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Characterization of ultrasonic impact on coir, flax and hemp fibers

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ABSTRACT

Ultrasonic processing appears as a promising way to modify lignocellulosic material composition. Herein, it has been used on coir, hemp and short flax fibers. The optimal degradation was obtained after 24 h of ultrasonic treatment. Fibers were only slightly degraded by ultrasounds: flax and hemp fibers underwent respectively 4.52% and 4.13% weight loss and were found less sensitive to the treatment than coir fibers which exhibited a 9.17% weight loss. Ultrasound effect on the different components of these fibers, i.e. lignin, pectins, cellulose and hemicellulose was investigated and the results obtained on fibers were confirmed using solutions of these compounds. We found that ultrasonic processing of coir, flax and hemp fibers only degraded hemicelluloses in the fibers.

1. Introduction

Coir, hemp and short flax are mainly constituted of cellulose, hemicellulose, pectin and lignin [1–3]. Lignocellulosic fibers are very promising because they show high mechanical properties, low weight and are extracted from renewable resources. Coir, flax and hemp fibers have been used to reinforce composite materials [4–7] and can also be used to manufacture geotextiles. The aim of this work consists in studying the possibility of substituting coir fibers by local byproducts of the flax and hemp industries to manufacture efficient geotextiles for soil stabilization. For this purpose, resistance of fiber to degradation is investigated.

Ultrasonic waves create pressure differences that travel through the liquid medium leading to high pressure (compression) and low pressure (rarefaction) regions. The rarefaction can stretch the liquid molecules apart and create cavities also known as bubbles. As the wave progresses through the liquid, the bubbles expand and contract according to the rarefaction and compression of the wave. Bubbles collapse during compression of the wave and result in the formation of free radicals which are capable of degrading the carbohydrates, through dissociation of the molecules within and around the bubbles. Moreover, a mechanical effect is exerted by high speed microjets shooting out the bubbles with the ability of breaking cell walls [8].

It has been showed that extraction of hemicellulose and lignin from lignocellulosic materials can be improved by applying ultrasounds [9,10]. In addition, ultrasounds have the ability to degrade cellulose or pectin [11,12]. Ultrasonic processing of hemp and flax could also induce flexibility improvement [13,14] which is of particular interest for both geotextiles and composites applications. The ultrasonic processing impact was investigated in order to compare its effect on flax, hemp and coir yarns.

2. Materials and methods

2.1. Chemicals

3–5 dinitrosalicylic acid (128848), cellulose (C6288), xylan (X4252) and lignin (370959) were purchased from Sigma, Citrus pectin from Kalys (France) and xyloglucan from Libios (France).

2.2. Biological material

Short flax fibers and hemp fibers were provided by Depestele Group (Le Bocasse, France). Coir fibers were provided by Ecobiotex (Thizy, France). Each kind of fibers was studied as a 1 cm long yarn.

2.3. Ultrasonic processing

Ultrasonication was carried out using an ultrasonic bath (Prolabo, 45KHz, 400W) and sonication time took up to 40 h. All

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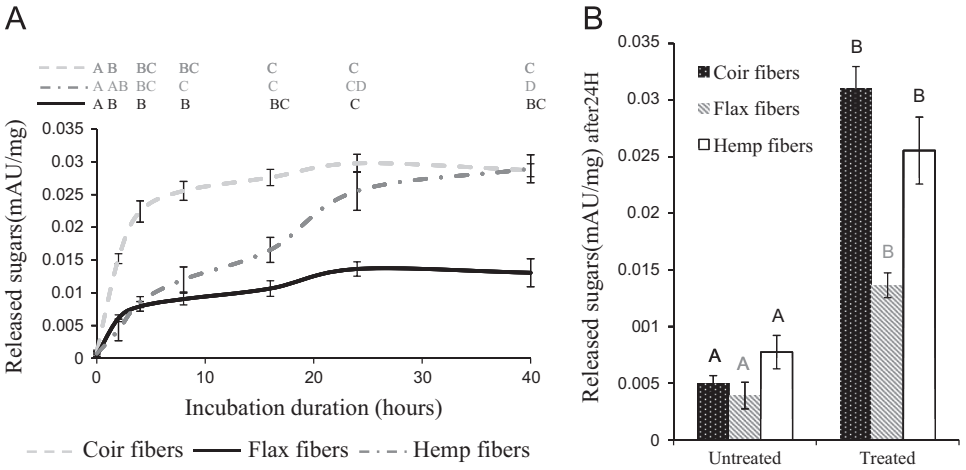

Fig. 1. (A) Kinetic of the release of sugars in extraction solvent (water) by coir, flax and hemp fibers during ultrasonic processing; (B) Release of sugars after 24 h, with or without ultrasonic processing. Measurements associated to different letters differ significantly one from another at $P < 0.05$ using Student test.

experiments were performed using samples (approximately 50 mg of dried fibers) immerged in 1 ml of distilled water without previous grinding.

2.4. Sugars quantification

Carbohydrates from the lignocellulosic fibers were quantified using 3–5 dinitrosalicylic acid (DNS) [15] after extraction performed in water. DNS acts on reducing sugar so that it forms 3-amino-5-nitrosalicylic acid whose concentration is measured by spectrophotometry at 490 nm. The results were expressed in arbitrary unit of absorbance per sample mass unit (AU/milligram).

2.5. Lignin purification and quantification

Lignin purification and quantification was carried out according to the protocol presented by Suzuki et al. (2009) [16]: lignothioglycolate derivates obtained by the action of thioglycolic acid on lignin were precipitated in HCl solution and resolubilized in NaOH solution for quantification. Lignin content was quantified by measuring absorbance at 280 nm, using a calibration curve determined from test performed on commercial lignin submitted to the same treatment.

2.6. Scanning electronic microscopy

Surface observations by SEM were carried out using a LEO 1430 VP electronic microscope at room temperature in variable pressure mode, allowing the observation of non-conductive samples with a resolution of several micrometers. In this way, the charged particles drifted away during measurement were unaffected.

2.7. ATR-FTIR analysis

Reflectance spectra were acquired using a Brucker V70 interferometer working under a dehydrated airflow in reflectivity mode, with an ATR accessory containing a gold crystal. The measurements were performed for wavenumbers situated in the middle of the infrared range (between 400 cm^{-1} and 4500 cm^{-1}). The instrumental resolution was about 4 cm^{-1} and measurements were averaged on 64 scans.

2.8. Statistical treatment of data

All data presented in this study are the mean and standard deviation of five independent replicates. Comparative statistical analysis of groups was performed using Student test. Statistical tests were considered to be significant at $p < 0.05$. Graphical and statistical treatment has been performed using Microsoft EXCEL 2010 software.

3. Results and discussion

3.1. Optimal duration of the treatment

The amounts of carbohydrates extracted from the studied lignocellulosic fibers, that represent fibers degradation, were measured by quantifying the degradation products i.e. released sugars using the DNS approach [15]. The impact of the ultrasonic kinetic on coir, flax and hemp fibers degradation was determined: results are presented in Fig. 1A. Coir fibers show the fastest ultrasonic degradation since the maximum extracted sugars quantity was reached after 4 h, whereas it took 16 h for flax fibers and 24 h for hemp fibers. As the maximum sugar release was reached for a time lower than 24 h for the three types of fiber, this duration was chosen for further experiments. The control extraction without ultrasounds remained significantly lower (Fig. 1B) indicating that, after 24 h, the release of sugars was mainly due to the ultrasonic treatment itself.

3.2. Surface properties and weight loss of fibers

Scanning electronic microscopy (SEM) revealed precipitations (probably of extracted components) on fibers surface (Fig. 2) as already observed on eucalyptus fibers by Aimin and coworkers [17]. These flake-like structures were more numerous and visible on coir fibers. However, no visible damage was observed on the global fiber's structure.

Ultrasonic processing induces a weight loss of about 9.17% for coir fibers, 4.52% for flax fibers, and 4.13% for hemp fibers (Table 1). The weight loss was found quite proportional to the published percentage of hemicellulose of 10–20% in coir fibers [1], 6% in flax [2] and 5.5–16% in hemp fibers [3].

Microscopy and weight loss data suggest a higher sensitivity to ultrasounds of coir as compared to hemp and flax fibers.

3.3. Ultrasonic impact on the composition of fibers

Cellulose, hemicellulose, lignin and pectin analysis performed on fibers and standard submitted to ultrasonic processing have been undertaken using FTIR in ATR mode and chemical quantification in