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Research Paper

Mathematical analysis of compound release during microwave assisted retting of flax stems

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Microwave-assisted retting was conducted at various power levels (1, 1.5 and 2 W g^{-1}) on pre-soaked flax stems (12, 24 and 36 h). The retted flax stems were dried and the fibres were separated. The amount of cellulose, hemicellulose and lignin presented in the flax fibres was established by NIR (near infrared) spectroscopy. Based on the rate of change of cellulose, hemicellulose and lignin at various levels of treatments, a kinetic model was developed and the model was validated by analysing the compositions of hemp fibres obtained from pre-soaked hemp stems at various microwave power levels. The rate of change of cellulose percentage in the model fitted with the observed values of cellulose percentage with an average R^2 value of 0.87 and an average RMSE (root-mean-square error) value of 0.0130. But in hemicellulose, the R^2 value was 0.936 and average RMSE value was 0.0135, and for lignin, R^2 value was 0.92 and RMSE value of 0.0181. The rate coefficient for all the treatments was increasing within the treatment limit, which indicated the increased reaction rate with an increase in microwave power. Validation of the model was successfully conducted by analysing the components of hemp fibres at various levels of microwave powers.

1. Introduction

Flax (*Linum usitatissimum*) is a fibrous plant where for processing the fibres are extracted from the stalk of the plant. Separation of fibres from the plant stems is usually a laborious process because of the adhesive nature of fibre bundles inside the stems with strong chemical bonds. Retting is the process of loosening of fibres from plant stems either by chemical or mechanical means (Akin, 2013). Various retting methods include water retting, dew retting, enzyme retting, steam explosion retting, microwave assisted retting etc. Fibres attached to the stems with a close matrix contain hemicellulose, pectin and lignin (Akin, Dodd, & Foulk, 2005). In water retting, aerobic bacteria attack the plant stems by eating down the soft part of plant stem and to break down the strong chemical bonds and release the cellulosic fibres from the plant-fibre matrix (Day et al., 2005). The breaking of bonds leads to the release of compounds like hemicellulose, lignin and increase in cellulose content in the fibres. The fibre bundles are embedded within the plant cell and they are attached to each other strongly with various layers. A fibre bundle consists of 10–40 single fibres of length from 25 to 150 mm, which varies with variety. These fibres are loosened due to retting. The main components that hold the fibres together inside the flax stem are waxes on the

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Nomenclature

R^2	R squared value
RMSE	Root-Mean-Square Error
W g^{-1}	Watts per gram
NIR	Near Infrared
X	The percentage of compound present in the fibre
X_i	The percentage of compound present in the untreated flax fibre
t	The time (min)
k	Constant dependent on the heterogeneity of the material
n	Constant dependent on the nature of the reaction
D	The dielectric loss tangent of the reagent
b	Constant dependent on the dielectric loss tangent of the reagent used

cell wall, pectin substances with traces of lignin inside the primary cell wall, while secondary cell wall consists of cellulose. The cambium cells separate fibres from shives (Kadla & Gilbert, 2000). The hemicellulose branches help to bind the microfibrils to one another and to other matrix components, particularly pectin. This cross links the cellulose microfibrils into a network of tough, fibrous molecules and is responsible for the mechanical strength of plant cell walls. Pectin amounts are often low in flax fibres, but they are strategically located within the plant tissues like cement for bricks (Akin, Gamble, Morrison Iii, Rigsby, & Dodd, 1996; Nair, Rho, Yaylayan, & Raghavan, 2013). Lignin is another non-cellulosic component of flax plant with a complex polyphenyl propanoid structure, which helps to oppose microbial degradation of plant carbohydrates tissues. The decomposition of the complex chemical bonds formed by these compounds results in the loosening of fibre bundles and hence further processing to achieve separation of fibres will be easier. Analysis of compounds present in fibres before and after retting is used as a method to explain the effect of retting. Microwave assisted retting of pre-soaked flax stem was conducted at Bioresource Engineering department, McGill University, and the compounds were analysed by the NIR spectroscopy method. The change in the composition of compounds showed a trend with respect to microwave power, soaking and treatment time.

There have been several studies conducted on the modelling of the bioconversion of lignocellulosic materials. Kinetic modelling is studied widely to investigate the compound analysis of biomaterials, while some of the researchers used non-kinetic or fuzzy inference models in complex systems like conversion of cellulosic biomass. Sun and Cheng (2002) and Mosier et al. (2005) have summarised the modes of actions, and the advantages and disadvantages associated with different biomass pretreatment methods to release lignin and hemicellulose (Mosier et al., 2005; Sun & Cheng, 2002).

Here, an attempt has been made to develop a mathematical model, which relates the rate of change of composition in the fibre with retting and the factors like microwave energy, treatment time and pre-soaking status. The objective of our study is to model the effect of microwave assisted retting on the relative changes in the components like cellulose, hemicellulose and lignin.

2. Materials and methods

2.1. Microwave-assisted retting

Non-retted flax stems were supplied by Lanaupôle Fibres, Montreal, Canada. Flax stem samples were prepared by cutting them in equal length of 80 mm. For experimental similarity, only middle portions of the stems were used. Flax stems were water-soaked for 12, 24 and 36 h intervals. Microwave-assisted retting was performed on the pre-soaked flax stems at various time intervals from 5 to 20 min with varying power levels of 1, 1.5 and 2 W g^{-1} (Nair et al., 2013, 2014). The flax stems after retting were dried and fibres were separated manually. The experiments were repeated 3 times at each level. The experimental design is as shown in Table 1.

The flax stems after retting were oven-dried and the fibres were separated manually using a hackling comb. Further compositional analyses were conducted by using the flax fibres obtained by the method described above (Nair et al., 2013).

2.2. Near infrared (NIR) analysis

The flax fibres were subjected to compositional analysis using an NIR spectrometer. The model used was Nicloet Antaris FT-NIR analyser (ThermoFisher Scientific Inc., Waltham, MA, USA). The percentages of compounds (cellulose, hemicellulose and lignin) were determined by comparing the spectrum of the sample with a calibration curve. A calibration curve was made by the using the values of the compounds (cellulose, hemicellulose and lignin) obtained by chemical extraction methods using principal component analysis (Nair et al., 2014). The NIR spectrometer results were used to analyse the changes in the composition of lignin, hemicelluloses (impurities) and cellulose (to analyse strength).

2.3. Kinetic model studies

The general form of kinetic model used in our study is that adopted from the work done by Dang and Nguyen (2006, 2007), who proposed a kinetic model to describe delignification and

Table 1 – Experimental design for microwave assisted retting of flax stems.

Factors	Levels	Description
Pre-soaking time	1	12 h
	2	24 h
	3	36 h
Microwave retting power	1	1.0 W g^{-1}
	2	1.5 W g^{-1}
	3	2.0 W g^{-1}
Microwave retting time	1	0 min
	2	5 min
	3	10 min
	4	15 min
	5	20 min