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A low-frequency sensor for determination of honey electrical properties in varying temperature conditions

Bartosz Paszkowski [a,*], Andrzej Wilczek [a], Agnieszka Szypłowska [a], Anna Nakonieczna [a,b], Wojciech Skierucha [a]

[a] Polish Academy of Sciences, Institute of Agrophysics, Department of Metrology and Modelling of Agrophysical Processes, Doświadczalna 4, 20-290 Lublin, Poland
[b] Institute of Physics, Maria Curie-Skłodowska University, Pl. M. Curie-Skłodowskiej 1, 20-031 Lublin, Poland

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ABSTRACT

The aim of the research was to test a prototype sensor used to determine the electrical properties of acacia honey. The sensor, placed inside a measurement cell, was connected to a precise LCR meter, which collected the impedance readings in the frequency range from 20 Hz to 2 MHz. The measurement results were analyzed using the electrical equivalent circuit (EEC) approach. The values of parameters describing impedances of four EEC elements, which were a capacitor, a constant phase element and two resistors, obtained at various temperatures from −10 °C to 35 °C during honey heating and cooling processes, were presented, as well as dielectric permittivity spectra of honey. The paper discusses the temperature dependence of the EEC electrical parameters and the functionality of the sensor.

1. Introduction

The complex impedance of a sample-sensor setup describes the behavior of the tested material in an alternating electric field. The impedance spectroscopy (IS) is an experimental technique relying on recording changes of the tested material impedance, which result from frequency variations of an applied voltage signal (Barsoukov and Ross Macdonald, 2005). It is successfully applied in many fields of science such as electrochemistry and materials science (Ahmed and Reifsnider, 2011), biophysics (Tura et al., 2007), pharmacology (Shervedani et al., 2006) and geophysics (Koelemeijer et al., 2012). In the case of sufficiently wide frequency range the impedance spectrum, which is a result of an IS measurement, incorporates the whole information about electric properties of an analyzed system (Harrington and van den Driessche, 2011).

The information contained in the frequency spectrum of impedance of the analyzed sample can be correlated with its physical and chemical parameters, which may serve as indicators of the material quality (Nelson, 2010). An interest in these aspects enhanced the development of rapid and non-destructive quality assessment dielectric measurement techniques, such as impedance spectroscopy, time-domain reflectometry (TDR) and frequency-domain reflectometry (FDR) (Jha et al., 2011; Skierucha et al., 2012). Nelson et al. investigated the correlations between a set of two quantities, i.e. the dielectric permittivity and the loss factor, and the pulp density along with the dissolved solids content of a honeydew melon (Nelson et al., 2006). These correlations formed a basis for the qualitative analysis of fruits for consumption. An attempt to differentiate honeys by measuring their conductivity, the dielectric permittivity and the loss factor was also made (Łuczycka, 2010). Moreover, the effort to distinguish fake and genuine honeys using electrical parameters measurements was presented (Łuczycka et al., 2012). It was also shown that impedance spectroscopy can be applied as an alternative method for the determination of floral origin of unifloral honey (Scandurra et al., 2013).

The physicochemical properties of honey are temperature sensitive. As temperature increases, the amount of 5-hydroxymethylfurfural raises and the diastase number decreases. The diastase number is connected with the decline in the honey enzymes activity. The enzymatic activity ceases when honey is overheated, what takes place at temperatures above 40 °C. The overheating changes electrical characteristics of honey, which is an important factor that may allow for distinguishing between overheated and not overheated honey. Additionally, the diastase inactivity in honey may indicate its adulteration with sugar syrup, which for the above reasons may be also detected with the use of electrical measurement techniques.

The purpose of this study was to test a prototype sensor for measuring the electrical parameters of honey and to determine the temperature effect on honey electrical parameters.

* Corresponding author. Tel.: +48 817445061x125.
E-mail address: b.paszkowski@ipan.lublin.pl (B. Paszkowski).

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2. Materials and methods

The acacia variety of honey was selected for the research because it sustains its liquid phase for a long period of time and crystallizes slowly due to high fructose content (Popek, 2002).

The examined honey was collected in the Lublin area in the year 2011. The results of analyses of physicochemical properties of the material under test, which were made by the Bee Products Quality Testing Laboratory in Puławy, are presented in Table 1.

A – the method accredited by the Polish Centre for Accreditation;
PN-88/A-77626, Miód pszczeli" ('Bee honey') – Polish Norm;
PB-02, PB-04, PB-06, PB-07 – test procedures in Bee Products Quality Testing Laboratory; E-03, E-05 – edition number;
HMF – 5-Hydroxymethylfurfural.
*One unit of diastase is equivalent to the activity of enzymes that are contained in 1 g of honey, capable of hydrolyzing 0.01 g of starch during 1 h at 40 °C (Waś et al., 2011).

According to the obtained assessment, in the honey sediment a high total number of pollen grains was found, which indicates the secondary enrichment of honey by pollen from the bee bread. The pollen analysis cannot determine the variety of honey in this case. The decisive factor for the classification is the glucose to fructose ratio, which in the studied case is equal to 1.39. This value confirms that the tested sample is the acacia honey. According to the International Commission for Honey, the norm which classifies the product to the acacia honey variety is the glucose to fructose ratio of about 1.4 (Council Directive, 2001). The parameters such as the 5-hydroxymethylfurfural content and the diastase number provide information that the honey was stored properly and it was not overheated during the packaging process (Council Directive, 2001). Moreover, the water content equal to 16.1% is in accordance with the standards, as it does not exceed 20% (Council Directive, 2001).

The schematic diagram of the sensor of custom design which was placed in the analyzed sample of honey is shown in Fig. 1. The sensor consisted of a 3.75 mm thick frame made of polycarbonate with two silver-plated wire closed loops of the diameter equal to 0.5 mm separated by a 6 mm distance and wound around the polycarbonate frame through the precisely carved notches.

The wire loops acted as electrodes since they were connected through coaxial cables to the BNC terminals of the LCR meter (Agilent E4980A). The scheme of the sensor-measuring device connection is depicted in Fig. 2.

The use of a silver-plated copper wire guarantees the reduction of measurement errors that could appear due to polarization effects on the surfaces of the electrodes (Barsoukov and Ross Macdonald, 2005). The frame with the electrodes was placed in a polycarbonate disc (Fig. 2), in order to maintain the steady position of the sensor during the measurements. The sensor was immersed in a sample of the tested material to the 36 mm depth. The investigated samples consisted of 100 ml of honey placed in a plastic container.

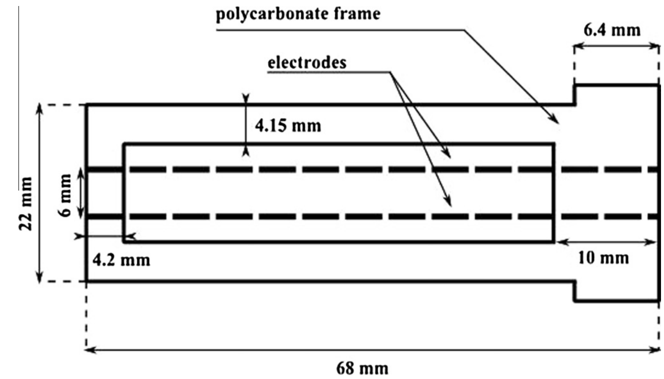

Fig. 1. The scheme of the sensor. The dashed black lines represent the electrodes, while the solid ones – the polycarbonate frame.

The complex impedance of the sensor-honey system is described by the following relation:

$$Z = R + iX_c \tag{1}$$

where Z denotes the impedance [Ω], R stands for resistance [Ω], X_c is reactance [Ω] and i is the imaginary unit.

The collected data were processed using the Frequency Response Analysis (FRA) method (Metrohm Autolab), i.e. an analysis of the frequency response in the frequency range 20 Hz–2 MHz (201 measurement points, logarithmic scale of frequency) was performed. The amplitude of the voltage signal generated by the LCR meter was equal to 100 mV. The collection of data from the LCR meter was controlled by the PC compatible computer with the use of a custom written application.

The research was conducted in two series of cooling and heating of the honey samples, which were performed in the climatic chamber (Weiss WKL100) with adjustable temperature stabilized with accuracy equal to ±0.1 °C. Stable thermal conditions were maintained to minimize the temperature change of honey electrical parameters during the readings.

The first measurement series included the temperature change from 21 °C to −10 °C, while the second one was performed from −10 °C to 35 °C. The temperature range was chosen to include storage temperatures of food in warehouses and households. The temperature was changed in steps by one degree every three hours during the first series and five degrees every five hours in the second series. The measurements were taken every ten minutes during the experiment. The honey heats up and cools down slowly, therefore the time interval appropriate for reaching the temperature equilibrium was carefully selected. After the sample temperature was stabilized, ten results were taken for elaboration and interpretation.

The results obtained during the experiment were analyzed using the computer software, specifically the 'ZSimDemo3.30 Demo' (EChem Software, 2001) and the 'EIS analyzer' (Bondarenko and Ragoisha, 2008). The analysis was based on

Table 1
Physicochemical properties of the acacia honey tested in the Bee Products Quality Testing Laboratory in Puławy.

Property	Norm/test procedure	Unit	Value
Water content	PN-88/A-77626 ptk. 5.3.3. refractometric method (A)	%	16.1
Fructose content	PB-02: E 05 from 17.02.2012 r. HPLC method	g/100 g	41.2
Glucose content	PB-02: E 05 from 17.02.2012 r. HPLC method	g/100 g	29.6
Sucrose content	PB-02: E 05 from 17.02.2012 r. HPLC method	g/100 g	<0.5
HMF content	PB-04: E 03 z 14.01.11 r. HPLC method (A)	mg/kg	10.9
Electrical conductivity	PN-88/A-77626 p. 5.3.10. conductivity method (A)	mS/cm	0.17
Diastase number	PB-06: E 03 from 14.01.11/Phadebas method (A)	Schade*	20.5
pH and free acids	PB-07: E 03 from 14.01.11/Potentiometric method (A)	meq/kg	pH 4.22 free acids 15.3