 XRD of processed agro-waste fibres using route A

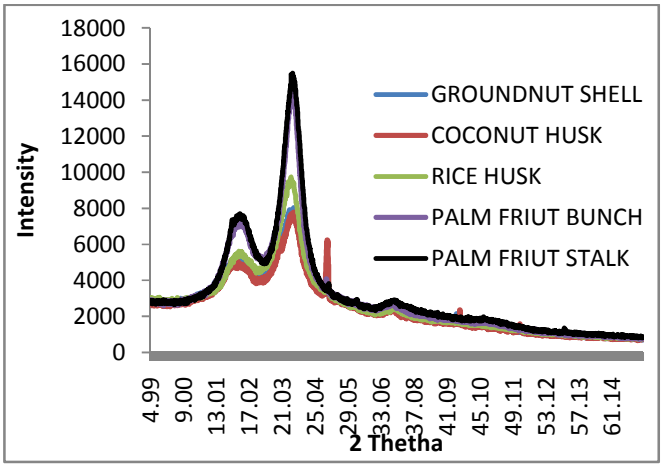


Fig. 12 XRD of processed agro-waste fibres using route B

IV. Conclusion

The study has established that amorphous cellulose near the surface of macro-fibrils can be hydrolysed readily by the two methods of treatments adopted as it exposes the fibril bundles in the macro-fibrils (as observed in SEM morphologies). XRD results indicate that cellulose apparent crystallinity improved tremendously (up to 20% increase) over those reported in literature. This suggests significant leaching of amorphous cellulose in the bulk fibres and evidence that the two methods examined are capable of producing quality technical fibres for composite applications.

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