Journal of Thermal Analysis and Calorimetry, Vol. 82 (2005) 253–260

MEASUREMENT OF MOISTURE IN GRAINS AT EXTREME TEMPERATURES
Very high frequency dielectric method

P. Mészáros[*] *and D. B. Funk*

Corvinus University of Budapest, Faculty of Food Science, Physics and Control Department, Somlói út 14-16, Budapest, Hungary 1118

This research has demonstrated that the dielectric method can be used successfully for grain moisture measurements for diverse grain types over wide ranges of temperatures if the grain moisture content is below certain (grain-specific) threshold values. These high-moisture limits were estimated. Optimum temperature correction coefficients for 149 MHz moisture measurements were determined for grain samples at different moisture levels. This information should be very helpful for grain moisture meter manufacturers and for moisture meter users who need to determine grain moisture contents at temperature extremes to be able to market grain more efficiently.

Keywords: *dielectric, grain, moisture, phase change, temperature, VHF*

Introduction

The radio-frequency (RF) dielectric method measures moisture content in grain by sensing the dielectric constant of grain samples. Since the dielectric constant of water is much higher than that of other grain constituents, the method is very sensitive to the amount of water present in the sample. Grain moisture meters based on the RF dielectric method are used widely around the world. In the investigation of foods by thermal analysis and calorimetric techniques, many physico-chemical effects can be observed in the temperature range between –50 and 300°C [1]. Sample temperature is one of the most important factors influencing dielectric grain moisture results since temperature affects the dielectric constant of water, the degree of hydrogen bonding between water and other molecules, and the magnitude of interfering factors such as ionic conductivity. The effects of sample temperature are further complicated by the phase change of ‘free’ water at 0°C. Ice has a much lower dielectric constant than water in the RF and higher frequency ranges. Since some types of grain are harvested during the onset of cold weather, the grain may be very cold when harvested. Storage in very cold weather (with or without prior drying) may also cause the grain temperature to be very low when the grain is removed from storage for sale. Other grain types, such as winter wheat, are typically harvested during the hottest periods of the summer. Waiting for grain to equilibrate to near room temperature to obtain accurate moisture measurements causes unacceptable delays in handling. Therefore it is very desirable to be able to make accurate moisture measurements on grain that is quite warm or very cold–even below the freezing point of water. The objectives of this research were to determine an appropriate temperature correction function and specific coefficients to permit accurate grain moisture measurements at 149 MHz over a wide range of temperatures, to estimate the upper moisture limits for measuring grain at temperatures below 0°C, and to investigate the differences in temperature sensitivity at 149 MHz and lower measurement frequencies that are currently used most widely for grain moisture measurements.

Materials and methods

Test samples

Bulk grain samples were obtained from grain receiving stations in Hungary (Herceghalom and Szombathely). The grain types tested included: soybeans (*Glycine soja*), soft wheat (*Triticum aestivum*), sunflower (*Helianthus annuus*), rapeseed (*Brassica napus*), autumn barley (*Hordeum vulgare*), and oats (*Avena sativa*). Moistened sand was used to compare the dielectric behavior of water in grain to that of ‘free’ water. The moisture contents of the samples and sand were adjusted as necessary by adding distilled water to the samples, mixing the samples thor-

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[*] Author for correspondence: peter.meszaros@uni-corvinus.hu

1388–6150/$20.00

Akadémiai Kiadó, Budapest, Hungary
Springer, Dordrecht, The Netherlands

oughly in sealed containers, and allowing them to equilibrate under refrigeration for at least one week prior to testing.

Instrumentation

Dielectric characteristics at 149 MHz for samples at different moisture levels were tested by a prototype moisture measurement system (Fig. 1a). This system included a signal generator type G4-107, a special parallel-plate transmission-line test cell (Fig. 1b), and three directional couplers to sample the drive signal level and the incident and reflected signals at the test cell. An Analog Devices type AD8302 Gain/Phase Detector integrated circuit was used to sense the relative gain and phase of the incident and reflected signals. An HP-3457A Digital Multimeter was used to measure the drive signal, gain and phase voltages from the AD8302, and the current from an Analog Devices type AD-590 semiconductor temperature sensor, which was mounted inside the test cell. The HP-3457A was connected to a PC-compatible computer through a GPIB interface to permit unattended data collection as controlled by a QBasic program.

An HP 4284A Precision LCR Meter and a parallel-plate test cell were used to measure the dielectric spectra of samples over the 30 Hz to 1 MHz frequency range. The test cell used for those tests was a modified GAC-II moisture meter test cell. (DICKEY-john Corporation, Auburn, Illinois, USA) The temperature sensor for that test cell consisted of two series-connected silicon diodes driven by a constant 1 ma current. The voltage across the diodes was measured by a HP 3457A Digital Multimeter. Both the HP 4284A and the HP 3457A were controlled by a PC-compatible computer (and a QBasic program) through a GPIB interface.

Test methods

The sample was loaded into the test cell (either the transmission-line test cell or the parallel-plate test cell) with a process intended to achieve a moderately high packing density so that settling was minimized during subsequent handling. Grain was poured slowly into the test cell while the test cell was vigorously shaken horizontally (approximately 1 cm displacement, 4 Hz frequency). After loading, the top of the test cell was sealed (with wide plastic adhesive tape) to minimize moisture loss. The test cell was placed in an insulating enclosure made of expanded polystyrene. The test cell (in the bottom half of the enclosure) and the (removed) top half of the insulating enclosure were placed in a laboratory freezer (set to approximately –25°C) and allowed to equilibrate for at least 12 h. After the equilibration period, the insulated test cell assembly was removed from the freezer, closed, transported to the laboratory area (approximately 22°C), connected to the measuring system, and allowed to warm slowly to room temperature. The temperature was automatically checked every 30 s and a set of dielectric measurements was initiated each time the sample temperature changed by more than 0.5°C from the temperature of the previous data set.

When the grain had warmed to near room temperature, the automatic data collection sequence was manually terminated. The test cell was placed in a laboratory oven, which was set to approximately 50°C, and was allowed to equilibrate for at least 6 h. Then the test cell (in its insulating enclosure) was connected again to the measuring system. When the grain had cooled to near room temperature, the automatic data collection sequence was terminated. The sample was emptied from the test cell and weighed so that the sample density could be determined.

The data were processed using custom-designed software written in Mathcad [6]. The measured gain

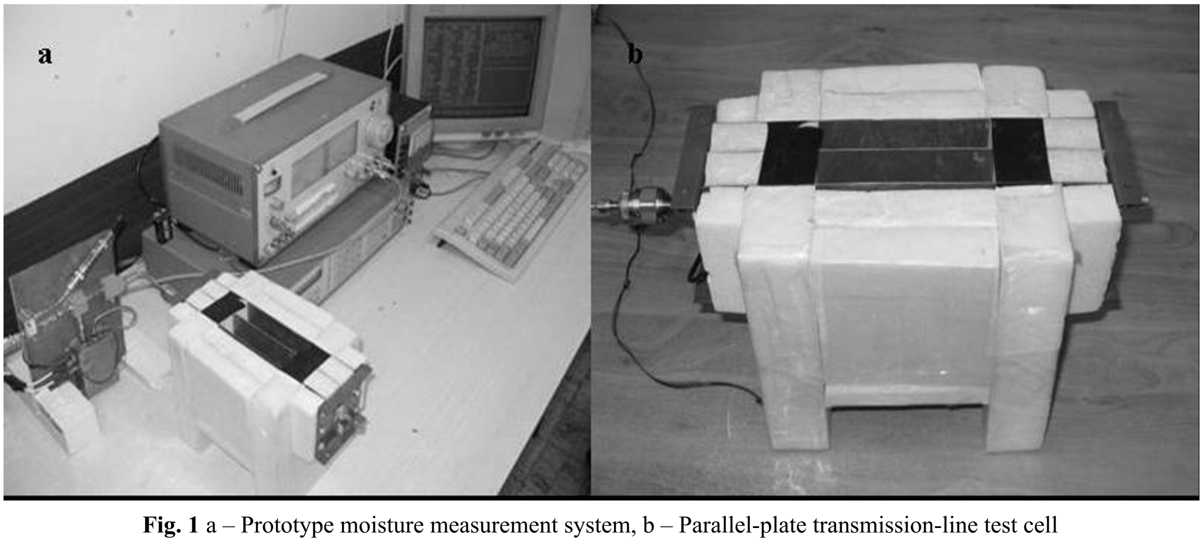

Fig. 1 a – Prototype moisture measurement system, b – Parallel-plate transmission-line test cell

and phase voltage levels from the gain/phase detector were converted to complex reflection coefficient (Γ). The complex reflection coefficient values were converted to complex dielectric constant (ε) with an iterative solver based on signal-flow graphs as shown by Funk [2]. The dielectric constant values were density-corrected and predicted moisture values were determined from the dielectric measurements by a fifth order polynomial equation [4]. The 'unified' calibration coefficients used for the predictions were calculated from dielectric data and reference moisture values obtained from research performed by the United States Department of Agriculture – Grain Inspection, Packers and Stockyards Administration. These data were developed based on samples representing all growing areas of the United States for the 1998 through 2002 crop years. The reference moisture value for the temperature test for each test sample was determined by the appropriate Hungarian Ministry of Agriculture standard air oven method (Hungarian Standard 6367/3-83).

Results and discussion

Figure 2 shows typical dielectric characteristics (at 149 MHz) that were measured as samples warmed slowly to room temperature. Moist sand (14% M) showed the expected discontinuity in dielectric characteristics at 0°C because of the dramatically different relaxation frequencies of ice and 'free' water. Grain samples did not show such a discontinuity. Much of the water in grain is associated more or less tightly with polar sites on grain constituents through hydrogen bonding. Such water does not show a dramatic discontinuity in the dielectric constant value at 0°C. For dry to moderately moist grain samples, where virtually all of the water is bound to grain constituents, the variation of dielectric constant with temperature is nearly constant over a wide range of temperature that extends well below 0°C. However, at high moisture contents, some of the water in the grain is capable of changing phase and becomes invisible to the dielectric method at temperatures below the freezing point of water [2].

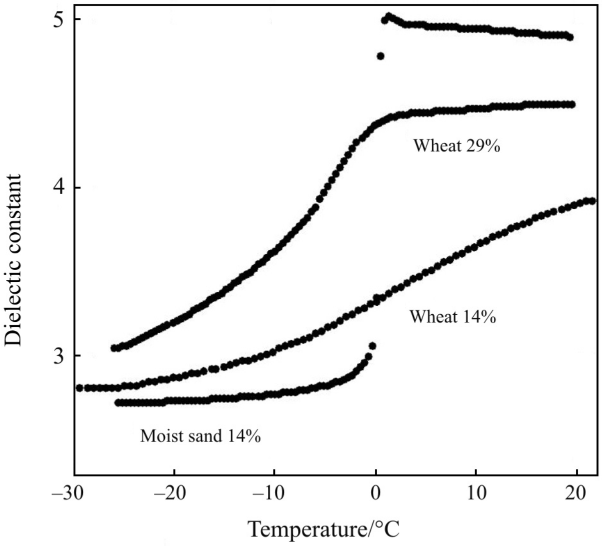

Fig. 2 Dielectric constant *vs*. temperature for moist sand and two wheat samples

Table 1 Optimum temperature correction coefficients (%moisture/°C) for six grain types at three measurement frequencies

Grain type	*KTC@* 2 MHz	*KTC@* 20 MHz	*KTC@* 149 MHz
Wheat	0.099	0.094	0.061
Barley	0.143	0.11	0.102
Soybean	0.167	0.14	0.075
Sunflower	0.088	0.088	0.052
Oats	0.244	0.127	0.126
Rapeseed	0.173	0.105	0.058

Prior research [3] showed a distinct moisture threshold for yellow-dent field corn. Below about 20% moisture, grain moisture could be determined accurately at temperatures as low as –25°C. However, for grain samples above 20% moisture, the moisture above 20% disappeared from the dielectric measurement when the grain was very cold. Therefore, the upper moisture limit for measuring 'frozen' corn by the dielectric method is approximately 20%. Mészáros and Funk [4] presented the moisture limits at low radio frequencies (200 kHz – 20 MHz) for six additional grain types (wheat, oats, rapeseed, barley, soybeans and sunflower).

Research collaboration between USDA-Grain Inspection, Packers and Stockyards Administration (GIPSA) and USDA-Agricultural Research Service (Athens, GA) over the period of 1995 to 2001 resulted in an improved RF dielectric method, the 'Unified Moisture Algorithm,' that effectively combines many diverse grain types into a single unified calibration. This method is capable of moisture measurement accuracy that is equal to or better than what is achievable for grain-specific calibrations with current instruments. In this method, similar grain types are placed in grain groups that can be measured without knowing the specific grain type. The method uses a single dielectric measurement at 149 MHz [2]. That study suggested the use of a simple moisture-independent correction function (Eq. (1)) for temperature, based on the limited data available, but stated that more research was needed to optimize the function.

$$\%M_{TC}=\%M-KTC(T-25) \qquad (1)$$

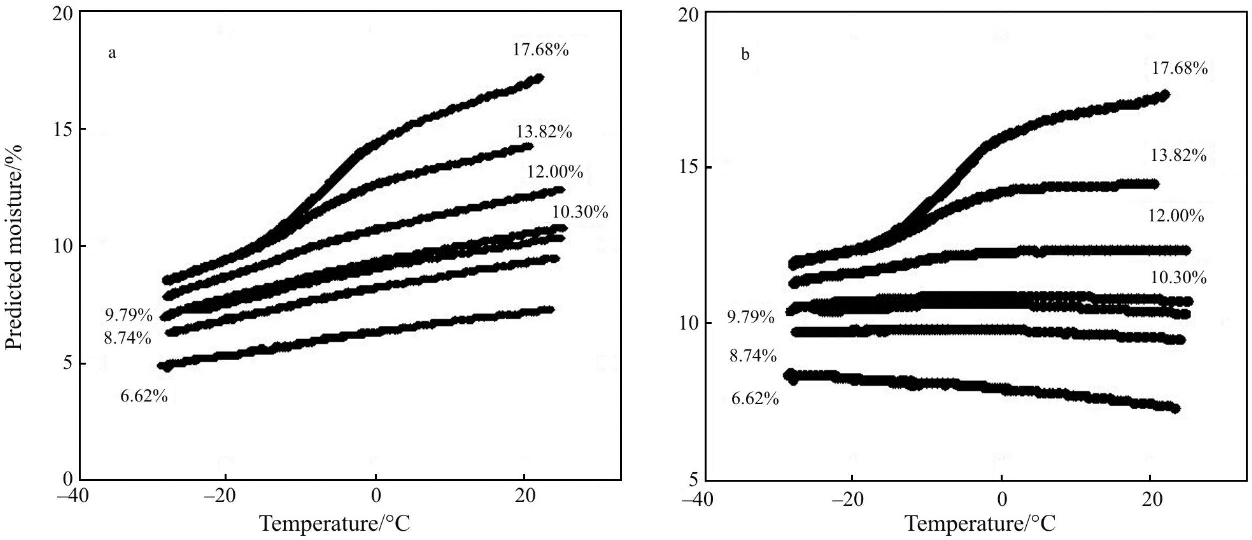

Fig. 3 Predicted moisture values for sunflower samples at 20 MHz, a – without temperature correction; b – with temperature correction

where $\%M_{\mathrm{TC}}$ is the temperature-corrected moisture content, $\%M$ is the predicted moisture content without temperature correction, *KTC* is temperature correction coefficient, T is the measured sample temperature in °C.

Figure 3 presents an example of the application of Eq. (1) to provide temperature correction for sunflower seed at 20 MHz. Predicted moisture values for dielectric measurements at 20 MHz are shown without and with temperature correction. Without temperature correction (Fig. 4a) the predicted moisture values change monotonically over the range of –30 to 20°C. Except for the two highest moisture samples at temperatures below 0°C, the curves appear nearly parallel – suggesting the use of a simple temperature correction function such as Eq. (1). Figure 4b shows the effectiveness of Eq. (1) for providing temperature correction for sunflower seed at 20 MHz. The moisture prediction error is considerably reduced, as seen by the nearly flat curves above 0°C; but the lowest moisture samples are over-corrected slightly and the highest moisture samples are under-corrected. That is, the single temperature correction (percent moisture per °C) is too large for the lowest moisture samples and too small for the highest moisture samples.

Because of the moderate effectiveness afforded by Eq. (1) at low frequencies, the same temperature correction strategy was applied to the 149 MHz data for sunflower seed (Fig. 4). Again, the temperature correction was moderately effective, but the high and low moisture samples still showed significant errors

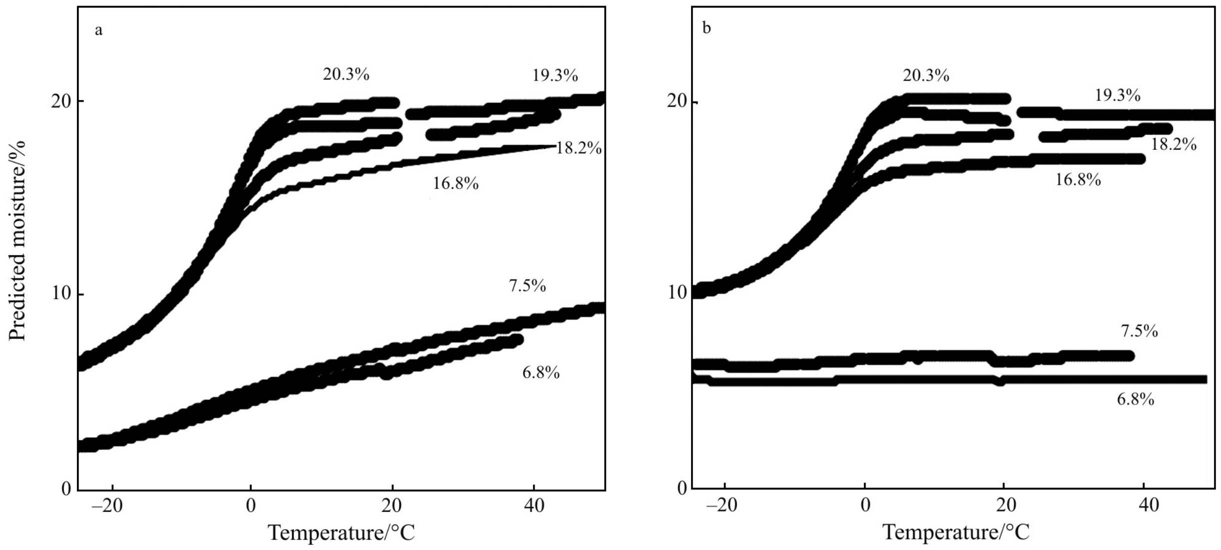

Fig. 4 Predicted moisture values for sunflower samples at 149 MHz, a – without temperature correction; b – with temperature correction (Eq. (1))

in predicted moisture content. At 149 MHz the low moisture samples were under-corrected and the high moisture content samples were overcorrected (above zero °C). This was opposite to the residual errors observed at 20 MHz and below.

These results suggested that a better temperature correction function was needed for moisture measurements at 149 MHz. The optimum temperature coefficients (percent per °C) for individual grain samples were calculated for three different measurement frequencies (2 MHz, 20 MHz and 149 MHz). Data from prior research [4] were used for the 2 MHz and 20 MHz calculations. The optimum temperature coefficient (*KTC*) for a sample was defined as the value that

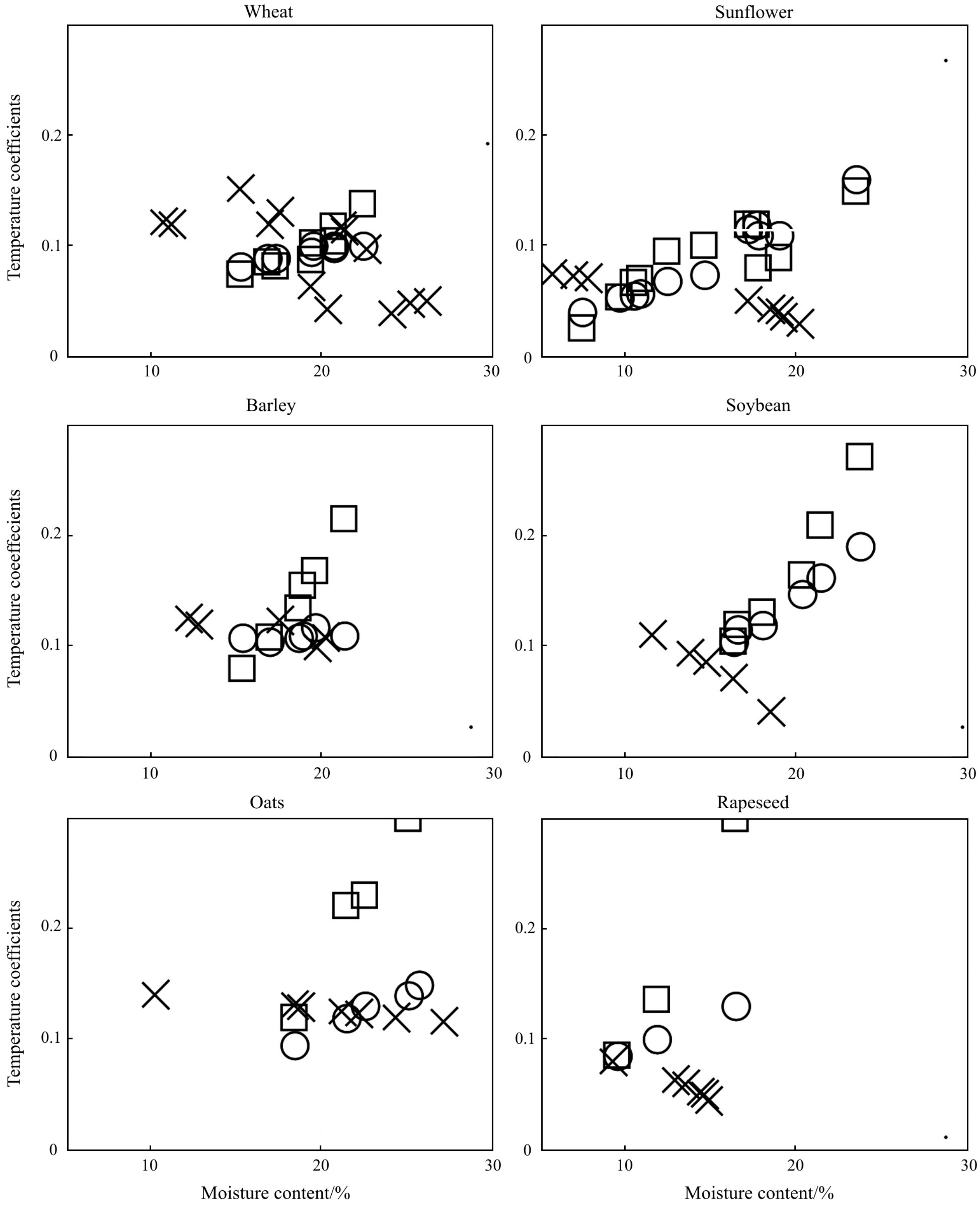

Fig. 5 Optimum sample temperature coefficients (*KTC*) *vs.* moisture content for six grain types at 2 MHz (□), 20 MHz (○), and 149 MHz (x)

minimized the variation in predicted moisture content (Eq. (1)) for all measurements at temperatures above 0°C. Figure 5 shows that the optimum sample temperature coefficients increased with moisture content for low frequency measurements and decreased with moisture content for VHF measurements. These figures also demonstrate greater variability and larger magnitude for *KTC* at lower frequencies.

Inspection of Fig. 5 revealed that the temperature correction was nearly linearly dependent on moisture at 149 MHz and suggested the functional form shown in Eq. (2). Linear regression was used to determine

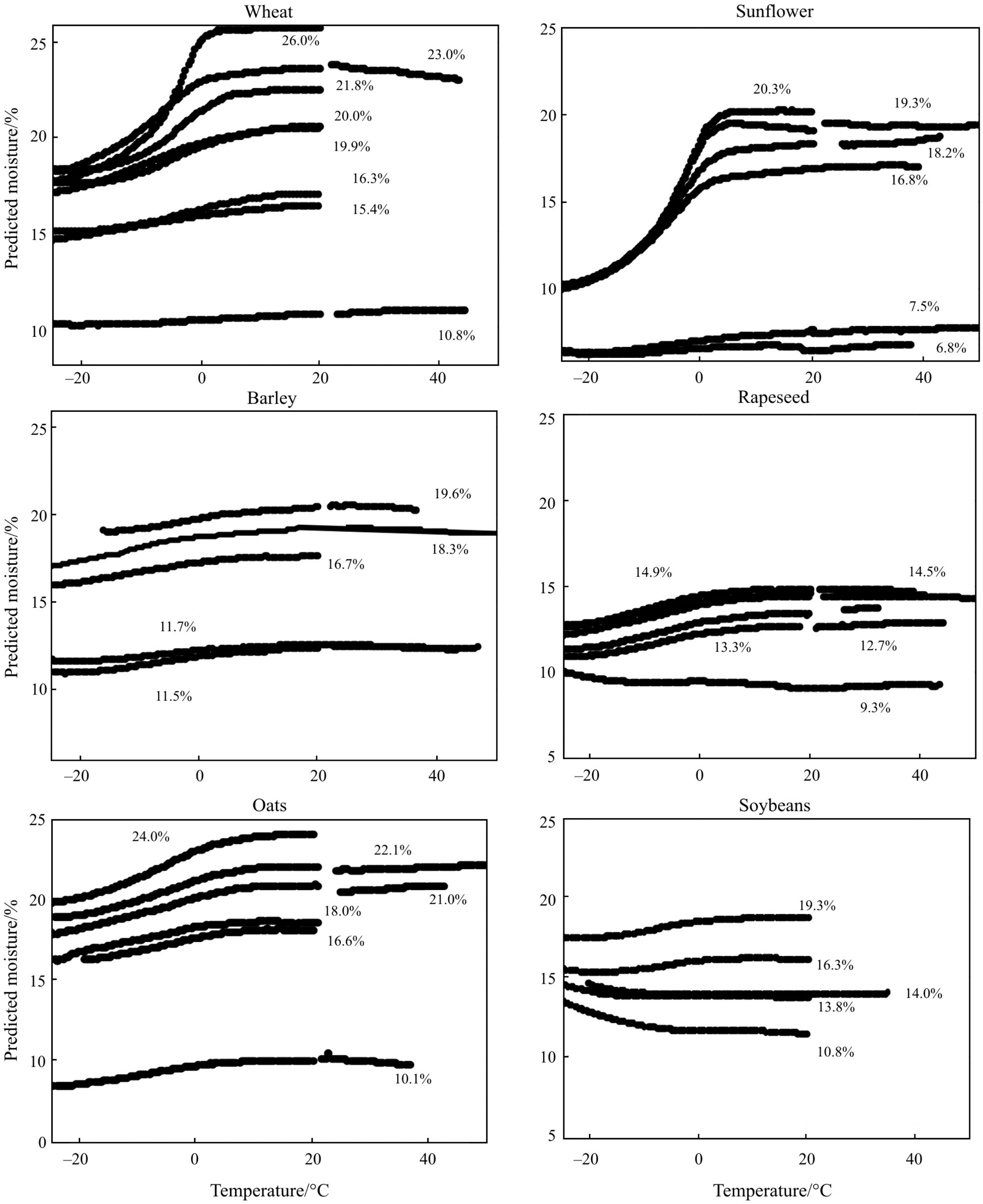

Fig. 6 Predicted moisture content *vs*. temperature for six grain types using the moisture-dependent temperature correction function (Eq. (4))

intercept and slope values for each grain type as shown in Table 2.

$$\%M_{\mathrm{TC}}=\%M-(KTC0+KTCS\cdot\%M)(T-25) \qquad (2)$$

where $\%M_{\mathrm{TC}}$ is the temperature-corrected moisture value, $\%M$ is the calculated moisture value without temperature correction, T is the sample temperature, $KTC0$ is the intercept value, $KTCS$ is the slope value.

The effect of using a moisture-dependent linear function (Eq. (2)) instead of Eq. (1) was tested. The success of the moisture-dependent temperature correction function (Fig. 6) can be appeared to be very nearly flat for all samples (above 0°C). With the moisture-dependent correction function, the predicted moisture errors (relative to moisture content at 25°C) were less than 0.5% moisture for all grain samples at temperatures above zero °C. This should be satisfactory for commercial grain moisture measurement applications.

The upper moisture limit (below which moisture can be determined below 0°C) for each grain type was estimated from Fig. 6. For some grain types, more data are needed to further refine the estimate. Table 3 gives the approximate moisture limit for each grain type.

An experiment was performed to investigate the source of the increase in the needed temperature correction with moisture content for lower frequency grain moisture sample for four different temperatures (Fig. 7). At –30°C, the loss factor is low and shows a maximum near 1 kHz. This is the expected frequency range for dipolar relaxation of ice [5]. The dielectric constant decreases slowly and gradually from 100 Hz to 100 kHz and is nearly constant from 100 kHz to 1 MHz. Note that the loss factor is very low at frequencies above 100 kHz.

At higher temperatures, there is a dramatic increase in the loss factor that is typical of dc conductivity through the sample. Due to electrode polarization, a portion of the dc conductivity is sensed as out-of-phase current and, therefore, contributes to the dielectric constant value. Electrode polarization causes a huge (50 times) increase in dielectric constant at 100 Hz for the sample shown. Note that the sloping dielectric constant and loss factor curves, which are characteristic of electrode polarization, ex-

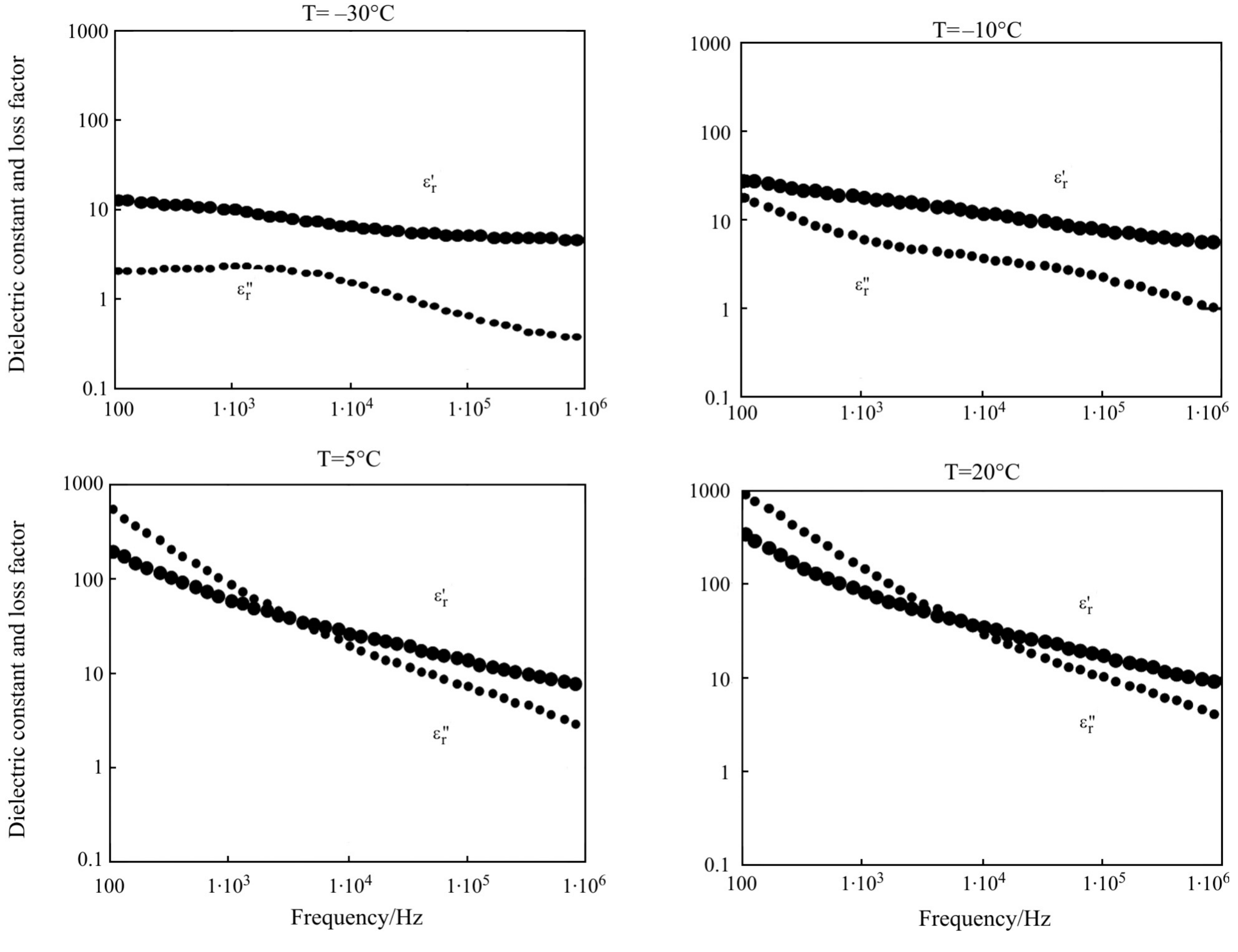

Fig. 7 Dielectric spectra of wheat 25% moisture wheat sample at four temperatures

Table 2 Slope and intercept values for the moisture-dependent temperature correction function

Grain type	Slope(10^{-3}) (%moist./°C%moist.)	Intercept (%moist./°C)
Wheat	–5.10	0.159
Barley	–3.47	0.159
Soybean	–9.91	0.226
Sunflower	–2.80	0.092
Oats	–1.47	0.155
Rapeseed	–5.83	0.134

Table 3 Estimated moisture limits for moisture measurements below 0°C for six grain types

Grain type	High moisture limit/%
Rapeseed	10
Barley	>19
Sunflower	10
Wheat	17
Oats	17
Soybean	>19

tend throughout the entire frequency range for the higher temperature measurements. Although the change at 1 MHz (about 2 times) is much smaller than at lower frequencies, the change is very large in terms of its effect on moisture predictions.

This test indicates that electrode polarization effects (though usually considered insignificant in the 2 to 20 MHz region) are actually large enough to contribute to the change in dielectric characteristics. The decrease in temperature coefficient with moisture at VHF frequencies is probably because electrode polarization is dramatically reduced in the VHF region and the negative coefficient of free water is more significant at higher moisture levels. ‘Bound water’ relaxation contributes to the observed temperature effects across the entire frequency range.

Conclusions

This research has demonstrated that the very high frequency dielectric method can be used successfully for grain moisture measurements for diverse grain types at temperatures well below 0°C if the grain moisture content is below certain threshold values. An improved temperature correction function was developed for moisture measurements at 149 MHz and appropriate temperature correction coefficients were estimated. Electrode polarization was found to contribute significantly to the temperature dependence of grain dielectric characteristics in the kilohertz and low MHz frequency range. This information should be very helpful for grain moisture meter manufacturers and for moisture meter users who need to determine grain moisture contents at very cold temperatures to be able to market grain more efficiently.

Acknowledgements

The research at Corvinus University of Budapest was supported in part by a grant and equipment loans and donations by the United States Department of Agriculture – Grain Inspection, Packers and Stockyards Administration. Also, the Agency provided the data that was used to create the calibrations for predicting moisture values at 149 MHz.

Viktor Jagasits wrote the QBasic program that was used to control the VHF prototype system to collect the temperature test data.

DICKEY-john Corporation donated the parallel-plate test cell that was used for some of the experiments.

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DOI: 10.1007/s10973-005-6896-0