UNIVERSAL CALIBRATION METHOD FOR MICROWAVE MOISTURE SENSING IN GRANULAR MATERIALS

S. Trabelsi, A. W. Kraszewski, S. O. Nelson

ABSTRACT: A feasibility study of a universal calibration method for nondestructive real–time sensing of moisture content in granular materials is described. The principle is based on measurement of the dielectric properties of granular materials at microwave frequencies and the use of a calibration function that is expressed in terms of these properties to predict moisture content from a single moisture calibration equation. Effectiveness of this method is shown for three granular materials exhibiting significant structural and compositional differences: wheat, oats, and soybeans. The resulting single moisture calibration equation, based on measurements of the dielectric properties at 9.46 GHz and 24 °C, provides moisture content in all three materials with a standard error of calibration of 0.46% moisture content, wet basis.

Keywords. Universal calibration method, Moisture content, Microwave sensors, Granular materials, Bulk density, Relative complex permittivity.

On–line, real–time sensing of moisture content and other physical properties of wet granular materials is crucial in many industries where these properties are used as quality control factors and/or indicators for the optimization of a given process, particularly when large quantities are involved. Methods for moisture content determination in natural and manufactured materials can be classified into two categories: direct methods and indirect methods. Direct methods rely on weight loss (oven drying method, ASAE Standards, 1999) or chemical titration (Karl–Fisher). Direct methods also require off–line testing of a few samples. They are accurate and are usually used as references for the calibration of other methods. The major disadvantages are their destructive nature and the time they require.

Indirect methods are based on the measurement of a property of the material that is directly correlated with moisture content. Nuclear radiation–, infrared–, and dielectric–based sensors are the most commonly used indirect methods. They have the advantages of being nondestructive and contactless, and most important, they provide a tool for on–line continuous measurement of moisture content. Moreover, with better sampling and averaging over the entire lot of the material being handled, they give a better estimate of the “true” moisture content. Nuclear radiation–based sensors are expensive and present potential hazards. Infrared sensors provide mainly surface moisture content. In contrast, with microwave moisture sensors, the spatial resolution of the electromagnetic waves provides information related to the volume rather than just the surface of the material.

In the grain industry, the emergence of precision farming concepts and computer–controlled processes requires development of a new generation of versatile sensing devices that can be easily integrated with routinely used equipment such as combine harvesters and grain conveyors. Fundamental limitations of most existing moisture sensors reside in the lack of transferability of grain moisture calibration across instruments and the need for an individual calibration for each type of grain. Some of the reasons are related to the fact that these calibrations are often established for instrument–specific parameters based on statistical analysis and without any physical foundation. Microwave moisture sensors based on measurement of the intrinsic properties of materials, such as the dielectric properties (Meyer and Schilz, 1981; Trabelsi et al., 1997b, 1998a), can be a suitable solution to these limitations and the new challenges cited above. They can continuously provide parameters such as bulk density, moisture content, and dry mass from nondestructive measurement of dielectric properties in static or dynamic situations. For instance, a method for simultaneous independent determination of bulk density and moisture content (Trabelsi et al. 1997b) can be the foundation for the development of a multiple–parameter microwave sensor.

The dielectric properties are represented by the relative complex permittivity (ε), which is an intrinsic electrical property of the material that characterizes the electric field/material interaction. The relative complex permittivity is a complex entity that is often written as ε = ε′ – jε″. The real part (ε′) reflects the ability of a material to store electric–field energy, and the imaginary part (ε″) is usually associated with the ability of a material to dissipate electric energy in the form of heat. j = \sqrt{-1} is the imaginary unit. The essence of
microwave moisture sensing is based on the polar nature of the water molecules, which translates into a high correlation between the dielectric properties of moist substances and their moisture content (Hasted, 1973), the absence of ionic conductivity, and the spatial resolution of the electromagnetic waves.

However, moisture is not the only variable affecting the dielectric properties. Temperature and bulk density (particulate and granular materials) have effects similar to that of moisture on these properties (Nelson, 1981, 1983; Kraszewski et al., 1996; Trabelsi et al., 1997a). Therefore, development of microwave moisture sensors based on the measurement of dielectric properties requires compensation for these effects. This compensation may be accomplished by two approaches: correction factors can be introduced, or the effects can be eliminated through identification of functions of the dielectric properties that are insensitive to temperature and density variations. The compensation/correction approach implies the combination of different types of sensors, which always complicates the moisture sensor design. It also complicates the calibration procedures and introduces a higher probability for errors in case of malfunction or failure of one or more of the devices. In addition, it increases the overall cost of the design and maintenance of such a system.

The second approach is more attractive technically and economically. Because temperature is a parameter that is easy to measure with relatively inexpensive and reliable devices, more efforts have been devoted to solving the bulk density problem (Kraszewski et al., 1977; Jacobsen et al., 1980; Meyer and Schilz, 1981; Menke and Knöchel, 1996) by defining density-independent calibration functions. Though density-independent, these calibration functions were either instrument-dependent or required individual calibrations for each type of grain or other material. Recently, a calibration method, based on measurement of the dielectric properties, was proposed that is both density-independent and material-independent (Trabelsi et al., 1997b, 1998a, 1999, 2000a).

It is the purpose of this article to demonstrate the “universal” character of this method through dielectric measurements on three granular materials exhibiting significant structural and compositional differences. Effectiveness of the method is shown for three granular materials, one of naturally low density (oats), and two of relatively higher density (wheat and soybeans). The resulting single moisture calibration equation, established from measurements of $\varepsilon'$ and $\varepsilon''$ in free space at 9.46 GHz and 24°C, provides moisture content in all three materials with a standard error of calibration of 0.46% moisture content, wet basis. To improve the measurement accuracy of the dielectric properties, a pair of horn/lens antennas, which provided a focused beam, was used and time-domain gating was applied.

**Materials and Methods**

**Sample Preparation and Sample Characteristics**

Because of timing and the nature of scheduling measurements, grain and soybean samples with a range of natural moisture contents were not available. Therefore, samples were tempered for these measurements. However, differences in the 1 to 50-MHz dielectric properties of naturally dried and tempered wheat were well within the range of variation noted among different cultivars of the same kind of wheat (Nelson and Stetson, 1976). These differences are also likely to be much smaller at microwave frequencies, which are well above the dielectric relaxations for bound water. Samples of hard red winter wheat (Triticum aestivum L.), winter oats (Avena sativa L.), and soybean (Glycine max L., Merrill) of different moisture contents were prepared and stored in sealed plastic bags for at least 72 hours at 4°C to equilibrate. When necessary, moisture was added by spraying distilled water on the materials before they were sealed. To obtain uniform moisture content throughout the entire sample, each sample was mixed periodically within the sealed bag. Before the microwave measurements were performed, the sealed samples were allowed to equilibrate to room temperature for at least 24 hours. For each sample, kernels or seeds were poured into a Styrofoam container of rectangular cross-section to provide a layer of material, which was placed in free space between two antennas. Measurements at three different bulk densities, ranging from loosely packed to compacted, were obtained by settling the sample mechanically.

For each sample, the sample mass ($m$) was measured with an electronic balance (Mettler PC 8000), and the volume ($v$) was determined from the dimensions of the sample holder used for each material. The bulk density is calculated as:

$$\rho = \frac{m}{v} = \frac{m_w + m_d}{v}$$  \hspace{1cm} (1)

where

$m = m_w + m_d = \text{mass of the moist material}$

$m_w = \text{mass of water}$

$m_d = \text{mass of dry matter of the sample}$

Geometry and dimensions of the kernels, along with surface characteristics, are determinant factors in the packing properties of a given material. For instance, wheat and oats kernels had ellipsoidal shape with lengths of 5 to 7 mm and 8 to 12 mm, respectively. The soybeans had a nearly spherical shape with diameters of 5 to 7 mm. These differences, along with those associated with their composition, engender differences in bulk density ranges (table 1).

A larger number of samples was prepared at different moisture levels for wheat than were prepared for soybeans and oats (table 1), but the number of moisture levels was adequate to show the necessary relationships. There was no need for repetition of the microwave measurements at each moisture, each bulk density, and each temperature.

Immediately after the microwave measurements, the temperature of each sample was measured with a digital thermocouple thermometer with a basic accuracy of 0.4°C. The moisture content of each sample was then determined (ASAE Standards, 1999) by drying triplicate 10-g samples of wheat and oats in a forced-air oven at 130°C for 19 hours and 22 hours, respectively. For soybeans, triplicate 15-g samples were dried at 103°C for 72 hours. The moisture content ($M$)

<table>
<thead>
<tr>
<th>Material</th>
<th>Number of Samples</th>
<th>Kernel Length (mm)</th>
<th>Shape</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wheat</td>
<td>15</td>
<td>5 – 7</td>
<td>Ellipsoidal</td>
</tr>
<tr>
<td>Oats</td>
<td>6</td>
<td>8 – 12</td>
<td>Ellipsoidal</td>
</tr>
<tr>
<td>Soybeans</td>
<td>9</td>
<td>5 – 7</td>
<td>Spherical</td>
</tr>
</tbody>
</table>

Table 1. Characteristics of grain and seed samples.
is determined as:

\[ M = \frac{m_w}{m_w + m_d} \times 100 \]  

(2)

**Measurement Technique**

Among all techniques for dielectric materials characterization (Bussey, 1967), free–space techniques are more suitable for implementation in many industrial processes. They can be used either in reflection or transmission modes, and thus they can be easily adapted and installed on line for almost any configuration, including conveyor belt, pipe, or chute. Figure 1 shows a general schematic diagram of wave/material interaction for a layer of granular material of thickness \( d \) in free space. The reflected wave at the front interface is characterized by the reflection coefficient \( \Gamma \), and the transmitted wave is characterized by the transmission coefficient \( \tau \). Usually, the dielectric properties are determined from measurement of \( \Gamma \) and/or \( \tau \) (Baker–Jarvis et al., 1990).

In free–space measurements, accuracy of the complex permittivity determination is related to a combination of factors. The most significant errors are related to near–field effects, scattering, diffraction at the edges of the sample, multiple reflections within the sample and between the different components of the measuring system, and interference with surrounding objects. Therefore, particular attention must be paid to the choice of frequency or frequency range, the sample size, distances between the antennas and the sample, and in some instances, distances from surroundings.

In this study, a free-space transmission technique was used for the measurement of the dielectric properties of wheat, oats, and soybeans. The measurement system (fig. 2) consisted of two linearly polarized, ultrabroadband (2 to 26 GHz) horn/lens antennas (BAE SYSTEMS model AHO–2077–N) that were connected with high–quality coaxial cables to the \( S \)–parameter test set of a Hewlett–Packard 8510B vector network analyzer (VNA). The VNA was calibrated in the transmission mode between 2 and 13 GHz with an empty sample holder located between the antennas. The antennas were mounted 61 cm apart on a stable wooden fixture that kept them well aligned. The horn/lens antennas collimate the electromagnetic energy in a relatively small beam and provide a plane wave a short distance from the transmitting antenna. With these features, the measurements were performed on samples of reasonable size in the far field without problems of edge diffraction or interference by reflections from the surroundings.

For each sample, the kernels or seeds were poured into a sample holder, which was placed midway between the transmitting and receiving antennas. The sample holder was made of 2.5–cm–thick Styrofoam sheet (\( \epsilon' = 1.03 \)) with a rectangular cross section. The dimensions of the sample material were 25 cm transverse, 25 cm in height, and 10.9 cm, 15.3 cm, and 11.1 cm in thickness for wheat, oats, and soybeans, respectively. The transverse and height dimensions were selected to minimize the diffraction effects at the edges of the sample by fulfilling the criterion of three times the \( E \)–plane 3–dB beamwidth over the whole frequency range (Ghodgaonkar et al., 1989).

For each material, the thickness was selected to ensure a 10–dB one–way attenuation through the sample to avoid problems from multiple reflections within the sample. In general, the selection of the sample thickness is a compromise among minimizing multiple reflections, reducing scattering effects, and optimum use of the dynamic range of the measuring system.

**Time–Domain Gating**

Post–calibration mismatches and multiple transmission paths cause oscilloscope traces observed in the frequency domain, the magnitude and phase of the scattering transmission coefficient in this instance, to exhibit ripples with variable magnitudes. This will translate into errors in the permittivity measurements. To remove such effects, the time–domain gating feature of the VNA was used (Hewlett–Packard, 1994). At first, the frequency–domain trace was converted to the time domain. After identifying the main response, a gate was applied to filter out the unwanted responses. Then, the gated response was converted back to the frequency domain. The trace was ripple free, indicating that the effects of responses outside the gate were removed. Effectiveness of time–domain gating relies mainly on appropriate selection of the gate parameters. However, given the fact that the examined samples were made of different materials of different bulk density, different moisture content, and different thickness, it was...
not easy to select the gate parameters. For each material, these parameters were selected after several trials on different samples for which the gated frequency–domain response was a good average of the ungated response.

**Measurement of the Relative Complex Permittivity**

From measurements of the complex scattering transmission coefficient ($S_{21}$), the attenuation ($A$) and the phase shift ($\varphi$) introduced by a sample of thickness $d$ were calculated as follows:

$$A = 20 \log |S_{21}|$$  \hspace{1cm} (3)

$$\varphi = \text{Arg}(S_{21}) - 2n\pi$$  \hspace{1cm} (4)

where $|S_{21}| = \text{modulus of } S_{21}$, $\text{Arg}(S_{21}) = \text{argument of } S_{21}$, and $n = \text{an integer to be determined}$ (Trabelsi et al., 2000b).

For low-loss materials ($\varepsilon'' \ll \varepsilon'$), the real and imaginary parts of the complex permittivity are calculated as (Trabelsi et al., 1998a):

$$\varepsilon' = \left( \frac{\beta}{\beta_0} \right)^2$$  \hspace{1cm} (5)

$$\varepsilon'' = \frac{2\alpha\beta}{\beta_0^2}$$  \hspace{1cm} (6)

where $\alpha = A / d = \text{attenuation constant}$, $\beta = \varphi / d + \beta_0 = \text{phase constant}$, $\beta_0 = 2\pi / \lambda_0 = \text{phase constant for free–space wavelength}$, and $\lambda_0$.

Figures 3 and 4 show variations of $\varepsilon'$ and $\varepsilon''$ for samples of wheat, oats, and soya beans of three different bulk densities with moisture content at 9.46 GHz at 24°C. At each moisture level and for each material, the effect of bulk density can be clearly distinguished. Although water content is a dominant component in the wave/material interaction at microwave frequencies, the relationship between the dielectric properties of these materials and moisture content is not unique. In general, the dielectric properties of soya beans are higher and have greater slope than those of wheat and oats. This is mainly related to their different density ranges (table 1), the fact that the complex permittivity of each material is the effective permittivity of a mixture of components exhibiting different dielectric behavior (i.e., air, dry matter, and water), and perhaps differences in the scattering properties of the kernels. For all three materials, the data in this form cannot be used for moisture content prediction without compensation for, or elimination of, the bulk density effect. Moreover, a universal sensor would require the elimination of the material effect.

**Universal Calibration Method**

At least two criteria have to be fulfilled by a dielectric calibration method for moisture determination in granular materials to be universal. First, it should be insensitive to bulk–density variations, and second, it should provide a single moisture calibration equation regardless of shape, size, and composition of considered materials. To satisfy these two criteria, a permittivity function that is exclusively dependent on moisture content at a given temperature and a given frequency was used (Trabelsi et al., 1997b, 1998a, 2000a). The definition of this function was based on the principle of the distribution of the electric–field energy between dissipated and stored energy within the dielectric material, and observations in the complex plane of the representation of the relative complex permittivity, normalized to bulk density $\rho$: $\tan \delta \rho = \varepsilon'' / (\varepsilon' \rho)$. Analytically, it is simplified in terms of $\varepsilon'$ and $\varepsilon''$ as:
where \( a_f \) is a frequency–dependent coefficient (Trabelsi et al., 1997b, 1998a, 1998b). Graphically (fig. 5), \( a_f \) is the slope of the complex plane representation of the complex permittivity divided by bulk density \((\varepsilon'/\rho, \varepsilon''/\rho)\). Linear regression computations for the data shown in figure 5 provide \( a_f \) values of 0.554 for wheat, 0.452 for oats, and 0.619 for soybeans at 9.4 GHz at 24°C. The \( \varepsilon'/\rho \)-axis intercept is characteristic of each material and represents the normalized dielectric constant of dry matter or that of a sample of given moisture content at very low temperature (Trabelsi et al., 1998a). For wheat, oats, and soybeans, the \( \varepsilon'/\rho \)-axis intercepts are at 2.58, 3.14, and 2.77, respectively. As shown in figures 3 and 4, the oats data are well separated from those of wheat and soybeans.

In figure 6 are shown the variations of \( \psi \) with respect to moisture content in wheat, oats, and soybeans. For all three materials, the effect of density is noticeably reduced, and the data points fall along the same straight line despite the structural and compositional differences among these materials. This has already been reported for other materials (Trabelsi et al., 1999) and confirms the exclusive dependence of \( \psi \) on water content. Consequently, a single calibration equation can be established and used for moisture content determination in different granular materials from measurements of their respective relative complex permittivities at microwave frequencies. In this instance, a linear fitting is used to correlate \( \psi \) with moisture content:

\[
\psi = 0.018 \times M + 0.083 \ \
\]  

From equation 8, the universal moisture calibration equation is determined as:

\[
M = 55.55 \times \psi - 4.61
\]  

In figure 7, the moisture content predicted in each sample of each material by equation 9 is plotted versus the moisture content determined by the oven–drying standard. The data points lie along the straight line that corresponds to the ideal relationship. The effectiveness of equation 9 in determining moisture content in wheat, oats, and soybeans can be evaluated by calculating the standard error of calibration \((SEC)\), which is defined as:

\[
SEC = \sqrt{\frac{1}{n - p - 1} \sum_{i=1}^{n} (\Delta m_i)^2}
\]  

where

- \( n \) = number of samples
- \( p \) = number of variables in the regression equation with which the calibration is performed
- \( \Delta m_i \) = difference between the predicted value and that determined by a standard method for the \( i \)th sample.

The \( SEC \), when using equation 9 to predict moisture content in wheat, oats, and soybeans, was 0.46% moisture content, wet basis. From equations 7 and 9, the accuracy with which \( M \) can be determined is dependent primarily on the accuracy with which \( \varepsilon' \) and \( \varepsilon'' \) can be measured at a given frequency and a given temperature, and to some extent, on the accuracy of the reference method against which the calibration was made, the oven–drying standard (ASAE Standards, 1999) in this instance.

**CONCLUSION**

The universal character of a dielectric calibration method for moisture content determination in wet granular materials has been demonstrated for wheat, oats, and soybeans. The calibration method is relatively independent of both the bulk density and the material type and exclusively dependent on water content. Results based on dielectric properties measurements were shown for three materials exhibiting signifi-
cantly structural and compositional differences (wheat, oats, and soybeans). The fact that the calibration function \((y)\) is analytically expressed in terms of the two components of the relative complex permittivity, which are intrinsic electrical properties of the material, rather than defined in terms of instrument–specific parameters, makes the calibration procedure transferable across instruments of the same and different designs, provided that \(\varepsilon'\) and \(\varepsilon''\) are measured accurately. In addition, it can be used regardless of the measurement technique, giving more flexibility in choosing the appropriate technique for a particular application. These features will constitute the basis for the development of a universal calibration algorithm that can be implemented in hand–held devices, laboratory instruments, and continuous–flow systems. Recent developments in microwave component technology, along with the findings presented in this article, provide incentives for the development of a new generation of universal microwave moisture sensors for granular materials and their common use in the agricultural, food, pharmaceutical, mining, and other industries.

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**REFERENCES**


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**Figure 7.** Predicted moisture content in each sample of wheat, oats, and soybeans by equation 9 versus oven–drying moisture content.