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Moisture content and bulk density prediction using dielectric properties for switchgrass and corn stover

by

Augusto César Magalhães de Souza

A thesis submitted to the graduate faculty in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Major: Agricultural and Biosystems Engineering

Program of Study Committee: Stuart Birrell, Major Professor Steven Hoff Brian Steward

Iowa State University

Ames, Iowa

2015

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DEDICATION

"What is true is that if you're not prepared to be wrong, you'll never come up with anything original."

- Sir Ken Robinson, Ph.D.

When I moved back to Iowa State University during the harsh winter of 2013, I left back in Brazil my family, my friends, my brothers, and many things that I love. Whenever I can communicate with them, I usually say that these are "small" sacrifices for a bigger reward in the future. Maybe, I am wrong, but I know I will always have their blessings to find out the truth. This work is dedicated to them!

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ABSTRACT

Dielectric measurements between 0.1 to 10 MHz were obtained using an HP 4192A impedance analyzer for switchgrass and corn stover. For each material, the measurements were obtained at four moisture content levels and three bulk densities. Dielectric properties of these materials could be calculated based on admittance readings and each variable was significant for dielectric constant and loss factor measurements. Dielectric variables were used to predict moisture content and bulk density for both materials. For switchgrass, moisture content was predicted with $R^2 = 0.94$ and RMSE = 0.052%, while for density was predicted with $R^2 = 0.90$ and RMSE = 0.0315%, whereas, for bulk density, the regression model had a $R^2 = 0.87$ and RMSE = 0.0068 g/cm³. The results indicated that dielectric measurements have good potential for predicting moisture content and bulk density although further investigation is required for a wider range of frequencies, moisture content, and bulk density levels.

1. Introduction

Moisture content (M.C.) and bulk density (B.D.) are important factors that affect harvesting, drying, and storage of biomass in production systems. During grain harvest, the kernel can be damaged if it is too wet, and its quality decreased. Additionally, damaged grain is more susceptible to fungal infection, leading to spoilage during storage and, consequently, becoming non-viable for human or animal consumption (Nelson and Trabelsi, 2004). Corn stover is typically harvested around 45% of M.C.; however, it has to be dried close to 20 - 25% in order to be consumed as supplemental feed for beef cattle and non - lactating dairy cattle. In addition, the moisture content of biomass materials such as corn stover and switchgrass has a significant effect on dry matter losses during storage and biomass supply chain costs (Shinners et al., 2007).

Standard methods for determining M.C. for grains and forage require weighing small samples and drying for a long period in controlled environment conditions (ASAE, 2012; ASAE, 2012). These procedures damage the sample and requires too much time, which makes impractical for on-line and real time determination of M.C. Therefore, instruments with electronic sensing have been developed to be a commonly-used means for agricultural products characteristics determination. Numerous instruments are based on the relationship between moisture content and electrical conductivity of biological a material. These systems measure the dielectric properties of the biomaterials and predict characteristics of a sample mass using a calibration model. However, different calibrations are necessary if different materials are being tested since dielectric properties depend on composition of the materials and amount of water present in the sample mass (Nelson, 2012).

Switchgrass (*Panicum virgatum*) is a native North American perennial herbaceous plant. Its primary use is associated to forage production, but switchgrass has been showing great potential as a sustainable energy crop, including the production of second-generation ethanol. According to McLaughlin and Kszoz (2005), switchgrass has higher biomass yield and broader adaptability than other cultivars tested, reaching average yields of 20.0 Mg.ha⁻¹ in some locations inside the United States. They also showed that carbon sequestration could occur under soils with switchgrass crops, increasing soil productivity and nutrient cycling. As a result, switchgrass was chosen as a model bioenergy crop by the Biofuels Feedstock Development Program (BFDP) funded by the U.S. department of Energy (Wright and Turhollow, 2010).

In 2014, corn production in U.S. reached approximately 14.2 billion bushels on 83.1 million acres of cultivated area (USDA, 2015). The amount of corn stover produced in a typical crop depends with type of soil, topography, and crop rotation, however, using a usual crop index of 0.45 (USDA, 2012), the annual estimated production is around 17.4 billion bushels of stover. Kadam (2003) estimated that only 76 - 82% of above ground-biomass could be harvested on a sustainable basis with current equipment and no-till farming, which results from 335.3 to 363.2 million tones at 15.5% of moisture content. The same study states that ethanol yield from stover can reach 80 gallons per ton of dry material. EIA (2014) predicts that energy from biomass should increase 1.4% until 2040

to supply human needs, thus, new techniques and more biomass feedstock should be available to reach this goal.

Even though corn stover and switchgrass are widely studied as feedstocks for biofuels and animal feed, there is a gap in the knowledge about how these materials interact with an electric field. Specifically, the dielectric properties in a wider range of frequencies under influence of different moisture content and bulk densities that have not been characterized. The purpose of this study was to, quantitatively, determine the dielectric properties of switchgrass and corn stover under the influence of different electrical signal frequencies, and, based on these relationships; predict moisture content and bulk density of these materials.

Nelson and Trabelsi (2004) showed that the dielectric properties of wheat, soybean, and corn are highly dependent of frequency, moisture content, bulk density, and temperature. Thus, in this study, the admittance of the switchgrass and corn stover with different levels of moisture content and bulk density was measured to determine the dielectric properties of these materials. Based on these characteristics, calibration models could be developed to predict moisture content and bulk density.

Improved measuring instruments and techniques can be developed by determining the dielectric properties of switchgrass and corn stover across a wider range of frequencies. In addition, microwave pyrolysis is a technique that is highly influenced by dielectric properties (Motasemi, 2014); thus, knowing more about the properties of the materials, would help understand the behavior of switchgrass and corn stover during pyrolysis. The measuring system consisted of a Hewlett – Packard 4192A LF impedance analyzer connected to a sample holder via an HP 16095 probe fixture. The sample holder was a parallel plate capacitor where the plates represented the electrodes of this capacitor. From the impedance analyzer, readings of the admittance of the system were taken to calculate the dielectric properties of the sample mass. Based on the calculated dielectric constant and loss factor, calibration models could be obtained for moisture content and bulk density for both switchgrass and corn stover.

1.1. Theory

Dielectric properties characterize the interaction between a material and an electrical field. They are used in several applications, including moisture sensors based on radio waves and microwaves. One of the properties is permittivity (ϵ) that measures the resistance of a material to form an electric field. Permittivity is a complex variable, where the real part corresponds to the dielectric constant ϵ ' and the imaginary part ϵ '' is the dielectric loss factor.

$$\boldsymbol{\varepsilon} = \boldsymbol{\varepsilon}' + \boldsymbol{j}\boldsymbol{\varepsilon}'' \tag{1.1}$$

The dielectric constant is associated with the energy storage of the electric field in the material while the dielectric loss is associated with energy dissipation in the material in the form of heat. For grains, both dielectric constant and loss factor are not only a function of the moisture content (M.C.); they are, also, dependent of the electric field frequency, the temperature, and the bulk density (B.D.) of the material (Nelson and Trabelsi, 2004). The loss tangent is defined as:

$$\tan(\delta) = \frac{\varepsilon''}{\varepsilon'} \tag{1.2}$$

Where δ is the loss angle. For a parallel-plate capacitor, capacitance is determined from the dielectric constant, area of the sensor plates, A, and the distance between the sensor plates, d, as it can be seen in figure 1.1.

$$C = \frac{\varepsilon' \times A}{d} \tag{1.3}$$

The dielectric constant is composed of a relative material dielectric constant, ϵ'_r , and the permittivity of free space, ϵ_0 , as shown,

$$\boldsymbol{\varepsilon}' = \boldsymbol{\varepsilon}_0 \times \boldsymbol{\varepsilon}_r' \tag{1.4}$$

where ε_0 is equal to 8.854x10⁻¹² F/m. The free space capacitance, C₀, for a particular parallel plate capacitor is determined as follows, where the relative dielectric constant of air, ε'_{air} , is equal to one.

$$C_0 = \frac{\varepsilon_0 \times \varepsilon'_{air} \times A}{d} \tag{1.5}$$

A material's capacitance, C_{mat} , is used when the sensing cell is filled with that material. Similar to air, the dielectric constant of the material, ϵ'_{mat} , will be used on equation 1.5 instead of ϵ'_{air} .



Figure 1.1. Representation of a capacitor

Impedance is defined as the effective opposition of an electrical circuit to alternating current when a voltage is applied. Impedance is a complex variable where the real part is the resistance of the circuit and the imaginary part is the reactance. The complex reciprocal of impedance is the admittance where the real part, G, is the conductance and the imaginary part, B, is the susceptance.

$$Y = G + jB \tag{1.6}$$

The real part of the admittance is defined as,

$$G = 2\pi f C_0 \varepsilon'' \tag{1.7}$$

Where f is the frequency, C_0 is the capacitance of the empty cell, and ϵ '' is the dielectric loss factor of the material. Similar, the imaginary part is related to the capacitance as shown,

$$B = 2\pi f C_{mat} \tag{1.8}$$

Combining equations 1.5, 1.7, and 1.8, dielectric constant and loss factor can be determined by the following equations,

$$\varepsilon' = \frac{d \times B}{2\pi f \varepsilon_0 A} \tag{1.9}$$

$$\varepsilon'' = \frac{G}{2\pi f C_0} \tag{1.10}$$

1.2. Objectives

The goal of this study was to determine the dielectric constant and loss factor of switchgrass and corn stover by measuring permittivity at different moisture content and bulk density levels. In addition, the study had an objective to develop a model capable of predicting moisture content and bulk density of these biomaterials based on the dielectric variables obtained. The specific objectives include:

- Determine dielectric constant and loss factor of corn stover and switchgrass based on readings of admittance and conductance at different moisture content and bulk density levels.
- 2. Use standard partial square regression to develop a model to predict dielectric constant and loss factor using moisture content, bulk density, and frequency as the independent variables.
- 3. Develop different models capable of predict moisture content and bulk density of corn stover and switchgrass based on dielectric properties measurements.
- 4. Select the best calibration models for the materials under test on this work for moisture content and bulk density prediction.

2. Literature Review

Early investigations into the measurements of dielectric properties for agricultural products focused on dehydrated carrots and dielectric properties were determined as a function of moisture content, temperature, frequency, density, and particle size. The frequency range used was between 18 kHz to 5 MHz. Carrots with moisture between 6 – 8% presented, small increases in dielectric constant compared to those with higher moisture content. In addition, signals with high frequencies showed better results in wetter materials. Hence, the higher frequencies are desirable to determine both dielectric constant and specific conductivity (Dunlap and Makower, 1945). There was also an increase in the dielectric properties with higher bulk density for all frequencies.

Several grains and seeds were tested to obtain their dielectric properties in frequencies varying from one to 50 MHz using a Boonton Q-meter and a coaxial testcondenser with a variable air capacitor (Nelson, 1965). In general, it was observed that dielectric constant increased with moisture content for all frequencies. However, a greater change was observed for both dielectric constant and loss factor as moisture content variation.

Moisture and frequency, followed by bulk density are the most influential factors on the permittivity in grains (Nelson, 1982). Nelson and Stetson (1976) studied the dependence of dielectric properties of seven varieties of hard red winter wheat with different moisture content (2.7 - 23.8%) and frequencies (250 Hz - 12.1 GHz) ranges using a coaxial sample holder. The values for dielectric constant varied from 100 for the lowest frequencies and around 2 for higher frequencies, while the loss factor vaired from 1000 to 0.1. Thus, the value for the real part of permittivity of wheat decreased with increasing frequency and decreasing moisture. Dielectric loss factor followed the same trend overall, although some exceptions were reported.

Lawrence (1993) developed "density-independent" moisture content equations of hard red winter wheat of dielectric constant at 10 MHz and loss factor at one MHz. The study showed that the equations performed better for higher moisture content materials than lower ones. However, they still presented reliable results measuring wheat M.C., even though only static measurements were taken.

Continuous measurements of several types of grain M.C. were made utilizing two kinds of electrodes: concentric and parallel plates. Zoerb (1993) reported that there were no problems with the variation of bulk density in the observed results under laboratory conditions. Regression models relating dielectric measurements to moisture content had coefficients of determination varying from 0.85 to 0.99 for parallel rectangular electrode plates.

Electrical resistance and resistivity were measured to determine an equation to predict moisture content of cotton lint (Byler, 1998). A number of test cylinders were constructed with cross-sectional varying from one to eight cm² with a 1 cm sample thickness. The electrical resistance varied from 5000 M Ω to 300M Ω for the M.C. range of 3% to 10% w.b., respectively, for samples with one cm² cross-section area. No significant results were found for materials with M.C. below 3%, and it was expected that the area to be inverse and linearly proportional to the resistance; however, the results were different from the theoretical analysis of resistance.

Eubanks and Birrell (2001) successfully developed a static multiple frequency parallel plate capacitor to determine the moisture content of hay and forages with unknown density, material volume, and material composition. Linear regression models were obtained to represent the moisture content of these materials, using results of sequential multiple frequencies at a rate of one frequency every three seconds. In general, the frequency range was from 900 kHz to 13 MHz, except for clover that used a 5 MHz frequency signal. The coefficient of determination varied from 0.65 to 0.95. The sensor predicted moisture content independent of density or the amount of material although calibration was necessary for each crop tested.

Nelson and Bartley (2001) studied the dependence of frequency and temperature over dielectric properties for some food materials. For this case, a temperature control assembly was design to be used with an open-ended coaxial line probe. The three materials tested were whey protein gel, whole-wheat flour, and apple juice. In general, both dielectric constant and loss factor increased together with temperature. However, there was a reduction on the permittivity at higher signal frequencies. For apple juice, there was an increase in the loss factor with temperature for higher frequencies. The author attributed this to the influence of ionic conduction in the material.

Simultaneous multiple frequency signals is a better alternative to sequential signals. This approach guarantees that the reading from each signal is passing through the same portion of flowing sample mass eliminating delay between signals. Based on this,

Benning et. al. (2004) determined that using a system of simultaneous multiple frequency signals is capable to measure hay and forage moisture content. In addition, extracted frequencies from a multi-frequency signal provided the same signal information as sequential scanned frequency signals. In this study, a multi-frequency signal composed of four frequencies took 326.8 microseconds whereas the bale moved only 3 mm.

Measurements of complex impedance of peanuts were taken to obtain the M.C. of small samples (Kandala e. al., 2007). For this study, a portable electric instrument was developed that measures the complex impedance of a parallel-plate capacitor with the sample between its plates. It is a high accuracy method, although the frequency range that it can be used is limited by circuit parameters. By using the radio frequency impedance method, the moisture content was estimated rapidly and nondestructively. The method applied is proper for small amounts of material and could be extended to other products depending on calibration.

Salema et al. (2013) measured the dielectric properties of oil palm biomass and biochar using a coaxial probe attached to a network analyzer with frequencies ranging from 0.2 to 10 GHz. The dielectric constant was inversely proportional to the frequency. However, the authors report that the loss factor was directly proportional to the frequency and did not obey the Debye equations, defined by:

$$\boldsymbol{\varepsilon}' = \boldsymbol{\varepsilon}_{\infty}' + \frac{(\boldsymbol{\varepsilon}_{s}' - \boldsymbol{\varepsilon}_{\infty}')}{1 + \omega^{2} \tau^{2}}$$
(2.1)

$$\varepsilon'' = \varepsilon'_{\infty} + \frac{(\varepsilon'_s - \varepsilon'_{\infty})}{1 + \omega^2 \tau^2}$$
(2.2)

which ε'_{∞} is the limiting high frequency relative dielectric constant, ε'_{s} is the limiting low frequency relative dielectric constant, ω is the angular frequency, and τ is the relaxation time.

The dielectric properties of switchgrass were measured at 915 and 2450 MHz from room temperature to around 450 °C under a nitrogen (N₂) environment (Matasemi et al., 2014). Dielectric constant and loss factor were measured during pyrolysis using the cavity perturbation technique. In general, the dielectric properties decreased during the drying and pyrolysis phases as shown in figure 2.1. The permittivity is almost independent from of frequency during both stages. The authors relate the decrease of the properties to the evaporation of water during the process; thus, any polar components are instantaneously volatized and cannot contribute to an increase in the energy storage and dissipation of the material. Opposite of drying and pyrolysis stages, both dielectric constant and loss factor increased with the temperature in the char region (beyond 450 °C), which can be related to the thermal decomposition of switchgrass and the phase change to char. Therefore, temperature is an important factor to be considered when measuring dielectric properties of biomass.

It can be concluded from the previous studies that the dielectric properties are highly influenced by the signal frequency applied and the characteristics of the material, as moisture content, bulk density, and its composition. Numerous techniques exist to determine permittivity of agricultural materials and it application depends on the frequency range that is used.



Figure 2.1. Dielectric properties of switchgrass vs. temperature under nitrogen environment at 915 MHz and 2450 MHz with initial density of 0.94 ± 0.050 g/cc; (a) relative dielectric constant, (b) relative loss factor, and (c) tangent loss (Matasemi et al., 2014).

3. Dielectric Properties Measurement

3.1. Materials and methods

Corn stover and switchgrass sample material were obtained from the BioCentury Research Farm at Iowa State University where the materials were ground using a 1.9 cm (¾ in) screen. The material was prepared in the laboratory in an attempt to set different moisture content levels. The material was separated in three different containers and water was add in excess to each containers for 24 hours. After this period, the excess water was removed and the wet material was place in other three containers to be dried. The measurements were collected throughout the drying process. A standard procedure was used to determine the moisture content after each measurement, where three replicate samples of approximately 80 grams of sample material were oven dried for 24 hours at 103 °C in a forced air oven (ASABE, 2012). The moisture content levels (standard deviation) for switchgrass were 10.3% (0.42%), 13.2% (0.45%), 21.4% (0.52%), and 35.4% (0.85%); and corn stover were 10.6% (0.77%), 13.8% (0.50%), 26.5% (1.3%), and 34.9% (0.66%), respectively. All moisture contents were calculated on a wet basis.



Figure 3.1. Switchgrass (left) and Corn stover (right) tested

To obtain the permittivity measurements for both switchgrass and corn stover, an impedance analyzer (model LF 4192, Hewlett – Packard, Palo Alto - CA) was utilized at several frequencies. The impedance analyzer was connected to a sample holder designed via probe fixture (HP 16095, Hewlett – Packard, Palo Alto - CA). The sample holder consisted of two parallel sensing walls and two insulating plates perpendicular to the sensing walls. On the top and bottom of the sensor, there were two additional insulating

plates closing the system. The insulating material chosen was polycarbonate due to its good electrical characteristics for this purpose.

3.2. Sample holder design and impedance analyzer assemble

The sensing wall was composed of four layers (15.24 cm x 15.24 cm in x 0.3175 cm) in which two intermediary layers were steel plates (S1 and S2) and the other two made of polycarbonate (P1 and P2). The layers were attached to each other with acrylic glue and silver epoxy was used to create the signal from S1 to S2 as it can be seen in figure 3.2 with the complete assembly of the sample holder.



Figure 3.2. Complete assembly of the sample holder and side view of the sensing wall

The outer guard and guard ring operated as an electrical shield, and the inner steel plate was responsible to conduct the signal through the material and back to the impedance analyzer, as shown in the figure 3.3. The objective of the shield was to minimize effects of stray electromagnetic fields by connecting the shield output to the ground. Two holes, with a diameter of 0.1875 cm (3/16 in), were made on S1; in the first one, an SMA connector was attached with silver epoxy to drive the input signal from the impedance analyzer to the center plate of S2, through P1 that had the same hole with the same diameter. Similarly, the second hole was used to lead the signal to the shield of the sample holder. P2 was used to cover S1 from outside interference and had the same dimensions of S1. The same characteristics were used on the opposite sensing wall but, instead, it was used as the output of the sample holder that led the signal to the ground. The two sensing walls were separated by a distance of 11 cm totalizing a volume of 2554.8 cm³ of free space. More details about the sample holder design can be found in Appendix A.



Figure 3.3. Each sensor wall consisted of an outer guard sheet (left), an inner electrode steel sheet (right, inner) and guard ring (right, outer). The outer guard and guard ring were connected to ground terminal, with the inner electrode connected to the measurement circuit.

SMA to BNC cables were used to make the connections between the impedance analyzer and sample holder. A banana plug was used to connect the output of the sample holder to the ground of the impedance analyzer, as seen in figure 3.4.



Figure 3.4. Empty sample holder (left) and impedance analyzer (right) forming the complete measurement system

3.3. Electrical measurements

To calculate the dielectric constant (ε_m ') and dielectric loss factor (ε_m ''), the complex admittance, Y = G + jB, was measured, where the real part, G, is the conductance and B represents the imaginary part, susceptance. These two quantities were obtained directly from the impedance analyzer using the parallel circuit mode. Expressions for ε_m ' and ε_m '' were derived from a schematic, as seen in Figure 3.5, similarly to methods used by Lawrence and Nelson (1993).



Figure 3.5. Equivalent circuit of the sample holder for dielectric measurements

Where,

R_{mat:} resistance related to the material inside the sample holder (Ohm)

- R_c: resistance related to the sensor cell (Ohm)
- R_f: fringe resistance of the system (Ohm)
- C_{mat}: capacitance of the material sample (Farads)
- C_c: capacitance of the sensor cell (Farads)
- C_f: fringe capacitance of the system (Farads)
- 1. Dielectric constant (ϵ '):
- a. Empty sample holder measurement:

$$Ca = Co + Cc + Cf$$
 (3.1)

Where:

Ca: measured air filled capacitance in pF.

Co: equivalent capacitance of the empty space of the sample holder pF.

b. Material filled sample holder measurement:

$$Cm = Cmat + Cc + Cf$$
 (3.2)

Where:

Cm: measured capacitance in pF of the sample holder filled with material.

By subtracting equation 3.1 from equation 3.2:

$$\mathbf{Cm} - \mathbf{Ca} = \mathbf{Cmat} - \mathbf{Co} \tag{3.3}$$

However, $Cmat = \varepsilon_m$ ' Co, substituting to 3.3 and rearranging,

$$\varepsilon_m' = \frac{Ca - Cm}{Co} + 1 \tag{3.4}$$

Co can be calculated from the given geometrical parameter of the capacitor.

$$Co = \varepsilon_a' \times \varepsilon'_0 \times \frac{A}{d}$$
(3.5)

Where:

 ε_a ' = dielectric constant of air (≈ 1).

- ε_0 ' = permittivity of space (8.84194 x 10⁻¹² F/m).
- $A_0 = capacitor area (m^2) = 0.1524^2 m^2 = 0.0232 m^2.$
- d = distance between capacitor plates (m) = 0.11 m.

In addition, since $B = C \ge \omega = C \ge 2\pi f$, equation 3.4 can be expressed in terms of the susceptance (mS) as follows:

$$\varepsilon_m' = \frac{Ba - Bm}{2\pi f \times 1.87 \times 10^{-12}} + 1 \tag{3.6}$$

- 2. Dielectric loss factor (ε_m "):
- a. Empty sample holder measurement:

$$Ga = Go + Gc + Gf$$
(3.7)

Where:

Ga: measured total conductance of empty sample holder in mS.

Go: conductance of the sample region with air as the dielectric material (Go~0)

Gc: conductance related to the sample holder in mS.

Gf: fringe conductance of the systems in mS.

b. Material filled sample holder measurement:

$$Gm = Gmat + Gc + Gf$$
 (3.8)

Where:

Gm: measured total capacitance of the material sample in mS.

Gmat: conductance of the material filled portion of the sample holder in mS.

Subtracting 3.7 from 3.8,

$$Gm - Ga = Gmat - Go \tag{3.9}$$

In addition, Gmat = $\omega Co\epsilon$ " and Go is negligibly small. Substituting in equation 3.9 and solving for ϵ_m ":

$$\varepsilon_m'' = \frac{Ga - Gm}{2\pi f \times 1.87 \times 10^{-12}}$$
 (3.10)

Measurements of conductance (G) and susceptance (B) were obtained for empty sample holder before each measurement of the sample holder filled with material. Five measurements were taken using four different moisture content and three bulk densities (0.08, 0.10, and 0.12 g/cm3) for each material in a frequency range of 0.1, 1, 2, 5 and 10 MHz. This procedure was repeated three times.

The impedance analyzer was turned on at least one hour before the beginning of the experiments to allow it to warm up. The samples were prepared by weigh according to each density utilized and stored in different containers. Before the start of sample readings, open-circuit measurements with the sample holder empty were taken in other to eliminate the effects of fringe capacitance and other probable unwanted disturbances that may have occurred during the readings of the other readings. Admittance was measured over the frequency range of 0.1 to 10 MHz. After the measurements were taken, the material was removed from the sample holder and stored again in plastic containers. The sample holder was cleaned after each procedure and used again for the next sample. The data measured were stored in data sheets awaiting analysis.

3.4. Statistical methods

3.4.1. Evaluation of dielectric properties response

Frequency, moisture content (M.C.), and bulk density (B.D.) are three variables that have high influence on dielectric properties of any agricultural material (Nelson and Trabelsi, 2004). Dielectric response models were developed for dielectric properties based on data treatments. Dielectric constant and loss factor were the response variables while frequency, moisture content, and bulk density were used as the predictor variables.

The dielectric response of the measured samples represents the net effect of these three factors. Therefore, the initial statistical analysis was used to check the significance and effect of each variable involved in the experimental design on the dielectric measurements. Dielectric constant and loss factor were assigned as the response variables being affected by M.C., B.D., and frequency.

3.4.2. Model analysis for M.C. and B.D. prediction

The following procedure was used to screen for the dielectric variables that had the best predictive capability, and to use those parameters to develop a calibration model that could predict moisture content and bulk density. The objective of this screening method was to reduce the numbers of parameters used without significantly interfering with the coefficient of determination of the models. The dielectric constant, loss factor, and calculated loss tangent at each frequency were considered as independent variable in the multivariate analysis and prediction analysis.

Multivariate stepwise regression was utilized to develop regression models with the goal of achieving a high coefficient of determination (\mathbb{R}^2) with the minimum number of parameters included in the model. Four different variations of models were used; 1) regression models based on the main factors with no interaction terms, and 2) second order factorial regression models based on the main factors and second order interaction term. In addition, regression models were determined for the moisture content and bulk density, and alternatively logarithm base 10 of moisture content and bulk density as the predicted variables. By using this variation, it was expected to increase the range of the screening and, consequently, increase the model fit.

Models were developed using only the dielectric properties previously described (original variables) and other regressions were created using related variables of the dielectric properties; for example the natural logarithm and cubic root of the dielectric constant and loss factor. Multivariate Standard Partial Squares Regression was used (α =

5%) and centered by the mean. Figure 3.6 shows the flow chart with the regression methods and variables used to develop the regression models.



Figure 3.6. Flow chart of all models developed for each material.

4. Results and Discussion

4.1. Dielectric properties and statistical analysis

The relationship between moisture content and dielectric properties for switchgrass is shown in figure 4.1 for switchgrass and figure 4.2 for corn stover. The points in the plot represent the average of the five replicate for each property and the error bars correspond to the standard deviation. Dielectric constant and loss factor were plotted against the moisture content individually for each bulk density, where loss factor is represented on the left and dielectric constant on the right. An increase in the dielectric constant and loss factor were observed for both materials with increase in moisture content. In general, the increase in the dielectric constant was more obvious for lower frequencies (1 MHz) than at higher frequencies (10 MHz), which suggests that dielectric constant is more sensitive to lower frequencies when trying to predict moisture content. In some cases, there was a decrease in the dielectric constant when increasing the moisture content. The opposite was observed for the loss factor, as shown in figure 4.3. The increase of this property was higher at 10 MHz compared to a 1 MHz signal.

The behavior of corn stover at different moisture content levels was similar to what was observed to switchgrass. In general, both dielectric constant and loss factor increased with higher amounts of water presented in the material, although, the increase was less attenuated compared to material at 0.12 g/cm³. For example, the loss factor increased, on average, 28 times from the driest sample to the wettest at 0.10 g/cm³, however, at 0.12 g/cm³, this same property increased 10 times comparing samples at similar moisture content level. For switchgrass, when comparing from driest to most wet, the loss factor increased 27 times at 0.10 g/cm³ and 41 times at 0.12 g/cm³.



Figure 4.1. Loss factor (left) and dielectric constant (right) of switchgrass at 1 MHz



Figure 4.2. Loss factor (left) and dielectric constant (right) of corn stover at 1 MHz

For the dielectric constant, when comparing values for the driest and wettest corn stover samples analyzed, this property increased 176% for 0.08 g/cm³, 275% for 0.10 g/cm³, and 260% for 0.12 g/cm³. When switchgrass was analyzed, the increase was 155% for 0.08 g/cm³, 317% for 0.10 g/cm³, and 377% for 0.12 g/cm³.



Figure 4.3. Dielectric constant and loss factor against the moisture content at 0.10 g/cm³ for two frequencies

It was concluded that the variation in the dielectric properties was due to a net effect of moisture content, bulk density, and frequency. Regression models were developed for both dielectric properties and materials based on data measured. Dielectric constant and loss factor were the responsible variable while frequency, moisture content, and bulk density were used as the predictor variables.

Two types of models were created. The first one involved the three variables (moisture content, bulk density, and frequency) as a second-degree polynomial. The second method used the variables in third-degree factorial regression. The method with the higher coefficient of determination was chosen as the best for each situation. The results obtained are shown on tables 4.1 to 4.4 and figures 4.4 to 4.7.



Figure 4.4. Regression model against actual loss factor for corn stover

1 able 4.1.	Loss lacu	or regr	ession model	IOF COFIL SLOV	/er

Term	Estimate	Std Error	t Ratio	Prob> t	F Ratio
Intercept	-3.19	0.474	-6.74	< 0.0001*	-
FREQ.	0.0224	0.0296	0.76	0.4485	0.576
$(FREQ 3.62)^2$	0.0152	0.00738	2.06	0.0404*	4.24
MC	7.93	0.757	10.48	< 0.0001*	109.73
$(MC - 0.215)^2$	177.57	12.49	14.22	< 0.0001*	202.21
BD	12.36	4.23	2.92	0.0037*	8.55
$(BD - 0.1)^2$	-160.5	366.1	-0.44	0.6614	0.192



Figure 4.5. Regression model against actual dielectric constant for corn stover

Term	Estimate	Std Error	t Ratio	Prob> t	F Ratio
Intercept	-0.263	0.384	-0.68	0.495	-
FREQ.	-0.241	0.0240	-10.07	< 0.0001*	101.41
$(FREQ 3.62)^2$	0.0447	0.00597	7.48	< 0.0001*	55.91
MC	5.58	0.613	9.10	< 0.0001*	82.80
$(MC - 0.215)^2$	74.70	10.11	7.39	< 0.0001*	54.59
BD	9.00	3.42	2.63	<0.0090*	6.92
$(BD - 0.1)^2$	326.8	296.4	1.10	0.2711	1.22

Table 4.2. Dielectric constant regression model for corn stover

The tables suggested that not all variables are highly significant to predict loss factor and dielectric constant for corn stover and switchgrass. The Standard Least Square regression to estimate loss factor for corn stover (Table 4.1) had an R^2 equal to 0.634. Dielectric constant (Table 4.2) had an R^2 of 0.525, for corn stover. For switchgrass, R^2 was equal to 0.802 for loss factor regression (Table 4.3), and 0.718 for the dielectric constant regression (Table 4.4).



Figure 4.6. Regression model against actual loss factor for switchgrass

1	Het Llobb Ide	tor regression	mouter for a			
Term	Estimate	Std Error	t Ratio	Prob> t	F ratio	_
Intercept	-3.27	0.365	-8.95	<.0001*	-	
FREQ.	0.141	0.0153	9.17	<.0001*	84.1	
MC	14.8	0.572	25.85	<.0001*	668.2	
(FREQ 3.62)×(MC - 0.202)	2.82	0.159	17.72	<.0001*	314.0	
BD	16.4	3.37	4.86	<.0001*	23.6	
$(FREQ 3.62) \times (BD - 0.1)$	3.86	0.939	4.11	<.0001*	16.9	
$(MC - 0.202) \times (BD - 0.1)$	204	35.1	5.82	<.0001*	33.9	
(FREQ 3.62)×(MC - 0.202)× (BD - 0.1)	63.0	9.76	6.45	<.0001*	41.7	

Table 4.3. Loss factor regression model for switchgrass



Figure 4.7. Regression model against actual dielectric constant for switchgrass

Term	Estimate	Std Error	t Ratio	Prob> t	F ratio		
Intercept	-0.690	0.379	-1.82	0.0698	-		
FREQ.	-0.324	0.0239	-13.58	<.0001*	184.5		
$(FREQ 3.62)^2$	0.0509	0.00596	8.54	<.0001*	72.89		
MC	15.9	0.822	19.29	<.0001*	372.3		
$(MC - 0.202)^2$	-43.3	9.23	-4.69	<.0001*	22.0		
BD	14.1	3.42	4.13	<.0001*	17.0		
$(BD - 0.1)^2$	-397.0	295.54	-1.34	0.1802	1.8048		

Table 4.4. Dielectric constant regression model for switchgrass

For all the models developed, the second-degree polynomial showed to be the best method to predict the dielectric properties, except for switchgrass loss factor where the R^2 would have been 0.552 using this method instead of using the factorial method.

Matasemi et. al. (2014) found similar results for switchgrass where the dielectric constant and loss factor were measured at different temperatures at a constant bulk density for a 915 MHz and 2450 MHz electrical signal. The objective of this research was to study the behavior of dielectric properties of switchgrass during pyrolysis, thus, temperature was the major variable in this work.

All three parameters (moisture content, bulk density, and measurement frequency) were highly significant, although, in general, frequency and moisture content had higher F values than bulk density. Since the F test is used to measure the relative significance of the independent variables on the response, for these regression models, frequency and moisture content were more significant than bulk density.

4.2. Variable selection and multivariate analysis

The dielectric constant, loss factor, and calculated loss factor at each frequency were considered as independent variable in the multivariate analysis and regression analysis.

Multivariate stepwise regression was utilized to develop regression models, to achieve a high coefficient of determination (\mathbb{R}^2) with the minimum number of parameters included in the model, as possible. Four different variations of models were used; 1) regression models based on the main factors with no interaction terms, and 2) second order factorial regression models based on the main factors and second order interaction term. In addition, regression models were determined for the moisture content and bulk density, and alternatively logarithm base 10 (log) of moisture content and bulk density as the predicted variables. Using this step, it was expected that any nonlinear effects of the response variables could be minimized and, consequently, increase the coefficient of determination. The adjusted coefficient of determination (\mathbb{R}^2 adj.) was calculated to find the ideal combination model with the best fit without over fitting the model; similarly, the root mean square error (RMSE) of the models was obtained as another measurement for model fit comparison.

Models were developed using only the dielectric properties previously described (original variables) and other regressions were created using related variables of the dielectric properties, for example the natural logarithm and cubic root of the dielectric constant and loss factor. Multivariate Standard Partial Squares Regression was used (alpha = 5%) and centered by the mean.

Figures 4.8 to 4.11 show the relationship between the numbers of parameters and the coefficient of determination for all different regression models developed as described

previously. It can be observed that R^2 increased together with the number of factors utilized. Each model was set to limit of 20 variables to avoid over fitting the regressions. The use of related dielectric variables helped to improve the coefficient of determination for some models, especially for corn stover that had higher R^2 when compared to models with only the original dielectric properties as variables. Some cases the regression models did not have more than two valid regression models matching the criteria of less than 20 variables; therefore, they were omitted from the charts in figures 4.8 to 4.11.

For moisture content determination, it can be observed from figure 4.8 and 4.9 that models with few variables were enough for a R^2 superior to 0.9. One switchgrass model with three parameters and using only original dielectric variables had a coefficient of determination equal to 0.9355 and adjusted R^2 of 0.9332. For corn stover, using original and related dielectric variables, a four parameters model obtained a R^2 of 0.9033 and adjusted of 0.8981, although, using more variables caused a higher determination for both materials.

Bulk density needed a higher number of parameters to describe models with coefficient of determination close to 0.9, as observed in figure 4.10. For switchgrass, 19 parameters were necessary using only the original dielectric variables for a R^2 equal to 0.9045 (adj. $R^2 = 0.8626$), although, a model with 16 parameters utilizing both original and related dielectric factors was observed for a R^2 of 0.8667 and adjusted R^2 of 0.8191. For corn stover, bulk density models did not achieve a coefficient close to 0.9 with 20 or less parameters. Using only original variables, the best fit used 20 parameters for a R^2 of 0.8959 and adjusted of 0.8465. Using 16 related and original variables, the best-fit model had an R^2 of 0.8737 and adjusted equal to 0.8306 as shown in figure 4.11.



Figure 4.8. Coefficient of determination against number of parameters for moisture content regression models using only plain dielectric variables.



Figure 4.9. Coefficient of determination against number of parameters for moisture content regression model using related dielectric variables.



Figure 4.10. Coefficient of determination against number of number of parameters for bulk density regression model using only plain dielectric variables.



Figure 4.11. Coefficient of determination against the number of parameters for bulk density regression models for corn stover.

The best models were created by JMP utilizing the Standard Least Square and significance level of 5% ($\alpha = 0.05$). The best models were selected based on those that had the fewest number of variables and closer to a coefficient of determination of 0.85 or higher.

The best model for switchgrass moisture content regression, as observed in figure 4.12 and table 4.5, had three parameters. Bulk density was described in a 16 factors calibration model as observed in figure 4.14 and table 4.7. It can be seen in table 4.6 and figure 4.13 that the best model for corn stover moisture content regression was a four variables model; it is described in figure 4.15 and table 4.8 the bulk density regression model utilizing 16 related dielectric factors.



Figure 4.12. Predicted model against actual MC for switchgrass.

I ADIC 4.3. MUSIULC CONCENT LESSON MUUCH IN SWILLISTA	Та	ıble	4.5.	Moisture	content	regression	model for	switchgras
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Tuble net i	ionstare com	tent regressi	on model	or switcing	400
Term	Estimate	Std Error	t Ratio	Prob> t	F ratio
Intercept	-1.067	0.0148	-72.09	<.0001	—
LF 5	0.0356	0.00558	6.39	<.0001	40.8
DC 0.1	0.0562	0.00406	13.86	<.0001	192.1

* LF = loss factor, DC = dielectric constant. Following number is the correspondent frequency used for this variables

The final model for switchgrass moisture content regression had an $R^2 = 0.94$ and RMSE = 0.052. It can be observed from table 4.5 that, as previously noted, low frequencies was more sensitive for dielectric constant measurement than high frequency and the opposite was observed for loss factor. Analyzing the F ratio for this model, the dielectric constant for a 0.1 MHz signal had higher potential to predict moisture content when compared to the loss factor. This model used the logarithm of the moisture content, thus the results obtained from the polynomial should be converted to moisture content in decimals or percentage.

The results obtained for corn stover moisture content regression are shown on table 4.6. Four parameters were necessary to explain 90% of the moisture content variation in corn stover with an RMSE equals to 0.0315 as observed in figure 4.13. One related and two original dielectric variable was used to describe this model. Loss factor of a 1 MHz signal was more significant than the dielectric constant to describe the correlation between this model and the dielectric properties.



Figure 4.13. Predicted model against actual M.C. (w.b.) for corn stover.

1 able 4.0. Wills	Table 4.0. Wolsture content (w.b.) regression model for corn stover							
Term	Estimate	Std Error	t Ratio	Prob> t	F ratio			
Intercept	-0.427	0.0768	-5.56	<.0001	_			
LF 0.1	-0.045	0.00370	-12.16	<.0001	147.9			
DC 0.1	-0.0178	0.0107	-1.67	0.1014	2.77			
DC ^{1/3} 0.1	0.528	0.07910	6.68	<.0001	44.6			

Table 4.6. Moisture content (w.b.) regression model for corn stover

* LF = loss factor, DC = dielectric constant. Following number is the correspondent frequency used for this variables

Density regression models require many more variables to develop a model that could explain the variation of density based on the dielectric properties. As observed in table 4.7, sixteen variables were necessary for a regression model with an R^2 equals to 0.85 and RMSE = 0.0323, according to figure 4.14. Related and original dielectric variables were involved on the model and, similar to the moisture content, the response for this regression was based on the log of bulk density. Among all the terms, the loss factor of 0.1 MHz signal had the most influence on this model when the F ratio is analyzed.

The results shown on table 4.8 and figure 4.15 used 16 terms to explain 87% of the variation of density for corn stover with a RMSE equals to 0.0068. For this model, both original and related dielectric variables were used to predict density for stover with loss factor being more influent than dielectric constant to explain this calibration according to the F ratio of the parameters.

The high number of variables necessary for predicting bulk density for both switchgrass and corn stover indicates the fact that more investigation is necessary for this factor and its interaction in an electric field. Density and moisture are two factors that are related to each other, thus, a wider range of moisture content could help also determining the dielectric properties of these materials.



Term	Estimate	Std Error	t Ratio	Prob> t	F ratio		
Intercept	-1.95	1.54	-1.27	0.2111	_		
LF 0.1	-1.34	0.204	-6.58	<.0001	43.3		
LF 1	0.129	0.0327	3.94	0.0003	15.5		
LF 10	0.0244	0.00770	3.17	0.0028	10.0		
DC 5	-0.195	0.0373	-5.24	<.0001	27.5		
DC ^{1/3} 10	-3.45	1.38	-2.51	0.0160	6.28		
LF ^{1/3} 0.1	5.41	0.791	6.84	<.0001	46.8		
ln(DC) 1	-1.78	0.261	-6.83	<.0001	46.6		
ln(DC) 2	2.09	0.314	6.66	<.0001	44.4		
ln(DC) 10	1.58	0.577	2.74	0.0089	7.49		
ln(LF) 1	-0.115	0.0257	-4.46	<.0001	19.9		
$(LF 0.1 - 1.83) \times (LF^{1/3})$ 0.1 - 1.15)	1.28	0.172	7.44	<.0001	55.3		
$(LF 1-1.37) \times (DC^{1/3} 10 - 1.27)$	-0.304	0.0845	-3.59	0.0008	12.9		
$(LF 10 - 2.96) \times (ln(LF) 1 + 0.237)$	-0.0204	0.00748	-2.73	0.0091	7.45		
$(DC 5 - 2.30) \times (ln(DC))$ 10 - 0.680)	0.237	0.0854	2.78	0.0080	7.71		
$(\ln(DC) 1 - 0.890) \times (\ln(DC) 2 - 0.837)$	0.290	0.129	2.25	0.0296	5.05		

Table 4.7. Bulk density regression model for switchgrass

* LF = loss factor, DC = dielectric constant, LT = loss tangent. Following number is the correspondent frequency used for the variable.



Term	Estimate	Std Error	t Ratio	Prob> t	F ratio
Intercept	0.310	0.0393	7.90	<.0001	_
LF 0.1	0.0492	0.0135	3.64	0.0007	13.3
LF 5	0.00356	0.00629	0.57	0.5745	0.320
DC 0.1	-0.0338	0.00508	-6.65	<.0001	44.2
DC 5	-0.0767	0.0213	-3.60	0.0008	13.0
DC 10	0.00930	0.00258	3.61	0.0008	13.0
LT 0.1	-0.347	0.0693	-5.00	<.0001	25.0
LT 5	-0.0160	0.0130	-1.23	0.2240	1.52
ln(DC) 5	0.169	0.0453	3.74	0.0005	14.0
ln(LF) 0.1	0.0475	0.00589	8.08	<.0001	65.2
ln(LF) 10	-0.00500	0.00177	-2.81	0.0073	7.92
$(LF 0.1-1.63) \times (LF 5-1.56)$	-0.0143	0.00173	-8.27	<.0001	68.4
$(DC \ 0.1 - 3.61) \times (LT \ 5 - 0.752)$	0.009512	0.00157	6.06	<.0001	36.8
$(DC 5-1.80) \times (LT 0.1-0.400)$	-0.142	0.0341	-4.18	0.0001	17.4
$(DC \ 10-1.98) \times (ln(LF) \ 10-0.140)$	-0.00172	0.00188	-0.92	0.3640	0.841
$(\ln(DC) 5-0.562) \times (\ln(LF) 0.1+0.221)$	0.0734	0.0111	6.62	<.0001	43.9

* LF = loss factor, DC = dielectric constant, LT = loss tangent. Following number is the correspondent frequency used for the variable in MHz.

5. Summary and Conclusions

Dielectric properties for switchgrass and corn stover were successfully calculated under the influence of different moisture content and bulk density. It could be observed that frequency, moisture content, and bulk density had a significant effect on measured dielectric constant and loss factor for both materials. Based on these readings, regression models could be developed to estimate both moisture content and bulk density. The main results of this research are:

- Both dielectric constant and loss factor increased proportionally to the amount of water present in the sample mass. For higher densities, the increase was more attenuated for switchgrass although, for corn stover, the same was not true. Dielectric constant was more sensitive at lower frequencies in this study, while loss factor had more sensibility for higher frequencies.
- 2. All three variables were highly significant for dielectric properties determination. Frequency and moisture content had a higher influence compared to bulk density when trying to predict dielectric constant and loss factor for both materials. Although, the response of the permittivity is a net effect of all the variables involved.
- 3. Dielectric properties variables could be successfully used to predict moisture content and bulk density of the materials tested. It was observed that the higher the number of variables used on a model, better is the coefficient of determination, although there is a risk of over fitting the data.

4. It is still necessary to investigate a wider range of bulk density, moisture content, and frequency levels to understand better the effect of these variables on determining the dielectric properties of the materials studied on this work.

6. Recommendations for further study

Auxiliary research is needed for a wider range of frequencies, moisture content, and bulk density levels. This calibration method can be studied for a real time sensor to be implemented in practice. Development of a sensor with flow material could be investigated as well to simulate the harvest process of these materials.

Dielectric properties could be used to measure other features besides moisture content and bulk density. This method could be used to measure the quality of the material depending on the reason that it will be applied.

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APPENDIX A – SAMPLE HOLDER BLUEPRINTS











