



# Characterization of radio frequency assisted water retting and flax fibers obtained



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## ABSTRACT

Water retting used to degrade pectin in the bast of plant, release and separate fibers normally takes one to two weeks. In this study, radio frequency (RF) was introduced to enhance the retting efficiency in different stages of water retting, and different RF treatment temperatures and durations were used. The pectin loss in phloem part of the RF-treated flax straws, defined as degumming rate, was measured to evaluate retting efficiency besides Fried test. The color, linear density and tensile properties of the resulting fibers were measured. The relationships between retting results and process parameters were established using the response surface methodology, and the way RF functioned in retting process was studied. It was found that RF best improved retting quality of the 2-day, and 6- and 10-day water retting at 40 °C and 90 °C for 60 min, respectively.

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## 1. Introduction

Flax (*Linum usitatissimum* L.) is one of the oldest fiber crops in the world. Flax fiber belongs to the natural bast fibers and is most frequently used in the higher value-added textile markets (Faruk et al., 2012) and paper-making industries, and nowadays is also widely used in the bio-composites (Bourmaud et al., 2015; Ehresmann, 2012) since flax fiber is a renewable and biodegradable natural resource with superior mechanical properties.

Flax fiber is found in the phloem region between the bark and inner core tissues, and exists as fiber bundles stuck and surrounded by pectin. The process that frees the fibers from non-fiber components, especially the pectin, separates fibers into smaller bundles and single fibers (Akin et al., 2000) and thereby facilitates further processing is called retting or degumming. Retting is thus important in processing flax since its quality is a main concern for both industries incorporating natural fibers into production (Fouk et al., 2011) and other applications which value the pectin removal of biomass (Deng et al., 2012; Pakarinen et al., 2012). Water retting is one of the traditional retting methods and achieved mainly by the

anaerobic bacteria, normally requiring 14 days to be completed. Although water retting is easy to operate and can produce high quality fibers, the long duration, and stench and water pollution generated have made this method less attractive (Tahir et al., 2011). In order to eliminate these problems while guaranteeing the quality at the same time, many efforts have been put into optimizing the traditional retting methods by adopting new formulations and new bacteria/enzymes (Akin et al., 2007; Das et al., 2012), and into developing new retting processes that apply and/or combine different technologies (Konczewicz et al., 2013; Lavoie and Beauchet, 2012; Nair et al., 2014).

The radio frequencies range from 300 kHz to 300 MHz in the electromagnetic spectrum, and RF dielectric heating has applications for drying, baking and pasteurizing in the food, textile and paper processing industries (Venkatesh and Raghavan, 2004). In the RF heating system, the material to be treated is placed between two electrodes where the RF generator creates an alternating electric field which causes polarization, i.e., the continuous reorientation of the polar molecules in the material to face the opposite poles at 27.12 MHz where RF works, the resulting friction among the molecules causes the material to heat up rapidly (Orsat, 1999). RF works well with large quantities because of its long wavelength in the order of 10 m.

The main purpose of the retting process is the removal of pectin. Pectin is a complex of acid polysaccharides with a backbone of galacturonic acid residues linked by  $\alpha$  (1–4) linkages and contains

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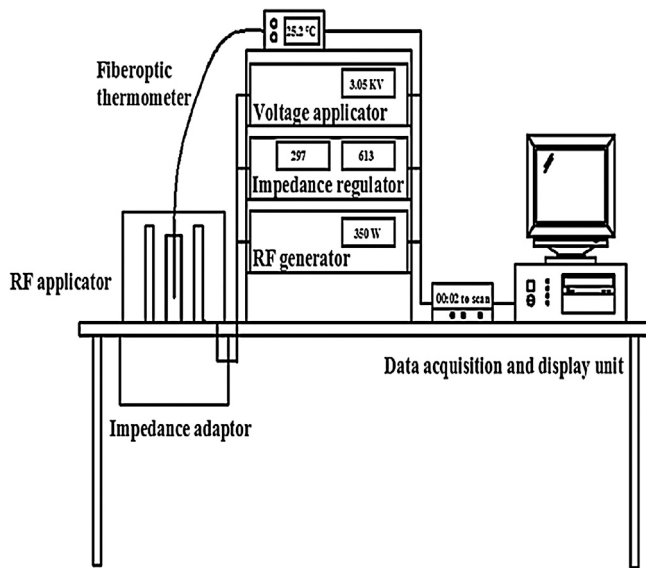


Fig. 1. RF heat treatment system.

pectinic acid as the main component (Kashyap et al., 2001). In water retting, some soluble components of pectin are first dissolved with other soluble materials, the rest are degraded by enzymes generated from microorganisms then dissolved as retting progresses. Pectin is also categorized as one of the anionic polysaccharides in the cell wall (Schols and Voragen, 1996), so there are regions in pectin which can be polarized, which makes RF function possibly in the retting process; in addition, polar water molecules introduced into flax straws by retting can allow more polarization movement, which also help disintegrate pectin.

The aims of this study were to improve the retting efficiency by incorporating RF dielectric heating technology into water retting and provide some useful information and references for further research into using RF heating technology to shorten retting time upon the point generating pollution. Therefore, the retting efficiency and the properties of flax fibers obtained were investigated, and the effects of RF treatment on these two issues were elucidated as well. The results of this study would also be of use for tailoring properties of natural bast fibers and for other applications that require pectin removal.

## 2. Materials and methods

### 2.1. Materials

Non-retted raw flax straws of Evelin fiber flax variety were kindly supplied by the National Research Council, Montreal, Canada, which were harvested from the research farm of Lanaupole Fiber, Quebec in 2011. To ensure the uniformity in all experiments, the middle part of the stem was chosen and cut into 100 mm length with a diameter of around 2.5 mm.

### 2.2. Experimental apparatus

The RF system used in this study was designed at Post Harvest Technology Lab, Bioresource Engineering Department of McGill University in Canada, is shown in Fig. 1. The system includes a power generator, a RF applicator, a data acquisition unit and control circuits. The generator operated at a frequency of 27.12 MHz and with a maximum power output of 600 W. The applicator adopted standard parallel electrodes spaced 60 mm apart, with a cylindrical Teflon container in between. To treat flax straws with RF, a

Table 1  
Experimental combination of RF assisted water retting of flax straws.

Run	Actual values of factors		
	Water retting days (day)	RF treatment temperature (°C)	RF treatment time (min)
1	2	40	10
2	10	40	10
3	2	40	60
4	10	40	60
5	2	90	10
6	10	90	10
7	2	90	60
8	10	90	60
9	2	65	35
10	10	65	35
11	6	65	10
12	6	65	60
13	6	40	35
14	6	90	35
15	6	65	35
16	6	65	35
17	6	65	35
18	6	65	35
19	6	65	35
20	6	65	35

glass tube containing the sample was put into the container and fixed by its rim, and an optical fiber sensor probe (NorTech EMI-TS series, Quebec City, Canada) was inserted directly into the sample to measure temperature. All the inputs, including power, voltage and sample temperature etc., were sent to the data acquisition unit (Agilent 34970A, Santa Clara, USA), which was connected to a computer.

### 2.3. Experimental procedures

#### 2.3.1. Experimental design

Three factors, water retting duration, RF treatment temperature and treatment time at the given temperatures, with each factor having three levels, were studied in this work. A central composite face-centered (CCF) method was used to design the experiment and to observe how the factors would affect the whole retting process. The experimental design is shown in Table 1. There were 20 runs in the table, including 6 replicates at center points (the treatment of 6 d-65 °C-35 min). Each run at non-center points was repeated twice. Design Expert 7.0.0, (StatEase Inc., USA) was used to generate the response surfaces.

#### 2.3.2. Water retting

Flax straw samples, 7 g each, were rinsed with distilled water then crushed twice using a 700 g rod to crack the stems for better retting performance (Henriksson et al., 1997). Subsequently, each sample was placed into a 250 ml glass bottle and distilled water was added in the ratio of 1:33. The bottles without lids were then placed at room temperature for various durations.

#### 2.3.3. Radio frequency treatment

After the required duration of water retting was completed, the flax straws were treated by RF. Each sample of flax straws was weighed then separated into two groups, one was rinsed and dried in an oven for measuring pectin content of water-retted flax, and another with a mass of around 16 g was subjected to RF treatment together with 64 g retting water at different temperatures for different periods of time.

To make sure the times required to heat the straws from room temperature to the different desired temperatures were the same, different output power levels were used. For 40 °C, 200 W was applied; for 65 °C, 350 W and for 90 °C, 450 W used, and all the RF

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