

Dielectric Properties of Bulk Materials and Restrictions to the Application of Two-Parameter Microwave Aquametry

Ihar Renhart¹, Boris Tsentsiper²

¹ The Byelorussian polytechnical academy, Minsk, 220119, Belarus, ren@microradar.com

² Agar Corporation, Houston, TX, USA

ABSTRACT. The application of two-parametrical methods for density - independent measurements of moisture content has been considered. Theoretical estimation of the accuracy of determination of moisture in the scope of linear model of mixture was discussed. The changes of dielectric properties with the increase of moisture content were studied for the substances containing free or bound water using resonator and waveguide methods. The theoretical and experimental investigation shows that the problems of moisture determination cannot be solved by the increase of the accuracy of measurements for some substances. The restrictions of the application of two-parametrical methods are discussed for different materials and sensors.

Keywords: moisture measurement, complex permittivity, two-parametrical method.

1 Introduction

Microwave moisture meters are an essential part of many industrial processes at present. In a number of these processes material moisture contents W are measured under the conditions of variable density ρ . In this case for W determination the measurements of density are necessary. The systems containing microwave moisture meters and isotopic densimeters are used for this purpose. However the problem of radioactive safety arises on their application. The methods invariant to density fluctuations are developed for moisture measurements under such conditions [1].

Two-parametrical methods of moisture determination based on measurements of complex permittivity $\varepsilon = \varepsilon' - i\varepsilon''$ or other two values connected with ε' and ε'' using only microwave technique are widely discussed [2-4]. Unfortunately, measured parameters are not always independent in experimental conditions. The relative contribution of permittivity of dry materials to one of “material – water” mixture diminishes with the increase of moisture content and can reach the limit at which this contribution can be negligible in the frame of experimental error. In this case the mixture formulae defining ε' and ε'' as functions of mixture component properties transform to the functions of $W\rho$ product and the determination of W and ρ from measured dielectric properties is impossible. This situation restricts the application of two-parametrical microwave methods. The aim of our study is the determination of boundaries of sensitivity of microwave two-parametrical methods for resonator, transmission and combined devices under moisture measurements for materials containing free and bound waters.

2 Theoretical

Within the scope of linear model [5] the complex permittivity for a 3-component mixture (material, water, air) is

$$\varepsilon'' = \frac{\varepsilon''_w}{\sqrt{\varepsilon'_w}} W \frac{\rho}{\rho_w} \sqrt{\varepsilon'}, \quad (1)$$

$$\sqrt{\varepsilon'} = W \frac{\rho}{\rho_w} (\sqrt{\varepsilon'_w} - 1) + (\sqrt{\varepsilon'_d} - 1) \frac{\rho}{\rho_d} (1 - W) + 1, \quad (2)$$

where indices d and w correspond to dry material and water parameters. With the moisture increase, $\sqrt{\varepsilon'} = f(W\rho)$. This situation is realized when

$$W \gg \left(\frac{\sqrt{\varepsilon'_d} - 1}{\rho_d} \right) \left/ \left(\frac{\sqrt{\varepsilon'_w} - 1}{\rho_w} + \frac{\sqrt{\varepsilon'_d} - 1}{\rho_d} \right) \right. . \quad (3)$$

In the typical conditions $\varepsilon'_d \cong 3$, $\rho_d = 1 \div 2 \text{ g/cm}^3$ and $\varepsilon'_w \cong 80$ we obtained $W \gg 4\%$. At these values of moisture within the scope of linear model the simultaneous determination of moisture and density using complex permittivity data is impossible.

We propose to carry out the hypothetical experiment. Assume that we conducted i measurements at different values of W_i . Complex permittivity ε_{ij} was defined at different values of density ρ_{ij} for each moisture. So we have a set of data (Tab.1). We can obtaine $\varepsilon'_{kl} = \varepsilon'_{mn}$ in k and m measurements for diverse values of moisture and density. In this case according to (1) variation of imaginary part of dielectric functions $\Delta\varepsilon''$ can be determined

Table 1 Data obtained as the result of i measurements in hypothetical experiment

1-th measurement	W_1	ρ_{11}	ε'_{11}	ε''_{11}
		ρ_{12}	ε'_{12}	ε''_{12}
		$\vdots$	$\vdots$	$\vdots$
		ρ_{1j}	ε'_{1j}	ε''_{1j}
2-th measurement	W_2	ρ_{21}	ε'_{21}	ε''_{21}
		$\vdots$	$\vdots$	$\vdots$
		ρ_{2j}	ε'_{2j}	ε''_{2j}
		$\vdots$	$\vdots$	$\vdots$
i-th measurement	W_i	ρ_{i1}	ε'_{i1}	ε''_{i1}
		$\vdots$	$\vdots$	$\vdots$
		ρ_{ij}	ε'_{ij}	ε''_{ij}

$$\Delta\varepsilon'' = \frac{\varepsilon''_w}{\sqrt{\varepsilon'_w}} \Delta W \frac{\rho}{\rho_w} \sqrt{\varepsilon'} + \frac{\varepsilon''_w}{\sqrt{\varepsilon'_w}} W \frac{\Delta\rho}{\rho_w} \sqrt{\varepsilon'}$$

or

$$\frac{\Delta \varepsilon''}{\varepsilon''} = \frac{\Delta W}{W} + \frac{\Delta \rho}{\rho} . \quad (4)$$

The variation of real part of dielectric function is

$$\begin{aligned} \Delta \varepsilon' = & 2\sqrt{\varepsilon'} \left(\frac{\rho}{\rho_w} (\sqrt{\varepsilon'_w} - 1) - (\sqrt{\varepsilon'_d} - 1) \frac{\rho}{\rho_d} \right) \Delta W + \\ & 2\sqrt{\varepsilon'} \left(W \frac{(\sqrt{\varepsilon'_w} - 1)}{\rho_w} + \frac{(\sqrt{\varepsilon'_d} - 1)(1 - W)}{\rho_d} \right) \Delta \rho \end{aligned} \quad (5)$$

Since ε' does not change under the conditions of our experiment, variation of density can be written in next kind

$$\Delta \rho = -\Delta W \frac{(\sqrt{\varepsilon'_w} - 1) \frac{\rho}{\rho_w} - (\sqrt{\varepsilon'_d} - 1) \frac{\rho}{\rho_d}}{W \frac{(\sqrt{\varepsilon'_w} - 1)}{\rho_w} + \frac{(\sqrt{\varepsilon'_d} - 1)(1 - W)}{\rho_d}} . \quad (6)$$

In this case the variations of ε'' depend on moisture changes in the following way:

$$\frac{\Delta \varepsilon''}{\varepsilon''} = \frac{\Delta W}{W} K(W) , \quad (7)$$

where

$$K(W) = \frac{\frac{\sqrt{\varepsilon'_d} - 1}{\rho_d}}{W \frac{(\sqrt{\varepsilon'_w} - 1)}{\rho_w} + \frac{(\sqrt{\varepsilon'_d} - 1)(1 - W)}{\rho_d}} .$$

The coefficient $K(W)$ shows the extent of influence of moisture variations on the changes of imaginary part of mixture permittivity on the assumption that $\varepsilon' = \text{const}$. The coefficient K depending on moisture was calculated for typical parameters of moist materials (Fig.1). K sharply diminishes with moisture increase for all considered parameters.

This result points out that the changes in ε'' diminish with W growth but the problem of W measurements can be solved by the increase of the accuracy of measuring system. We obtained the theoretical estimation (within the scope of linear theory for mixtures) of the accuracy with which we should carry out measurements for determination of moisture. To confirm this outcome we carried out experimental investigation for materials containing free or bound water.

3 Experimental

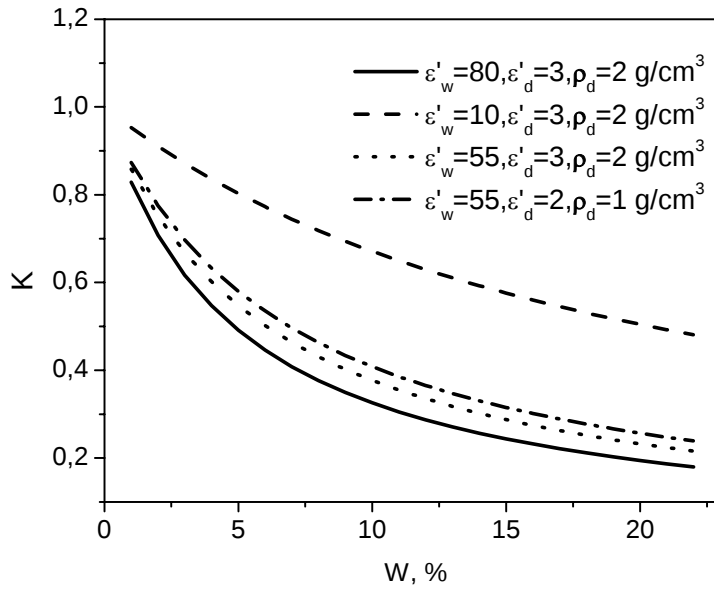


Fig.1. Coefficient K in dependence on moisture for different material parameters.

We studied the possibilities of the measurements of moisture by two-parametrical method for different materials and for different measuring systems. We used quartz sand as material containing practically free water in moist state and wheat flour containing up to $W=17\%$ exceptionally bound water. Samples of moist sand were prepared by mixing of water and dry sand, the samples of flour were obtained by drying flour with $W \approx 17\%$ up to required moisture. The moisture content of the prepared samples of sand and flour are determined by drying to

constant weight at temperature 103°C in a drying oven. To decrease the moisture fluctuations the samples were kept in sealed jars for 72 hours at room temperature. We also carried out measurements with potato starch and kaolin powder. The samples were prepared similarly to the experiment with flour.

The measurements were carried out by resonator and waveguide methods. In our experiments we defined the following couples of parameters: 1) shift of resonance frequency $\Delta f = f - F$ (f and F is a resonance frequency of filled with matter or empty resonator) and Q-factor Q ; 2) Δf and transient attenuation A for resonator methods; or 3) A and phase incursion β for waveguide methods. All these parameters are the functions of dielectric properties of materials studied.

The samples of moist sands are located in a bulk cylindrical resonator. The measurements were carried out at the resonator frequencies of $F_1=1.5$ and $F_2=2.5$ GHz. In each measurements with fixed $W = W_n$ we determined the values of f_{nk} and Q_{nk} for different densities ρ_{nk} (Tab.2). Using our experimental data and regression analysis we obtained functions $Q_n = Z_n(f_n)$. Then we calculated the values of Q for different moisture contents at the fixed value $f_c = 1595 \text{ MHz}$ for F_1 and at $f_c = 2595 \text{ MHz}$ for F_2 . Note that resonance frequency shift and Q –factor is the functions of complex permittivity and to some extend the situation with fixed frequency in real experiment is the analogue of our hypothetical experiment, where the fixed value was ε' . We also carried out the measurements of these parameters for different moisture at fixed densities $\rho_c = 1.45 \text{ g/cm}^3$. Dependence of Q-factor on moisture in cases of fixed density or fixed shift of resonance frequency are shown in Fig.2.

Table 2 Experimental data and data analysis

1-th measurement	W_1	ρ_{11}	Δf_{11}	Q_{11}	$Q_1 = Z_1(f_1)$	$Q_1 = Z_1(f_c)$
		ρ_{12}	Δf_{12}	Q_{12}		
		$\vdots$	$\vdots$	$\vdots$		
		ρ_{1j}	Δf_{1j}	Q_{1j}		
2-th measurement	W_2	ρ_{21}	Δf_{21}	Q_{21}	$Q_2 = Z_2(f_2)$	$Q_2 = Z_2(f_c)$
		$\vdots$	$\vdots$	$\vdots$		
		ρ_{2j}	Δf_{2j}	Q_{2j}		
		$\vdots$	$\vdots$	$\vdots$		
i-th measurement	W_i	ρ_{i1}	Δf_{i1}	Q_{i1}	$Q_i = Z_i(f_i)$	$Q_i = Z_i(f_c)$
		$\vdots$	$\vdots$	$\vdots$		
		ρ_{ij}	Δf_{ij}	Q_{ij}		

The similar measurements were carried out for wheat flour by the use of a bulk cylindrical resonator. But in this case we determined Δf and transient attenuation A . Measurements were carried out at the resonator frequencies of $F_1=1.1$ and $F_2=2.5$ GHz. The procedure described above was used for determination of dependences of transient attenuation on moisture in the conditions of fixed shift of resonance frequency or fixed density (Fig.3). We also studied the dependence of A and f on moisture increase in the experiments at $F=2.5$ GHz with potato starch by the same way (Fig.4).

The waveguide method was used in experiments with moist kaolin powder. The measurements were carried out at $F=10$ GHz. Attenuation A and phase incursion β in the layer of moist matter was used as measured parameters. For each moisture function $A_i = \Psi_i(\beta_i)$ was determined using experimental data on A and β and regression analysis. Results of investigation are shown in Fig.5.

4 Results and Discussion

The determination of sensitivity of measurements to moisture and density variations is very important task in development of microwave moisture meters. As follows from our theoretical consideration in the approximation of linear model of mixtures that with W increase, moisture variations result in very small changes in ε'' at fixed ε' . This points out on very high requirements to accuracy of microwave moisture meters and maybe, on impossibility of distinctive determination of moisture and density by use of exclusively microwave measurements. Our experimental study confirms this outcome.

According to our measurements with quartz sand by resonator method, Q-factor monotonically decreases with increase of moisture at fixed density (Fig.2,a,b). This behavior is typical for the most of large disperse materials. But Q-factor diminishes with W increase when $f_c = \text{const}$ at first and then Q does not change. It takes place at $W>4\%$. We can conclude that this moisture magnitude is the boundary value of W up to which we can use microwave methods. The sensitivity of measurements at $W = 4\%$, which follows for quartz sand from

expression (7), gives the value of $\sim 0.3\%$. Thus our measurements for moist quartz sand confirm conclusion obtained within the scope of linear theory of mixture.

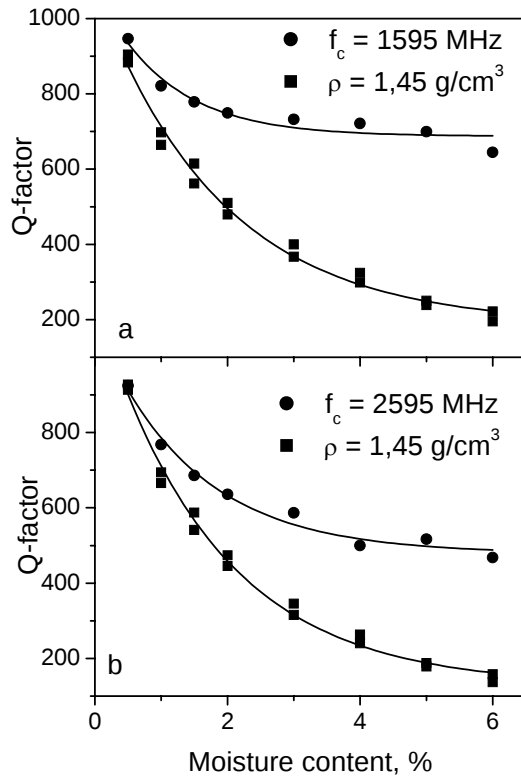


Fig.2. Dependences of resonator Q-factor on moisture content at resonator frequency (a) 1600 MHz and (b) 2600MHz for quartz sand

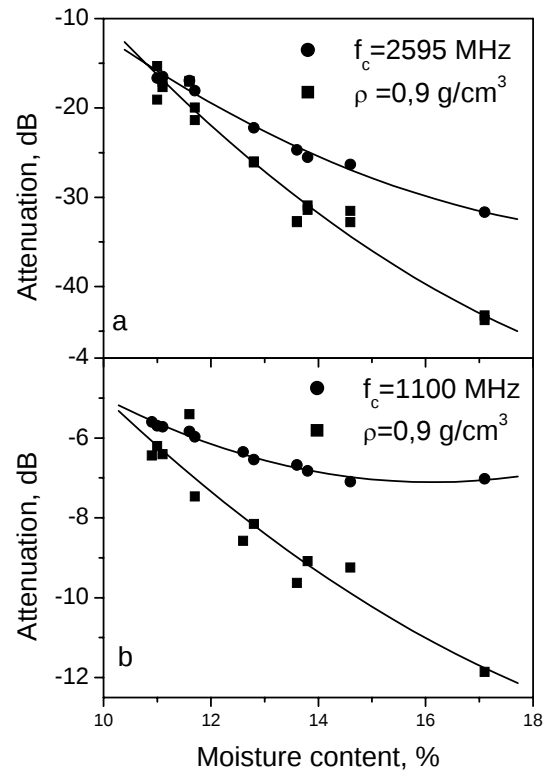


Fig.3. Dependences of transient attenuation on moisture content at resonator frequency (a) 2600 MHz and (b) 1100 MHz for wheat flour

We observed the same situation under measurements with wheat flour. We have typical dependence for $A(W)$ at fixed densities. The modulus of A grows with W increase at fixed ρ . When $f_c = \text{const}$, for both resonant frequencies $|A|$ increases with moisture growth up to $W \sim 15\div 20\%$ and with further W growth, attenuation does not change (Fig.3,a,b). But in contrast to large disperse matter (quartz sand) this is observed for moisture values greater than $15\div 20\%$. So we again obtained that there is the restriction of application of two-parametric method or microwave techniques but in this case this restriction is reached at the greater values of the moisture in comparison with quartz sand. It means also that for moisture measurements in wheat flour the sensitivity requirements for measuring devices are not so strong like in the case of large disperse materials. This advantage of small disperse matter in respect to large disperse matters can be explained by presence of bound water in small disperse matters.

It should be noted that we observe monotonically lessening Q and A dependence on moisture for both large disperse or small disperse materials. This behavior is typical for a number of materials.

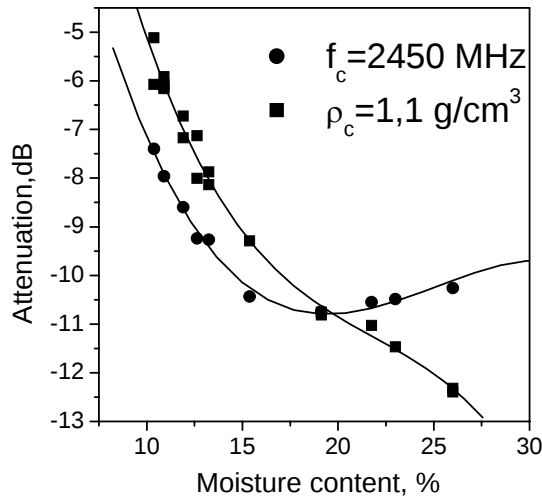


Fig.4. Dependences of transient attenuation A of a cylindrical resonator at $F=2500$ MHz for potato starch.

With moisture increase along with bound water free water appears. This results in faster growth of ε' in comparison with the imaginary part and leads to change in behavior of dependence of measured parameters on moisture.

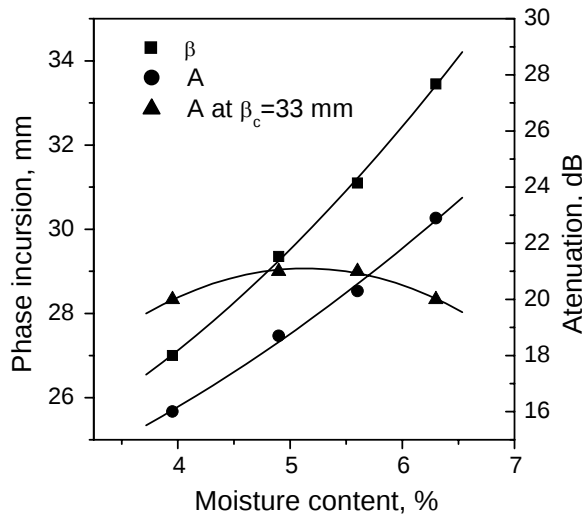


Fig.5. Phase incursion β and attenuation A and A when $\beta=\text{const}$ at $F=10$ GHz for kaolin powder.

However, our study shows that in the experiments with potato starch or kaoline powder the dependences $A(W)$ have an extremum or the dependence curves have inflexion (Fig.4 and Fig.5). In this situation the equal values of attenuation correspond to different values of moisture. It is evident that the measurements of moisture are impossible in this case. For potato starch we can carry out these measurements up to values of $W \sim 15\%$, where $A(W)$ monotonically lessens at fixed frequency shift. As for kaolin powder, the measurements are not already possible at $W \geq 4\%$. This change in behavior of $A(W)$ (we mean the appearance of an extremum) can be explained by formation of free water. As is known both these materials (flour and starch) contain bound water.

5 Conclusion

Our theoretical study within the scope of linear theory of mixture shows that the application of two-parametrical microwave methods for moisture determination has essential restrictions. The experiments carried out with materials containing free and bound water confirm this conclusion. We obtained that the application of this method for materials containing free water (quartz sand) is restricted by the values of moisture less than 4%. In the case of materials containing bound water (wheat flour and potato starch) the measurements of W by two-parametrical method are possible up to $W \sim 15 \div 20\%$. Our theoretical and experimental results show that the simple increase of accuracy of measuring system can solve the

problem of moisture measurements not always. The dependences of measured parameters on moisture have inflexion for some materials (potato starch and kaoline powder). In this case different values of moisture correspond to equal measured parameters and the use of two-parametrical methods for moisture measurements are impossible.

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Contact point: Dr. Ihar Renhart, MICRORADAR LTD; Novatorskaya 2A; 220104 Minsk; Belarus. Phone: + 375-172-532183; Fax: + 375-172-343776; E-mail: ren@microradar.com