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Environmental friendly method for the extraction of coir fibre and isolation of nanofibre

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ABSTRACT

The objective of this work was to develop an environmental friendly method for the effective utilization of coir fibre by adopting steam pre-treatment. The retting of the coconut bunch makes strong environmental problems which can be avoided by this method. Chemical characterization of the fibre during each processing stages confirmed the increase of cellulose content from raw (40%) to final steam treated fibres (93%). Morphological and dynamic light scattering analyses of the fibres at different processing stages revealed that the isolation of cellulose nano fibres occur in the final step of the process as an aqueous suspension. FT-IR and XRD analysis demonstrated that the treatments lead to the gradual removal of lignin and hemicelluloses from the fibres. The existence of strong lignin–cellulose complex in the raw coir fibre is proved by its enhanced thermal stability. Steam explosion has been proved to be a green method to expand the application areas of coir fibre.

1. Introduction

The promising performance of cellulose nanofibres and their abundance encourages the utilization of agricultural waste residue, which acts as the main source of cellulose. In nature, a large number of plants and animals synthesize extra-cellular high-performance skeletal biocomposites consisting of a matrix reinforced by fibrous biopolymers (Mohanty, Misra, & Hinrichsen, 2000). Cellulose is a classical example where the reinforcing elements exist as whisker-like microfibrils that are biosynthesized and deposited in a continuous manner (Itoh & Brown, 1984). Coir fibre obtained from coconut husk is one of the major underutilized raw material which is composed of cellulose nanofibre which constitutes 32–43% of its dry weight (Ayrilmis, Jarusombuti, Fueangvivat, Bauchongkol, & White, 2011). Total world coir fibre production is 250,000 tonnes per year and out of it, 80% of the fibre is contributed by the coastal region of Asia (Hon, 1994). The raw fibres have been reported to be used in the field of polymer composite application (Geethamma, Thomas Mathew, Lakshminarayanan, & Thomas, 1998). Efforts are going on for exploring wider export markets for coir and coir products but still most of the raw coir fibre remain underutilized. Judged from the increase in production and employment, the progress has been rather slow and exports in physical terms have remained mere or lets static.

Another major issue is related to the pollution originated during the retting and processing of raw coir fibre (Narayanan, 1999). One of the striking features of southern India is the continuous chain of lagoons or backwaters existing along the coastal region. The backwaters support rich and diverse life forms and provide crucial nurseries for shrimps and fishes as well as habitat for oysters, clams and mussels which later enrich the ocean. The shallow fringes of the backwaters and the channels drawn from them are used for retting of coconut husk. It adversely affects the productivity of the backwaters and is harmful to marine fisheries. The retting process is brought about by the pectinolytic activity of micro organisms especially bacteria, fungi and yeasts degrading the fibre binding materials of the husk and liberating large quantities of organic matter and chemicals into the environment, including pectin, pentosan, tannins, polyphenols, etc. Consequently hydrogen sulphide, phosphate and nitrate contents increase while dissolved oxygen and community diversity of plankton decrease in the ambient waters during the retting process. Here we report a novel environmental friendly method to use the raw coir fibre as they exist in the coconut husk, and thereby avoiding the retting steps of the coir fibre.

In an earlier work, nanocellulose was prepared from banana fibres by steam explosion (Abraham et al., 2011). Coir husk is

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another important source of cellulose. The coir fibre comprise mainly of cellulose, lignin and hemicellulose. The cellulose part is crystalline and the lignin part is completely amorphous in nature (Mohanty et al., 2000). Coir fibre industry mainly uses the fibre as it is obtained from the coconut fruit and no other extraction procedure has been reported yet. However, such separation is mandatory for its effective utilization in the current nanotechnological areas. The extracted lignin is a suitable source for renewable energy production. In this paper we report on a novel technique, steam explosion, for the separation of nanocellulose from coir fibre and there by expanding its application fields. In the various pretreatment technologies for the isolation of nanocellulose fibres, steam explosion is an attractive choice. We used mild acids with low concentration which avoids toxicity. Since nanocellulose is a hot topic in the current research field (Abraham et al., 2011; Zimmermann, Pohler, & Geiger, 2004; Goetz, Mathew, Oksman, Gatenholm, & Ragauskas, 2009), and its generation by a low cost method by a green pathway makes this work more important in this current environmental scenario.

2. Experimental

2.1. Materials

Coconut husk which is the principle source for the coir fibre were collected from locally available waste bunch of the coconut fruit. All the various chemicals used for extraction of the fibre are $NaClO_2$, NaOH and oxalic acid are of analytical grade obtained from Nice chemicals, Cochin, India. The chemicals used are in mild concentration to accomplish the environmental friendliness.

2.2. Chemical analysis of the fibres

Chemical constituents of fibres were determined according to ASTM standards. α-cellulose (ASTM D 1103-55 T), hemicellulose (ASTM D 1104-56), lignin (ASTM D 1106-56), Moisture content (ASTM D 4442-92). The cellulose, hemicelluloses, lignin and moisture content of the fibre in the untreated raw, steam exploded and acid hydrolysed stages of the coir fibre were determined.

2.3. Methods for the isolation of nano cellulose from raw fibres

2.3.1. Alkali treatment of the fibre

The alkali treatment will make the coir fibre more exposure to bleaching and acid hydrolysis. The optimum caustic soda treatment is both a very effective surface modification and a low cost surface treatment for coir fibres. Locally available coconut husk were cut it into pieces and subjected for treatment. Coir fibres were soaked in 2% caustic soda and placed for six hours at a temperature of 25 °C.

2.3.2. Steam explosion of the alkali treated fibre

A laboratory autoclave which can work with 137 Pa (20 lbs) pressure was used for steam treatment. Steam explosion technique was applied on the alkali treated fibre for one hour. Steam pretreatment was performed by loading the lignocellulosic material directly into the steam gun and treating it with high pressure steam at temperatures within 100–150 °C. The term used 'steam exploded fibre' means the alkali treatment followed by steam exploded coir fibre.

2.3.3. Bleaching of the steam exploded fibre

Alkali treated and steam exploded samples are then subjected to bleaching. The bleaching treatment with sodium chlorite ($NaClO_2$) solution (pH 2.3) for 1 h at 50 °C was performed to remove the remaining lignin. In the bleaching step, the absence of elemental chlorine which causes strong environmental problems is accomplished by using $NaClO_2$.

2.3.4. Acid treatment followed by steam explosion

The bleached sample is then subjected to mild acid treatment. 5% oxalic acid was used for the acid hydrolysis followed by second step of steam explosion for one hour. A pressure of 137 Pa (20 lbs) was used, followed by sudden release of pressure. The fibres were then washed thoroughly by water and then subjected to mechanical stirring followed by sonication.

2.4. Fourier transform-infra red spectroscopy (FTIR)

The FTIR spectra were recorded on an attenuated total reflection fourier transform infrared (ATR-FTIR) instrument (Nicolet 5700, Thermo Electron Corp., USA) in the range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1}. The samples were ground into powder by a fibre microtome and then blended with KBr followed by pressing the mixture into ultra-thin pellets.

2.5. X-ray diffraction technique (XRD)

X-ray equatorial diffraction profiles of the fibres were collected by a JEOL diffractometer, Model JDX 8P, using CuKα radiation at the operating voltage and current of 30 kV and 20 mA, respectively. The diffraction intensities were recorded between 2 and 80° (2θangle range).

2.6. Scanning electron microscopy (SEM)

Morphological analysis of untreated and steam exploded fibres were done by scanning electron microscopy. SEM micrographs of fibre surface were taken using a scanning electron microscope model JEOL JSM-35 C and Cambridge 250 MK_3 stereo scan operated at 10–20 kV. Prior to SEM evaluation, the samples were coated with gold for 5 minutes by means of a plasma sputtering apparatus.

2.7. Scanning probe microscopy (SPM)

The morphological study of the final nano cellulose fibrils were done by scanning probe microscopy. The scanning probe microscopy images of the nano fibres were made with a Multimode SPM (Veeco Inc., Santa Barbara, USA) with a Nanoscope IV controller in tapping mode. A dilute solution of nano dispersion which was sonicated just before the experiment was conducted.

2.8. Dynamic light scattering (DLS) and zeta potential

The particle sizing of nanocellulose dispersion in water was recorded by dynamic light scattering (DLS) system Inc., Santa Barbara, CA, USA. The principle of the DLS is based on the illumination of the sample by a laser beam followed by detection of the resultant fluctuations of the scattered light at a known scattering angle θ by a fast photon detector.

The concentration of the solution was 0.1 g of nanocellulose dispersed by sonication in 1 litre of water. Run time of the experiment was 5–7 min. The measurements were performed with an angle of 160° by using laser (4 mW) and the wavelength of the scattering light used was 639 nm and the count rate was 284 KHz. The temperature of the system was 20 °C. The channel width was 200.0 μs and the index of refractive was 1.347. Zeta potential measured as function of concentration of original aqueous coir cellulose suspension in 0.1 mM KCl electrolyte.