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Microwave-assisted retting and optimization of the process through chemical composition analysis of the matrix

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ABSTRACT

Pre-soaked flax stems were subjected to microwave assisted retting at different power levels and the effectiveness of microwave assisted retting was studied through the analysis of chemical compounds presented in the retted flax fibers processed from the stems. Response surface statistical design was used for this study with JMP® 10 software. Chemical analyses were performed by gravimetric methods to find out percentage of compounds. Cellulose, hemicellulose, lignin and pectin concentrations and the sugar content in the solution obtained after the treatment were subjected to analysis. Cellulose percentage in the fiber was increased significantly ($p < 0.0001$) with the increase of microwave power at various soaking levels, which proved the release of cellulosic fiber with the application of microwave energy during retting. Hemicellulose, lignin and pectin concentrations decreased significantly after microwave-assisted retting which explained higher degree of retting after the treatment. This study noted change in chemical composition of the fibers, which can be used as a tool to estimate the effectiveness of microwave-assisted retting and the results could lead to optimization of the process.

1. Introduction

Bio-fibers are widely used as a replacement for synthetic fiber in industries making speciality paper, bio-composite materials, apparel, etc., which contribute to the successful commercial production of bio-fibers (Akin et al., 2005). The environment-friendly bio-fibers are now being used instead of glass fiber in the making of almost all the composites. But bio-fibers are costly in the market when compared to other similar materials, which is due to its difficulty in processing and problems with the availability of fibers with similar physical qualities (Akin et al., 2001). Flax fiber is one of the most available bio-fiber and is cultivated all over the world (Sharma and Van Sumere, 1992b). Flax fibers are processed from its stems. The height of fully grown flax plant varies from 50 cm to 150 cm with slender stems of 1–2 mm in diameter. The life of a flax plant is 90–180 days from sowing till cultivation, which depends on the weather and soil type (Day et al., 2005). In flax stems, fiber bundles are embedded within the plant cell wall and they were arranged in concentric layers. A fiber bundle consists of 10–40 single fibers of diameter ranging from 5 μm to 40 μm (Coroller et al., 2013). These fiber bundles should be primarily loosened in order to separate the fibers. The main components that hold the fibers together inside the flax stem are waxes on the cell wall and pectin with traces of lignin in the primary cell wall. In the secondary cell wall, cellulosic microfibrils are embedded in the matrix consisting of proteins, lignin, hemicelluloses and pectin (Kadla et al., 2000). Retting is the process of the separation of fiber bundles from the non-fiber tissues in the stem (Pallesen, 1996). Plant stems are subjected to retting which enables the easy separation of fibers from it. Water retting and field retting are the most commonly used retting methods (Akin, 2013). Under-retting leads to coarse fibers with large quantities of impurities and overly retted fibers are thin with poor physical qualities (Morrison Iii et al., 1999). But now, in order to meet the strict demands of modern industries in terms of quantity, quality and consistency, new technologies like enzyme retting, steam explosion retting, and microwave assisted retting have been developed by various scientists (Akin et al., 2000; Kessler and Kohler, 1996). After retting process, fibers are dried and subjected to further processing, which includes breaking, scutching and hackling to obtain the clean fibers without any impurities.

Studies were conducted on microwave assisted retting at Bioresource Engineering Department, Macdonald Campus, McGill University and process was established by estimating the quality

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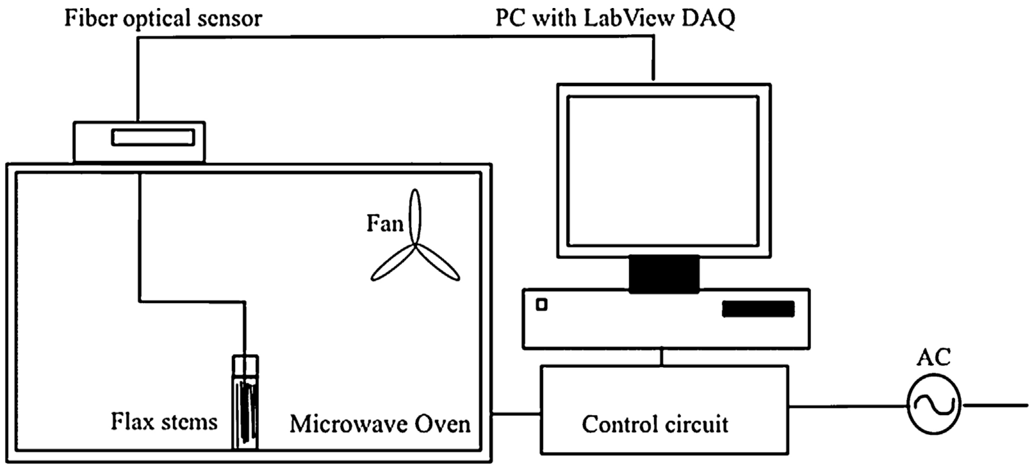

Fig. 1. A schematic of the experimental set up.

and physical properties of the retted flax fibers. Microwave energy was passed through pre-soaked flax stems, and due to the combined effect of microwave energy and heat energy released due to the dipole rotation, the fibers were separated by breaking the covalent bonds which were responsible for holding the fiber bundles together (de la Hoz et al., 2005; Yin, 2012). Flax fiber after retting process was clean and without any impurities. One of the ways of analyzing the degree of retting was to find out the chemical composition of the retted fiber. As discussed, non-retted flax fibers are closely bound by strong chemical bonds formed by pectin and non-methoxylated carboxyl groups on galacturonic acid which are often cross linked by Ca++ or other cations that form stable bridges across pectin molecules (Sakai et al., 1993). Shives are from the woody core tissues in the central region of the stem where highly lignified cells are present (Akin et al., 1996). Lignin contains recalcitrant compounds with a complex polyphenyl propanoid structure, which is a major cause for the limited degradation of plant carbohydrates as well as retting (Akin, 1989). Hemicellulose is also found in the secondary cell wall of the non-retted flax fiber. In retted and clean flax fibers, pectin, hemicellulose and lignin are removed from the primary cell wall and the percentage of cellulose content is higher than that of non-retted fibers. The changes in the composition of flax fiber of various retting levels can be analyzed and the degree of retting can be found on the basis of its chemical composition (Mooney et al., 2001). Hence the objective of this study was to find out the efficiency of microwave-assisted retting of flax stems and to optimize microwave assisted retting by analyzing the chemical composition of flax fibers obtained after various treatment combinations.

2. Materials and methods

2.1. Flax stems

Non-retted, dried flax stems of Evelin fiber flax variety were obtained from Lenaupole Fiber, Montreal and the samples were prepared by cutting 5 cm of length from the middle portion in order to fit that into a 50 ml test tube and to ensure the uniformity of experiments. The initial moisture content of flax stems was estimated by oven dry method, which was 3.93% (wet basis).

2.2. Microwave apparatus

Retting experiments were conducted by using a microwave apparatus designed in the post-harvest technology lab, Macdonald Campus, McGill University (Fig. 1). The microwave generator frequency was 2450 MHz with a variable power from 0 to 750 W. An optical fiber probe was used to measure the temperature of the flax samples (Nortech EMI-TS series, Quebec City, Canada) and temperature probes were connected to an Agilent 34970A data acquisition unit connected to a computer. A hot air supply was made possible into the microwave cavity to enable hot air to remove the moisture generated by the samples (Nair et al., 2011).

2.3. Experimental procedure

Experimental procedures of microwave assisted retting and chemical analyses are presented below.

2.3.1. Microwave assisted retting

Flax stem samples (5 g) were subjected to soaking in a 50 ml test tube. The solvent used for soaking was water, which is the most common, readily available and environmental friendly solvent in the world. Water soaking ensured removal of inorganic salts, colored materials and soil particles from the flax stems (Akin, 2013). The response surface based retting experiments were designed by using JMP® 10 software. The upper and lower limits of soaking were fixed as 12 h and 36 h where as the treatments were set between the intervals 0 to 3, where 0 was the treatment with hot water at a temperature of 100 °C for a duration of 20 min and 3 was microwave assisted retting fixing the microwave power at 3 W/g. Treatment time and temperature were kept constant throughout the study and the total number of experiments was 40.

2.3.2. Analysis of components

The flax stems obtained after different conditions of retting were dried and the fibers were separated manually. Separated fibers were cleaned to ensure that there were no impurities and cut into very short pieces to perform the chemical analyses for obtaining the concentration of cellulose, hemicellulose, lignin and pectin. The solutions obtained after various soaking and retting treatments were analyzed to estimate its sugar content to determine the efficiency of retting in terms of sugar released during the treatments. Gravimetric methods were adopted to find out the compounds, which are simple and convenient for routine analysis of compounds present in biomaterials where the compounds are separated by processes of elimination (Deschatelets and Yu, 1986). Tukey's tests were performed to analyze the statistical significance of various treatments. Neutral detergent fiber (NDF) in flax is the cell wall components such as cellulose, hemicellulose, lignin and minerals.