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Dielectric characterization of corn stover for microwave processing technology

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1. Introduction

Corn is one of the major agricultural crops in the United States [1] and Canada [2] and in several other countries around the globe. Large amount of corn residues is generated every year which either needs to be utilized or disposed of safely. It also attracted a lot of attention as a feedstock for bioenergy production especially in North America [3–5]. One of the potential utilizations of biomass materials such as corn is the production of fuels and chemicals [6,7].

Several pyrolysis technologies such as fixed bed, fluidized bed, transported bed, augur or screw, ablative, rotating cone and chain gates [8] are available in order to process corn residues into value added products. Most of these technologies use traditional type of heating methods in which the heat is transferred from the surface towards the center of the material by conduction and convection. Alternative to traditional heating, microwave heating can be beneficial in terms of saving time and energy because the microwave energy is transformed directly into heat within the material [8,9]. Generally, microwave provides rapid and selective heating [10] with the overall system efficiency of 80–85% [11] which is reasonable compared to conventional heating techniques.

Microwave pyrolysis process has attracted a considerable attention during the past decade because of several advantages it offers as compared to other methods [12]. This technique has been reviewed throughly by [13] and has been applied to different types of biomass materials such as agricultural [14] and forestry wastes [15]. As the material undergoes microwave pyrolysis, the dielectric properties of

$A \hspace{0.1in} B \hspace{0.1in} S \hspace{0.1in} T \hspace{0.1in} R \hspace{0.1in} A \hspace{0.1in} C \hspace{0.1in} T$

The aim of this paper was to investigate the microwave dielectric properties of corn stover at 915 and 2450 MHz from room temperature to 700 °C under nitrogen (N₂) environment. Cavity perturbation method was used to measure the dielectric constant and loss factor. The heating process was divided into three distinct stages, namely drying, pyrolysis, and char stage. Dielectric properties during drying and pyrolysis stages were inversely proportional to the temperature, whereas directly proportional to the temperature in char stage. The maximum microwave penetration depth of 8.5 m was obtained at 392 °C and 915 MHz, just at the end of the pyrolysis reaction zone. The experimental data were fitted using regression fit and based on this the dielectric properties model related to the temperature was developed.

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the material change with the temperature [16,17]. Hence, it is important to know how the dielectric properties and penetration depth of microwaves in the material change according to the temperature [11]. This knowledge is crucial to design microwave processing systems [18].

Investigation of dielectric properties for biomass materials has been reported in the literature such as rubber wood [19], oil palm fibers [20], rice husk, rice straw, kenaf, and resin [21], peanut hull pellets [22], woody biomass [23,24], hardwood [25], Aleppo pine, Holm oak and Thuja burl woods [26], coal [27], softwoods (black spruce, balsam fir, and tamarack) [28], oil palm biomass [29], Jordanian oil shales [30], empty fruit bunch biomass [31], switchgrass [16] and wood and wood-based products [32].

Although extensive research work has been carried out on microwave pyrolysis of corn stover [33–35], there is a clear lack of fundamental information on its dielectric properties during pyrolysis. Thus, the main objective of this study was to determine the microwave absorption capacity of corn stover in addition to dielectric properties (dielectric constant and loss factor) and penetration depth. The dielectric properties were investigated from room temperature to ~700 °C and at two commercial microwave frequencies (915 MHz and 2450 MHz). Lastly, the microwave absorption behavior during pyrolysis was fitted to a numerical function in order to develop dielectric properties model.

2. Materials and methods

2.1. Materials

The corn stover studied had a composition of 50% stalks, 22% leaves, 15% cob, and 13% husk [36,37]. It did not include the crown or surface roots. Table 1 shows the proximate and ultimate analysis of corn stover.

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Table 1Proximate and ultimate analysis of corn stover.

| Parameters | wt.%, dry basis |
|------------------|-----------------|
| Volatile | 76.2 |
| Fixed carbon | 17.0 |
| Moisture content | 1.5 |
| Carbon | 46.67 |
| Nitrogen | 0.02 |
| Hydrogen | 6.01 |
| Sulfur | 0.02 |
| Oxygen | 47.28 |

The sample was prepared by grinding in a Wiley Mill to particle size less that 2 mm, and then the grinded sample was dried at 105 °C for 15 h in an oven. The dielectric properties were investigated on pelletized samples which were formed by uniaxial press in a die lined with tungsten carbide at ~135 MPa. Three corn stover pellets with almost similar dimension were stacked on top of each other to form one test sample with diameter (3.700 \pm 0.050, mm), length (15.200 \pm 0.050, mm), mass (0.146 \pm 0.002, g), initial density of 0.890 \pm 0.050 (g/cc), and final density of the sample at the end of experiment was 0.300 \pm 0.040 (g/cc). The purpose of pressing the samples into pellet form was to reduce the air fraction in the pellet, thus increasing the accuracy of the data.

2.2. Measurement of dielectric properties

The dielectric response of a material is commonly presented as permittivity (ε), which is be given by:

$$\varepsilon = \varepsilon_0 \ \varepsilon_r = \varepsilon_0 \left(\varepsilon_r' - j \ \varepsilon_r'' \right) \tag{1}$$

where, ε_0 is permittivity of free space (8.854 × 10⁻¹² F/m), ε_r is complex relative permittivity, and *j* is imaginary unit ($j^2 = -1$). As indicated by Eq. (1), the complex relative permittivity is comprised of two components: $(\varepsilon_r^{'})$, the real part (which is generally known as the relative dielectric constant and it is a measure of the ability of the dielectrics to store electrical energy) and $(\varepsilon_r^{''})$, the imaginary part (which is also called the relative dielectric loss factor and represents the loss of electrical energy in dielectrics).

The dielectric properties (ε_r ["] and ε_r [']) of the samples were measured during pyrolysis using the cavity perturbation technique. The samples were heated to the desired temperatures with a ramp rate of 5 °C/min, starting at ~30 °C and progressing in ~25 °C steps up to 150 °C, then 50 °C steps to ~700 °C. The pyrolysis reaction was done with the sample

2.3. Penetration depth

The penetration depth (D_p) is a very important factor in the design and scale-up of a microwave heating system. It is defined as the depth in the material at which the power carried by a forward-traveling electromagnetic wave of the specified frequency falls to 1/e of the value just inside the surface. The penetration depth can be calculated using the following equation [11] (λ_0 is the microwave wavelength in free space):

$$D_{p} = \frac{\lambda_{0}}{2\pi (2\varepsilon_{r}^{'})^{1/2}} \left\{ \left[1 + \left(\frac{\varepsilon_{r}^{''}}{\varepsilon_{r}^{'}}\right)^{2} \right]^{1/2} - 1 \right\}^{1/2} .$$
(2)

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3. Results and discussion

The thermal degradation of corn stover was divided into three distinct stages, i.e. drying (from room temperature to ~200 °C), pyrolysis (from ~200 °C to ~450 °C), and char stage (from ~450 °C to ~700 °C).

3.1. Dielectric properties in drying and pyrolysis stages

The relative dielectric constant and loss factor of corn stover were almost independent of the microwave frequency in drying and pyrolysis stages as shown in Fig. 1. However, the dielectric properties decreased sharply from room temperature to 75 °C and then decreased slightly until 200 °C due to removal of free and bound water [38-40]. This water in form of moisture is a strong absorber of microwave. The dielectric properties further dropped between 250 °C and 300 °C, then while it remained constant until 450 °C. During this temperature range, thermochemical decomposition of biomass takes place and the volatile matters are released. The polar components released during "thermal cracking" of biomass are immediately volatized, therefore they might not contribute to an increase in dielectric properties [17,40]. Thus, according to Rajeshwar and Inguva [36], when the temperature reaches a point where the polar molecules in the organic fraction start to mobilize and due to this the dielectric properties will begin to change. Typically, corn stover can be considered as a poor microwave absorber during drying and pyrolysis stages. Hence, in order to induce pyrolysis reaction under microwave system, certain high microwave absorbing materials



Fig. 1. Dielectric properties of corn stover against temperature under nitrogen environment at 915 MHz and 2450 MHz with initial density of 0.89 \pm 0.05, g/cc; (a) relative dielectric constant and (b) relative loss factor.

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