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Cellulose Whiskers Derived from Banana Pseudo-Stem as Potential Reinforcement Filler for Tyre Tread Compounds

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ABSTRACT

In this study, cellulose fibre was extracted from banana pseudo-stem and converted into cellulose whiskers (CWs) with high-pressure defibrillation method. The CWs were characterized using FTIR, XRD and the results conform the structure of cellulose. XRD studies showed that the percentage crystallinity of bleached fibres is about 93%. The particle size analysis showed a bimodal distribution for CWs where approximately 21% of the sample has average size of 110 nm. The rubber compounds were prepared according to a general tyre tread formulation while incorporating the purified CWs in diluted centrifuged latex (NR) followed by converting them into rubber sheets to produce cellulose whiskers/natural rubber CW/NR composite. Meanwhile, the control was prepared without incorporating CW in the diluted centrifuged latex. The lowest Mooney viscosity and the highest stress relaxation coefficient of CW/NR composites ensured higher processability. Curing characteristics of the compounds were evaluated at 120 °C and physical properties were evaluated on par to the ASTM standards. The highest cure rate and better scorch time were observed in CW/NR composite. Most of the physical properties were superior. In overall, it is obvious that cellulose whiskers have a potential to use as reinforcing material for natural rubber tyre tread compounds.

1. Introduction

Natural rubber latex is a renewable biopolymer, tapped from *Hevea brasiliensis* with a wide spectrum of applications, which can be gained after vulcanization. However, vulcanization itself cannot gain the required properties in a respected rubber product. Therefore, it is usually modified for further increment of its targeted uses. Nevertheless, natural rubber is an indispensable and very important material in both industrial and household uses while performing as an outstanding material for better tensile properties, good crack growth resistance due to its strain induced crystallization [1]. With respect to achieve the intended properties, reinforcement of a polymer composite material is highly considered. According to Bokobza and Chauvin [2], it has been suggested that reinforcement of the elastomeric compound was defined as the elevation of elastic modulus and elongation at break by addition of particular filler ingredients.

Reinforcement of elastomers is commonly done by the carbon black (CB) and silica in the rubber industry and it is said that any finely divided particles can enhance the tensile properties where chemical and physical bonds fixes the filler in the polymer matrix and allows the stress delocalization through facial slippage [2]. Comparatively, with gum rubber (without any filler), CB filled rubber composites are superior in high modulus, strength and abrasion resistance. However, with the green concepts, the controversial argument is there against CB and also it is said that the effect of the CB on such composites is not negligible [3] and cannot be replaced by any other existing low-cost fillers. Silica is another filler which is more capable in reducing the heat buildup and possess low rolling resistance which makes it indispensable in rubber composites, especially in tyre manufacturing. However, high density (2.5 g/cm^3) of silica increases the density of composites, longer the processing time and higher the energy requirement of mixing [4]. Not only that, but also a study done by Kirushanthi et al. [5] has suggested that the silica derived from rice husk ash, another agricultural waste, can comparatively improve the crumb rubber-filler interaction to provide better reinforcement. Kondarage et al. [6] has shown that fired waste ceramics can be incorporated with natural rubber and obtained substantial improvement of mechanical properties. Also, short natural fibres and clay materials are used as green fillers in rubber manufacturing [7].

According to the study done by Bras et al. [8], cellulose have been extracted from bleached sugarcane bagasse craft pulp and have added the cellulose whiskers as the reinforcing elements into NR without any surface modification, and the effect of loading of cellulose whiskers has been studied. Significant improvement of Young's modulus and tensile strength have been observed as a result of adding cellulose whiskers to the rubber matrix especially at high whiskers loading levels. Also, according to the study of Pasquini et al. [9], they have succeeded in obtaining cellulose from cassava bagasse, a byproduct of cassava starch production where sulfuric acid hydrolyzation treatment has been practiced. High aspect ratio as well as good physical properties after NR compounding has been observed after dynamic mechanical tests. And the storage tensile modulus has been increased significantly upon the addition of cellulose. Positive properties of tensile strength, elongation modulus, storage modulus, and thermal stability have been observed [10], by incorporating bamboo based cellulose whiskers derived from bamboo pulp wastes of paper production. Also, it is succeeded in separating cellulose from agricultural wastes like straw and tunicin and they have succeeded in using the same cellulose as reinforcement in polymer composites. It has been reported that cellulose fibres in both nano and micro sizes can be used to replace the man made synthetic fibres or any other materials which can supposed to be harmful to the environment also it can reduce the over dependence of petroleum based treacherous reinforcing materials like CB [11].

Currently, natural plant fibres such as kenaf, jute, coir, rice husk, banana, bamboo, sisal, etc. are predominantly available in developing countries such as Malaysia, Indonesia, Thailand, and other Asian countries [12, 13]. Indeed, natural cellulosic fibres are rapidly emerging as novel low-cost materials for several industrial applications while large volumes of cellulosic waste materials originating from agricultural and industrial activities are often considered as worthless staffs [14].

In the plants fibres, cellulose is available in its very natural form and often with other components such as hemicellulose, lignin, and pectin, etc. Altogether, the source of the cellulose is lignocellulosic material in wood [15].

Among the above stated plants, banana is recorded as one of the oldest cultivated plants [13] in which pseudo stem is commonly known as trunk which is fibrous in nature and enriched in cellulose content [16] are left

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behind after harvesting the ripened fruit bunches [17]. Those pseudo stems are currently used only for the compost preparation or some time burnt resulting in the environmental pollution. The outer sheaths of pseudo-stem are composed of the coarsest fibre which are very brittle and can be broken at a slightest tension. Since the innermost sheaths contain pulpy matter only the intermediate sheaths have been utilized for obtaining banana pseudo-stem fibres. Extraction of banana fibres from the pseudo stem can be done manually as well as mechanically using banana fibre extractor machines or it can be also done in laborious manual processes [16].

According to Cherian et al. [18], based on the cellulose derived from banana pseudo stem fibre, they suggested that cellulose fibre has gain much attraction due to its large surface to volume ratio, high surface area, good mechanical properties including a high Young's modulus, high tensile strength and a very low coefficient of thermal expansion, and formation of highly porous mesh as compared to other commercial fibres. Moreover, it reveals that banana fibre cellulose can be chemically extracted and purified by using chemical methods. These raw banana fibre can be used to synthesis cellulose whiskers that can be used as the reinforcing filaments in rubber compounds without any tedious surface modifications [8].

In this work, the major objective was to extract cellulose from banana pseudo-stem and utilize the derived cellulose in rubber tyre tread compound preparation. These banana fibres were undergone high pressure defibrillation followed by rigorous chemical treatments.

2. Experimental Methods

For this study, manually extracted dried banana fibres, analytical grades of sodium hydroxide, acetic acid, sodium hypochlorite and oxalic acid were used.

2.1 Banana Fibre Extraction

Banana fibres were manually extracted after boiling the banana pseudo-stem sheaths in an Aluminium container in the presence of wood ash for 2 hours and the mucilaginous substances were removed using a wire brush and the banana pseudo stem fibres were extracted. For that the middle most sheaths were only taken for the boiling purpose. The extracted fibres were sun dried to reduce the moisture content of the fibre and then the dried fibres were packed in a sealed transparent polythene bag.

2.2 Cellulose Whiskers Preparation

Firstly, the dried banana fibres were ground using a high-speed universal disintegrator and sieved through 500 µm mesh. Secondly, during the steam exploding, ground banana fibres were treated in 2% NaOH (fibre to liquor ratio 1:10) in an autoclave under 20 lb for one hour. Then the pressure was released immediately. After, treated fibre was allowed to drain the water followed by rigorous washing with distilled water until it was got rid of alkalinity. Thirdly, in order to prepare the steam exploded bleached fibres, pretreated fibres were bleached using NaOH solution (27 g in 1 liter of water), acetic acid solution (78.8 g in 1 liter of water) and a mixture of 1:3 sodium hypochlorite solution repeatedly for six repetitions. Then the treated fibres were washed rigorously in distilled water. Fourthly, the treated fibres were treated in 11% oxalic acid in autoclave until it took 20 lb and the pressure was released immediately. Then the pressure in the autoclave was allowed to obtain 20 lb and the medium was allowed to be there for 15 minutes and the process was repeated for 8 times. Then the fibres were taken out and they were washed using distilled water until the washings does not decolourized the $KMnO_4$ solution. And finally, fibres were suspended in distilled water and it was emulsified at 8000 rpm for 4 hours using a Silverson High Shear Mixer (L4RT) to prepare the cellulose whiskers [18].

2.3 Fibre Characterization

FTIR spectrometer (Bruker, Alpha-T, German) was used to characterize the fibre samples from raw banana fibre, alkaline treated banana fibre and the bleached banana fibres using a deuterated triglycine sulphate (DTGS) as a detector. It was interfaced to a computer operating under Windows-based, and connected to OPUS (Version 7.5) software. FTIR spectra were collected in frequency 4000 - 500 cm^{-1} by co-adding 64 scans and at resolution of 4 cm^{-1}. All spectra were rationed against a background of air spectrum. Resulted fibres from each step was ground and mixed with dehydrated KBr to prepare pastilles.

Rigaku Ultima IV-X-Ray Diffractometer was used to investigate the relative crystallinities of steam exploded banana fibre, bleached fibre as well as raw banana fibres. The percentage crystallinity was determined using a powder X-ray diffraction method (PXRD). Percentage crystallinity (C_I) was measured where I_{002} is the maximum intensity (in arbitrary units) of the 0,0,2 lattice diffraction and I_{am} is the intensity of diffraction in the same units at the nearest depression.

$$C_I = \frac{I_{002} - I_{am}}{I_{002}} \times 100$$

Particle size was analyzed using a Malvern Particle Size Analyzer (Zetasizer Nano ZS). There, the bleached cellulose aqueous medium was investigated considering the refractive index of water as 1.333.

2.4 Rubber Compounding

In dry rubber compounding, 3 phr of the CWs were mixed with diluted centrifuged latex up to 30% dry rubber content (DRC) and the raw rubber sheets were prepared. According to Tables 1-4, CW/NR composite and the control were prepared. Raw rubber sheets were evaluated for their Mooney viscosities using the Mooney Viscometer (MV 2000, Alpha Technologies, USA) using a large rotor at 100 °C under ML (1+4) 100 °C test condition. Following a general tread formulation, both 1st stage and the 2nd stage compounds of control and CW/NR composite were prepared.

Table 1 First stage compound formulation

Ingredient	Weight (g)
Rubber with 3 phr of CWs	781
CB	356
Activator System	49
Protection System	12

Table 2 Second stage compound formulation

Ingredient	Weight (g)
1st Stage Compound	1200
Curing System	17

Table 3 First stage compound formulation of the control compound

Ingredient	Weight (g)
Rubber without 3 phr of CWs	773
CB	363
Activator System	50
Protection System	12

Table 4 Second stage compound formulation of the control sample

Ingredient	Weight (g)
1st Stage Control Compound	1200
Curing System	17

Curing characteristics of the second stage compounds were measured using an ODR Rheometer (Alpha Technologies, USA) at 150 °C for 30 minutes of testing time and the physical and mechanical properties were measured following ASTM standards.

Density of compounds was measured by electronic densimeter MD-3003 in accordance with ASTM D-297 standard. Resistance to indentation (Hardness) was measured in accordance with ASTM D-2240 standard. Evaluation of tensile strength, elongation and modulus of the vulcanizates were measured according to ISO 37-1994 standard. Evaluation of tear strength of rubber was measured in accordance with ASTM D-624-86 standard. Determination of abrasion resistance was measured on par with the volume loss of the rubber vulcanizate in accordance with DIN 53516 standard. In evaluation of rebound resilience, it was measured in accordance with ASTM D-2632 standard.

3. Results and Discussion

FTIR spectra of raw banana fibre and its derivatives have been represented from Figs. 1-4.

According to the Fig. 1, at the left of the FTIR spectrum, the peak which arises from the 3400 cm^{-1} area is due to stretching of OH bond of absorbing moisture [19]. Next to this region is the C-H bonding which appears between 3000-2800 cm^{-1} region. Then the 2915 cm^{-1} peak is due to the alkyl C-H stretch functional groups mostly from the cellulose and a bit from the hemicellulose presenting in the purified cellulose. The peak at 1635 cm^{-1} is due to amide C=O Stretching because of the dissolution of hemicellulose during the steam explosion and bleaching while the amide C=O Stretching of the remaining pectin gives the same peak having methyl

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esters and carboxylic acids [20]. In the Figs. 2 and 3, this peak is absent or less due to the partial de-esterification process happens in the process of alkaline treatment and the bleaching. The peaks at 1629 cm^{-1} - 1630 cm^{-1} is due to the alkenyl C=C stretching vibration, the indicative of the presence of lignin. The absence of the peak conforms that this sample is not contaminated with lignin. In the Fig. 1, the bands in the region of 1250-1056 cm^{-1} wavenumber involving the C-O stretching vibrations of aliphatic primary and secondary alcohols in cellulose, hemicellulose, lignin, and extractives [18].

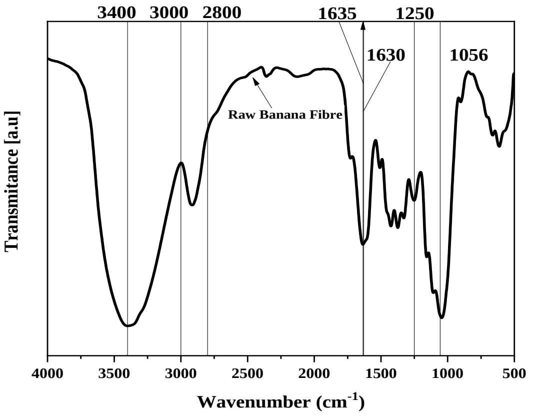

Fig. 1 FTIR spectra of the raw banana fibre

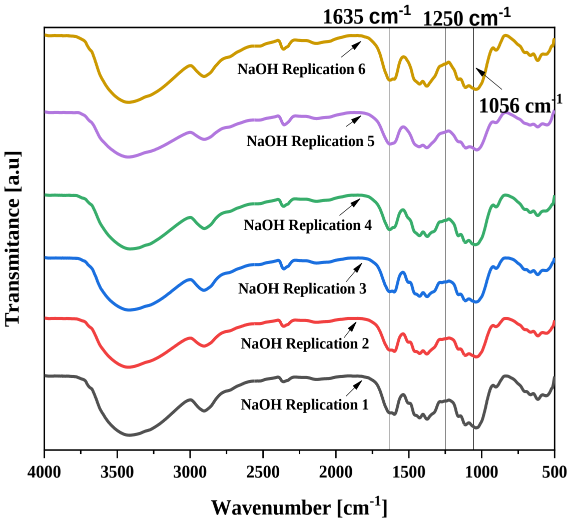

Fig. 2 FTIR spectra for six-times purification of BF with NaOH

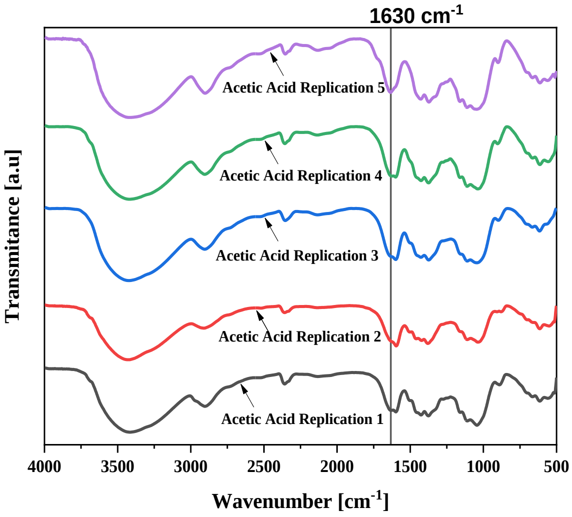

Fig. 3 FTIR Spectra for five-times purification of BF with acetic acid

According to the Fig. 4, the intensity of the peak at 1250 cm^{-1} is sharply weakened after bleaching treatment because of the removal of the hemicellulose material. The peak area of the peaks due to the lignin components is found to be decreased from raw fibre to the bleached fibres. This proves that most of the lignin components were dissolved during steam explosion and on further bleaching processes [20].

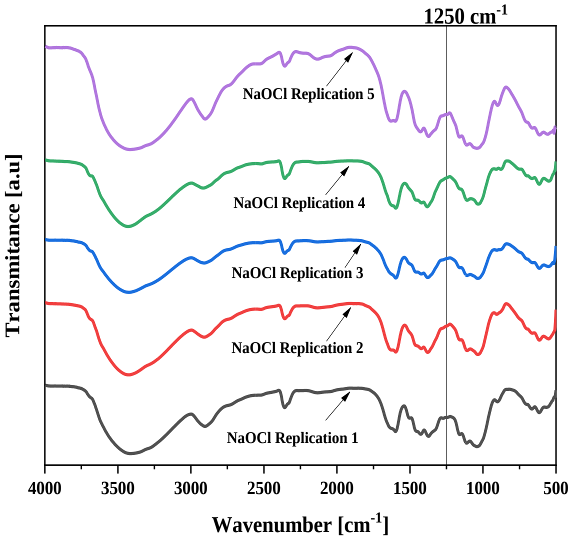

Fig. 4 FTIR spectra for five-times purification of BF with NaOCl

According to the Fig. 5, banana fibres reveal a semi crystalline nature [21]. It was vividly clear that each and every purification step were able to improve the crystallinity except NaOH treatment. According to the diffractogram, it shows a very little crystallinity while the majority was in amorphous phase [18]. Raw banana fibres showed 81.53% of the percentage crystallinity at 2θ value of 22.23°. However, with the alkaline treatment (steam exploded banana fibres), the percentage crystallinity was recorded as 68.28% showing an amorphous nature at the 2θ of 21.82°. But after the bleaching process, the percentage crystallinity of the cellulose has increased up to 92.72% at 2θ value of 22.72°. It was due to more efficient removal of non-cellulosic polysaccharides and dissolution of amorphous zones [22].

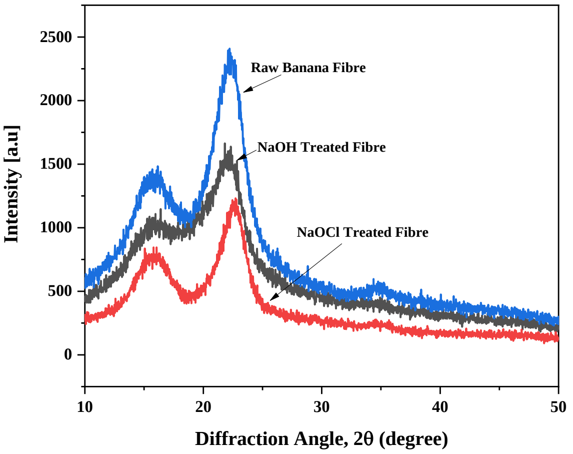

Fig. 5 XRD curves of raw, steam exploded and bleached banana fibres

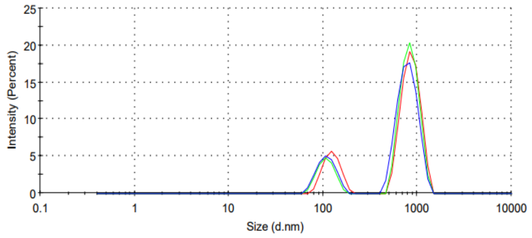

Fig. 6 Particle size distribution of CWs

According to the Fig. 6, it revealed a bimodal distribution where approximately 21% of the sample has average size of 110 nm and the rest has the size of 795 nm. However, the Z-Averaged particle size was 1026.14 nm.

3.1 Mooney Properties of Rubber Compounds

According to the Table 5, the lowest initial Mooney Viscosity (MI), the lowest Mooney Viscosity (ML) and final Mooney Viscosity (MF) of CW/NR compound and control have been recorded. Also, the elastic coefficient (a) of CW/NR compound is higher (-0.28) than the control (-0.25). Therefore, the highest processability is shown by the CW/NR composite.

Table 5 Mooney viscosity of the control and CW/NR

Material	MI	ML	MF	a	k
Control	176.66	91.21	91.21	-0.25	69.23
CW/NR	115.02	75.45	75.45	-0.28	58.19

3.2 Curing Characteristics

According to the curing characteristics (Table 6), it revealed that CW/NR composite has the highest ts2 time (122.3s), with the lowest t90 (203s), where the 90% of the cross links are created, which enables the CW/NR composite to take the highest curing rate (124.4 s^{-1}).

Table 6 Curing characteristics of rubber compounds (150 °C, 30 Min)

Material	MI (dNm)	MH (dNm)	ts2 (S)	t90 (S)	Curing Rate (S^{-1})
Control	27.2	105.4	120.6	297.6	56.5
CW/NR	24.9	76.1	122.4	202.8	124.4

3.3 Density and Hardness Properties

After the preparation of the cured compound CW/NR composite showed the highest density of 1.13 g/cm^3 while it is 1.12 g/cm^3 in the control. Both compounds had its Shore A hardness values as 68 nevertheless the lowest rebound resilience (53) showed in CW/NR composite and it was 58 in the control. However, the both materials were in the desired range of the respected tyre compound properties.

3.4 Tensile Properties

An improved tensile strength has been recorded in the CW/NR composite (245.12 MPa) compared to the control (158.68 MPa). The modulus at 300% level is inferior in CW/NR compound. However, CW/NR composite showed the superior elongation at break (425%) at the same time and it was 331.15% in the control (Table 7).

Table 7 Modulus 300%, tensile, tearing, abrasion

Material	Modulus 300% (MPa)	Tensile (MPa)	Elongation at break%	Tearing (N/mm)	Abrasion Volume loss (mm^3)
Control	177.6	209.7	331.1	47.3	99.0
CW/NR	158.6	245.1	425.0	37.1	110.0

After taking the abrasion volume loss, CW/NR was inferior because it showed 110 mm^3 abrasion volume loss. However, the control showed the abrasion volume lose as 99 mm^3 nominating the control as the superior at abrasion resistance. Surprisingly, both materials showed the less tearing values but from that the control was the superior marking its tearing value as 47.43 N/mm.

3.5 Cut and Chip Properties

According to the results of cut and chip test, it revealed that CW/NR compound has superior properties than the control in both percentage weight loss and averaged percentage diameter loss (Table 8).

Table 8 Averaged percentage weight loss and averaged percentage diameter loss

Material	Percentage Weight loss (%)	Percentage Diameter loss (%)
Control	11.5	14.4
CW/NR	9.3	12.5

4. Conclusion

In summary, CWs were extracted from banana pseudo-stem suggesting that the under-utilized banana pseudo-stem waste can be effectively utilized by extracting cellulose from its ligno-cellulosic biomass by steam explosion technique. Both FTIR and XRD studies conformed the structure of cellulose as well as its purification trends. Also, from the particle size analysis, it showed that a proportion of CWs was almost in the nano scale. The prepared CWs were incorporated at 3 phr of loading level with diluted centrifuged latex. From the Mooney properties of the raw materials, it showed that CW/NR composite had the higher processability over the control. By considering the physio-mechanical properties such as modulus at 300%, tensile strength and cut and chip values concluded that the cellulose whiskers have a potential to slightly replace CB in tyre tread manufacturing.

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