New Density-Independent Calibration Function for Microwave Sensing of Moisture Content in Particulate Materials

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Abstract—**Microwave techniques have been considered for a long time for moisture sensing in many food processing and agriculture-related industries. They are suitable for on-line real-time monitoring and control. However, with particulate materials, bulk density fluctuations cause significant errors in moisture content determination. To overcome this shortcoming, density-independent calibration functions are needed. In this paper, a new approach is presented in which both bulk density and moisture content are determined directly from measured microwave dielectric properties. A simple relationship between bulk density and the dielectric properties is identified, and a new density-independent function for moisture content prediction, exclusively dependent on the dielectric properties of the material under test (ϵ', ϵ''), is proposed. The validity and applicability of this function are demonstrated with an extensive data set obtained from measurements on a granular material (wheat), over wide ranges of frequency (11–18 GHz), temperature (-1 °C–42 °C), moisture content (10.6%–19.2%, wet basis), and bulk density (0.72–0.88 g/cm^3). Explicit calibration equations for moisture prediction at different frequencies and temperatures are provided. Although data obtained by a transmission microwave measurement technique were used, this new approach remains valid in general for other techniques, provided that ϵ' and ϵ'' are determined accurately.**

Index Terms—**Bulk density, calibration functions, density independence, dielectric properties, microwave sensing, moisture content, particulate materials, permittivity.**

I. INTRODUCTION

MOISTURE content is a key parameter in many food processing and agriculture-related industries. It is often defined on a wet basis as the ratio of mass of water m_w to the total mass $m_w + m_d$, where m_d is the mass of dry material. When expressed in percentage, it can be written as

$$M = \frac{m_w}{m_w + m_d} \times 100. \tag{1}$$

Conventional techniques for moisture determination are based either on oven drying and weighing of samples according to a well-defined protocol [1] or chemical titration (Karl Fisher test). Therefore, they are destructive and time- and energy-consuming.

A few decades ago, methods based on the interaction of radiation and moist substances were developed for indirect determination of moisture content and, in some instances, determination of both density and moisture content [2], [3]. These methods have the advantages of being nondestructive, allowing continuous monitoring and control, and, in most instances, not requiring direct contact with the material under test. Among these methods, microwave techniques are attractive because at these frequencies the electric energy is strongly absorbed by water due to the dipolar character of water molecules, and the effect of ionic conductivity is negligible [4]. Also, microwaves are distinguished by their spatial resolution and their safe use. Furthermore, the recent development of reliable low-cost and reduced-size microwave components make microwave methods more competitive with other techniques. Taking advantage of all these features, many moisture meters have been developed and successfully used in various industries [2]. However, more improvements are needed for optimum design of such instrumentation. Besides the economic challenges, calibration and accuracy can be considered as the main tasks in microwave moisture meter development. For this purpose, more research has been devoted to a better understanding of the wave interaction with moist substances by measurements of their dielectric properties [2], [3].

The dielectric properties of a material are intrinsic properties usually expressed by the relative complex permittivity, $\epsilon = \epsilon' - j\epsilon''$, where ϵ' is the dielectric constant and represents the ability of a material to store electric energy, and ϵ'' is the loss factor and represents the loss of electric-field energy in the material. Another parameter often used to describe the amount of loss is the loss tangent, $\tan \delta$, defined as the ratio ϵ''/ϵ'. The dielectric constant and loss factor, as well as the loss tangent, of moist substances are generally dependent on frequency, temperature, density, moisture content, and other factors, such as the material structure and composition and the binding modes of water molecules, which are known to be important but for which no data are available for quantification. The influence of frequency, temperature, density, and moisture content have been explored and reported for many materials [5]–[15]. Knowledge of these dependencies is important in optimum design of meters for industrial use. For instance, when the dielectric properties are used to predict moisture content, effects of other factors such as density and temperature should be either accounted for or canceled out by identifying temperature-insensitive and density-independent calibration functions over a certain frequency range. Ideally, a moisture meter operating at a single frequency or over

a broad frequency range should integrate the two concepts. The temperature-insensitive concept has been considered [8], [12], but no particular relations have yet been identified. In fact, in many industrial applications, measurement of temperature is much easier than measurement of density. Therefore, corrections based on empirically determined relations should be sufficient. It is for this reason that, for the last two decades, more efforts have been dedicated to the development of meters for moisture sensing independent of density. In this paper, a new density-independent calibration function for moisture content determination in particulate materials is defined based on experimental observations, the existence of a simple relationship between the bulk density, and the two components of the relative complex permittivity and the distribution of dissipated and stored energy in dielectrics expressed by $\tan \delta$. The new density-independent function is totally defined in terms of ϵ', ϵ'' and a frequency factor (a_f). Its frequency, density, temperature, and moisture content dependence are presented for a granular material (wheat), and explicit calibration equations for moisture prediction are given along with the standard errors of performance.

II. MATERIALS AND METHODS

A. Measurement Technique and Sample Preparation

Measurements of the relative complex permittivity were performed by using a free-space transmission technique [19]. A sample holder, with 0.1-cm polyethylene walls, rectangular in cross section, providing a sample 10.4 cm thick, 12.4 cm wide, and 15.4 cm high, was filled with wheat kernels and inserted between two WR-62 horn antennas. As the wave propagates through the layer of material, it is attenuated and the phase is shifted. The attenuation ΔA and phase shift $\Delta \Phi$ were measured relative to an empty sample holder between the two antennas by means of a vector network analyzer (Hewlett-Packard[1] 8510B) calibrated in the transmission mode with a response type calibration. To avoid multiple reflections within the sample, the sample thickness, $d = 10.4$ cm, was selected to ensure at least a 10-dB one-way attenuation. The measurements were performed at selected frequencies between 11.3 and 18 GHz which correspond to the best matching of the two antennas with the empty sample holder between them (typically, VSWR <1.2).

Two cultivars of hard red winter wheat, *Triticum aestivum* L., 'Karl' and 'Arapahoe,' grown in Nebraska in 1992 and 1994, respectively, were used in this study. Sublots of different moisture contents were prepared by spraying distilled water on the wheat kernels and storing them in sealed jars for 72 h at 4 °C to equilibrate. Each sample was mixed periodically by rotating the sealed jar so that the moisture content was uniform throughout the entire sample. Before the attenuation and phase shift measurements were performed, the sealed samples were allowed to equilibrate to room temperature (24 °C ± 1 °C) for at least 24 h. The moisture content of each sample was determined by a standard oven method [1] immediately after

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[1] Mention of company or trade names is for purpose of description only and does not imply endorsement by the U.S. Department of Agriculture.

the microwave measurements. This standard specifies drying unground 10-g samples of wheat for 19 h at 130 °C.

Seventy-one moisture levels were measured for the two wheat cultivars. For each sample, the bulk density was gradually increased by settling the grain in the sample holder and adding more kernels. The attenuation and phase shift measurements were repeated for at least three different bulk densities ranging from loosely packed to compacted. Measurements were also taken at temperatures below and above room temperature, 24 °C. Sealed samples of various moisture contents were allowed to stabilize for three days in a chamber where the temperature was adjusted to the desired level. For some samples, temperature was checked before and after the microwave measurement. On average, temperature was stable within ±0.5 °C. An extensive data set was obtained over wide ranges of frequency (11–18 GHz), moisture content (10.6–19.2%, wet basis), bulk density (0.72–0.88 g/cm^3), and temperature (−1 °C–42 °C).

B. Relative Complex Permittivity Determination

When an electromagnetic plane wave is transmitted through a dielectric slab of thickness d placed in air, the complex transmission coefficient τ for perpendicular incidence can be expressed as

$$\tau = |\tau| e^{j\varphi} = \frac{(1 - \Gamma^2)e^{-\gamma d}}{1 - \Gamma^2 e^{-2\gamma d}} \qquad (2)$$

where Γ is the complex reflection coefficient at the air–slab interface and γ is the complex propagation constant.

The complex reflection coefficient Γ at the air–slab interface is

$$\Gamma = \frac{1 - \sqrt{\epsilon}}{1 + \sqrt{\epsilon}}. \qquad (3)$$

The complex propagation constant γ is

$$\gamma = \alpha + j\beta \qquad (4)$$

where α is the attenuation constant and β is the phase constant. They are expressed as functions of the dielectric constant ϵ' and loss factor ϵ'' as follows:

$$\alpha = \frac{2\pi}{\lambda_0} \sqrt{\frac{\epsilon'}{2}\left(\sqrt{1 + \frac{\epsilon''^2}{\epsilon'^2}} - 1\right)} \qquad (5)$$

$$\beta = \frac{2\pi}{\lambda_0} \sqrt{\frac{\epsilon'}{2}\left(\sqrt{1 + \frac{\epsilon''^2}{\epsilon'^2}} + 1\right)} \qquad (6)$$

where λ_0 is the free-space wavelength.

Because the modulus $|\tau|$ and argument φ of the complex transmission coefficient τ are coupled nonlinear functions of α and β, it is not possible to compute α and β directly from $|\tau|$ and φ. Assuming that $\Gamma = 0$ in (2), the complex transmission coefficient reduces to

$$\tau = e^{-\gamma d}. \qquad (7)$$

For low loss materials ($\epsilon'' \ll \epsilon'$), the dielectric constant ϵ' and loss factor ϵ'' are expressed as functions of α and β as

follows [2]:

$$\epsilon' = \left(\frac{\beta}{\beta_0}\right)^2 \qquad (8)$$

$$\epsilon'' = \frac{2\alpha\beta}{\beta_0^2} \qquad (9)$$

where $\alpha = \Delta A/d$, $\beta = \Delta\Phi/d + \beta_0$, and $\beta_0 = 2\pi/\lambda_0$. The attenuation and phase shift are related to the modulus $|\tau_m|$ and argument φ_m of the measured transmission coefficient as follows:

$$\Delta A = 20\log|\tau_m| \qquad (10)$$

$$\Delta\Phi = \varphi_m - 2\pi n \qquad (11)$$

where φ_m is the reading of the instrument $(-\pi \leq \varphi \leq +\pi)$ and n is an integer to be determined when the thickness d of the layer is greater than the wavelength in the material. The integer n can be obtained by repeating the measurement with samples of different thickness or by taking measurements at two frequencies [20].

For $\Gamma \neq 0$, an iterative procedure was used to compute ϵ' and ϵ'' from (2). First the intervals for ϵ' and ϵ'' were selected with their mean values equal to those given by (8) and (9), respectively. Then, for each couple (ϵ', ϵ''), the complex transmission coefficient (2) and the differences $\delta A = |20\log|\tau| - 20\log|\tau_m||$ and $\delta\Phi = |\varphi - \varphi_m|$ were calculated. The iteration process was continued until δA and $\delta\Phi$ converged to the values of uncertainties in attenuation and phase-shift measurements specified for the type of instrument used in this study. In this instance, the iteration procedure was terminated when $\delta A \leq 0.1$ dB and $\delta\Phi \leq 1°$ were achieved. Calculations based on the measurements of the complex transmission coefficient at a midband frequency (14.2 GHz) and room temperature (24 °C), for the investigated moisture content and bulk density ranges, showed that the differences in the values of ϵ' and ϵ'' were less than 2% when compared to those determined with (8) and (9). Therefore, in this study, the dielectric constant and loss factor were calculated according to (8) and (9). Data available in the literature are very limited for this type of material and often are restricted to a single frequency and a single temperature. However, values of ϵ' and ϵ'' compare quite well with those obtained for the same material using a reflection measurement technique where the sample holder was either a short-circuited coaxial line or WR-90 waveguide [5], [11]. According to (8)–(11), the accuracy of the dielectric constant and loss factor measurements is based on the accuracy with which ΔA and $\Delta\Phi$ can be measured. The measuring system performance was improved by appropriate calibration and satisfying requirements for selection of sample thickness and frequencies of best matching of the two antennas for the frequency range under consideration [19]. Repeatability of the measurements was checked by measuring the attenuation and phase shift of two samples of wheat ten times each. Results, at 14.2 GHz and 24 °C show that, for a sample of wheat of 11.8% moisture content, w. b., the mean value of the attenuation was 21.96 dB with a standard deviation of ±0.02 dB and the mean value of the phase shift was -1176.08 ° with a standard deviation of ±1.06 °. For a sample of 18.5% moisture content,
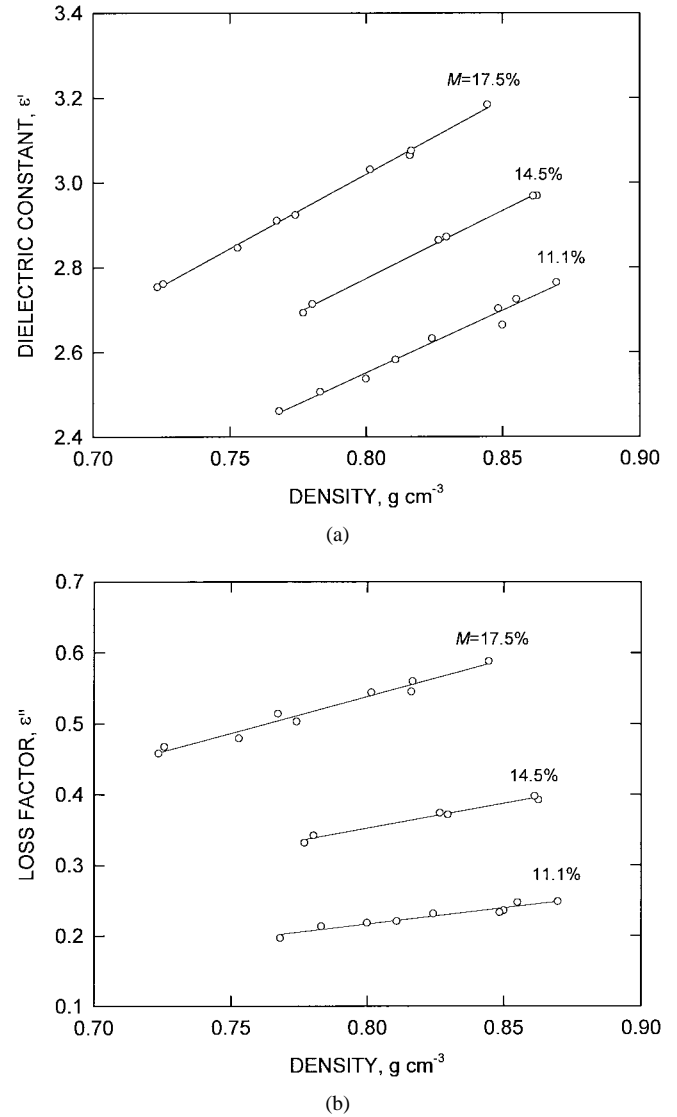

(a)

(b)

Fig. 1. Density dependence of (a) the dielectric constant and (b) the loss factor of hard red winter wheat at 14.2 GHz, 24 °C, and indicated moisture contents.

w. b., the mean value of the attenuation was -46.50 dB with a standard deviation of ±0.1 dB and the mean value of the phase shift was -1372.92 ° with a standard deviation of ±1.21 °. These values are within the ranges specified for this type of instrument.

III. DENSITY AND MOISTURE DEPENDENCE OF THE RELATIVE COMPLEX PERMITTIVITY

In this section, both the density and moisture dependence are considered at a midband frequency of 14.2 GHz and room temperature (24 °C $\pm$ 1 °C).

A. Density Dependence

Bulk density is usually defined as the ratio of the total mass to the total volume. Fig. 1 shows the variations of ϵ' and ϵ'' with bulk density for different moisture contents. Both ϵ' and ϵ'' increase linearly with density. When analyzing these variations, one should keep in mind that the relative
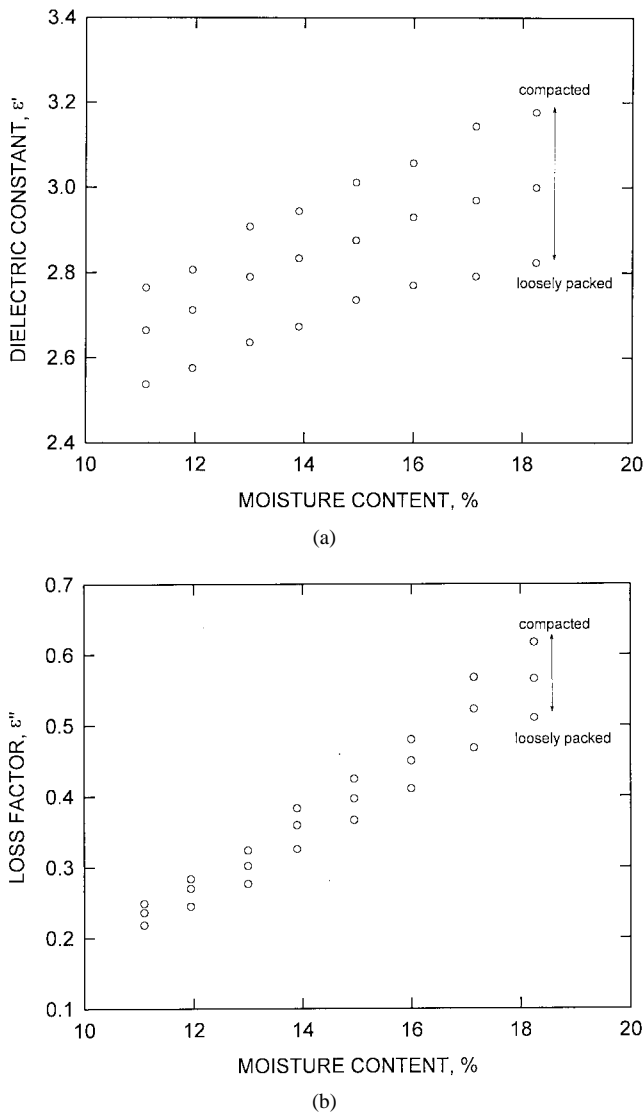
Fig. 2.   Moisture dependence of (a) the dielectric constant and (b) the loss factor of hard red winter wheat at 14.2 GHz, 24 °C, and three different bulk densities ranging from loosely packed to compacted.

complex permittivity considered here is the average effective permittivity measured for a mixture consisting of air and wheat kernels. For a sample holder of fixed volume, by changing the density, the air and kernels proportions are changed, and so the amount of water interacting with the incident wave is also changed. However, at the kernel level, the dielectric properties are constant and assumed equal within the same sample of a given moisture content.

B. Moisture Dependence

The variations of ϵ' and ϵ'' with moisture content are illustrated in Fig. 2 for three different densities ranging from loosely packed to compacted. Since the permittivity of water is large compared to that of dry material (typically the real part is about 2 and the imaginary part is expected to be very small [4]), the measured dielectric constant and loss factor show significant variations with moisture content. A change of about 8% in moisture content produces, on average, a variation

of 12% for ϵ' and, as expected [4], a more important variation of about 140% for ϵ''. Similar trends were found at other temperatures over the investigated frequency range.

IV. DENSITY-INDEPENDENT CALIBRATION FUNCTION

A. Density-Independent Concept

The concept of a density-independent function that is insensitive to bulk density fluctuations in particulate materials was first introduced by Kraszewski et al. [13]. It is based on a two-parameter measurement, namely, the attenuation ΔA and phase shift $\Delta\Phi$ of microwaves passing through a moist substance at given frequency and temperature. They identified empirically the ratio $\Delta\Phi/\Delta A$ as being density-independent over a certain range of moisture contents. Later, Meyer and Schilz [8], [14] expressed this ratio in terms of dielectric constant and loss factor as $[(\epsilon' - 1)/\epsilon''] \times [2\sqrt{\epsilon'}/(\sqrt{\epsilon'} + 1)]$, where the term $2\sqrt{\epsilon'}/(1+\sqrt{\epsilon'})$ was then omitted because of its negligible influence for small ϵ'. They have shown the density-independent character of $(\epsilon' - 1)/\epsilon''$ for different types of materials. Jacobson et al. [7] used these results and developed several industrial moisture meters. Kress-Rogers and Kent [12] and Kent and Kress-Rogers [15] confirmed the consistency of the density-independence of these ratios by measurements on powders (coffee and milk). However, they have demonstrated that the ratio $\Delta\Phi/\Delta A$ was less sensitive to density variations than $(\epsilon' - 1)/\epsilon''$. Recently, Menke and Knöchel [16] extended the use of the ratio of phase shift and attenuation to higher and lower moisture contents by considering the ratio $(\Delta\Phi/\Delta\omega)/(\Delta A/\Delta\omega)$, where $\omega = 2\pi f$ was swept over a broad range of frequency f. In general, applications based on the principle of a two-parameter measurement and the ratio $\Delta\Phi/\Delta A$ [17], are limited to transmission techniques. It is more attractive to define a density-independent calibration function that is directly related to the dielectric properties of the material under test and therefore can be applied regardless of the measurement technique.

Figs. 1 and 2 show that density and moisture content affect the measured dielectric properties in a similar fashion. Therefore, when moisture content is the target parameter, the density fluctuations will cause undesirable errors in moisture determination. These errors can be reduced by a separate measurement of density and correcting the meter output, or by identifying density-independent entities exclusively dependent on moisture content. A separate density measurement always involves an additional cost for a practical moisture meter and adds more technical complications in the conception and installation of the measuring system. The density-independent concept is considered to be a better alternative.

B. Relationship Between the Bulk Density ρ and ϵ' and ϵ''

Fig. 3 shows the loss factor plotted against the dielectric constant, at 14.2 GHz and 24 °C, for different moisture contents and bulk densities. A cluster of data points is obtained in the complex plane. The nature of the dependence of the two components of the relative complex permittivity on bulk density (see Fig. 1) implies that dividing both ϵ' and ϵ''
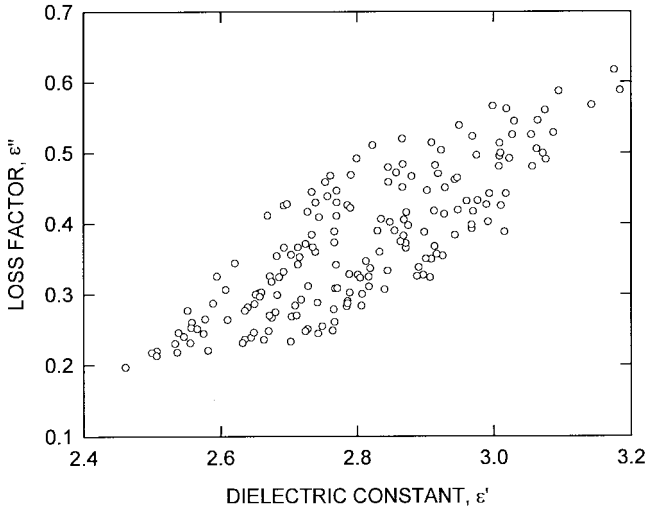
Fig. 3. Locus in the complex plane of the relative complex permittivities of samples of hard red winter wheat of different moisture contents and bulk densities at 14.2 GHz and 24 °C.

Fig. 4. Locus in the complex plane of the relative complex permittivities divided by bulk density for samples of hard red winter wheat of different moisture contents at 14.2 GHz and indicated temperatures.

by the bulk density should reduce the density effect. This is illustrated in Fig. 4 where the points are now located along the same straight line in the complex plane. The same line is obtained with data collected at −1 °C and +42 °C. These points overlap those measured at 24 °C, with the data corresponding to the lowest temperature located in the lower region of the graph. Therefore, at a given frequency, the temperature effect corresponds to a translation along the line. Similar dependence was observed at other frequencies. This reflects the thermal behavior of "bound" water in organic materials, where the water molecular dipoles occupy well-defined sites and are not free to rotate because of their bonds with surrounding neighbors and the nature of forces acting on these dipoles. There are different degrees of binding, and each water molecule may have up to three bonds (ice) depending on the structure and composition of the material and the amount of water available. As the temperature increases, the mobility of the water molecules increases, making their contribution to the polarization of the medium higher and increasing the losses at the same time. In contrast, the lower the temperature, the slower the action of the molecular dipoles, and the losses tend to be negligible. By extrapolation, the losses reach the zero value at a temperature where the electrical behavior of the material tends toward that of the dry material (see Fig. 4). From the complex-plane representation, the effects of temperature and moisture content seem to be perfectly interchangeable. The normalized dielectric properties for a given moisture content at high temperatures are those for a higher moisture content at lower temperatures.

At each frequency, a data set is formed by data points, corresponding to measurements at all temperatures and moisture contents (396 data points), which can be fitted with a linear regression of the form

$$\frac{\epsilon''}{\rho} = a_f \left(\frac{\epsilon'}{\rho} - k \right) \qquad (12)$$

where a_f and k are the slope and x-axis intercept, respectively. The values of a_f and k and the coefficients of determination r^2
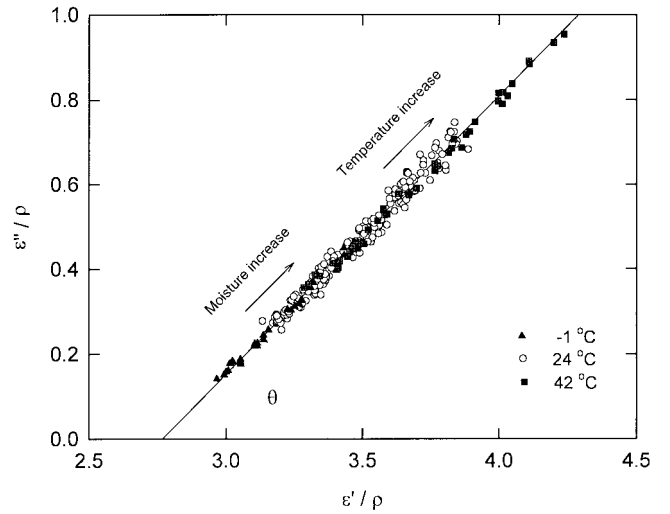
TABLE I
SLOPE a_f, INTERCEPT CONSTANT k, AND COEFFICIENT OF DETERMINATION r^2 OF THE LINEAR REGRESSIONS PLOTTED IN THE COMPLEX PLANE (SEE FIG. 4) FOR DIFFERENT FREQUENCIES. DATA IS COMPOSED OF MEASUREMENTS AT ALL MOISTURE CONTENTS AND TEMPERATURES

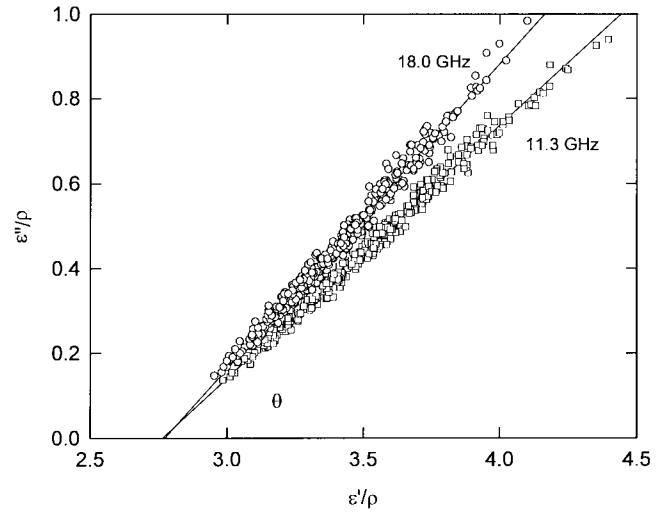| Frequency, GHz	11.3	12.3	13.3	14.2	15.2	16.8	18
a_f	0.5960	0.6060	0.6255	0.6474	0.6596	0.6902	0.7187
k	2.765	2.776	2.776	2.758	2.747	2.756	2.773
r^2	0.9907	0.9875	0.9888	0.9900	0.9884	0.9891	0.9868

Fig. 5. Locus in the complex plane of the relative complex permittivities divided by bulk density for samples of hard red winter wheat of different moisture contents at two extreme frequencies and different temperatures $(-1 \leq T(°C) \leq +42)$. Also shown is the angle in the complex plane, $\theta = \arctan((\epsilon''/\rho)/(\epsilon'/\rho) - k)$, with θ independent of moisture content and temperature, and a function of frequency alone.

for different frequencies are given in Table I. In Fig. 5, ϵ''/ρ versus ϵ'/ρ is plotted for all temperatures and moisture contents for the lowest and highest frequencies considered in this study. Similar lines are obtained at the other frequencies with a slope increasing as the frequency increases (see Table I). The

intercept constant k is essentially constant for all frequencies and has an average value of 2.76. All the lines cross the x axis at a common point (2.76, 0). This point corresponds to $(\epsilon''/\rho) = 0$ and can be considered as the coordinates, in the complex plane, of the dielectric properties of a totally dry air-kernel mixture or that of a sample of any moisture content at very low temperature. Measurements carried out on dried samples of different bulk densities over the same frequency range and at room temperature provided a value of 2.74 for ϵ'/ρ, which agrees well with that obtained by graphical extrapolation. As expected, the dielectric properties of the dry material are nondispersive and thus showed no variation with frequency. Although the microwave measurements on dry samples were made at room temperature, there is no reason to expect these properties to be temperature-dependent. The frequency effect can be described as a rotation of the line about the point $(k, 0)$ making an angle θ in the complex plane with the $\epsilon''/\rho = 0$ axis

$$\theta = \arctan\left(\frac{\dfrac{\epsilon''}{\rho}}{\dfrac{\epsilon'}{\rho} - k}\right). \tag{13}$$

Thus, θ is independent of moisture content and temperature and is a function of the frequency alone. Therefore, a relationship between the slope a_f and the frequency can be established empirically. A linear regression shows that a_f is related to the frequency f as follows:

$$a_f = 0.0184\,f + 0.3826 \quad r^2 = 0.993 \tag{14}$$

where f is in gigahertz.

As a result, from (12), the bulk density can be expressed in terms of ϵ' and ϵ'' as follows:

$$\rho = \frac{1}{k}\left(\frac{a_f\epsilon' - \epsilon''}{a_f}\right). \tag{15}$$

Equation (15) shows that a simple relationship exists at any given frequency between the bulk density and the two components of the relative complex permittivity. Therefore, from the measured dielectric constant and loss factor and knowledge of the intercept constant k and relation (14), the bulk density can be determined without prior knowledge of moisture content and temperature.

C. Definition of the Density-Independent Calibration Function Ψ

The loss tangent is the entity that best describes the distribution between dissipated and stored energy in dielectrics [21]. At high frequencies, when an electromagnetic wave interacts with a moist substance, water is the dominant factor associated with the energy dissipated in the material. Therefore, a density-independent function based on the loss tangent should better describe the wave–material interaction from the energy point of view. Fig. 6 shows the variations of $\tan\delta$ with moisture content at 14.2 GHz and 24 °C. As with ϵ' and ϵ'', $\tan\delta$ increases with moisture content. However, the density effect is still visible at each moisture level. To reduce this effect,
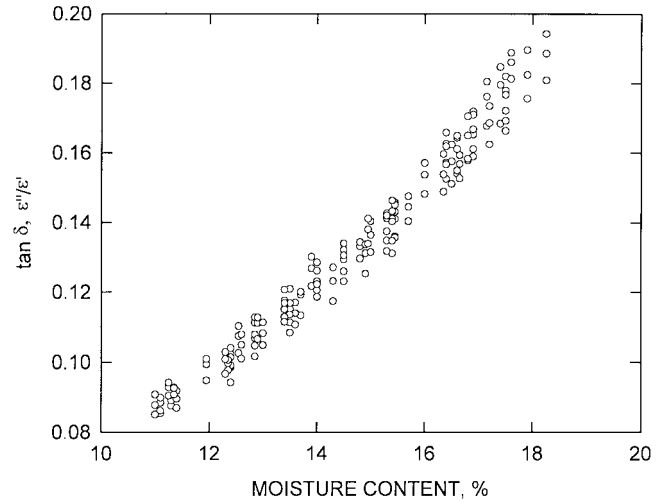

Fig. 6. Moisture dependence of $\tan\delta$ for hard red winter wheat at 14.2 GHz, 24 °C, and different bulk densities ranging from loosely packed to compacted.

a density-independent function Ψ is defined as the ratio of $\tan\delta$ to the bulk density from (15). The following expression is obtained for Ψ:

$$\Psi = \frac{ka_f}{a_f\epsilon' - \epsilon''}\left(\frac{\epsilon''}{\epsilon'}\right). \tag{16}$$

Ψ is fully defined in terms of dielectric properties of the material, the intercept constant k, and a frequency factor a_f independent of moisture content and temperature. For practical purposes, the fewer extrinsic parameters involved in the calibration equation, the easier is the calibration procedure. Examination of (16) indicates that for a given material at a given frequency the product $k \cdot a_f$ is a constant and thus can be omitted. Therefore, the density-independent calibration function Ψ when used in calibrating a measuring system operating at a single frequency can be simplified to

$$\xi = \frac{\epsilon''}{\epsilon'(a_f\epsilon' - \epsilon'')}. \tag{17}$$

In this simplified form only one extrinsic parameter is needed, namely the frequency factor a_f.

D. Density and Moisture Dependence of ξ

The density dependence of ξ is shown in Fig. 7 for the same data used in Fig. 1. The straight lines are now essentially parallel to the ρ axis, thus demonstrating the density-independent character of ξ. Variation of $\sqrt{\xi}$ with moisture content is shown in Fig. 8, for the same data used in Fig. 6. The square root of ξ increases linearly with moisture content. The spread of points related to bulk density variation at each moisture level is significantly reduced. Both Figs. 7 and 8 confirm the density-independent character of ξ.

E. Frequency and Temperature Dependence of ξ

To complete the study of ξ, the frequency and temperature dependence have to be considered. The frequency dependence is illustrated in Fig. 9 for two levels of moisture. ξ decreases
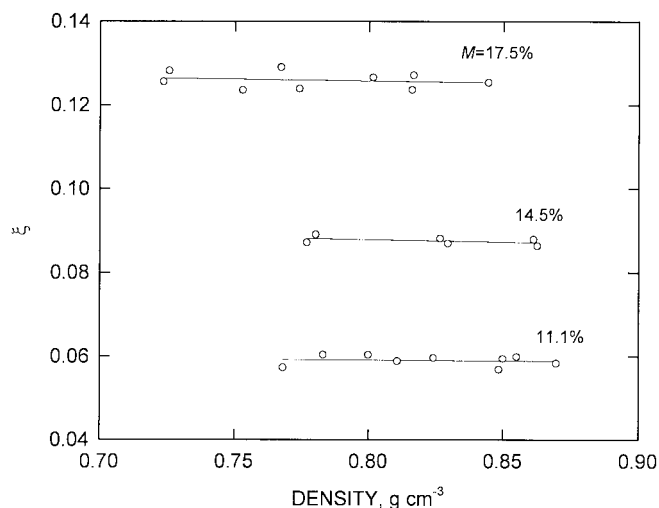
Fig. 7. Density dependence of ξ at 14.2 GHz, 24 °C, and indicated moisture contents for hard red winter wheat.
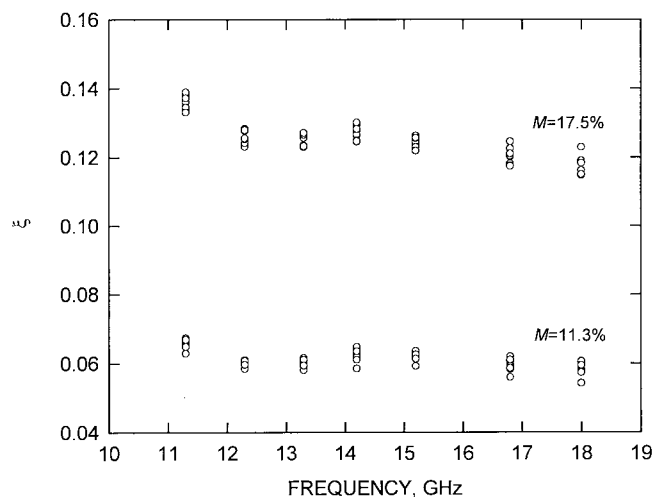

Fig. 9. Frequency dependence of the density-independent calibration function (ξ) at 24 °C and indicated moisture contents for hard red winter wheat.
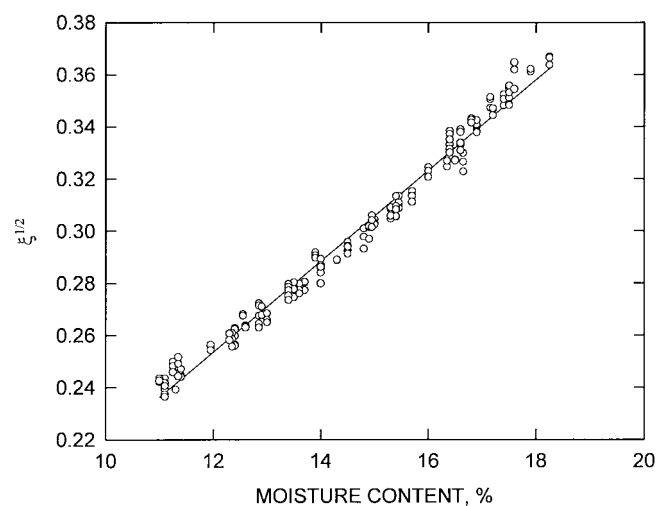

Fig. 8. Square root of the density-independent calibration function (ξ) versus moisture content at 14.2 GHz and 24 °C for hard red winter wheat.

Fig. 10. Temperature and moisture dependence of ξ at 14.2 GHz for hard red winter wheat.

slightly with increasing frequency, in a way similar to the variation with frequency noted for ϵ' and ϵ'' [19]. Therefore, for a moisture sensing instrument operating at a single frequency, the frequency selection should be based on other criteria, such as the dynamic range and sensitivity of the system, the thickness of the layer, the dimensions and shape of the kernels (scattering phenomenon), etc.

The temperature dependence of $\sqrt{\xi}$ at 14.2 GHz is shown in a three-dimensional illustration (Fig. 10) along with the moisture dependence. A network of essentially parallel lines is obtained. As expected, $\sqrt{\xi}$ increases linearly with moisture content and temperature. The increase of $\sqrt{\xi}$ with temperature reflects the thermal behavior of "bound" water as interpreted above.

V. CALIBRATION EQUATION FOR MOISTURE DETERMINATION

Based on Fig. 8, at 14.2 GHz and 24 °C, a change of about 8% in moisture content produces a variation of 50% for $\sqrt{\xi}$, which is between that of the dielectric constant and loss factor
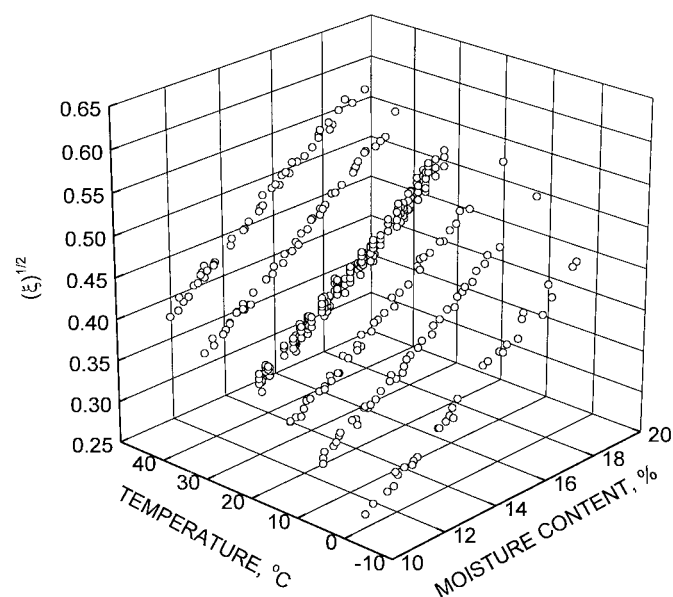
(Fig. 2). At each temperature, a linear regression of the form

$$\sqrt{\xi} = A \cdot M + B \tag{18}$$

was used to fit the data, where A is the slope, M is the moisture content in percent, and B is the intercept. The values of A and B and the coefficient of determination r^2 are given in Table II(a)–(c) at different frequencies and temperatures. The slope A generally shows very small temperature dependence, and considering the errors in ϵ' and ϵ'', A can be assumed constant. In contrast, B increases with temperature. An explicit relation can be established empirically between B and the temperature. For example, at 14.2 GHz and $9 \leq T(°C) \leq 34$,

TABLE II
SLOPE A, INTERCEPT B, AND COEFFICIENT OF DETERMINATION r^2
OF THE LINEAR REGRESSION REPRESENTING $\sqrt{\xi}$ VERSUS M
(SEE FIG. 8) AT DIFFERENT FREQUENCIES AND TEMPERATURES.
(a) $f = 11.3$ GHz. (b) $f = 14.2$ GHz. (c) $f = 18$ GHz.

Temperature, °C	-1	9	16	24	34	42
A	0.0173	0.0176	0.0186	0.0187	0.0191	0.0174
B	-0.0023	0.0200	0.0242	0.0392	0.0546	0.0933
r^2	0.9858	0.9916	0.9911	0.9883	0.9946	0.9924

(a)

Temperature, °C	-1	9	16	24	34	42
A	0.0161	0.0160	0.0172	0.0174	0.0183	0.0177
B	0.0076	0.0303	0.0348	0.0446	0.0540	0.0800
r^2	0.9811	0.9905	0.9878	0.9855	0.9913	0.9921

(b)

Temperature, °C	-1	9	16	24	34	42
A	0.0144	0.0148	0.016	0.017	0.0179	0.0182
B	0.0187	0.0362	0.0376	0.0430	0.0488	0.0636
r^2	0.9843	0.9888	0.9845	0.9772	0.9852	0.9857

(c)

a linear regression gives

$$B(T) = 9.77 \cdot 10^{-4} \cdot T + 0.0206 \qquad r^2 = 0.99 \qquad (19)$$

where T is the temperature in degrees Celsius.

The high values of r^2 show the high correlation between $\sqrt{\xi}$ and M and demonstrate that the density-independent calibration function ξ can be of potential use for moisture content prediction. Therefore, when (18) is used, the moisture content in percent, at a given frequency and temperature, can be calculated from the following calibration equation:

$$M(\%) = \frac{\sqrt{\xi(a_f, \epsilon', \epsilon'')} - B(T)}{A}. \qquad (20)$$

In Fig. 11, the moisture predicted by (20) at room temperature and 14.2 GHz is plotted versus oven moisture content. The data points lie along the straight line which corresponds to the ideal relationship. The effectiveness of (20) in predicting moisture content can be evaluated by calculating the standard error of performance (SEP) which is defined as

$$\text{SEP} = \sqrt{\frac{1}{N-1} \sum_{i=1}^{N} (\Delta M_i - M)^2} \qquad (21)$$

where N is the number of samples, ΔM_i is the difference in moisture content predicted from that determined by the reference method for the ith sample, and
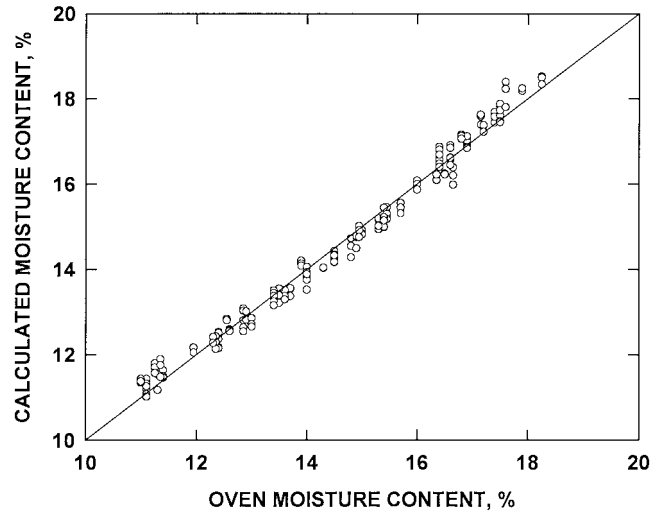
$$M = (1/N) \sum_{i=1}^{N} \Delta M_i.$$

Fig. 11. Predicted moisture content calculated by (20) versus oven-drying moisture content for hard red winter wheat.

TABLE III
SEP OF MOISTURE CONTENT PREDICTION AT DIFFERENT FREQUENCIES AND 24 °C

Frequency, GHz	11.3	12.3	13.3	14.2	15.2	16.8	18.0
SEP, %*	0.24	0.22	0.23	0.26	0.26	0.30	0.32

For the SEP calculation, at each frequency, the data set was split into two subsets covering the same moisture range. One subset was used for calibration, i.e., to determine the coefficients A and B in (18). The second one was used for validation, i.e., to calculate moisture content according to (20). Values of the SEP for different frequencies and room temperature are given in Table III. Their increase with frequency might be related to the scattering effect and/or errors in measurement of attenuation and phase shift, both more pronounced at higher frequencies. At the 95% confidence level, the uncertainty for moisture content determination over the investigated frequency and bulk density ranges at room temperature can be taken as twice the SEP value. On average, in this instance, moisture content can be determined from dielectric properties measurement with $\pm 0.5\%$ error (percent moisture content). The accuracy with which M is determined depends mainly on the accuracy with which the dielectric properties of the material are measured. In this respect, the choice of the measurement technique is important. Moreover, as in any calibration procedure for indirect determination of a particular parameter, moisture content in this instance, the choice of the reference method is an important factor. The oven-drying technique was used in this paper.

VI. SUMMARY AND CONCLUSIONS

A density-independent calibration function for moisture sensing in particulate materials was defined, based on experimental observations, a complex-plane representation of the normalized dielectric properties, and the energy distribution in dielectrics expressed by $\tan \delta$. Both the bulk density and new density-independent calibration function are expressed in terms of the two components of the relative complex permit-

tivity (ϵ' and ϵ'') and a constant (k) and a frequency factor (a_f), proper to each particular material. The new density-independent calibration function for moisture determination takes into account the energy distribution and integrates effects of both frequency and temperature. Its density independence was shown for wheat over wide ranges of density, frequency, and temperature. Explicit calibration equations for bulk density and moisture content determination can be generated at different frequencies and temperatures from Tables I and II. The accuracy with which both entities can be determined is based mainly on the accuracy with which ϵ' and ϵ'' are determined. Values of the SEP, given in Table III, indicate that, at the 95% confidence level, moisture content in the range 10–19%, wet basis, can be determined within $\pm 0.5\%$ error on average. Since this approach is independent of the measurement technique, it provides more freedom of choice for the appropriate technique for nondestructive sensing of either entity, i.e., bulk density or moisture content. Therefore, it can be of potential use in many applications, particularly for on-line configurations for monitoring and control of such entities.

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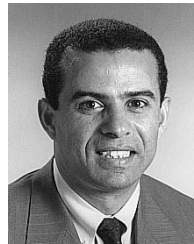
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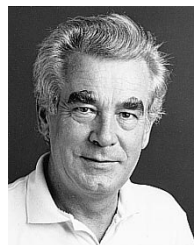
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