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DETERMINATION OF FLUOR MOISTURE BY MEASURING THE DIELECTRIC LOSS COEFFICIENT tgδ

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Key words: dielectric loss coefficient, flour moisture determination.

The suspension method of measuring the dielectric loss coefficient tg δ in flour was used. Formerly the method was applied to the measuring of inorganic dielectrics. The results were used to determine the correlation between flour moisture measured with the dryer method and log tg δ values.

The dryer method is presently the only one approved by the Polish Standards for measuring levels of moisture in fluor [2]. The method provides satisfactory degrees of accuracy but its shortcoming is the length of time required for making the determinations [2, 3].

In recent years there has been a growing interest among researchers and practitioners in measuring dielectric permeability ε and the dielectric loss angle tgδ in order to find any relationship between the two and moisture content in a tested product. A test sample is poured freely into a measuring condenser, or depending on the test apparatus construction and design, it is pressed into a tablet form. Condenser sensors adapted to measure tablet samples provide better repetitiveness of results [4, 5].

The powder structure of flour is the reason why the obtained results show a high level of error. In the course of measuring flour particles are separated by air of a given humidity which differs from sample to sample as to quantity. When powdered materials are measured intensive exchange of moisture with environment takes place. This means that results of measurements depend on conditions in which they are performed [6]. The problem of measuring the dielectric loss angle tgδ in powdered materials is handled effectively with the suspension method that was worked out for metal oxides [7, 8].

The aim of the present study was to adapt the suspension method of measuring the dielectric loss coefficient tgδ for determination of water content in flour.

MATERIALS AND METHODS

The test flour was from two sources: our own laboratory mill and a foodshop in Warsaw. The tgδ coefficient was measured with a conductivity gauge Type VLU Rhode Schwarz within a frequency range between 0.1 and 10 MHz. The tested sample was placed between electrodes of a flat 15 mm condenser (capacitor). The distance between electrodes — 0.5 mm. The following parameters were measured:

C_0 — capacity of condenser/capacitor with air,
C — capacity of condenser with a dielectric,
R — resistance.

The tgδ coefficient was then calculated with the following formula:

$$\mathrm{tg}\delta = \frac{X}{R} \tag{1}$$

where

$$X = \frac{1}{\omega_c} \tag{2}$$

where $\omega = 2\pi f$

R — resistance,
C — capacity of condenser with a dielectric,
f — frequency.

PROCEDURES

Flour samples were conditioned over water solutions of salt in exicators. The measurements were performed at the following frequencies: 10; 5; 2; 1; 0.5; 0.2; 0.1 MHz. The tested samples of flour were put into a suspension liquid: silicone oil Silon 350, an anhydrous oil characterized by its dielectric loss coefficient tgδ of the order of 10^{-5} the value exceeded the separation capability of the instrument).

During the first phase of the analysis the tgδ coefficient values were determined for flour concentration in oil at 50%, 40%, 30%, 20% and 10% flour weight. Samples of the flour in silicone oil were mechanically put on the upper and lower electrodes of the measuring capacitor, separately for each measurement (a brush was used). The measurement was carried out immediately after a sample was placed between electrodes of the measuring capacitor. The manner of doing it prevented formation of air bubbles between electrodes, thus securing correct repetitiveness of measurements.

The obtained results on tgδ made it possible to select for further analytical work the optimum concentration of flour in the silicone oil. Results on tgδ for the above mentioned range of frequencies and on different levels of water content in a given flour concentration in the silicone oil were used to determine such a measuring frequency at which there was a directly proportional relationship between flour moisture and values of the dielectric loss coefficient tgδ.

The selected frequency was used for measuring tgδ of different flours and different levels of water content in them. The moisture in the tested samples was also measured with the dryer method.

RESULTS

In the first stage of analysis the coefficient tgδ was measured in a flour with fixed mosture content of 12.28%. For that purpose suspensions of the flour in Silon 350 at the following concentrations had been prepared: 50%, 40%, 30%, 20%, 10% of flour weight. The measurement frequency range: 0.1; 0.2; 0.5; 1.0; 2.0; 5.0; 10 MHz. In order to give a better presentation of the results the log scale was used. The obtained results are in Table 1 and in Fig. 1.

Table 1. Logarithmic values of the dielectric loss coefficient tg δ for flour with constant moisture content of 12.28% as dependent on frequency and concentration of flour in silicone oil

Frequency log Hz	Percentage of flour concentration in silicone oil				
	50%	40%	30%	20%	10%
	log tg 2	log tg 2	log tg 2	log tg 2	log tg 2
7.00	8.3	7.6	6.15	4.3	1.74
6.90	7.8	6.9	5.8	3.8	0.38
6.30	7.1	6.2	4.9	2.8	0.099
6.00	6.6	5.5	4.3	2.8	0.098
5.69	6.4	5.7	4.8	2.9	0.099
5.30	7.1	6.2	4.9	3.6	1.13
5.00	9.1	8.4	6.8	4.9	3.7

The figures in Table 1 and in Fig. 1 indicate that the value log tgδ is directly proportional to per cent of flour concentration in silicone oil. The log tgδ value reaches maximum in the samples with 50% flour concentration in silicone oil. This was also the upper limit of concentration providing a suspension with consistence permitting correct spreading of a thin layer of it on electrodes of the measuring capacitor.

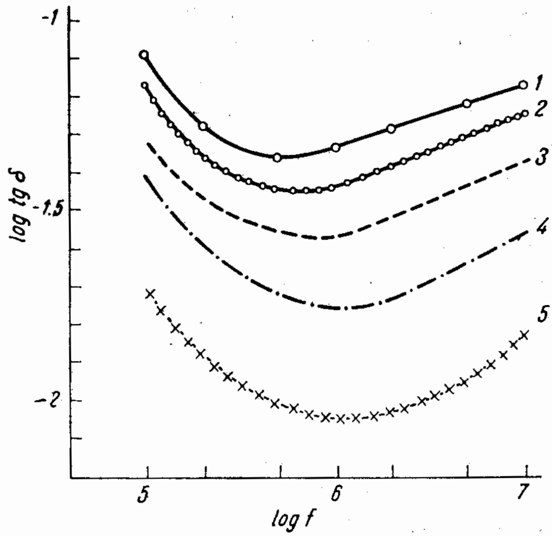

Fig. 1. Relations between log tg δ, log Hz and concentration percentage of constant moisture flour in silicone oil "Silon 350"; percentage of flour concentration in silicone oil; 1 — 50%, 2 — 40%, 3 — 30%, 4 — 20%, 5 — 10%

On the basis of the obtained results selection was made as to the 50% flour in oil samples for further analytical procedures. The samples were subsequently conditioned to obtain differentiated levels of moisture in

Table 2. Logarithmic values of the dielectric loss coefficient tg δ as dependent on frequency and flour moisture at constant concentration of 50% flour weight in silicone oil „Silon 350"

Frequency log Hz	Moisture				
	log tg $\bar{2}$	log tg $\bar{2}$	log tg $\bar{2}$	log tg $\bar{2}$	log tg $\bar{2}$
	16.3%	14.85%	12.9%	12.73%	9.35%
7.00	8.2	7.8	8.3	8.3	8.7
6.69	7.8	7.6	7.6	7.7	8.3
6.30	7.4	7.4	6.9	7.0	7.2
6.00	7.6	7.2	6.6	6.8	6.4
5.69	8.7	7.7	7.0	6.5	5.8
5.30	9.8	9.1	8.0	7.4	5.3
5.00	23.0	16.5	9.7	9.0	6.3

them. The moisture of the samples was determined with the dryer method, and parallelly, the value of the tgδ coefficient for the 50% flour suspension in 'Silon 350' was measured.

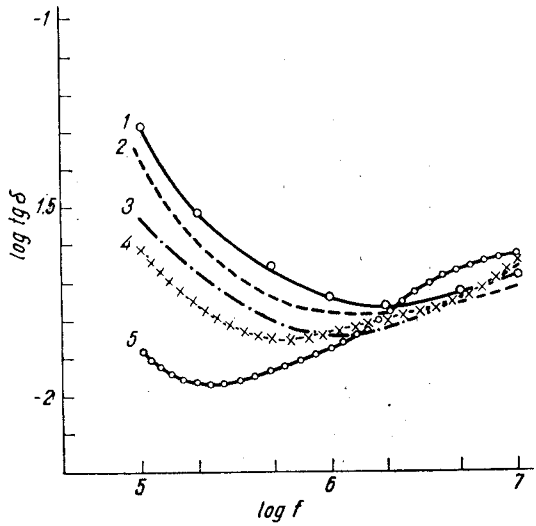

Fig. 2. Relations between log tg δ and percentage of moisture in flour within 100 Hz to 10 000 000 Hz for 50% flour concentration in silicone oil "Silon 350"; 1 — 16.30%, 2 — 14.85%, 3 — 12.90%, 4 — 17.73%, 5 — 9.35%.

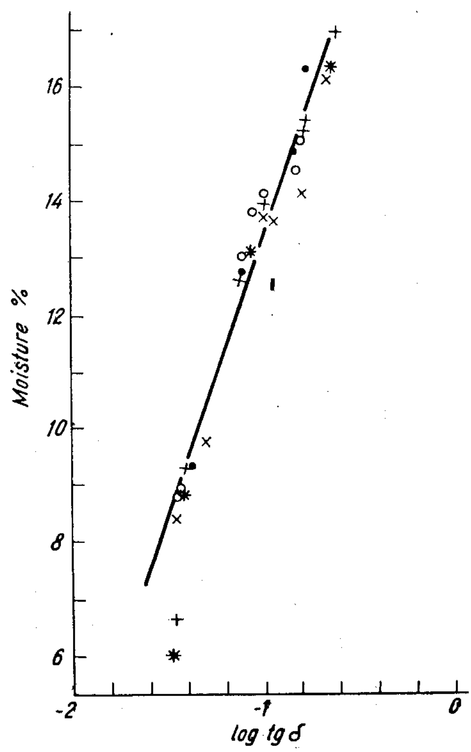

Fig. 3. Relations between log tg δ and moisture levels in the flours at 0.1 MHz frequency; +++ — wheat flour type "Wrocławska" ●●● — wheat flour type "Tortowa I" ××× — wheat flour type "Krupczatka", ooo — wheat flour type "Tortowa II", *** — wheat flour from laboratory mill.

Table 3. Relations of log tg δ to moisture content of selected wheat flours at 0.1 MHz

Wheat flour type „Tortowa I"		Wheat flour type „Krupczatka"		Wheat flour type „Tortowa II"		Wheat flour from laboratory mill		Flour type „Wrocławska"	
moisture %	log tg δ	moisture %	log tg δ	moisture %	log tg δ	moisture %	log tg δ	moisture %	log tg δ
9.35	$\bar{2}.65$	8.40	$\bar{2}.556$	8.83	$\bar{2}.566$	6.02	$\bar{2}.530$	6.77	$\bar{2}.540$
12.73	$\bar{2}.90$	9.70	$\bar{2}.710$	8.95	$\bar{2}.574$	8.82	$\bar{2}.596$	9.26	$\bar{2}.600$
12.90	$\bar{2}.97$	13.71	$\bar{1}.061$	13.00	$\bar{2}.905$	13.08	$\bar{2}.942$	12.64	$\bar{2}.883$
14.85	$\bar{1}.165$	13.83	$\bar{1}.045$	14.17	$\bar{1}.042$	13.80	$\bar{1}.017$	13.85	$\bar{1}.033$
16.30	$\bar{1}.230$	14.07	$\bar{1}.11$	14.54	$\bar{1}.184$	16.30	$\bar{1}.373$	15.30	$\bar{1}.212$
—	—	16.17	$\bar{1}.35$	15.17	$\bar{1}.210$	17.00	$\bar{1}.390$	15.35	$\bar{1}.218$

The results are presented in Table 2 and in Fig. 2. The obtained results show that for the 0.1-0.5 MHz frequency range there is a direct proportionality between log tgδ and flour moisture. Within the 1.0-10 MHz range this relationship does not occur. For further work the 0.1 MHz frequency was chosen. At this frequency small variations in flour moisture cause considerable alterations in the tgδ coefficient values. This 0.1 MHz was used to measure the dielectric loss coefficient values for different flours: Tortowa I, Krupczatka I, Tortowa II, Wrocławska as well as our own laboratory milled flour. They were all wheat flours with different levels of water content. Results of the measurements are given in Table 3 and in Fig. 3. Fig. 3 shows the points at which the relationship between log tgδ and mosture levels in particular flours is expressed. The distribution of the points indicates that there is a correlation between log tgδ and wheat flour moisture content regardless of the type of flour.

The relationship between flour moisture and log tgδ is correct with the 8 to 17% moisture content. Below the level of 8% big changes in flour moisture are accompanied by limited changes in log tgδ. The analyzed range between 8 to 17% finds practical application on account of the real water content in flour. The empirically obtained data in Table 3 were used to compute the function:

$$y_i = a' x_i + b' \qquad (4)$$

$i = 1,2,3, ...k$ — number of observations,
x_i — independent variable — log tgδ,
y_i — dependent variable — moisture in flour,
a' and b' were calculated with appropriate formulas:

$a' = 9.859$ $b' = 3.389$

The obtained a' and b' values were used in Formula (4) to compute this function:

$$y_i = 9.859x_i + 3.389 \qquad (5)$$

In order to establish the level of confidence for the obtained results from function (5) mean average deviation of regression S_{xy} was calculated after the formula:

$$Sxy = \sqrt{\frac{\sum_{i=1}^{i=k} (y_i - y_i)^2}{n-2}} \qquad (6)$$

y_i — the observed value of variable y
y_i — value of the variable determined with equation
$y_i = 9.859x_i + 3.389$ for a given value of x_i
n — number of observations of the variables x and y.
Introducing appropriate values into the formula the result was obtained:

$S_{xy} = 0.4215$

The significance of regression R was also calculated:

$$R = \frac{SS}{MR_s} \tag{7}$$

where
R — regression significance
SS — sum of the squares of deviations anticipated by regression
MR_s — sum total of deviation squares
Following the computation and introduction of the results for SS and MR_s to Formula (7) the following was arrived at:

$$R = 97.63\%$$

Regression significance equals 97.63%.

CONCLUSIONS

The results of investigations concerning determination of flour water content by means of the dielectric loss coefficient tgδ make it possible to conclude as follows:

1. The applied suspension method for measuring the dielectric loss coefficient tgδ during flour testing was useful. The method provides for elimination of measurement errors resulting from the powder form of flour.

2. The analysis of relationship between log tgδ and water content in wheat flour, regardless of type, proved that the presented method finds practical application.

3. Statistical analysis of the results proved that determination the water content in flour by means of measuring the tgδ coefficient guarantess high accuracy. Regression significance is 97.63%.

4. In effect of the analytical procedures it was demonstrated that the proposed method makes it possible to determine the level of moisture in flour within several minutes with high precision, which is a good evidence that the method is more convenient in practice than the dryer method.

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WYZNACZANIE WILGOTNOŚCI MĄKI METODĄ MIERZENIA WSPÓŁCZYNNIKA STRATNOŚCI DIELEKTRYCZNEJ tg δ

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Streszczenie

Stosowana dotychczas metoda pomiaru wilgotności mąki jest pracochłonna i wymaga dużo czasu. Jednocześnie przemysły: piekarniczy, zbożowo-młynarski itp. wymagają szybkiej i dokładnej metody pomiaru wilgotności mąki. W związu z tym postanowiono opracować warunki adaptacji metody suspensyjnej pomiaru współczynnika stratności dielektrycznej tg δ stosowanej do pomiaru dielektryków nieorganicznych w formie sproszkowanej, w celu wyznaczenia wilgotności mąki. Opracowana metoda suspensyjna polega na zawieszeniu badanych próbek mąki w oleju silikonowym silon 350 F bezwodnym, o małym współczynniku stratności dielektrycznej, o wartości niższej niż czułość pomiarowa przyrządu stosowanego do badań. Stężenie mąki w oleju silikonowym wynosiło 50%. Pomiar przeprowadzono umieszczając próbkę pomiędzy okładkami kondensatora pomiarowego miernika przewodności typu VLU firmy Rhode Schwarz.

Wskutek przeprowadzonych badań ustalono, że optymalne wyniki otrzymuje się w zakresie częstotliwości pomiarowej 0,1 MHz. Równocześnie przeprowadzono oznaczenia wilgotności badanych próbek metodą suszarkową. Uzyskane wyniki pomiaru dla różnych mąk pszennych (niezależnie od ich jakości) przyjęto jako podstawę do wyznaczenia równania regresji:

$$y_i = a'x_i + b'$$
$$a' = 9.859 \qquad b' = 3.389$$

gdzie: x_i — zmienna niezależna — log tg δ
y_i — zmienna zależna — wilgotność badanej mąki.
Następnie wyliczono istotność regresji.

Uzyskane wyniki badań wykazały, że proponowana metoda pozwala na skrócenie czasu pomiaru wilgotności mąki do kilku minut i zapewnia stosunkowo dużą dokładność wyników (istotność regresji = 97,63%).

W konsekwencji przeprowadzonych badań ustalono, że metoda dielektryczna pomiaru wilgotności mąki nadaje się do stosowania w praktyce zamiast stosowanej dotychczas metody suszarkowej.