



Dielectric properties of honey: the potential usability for quality assessment

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Abstract

Honey, as a valuable product of high price, is subject to illegal adulteration by adding water or inexpensive sweeteners as well as overheating. Therefore, cheap, quick, and reliable methods of honey quality assessment are of interest to the food industry and consumers alike. In this study, two dielectric honey parameters: relative permittivity and dielectric loss coefficient measured at frequencies below 1 MHz and at temperatures from 20 to 40 °C were considered as potential indicators of honey quality. For the data analysis, chemometric methods (artificial neural networks and two methods of extracting the contribution of independent variables) were employed. No significant differences in results of the relative contribution of input variables were found depending on frequency. Results lead to the conclusion that relative permittivity, which is significantly affected by pH, can be potentially useful for honey microbial contamination detection and dielectric loss coefficient, influenced mostly by HMF content, can be considered as an indicator of honey freshness.

Keywords Honey · Relative permittivity · Dielectric loss coefficient · Neural Network · Sensitivity analysis

Introduction

Honey is a natural food produced by honey bees (*Apis mellifera*), mainly composed of sugars. Monosaccharides represent about 75% of the honey sugars, 10–15% are disaccharides and the rest are small amounts of other sugars [1]. The other honey components are enzymes (invertase, glucose oxidase, catalase, and phosphatases), amino acids, organic acids, lipids, vitamins, minerals, carotenoids, phenolic acids, aromatic substances, and solid particles derived from honey collection [2]. The strong biological effects of honey and its positive influence on human health caused by prebiotic, antioxidant, antibacterial, and/or antimutagenic functionalities of certain constituents are very well proven [3–5]. Many parameters such as pollen source, geographical origin, climate and weather conditions, honeybee species, and also processing conditions and storage time affect composition, color, aroma, and flavor of honey [6]. Honey is very often

adulterated or improperly treated causing quality decrease and reduction of therapeutic value. The high price of honey and its limited availability results in adulteration by adding water or inexpensive sweeteners such as corn syrups, invert syrups or high fructose inulin syrups or bee feeding with sugars and/or syrups or artificial honey. Since customers often prefer fresh liquid honey and a lot of honey types crystallize rapidly, many beekeepers and factories heat honey at high temperatures for melting. This may result in unfavorable chemical processes [reduction of α -amylase activity and increase of 5-hydroxymethyl furfural (HMF) content] [7]. Therefore, honey quality assessment is crucial at every stage of the production process. Codex Alimentarius Standard [8] presents several international quality indicators such as moisture content, mineral content, pH, invertase, HMF content, diastase activity, and specific conductivity. HMF content and diastase activity can be used as indicators for honey freshness and overheating [9, 10]. HMF is almost entirely absent in fresh honey but it is naturally generated during heat treatments or long storage. Additionally, HMF can be formed even at low temperatures in acidic conditions by dehydration of sugar [10]. Moisture content is a parameter crucial for honey shelf-life and resistance against yeast fermentation. High moisture content causes honey fermentation, spoiling, and loss of flavor and water activity is a

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major factor which governs microbial growth [6]. The pH is considered as an indicator of possible microbial contamination [11]. The use of chemical parameters for honey quality assessment is time-consuming and requires special apparatus and equipment. Therefore, other effective, fast, and non-destructive techniques are developed by researchers. In the case of food of low conductivity such as honey, methods based on impedance spectroscopy seem to be promising for quality determination [12]. The high impact of HMF content and pH on honey complex impedance was previously proven, leading to the conclusion that this parameter can be potentially useful for honey quality deterioration detection [13].

Chemometric tools such as artificial neural networks (ANNs) have been described as helping in processing complex or imprecise data [14]. ANNs were employed by some researchers for honey parameters analysis, in particular for prediction of the origin of honey samples or their classification based on certain chemical parameters. The multi-layer perceptron (MLP) is the most commonly used ANN for solving prediction tasks. MLP compared to polynomial model was employed by Oroian et al. [15] for modeling relationships between honey chemical composition and textural properties. ANN model accuracy determined by R^2 values ranged between 0.92 and 0.99, and was slightly worse than accuracy of polynomial model. Shafiee et al. [16] used the combination of computer vision system and artificial neural networks for honey colour assessment and prediction of its total phenolic content (TPC), ash content (AC) and antioxidant activity (AA). In this case, ANN converted RGB values from computer vision system to CIE L^* , a^* , b^* colourimetric parameters and additionally predicted AC, AA and TPC with high accuracy (0.99, 0.98, and 0.87, respectively). Özbalci et al. [17] reported that ANN can be successfully adopted for the estimation of honey sugar contents using Raman spectra of the samples. This approach is an alternative for chromatographic methods. MLP was also used for modeling relationships between honey chemical and electrical parameters [18]. The literature review shows many application of ANNs for honey classification. Cajka et al. [19] reported 96.5% ability of the MLP model for honey samples classification according to geographical origin on the basis of the pattern of volatiles. An excellent separation (100%) among honey samples according to their botanical origin in MLP model with amino acids content as input parameters was presented by Chen et al. [20]. About 95% accuracy was achieved by Anjos et al. [21] when MLP classified honey samples according to botanical origin on the basis of the colorimetric information and the electrical conductivity. MLP was adopted by Zhu et al. [22] to correctly (90.2%) classify pure and adulterated honey samples with different NIR spectral data. Four types of neural network models: MLP, probabilistic neural network, recurrent neural network, and modular

neural network were developed by Oroian and Sorina [23] for the classification of honey based on their physicochemical parameters and phenolics. The MLP with 2 hidden layers was the most suitable ($R^2 = 0.877$) model.

So far, only limited information is available on dielectric properties of honeys, which can be considered as applicable for fast, non-destructive methods of honey quality assessment. Other fast techniques are also presented in literature. Electronic nose was proposed as a rapid and non-invasive determination of the botanical origin and quality of honey [24, 25]. This tool is also potentially useful for honey adulteration detection [26]. However, electronic nose requires proper sensor selection to reduce redundancy and improve classification process. Similarly, electronic tongue was reported as potential tool for the rapid determination of sugars content and for the identification of honey according to botanical and geographical origin [27, 28]. Nevertheless, both these techniques (contrary to dielectric properties measurements) need specifically-prepared samples.

Therefore, the aim of this research is to determine the potential usability of two dielectric parameters (relative permittivity and dielectric loss coefficient) for honey quality assessment. For this purpose, a chemometric approach employing ANNs for data interpretation was used. Based on ANN models, the level of the influence of honey chemical parameters such as glucose/fructose content ratio, water activity, HMF content, pH, diastase activity, as well as the temperature on dielectric honey parameters was calculated. Considering the dependency of dielectric parameters on frequency, the investigation has been performed for two selected frequency values.

Materials and methods

Honey samples

A total of 50 honey samples that were harvested in the year 2011 in Poland were used for this research. 39 samples of nectar honeys [acacia-*Robinia* L. (3 samples), rape-*Brassica napus* L. (10 samples), phacelia-*Phacelia Juss.* (3 samples), goldenrod-*Solidago* L. (3 samples), buckwheat-*Fagopyrum esculentum* (3 samples), heather-*Calluna vulgaris* (L.) Hull (1 sample), willow-*Salix* L. (1 sample), and multiflower (15 samples)], four samples of nectar-honeydew honeys, and seven samples of honeydew honeys (conifers and deciduous) were collected. Honey types were verified based on a pollen analysis which was accomplished in an accredited laboratory according to Polish Standard (PN-88/A-77626, 1998 based on Louveaux et al. [29]). The average percentage of major pollen species in monofloral honey samples were as follows: acacia—32.75, rape—76.14, phacelia—79.48, goldenrod—66.14, buckwheat—48.56, heather—73.39,

willow—84.83. Additionally, the classification of honey samples to nectar-honeydew or honeydew honey group was based on the value of a 20% honey aqueous solution conductivity at 20 °C. Samples described by pollen analysis as nectar-honeydew with electrical conductivity higher than 0.8 mS cm^{-1} were classified as honeydew. For each sample, the following chemical parameters were measured in single repetition: glucose/fructose content ratio [%], water activity of liquid honey, HMF (5-hydroxymethyl furfural) content [mg/kg], pH and diastase activity. For testing these parameters, the methods compiled by the International Honey Commission [30] were used. The methods for measurement of HMF content [31] and chromatographic sugars content [32] were modified according to the conditions of the Bee Products Quality Testing Laboratory. Chemical characteristics of each honey group are presented in Table 1.

For each sample, complex impedance was measured by means of ATLAS 0441 HIA apparatus with an electrode installed in a climate chamber. Measurements of impedance were taken at a frequency ranged from 1 kHz to 1 MHz and at five temperatures: 20, 25, 30, 35, and 40 °C. Based on impedance values, relative permittivity (ϵ') and dielectric loss coefficient ($tg\delta$) were calculated. The calculation method was explained in detail by Łuczycska [33].

ANN prediction models

ANNs are mathematical algorithms that allow solving of regression problems by simulating the human brain functioning. ANNs work effectively even if interrelations are

complex, multidimensional and highly nonlinear. They consist of an array of artificial neurons linked by weighted connections. The multi-layer perceptron used in this research is made up of neurons arranged into one input layer, one output layer, and at least one hidden layer. After the training process, the weights are adjusted to minimize the difference between values of output signals expected and calculated by the network.

The MLP with one hidden layer as a network topology was chosen in this work. Simulations were made up using the Statistica v.10 environment. Experimental data (244 vectors) were the subject of a scaling procedure into a new range of $<0.1-1>$. In the hidden and output layer, neurons with sigmoidal activation function were implemented.

The following parameters were used as an input data set:

- glucose/fructose content ratio (GF),
- water activity of liquid honey (WA),
- 5-hydroxymethyl furfural content (HMF),
- pH,
- diastase activity (D),
- temperature (T).

Four independent neural models were developed. In each model, there were six input nodes and one neuron in the output layer corresponding to the relative permittivity or dielectric loss coefficient measured at a certain frequency. For the determination of the contribution of variables, the group with the best ANN architectures was produced through the following procedure: (1) The 200 ANNs with

Table 1 Chemical properties (maximum, minimum, mean and standard deviation) of honey samples [13]

Chemical parameter	Minimum	Maximum	Mean	Standard deviation
Nectar				
Glucose/fructose content ratio [%]	0.60	1.10	0.86	0.09
Water activity of liquid honey	0.53	0.65	0.57	0.02
HMF content [mg/kg]	1.80	73.00	16.84	14.64
pH	3.67	4.50	4.04	0.19
Diastase activity	5.80	69.30	26.82	13.23
Nectar–honeydew				
Glucose/fructose content ratio [%]	0.73	0.89	0.83	0.06
Water activity of liquid honey	0.56	0.61	0.58	0.02
HMF content [mg/kg]	2.80	23.50	12.45	9.34
pH	4.06	4.69	4.40	0.27
Diastase activity	21.60	31.50	24.38	4.13
Honeydew				
Glucose/fructose content ratio [%]	0.80	0.89	0.85	0.03
Water activity of liquid honey	0.53	0.60	0.56	0.02
HMF content [mg/kg]	1.40	13.40	7.03	3.57
pH	4.39	4.82	4.57	0.12
Diastase activity	23.40	47.90	31.97	7.65

a number of neurons in the hidden layer changing from 10 to 22 were created. The number of neurons in hidden layer was incremented to minimize error in training stage and until the overfitting occurred (2) The type of training was defined. During training, the three methods of supervised learning were used: Broyden-Fletcher-Goldfarb-Shanno, Scaled Conjugate, and Gradient Descent algorithms. (3) The networks were trained with the data, considering a 70% dataset (170 vectors) for learning and 15% (37 vectors) for monitoring. (4) The networks were validated using the validation data (15% of the dataset—37 vectors), and (5) The 20 best ANN architectures were chosen on the basis of a correlation index between values calculated by the model and the expected values for validation dataset. The 20 networks which had maximum correlation coefficient values between experimental and the estimated data were selected as the optimal for the determination of contribution of variables.

Methods for determining the contribution of variables

In this work, two methods of extracting the contribution of independent variables were used. Both methods can be applied only to the MLP networks and require the knowledge of the connection weights matrix. The first method, the partial derivatives method (PaD Method), uses all training data and requires the knowledge of connection weights and the biases matrix [34]. Based on weights and biases, the percentage influence of *i*th input parameter is calculated. The second method is the connection weights method. In this method, the product of connection weights between input nodes and neurons in a hidden layer, as well as the connection weights between neurons in a hidden layer and neurons in the output layer, is calculated. In this work, the method of weights' product determination proposed by Garson [35] was employed.

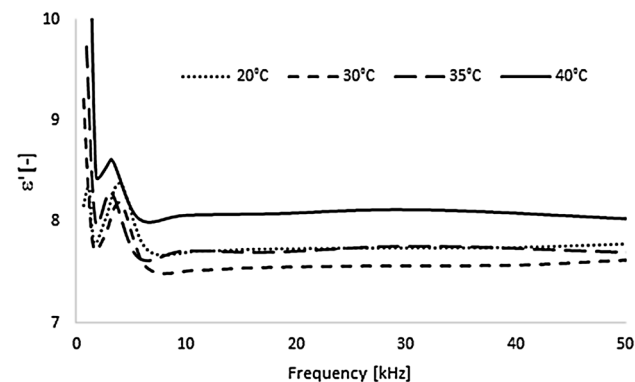


Fig. 1 The frequency dependence of the relative permittivity at various temperatures

Results and discussion

Many scientific works concerning large numbers of agricultural products and foods show that the frequency, temperature, and food composition significantly influence a material's dielectric properties [36–38]. Hence, honey relative permittivity (ϵ') and dielectric loss coefficient ($\text{tg}\delta$) depend on frequency, temperature, and honey physicochemical parameters. Figures 1, 2 depict dependence of dielectric parameters of rape honey on frequency at different temperatures and Figs. 3, 4 show the effect of frequency on dielectric parameters for different honey types. Figures 3, 4 depict dielectric parameters measured at a temperature of 20 °C, the frequency dependencies for temperatures of 25, 30, 35, and 40 °C are analogous. The frequency in figures is limited to 50 kHz to make graphs more readable (in higher frequencies dielectric parameters do not change significantly).

A subject of further analysis (neural modeling) is the relationship between chemical honey parameters as well as

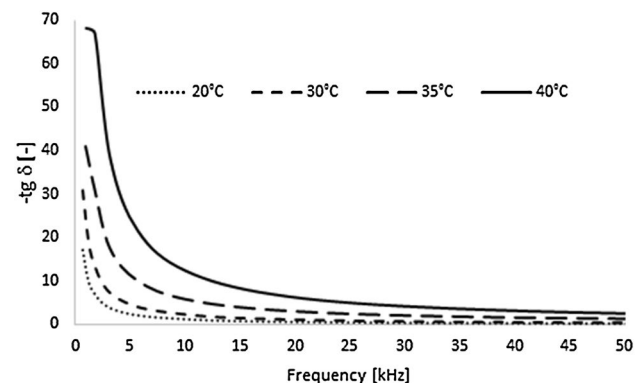


Fig. 2 The frequency dependence of the dielectric loss coefficient at various temperatures

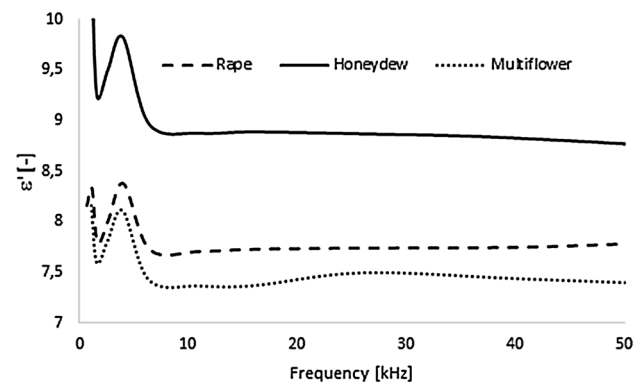


Fig. 3 The frequency dependence of the relative permittivity for various honey types at a temperature of 20 °C

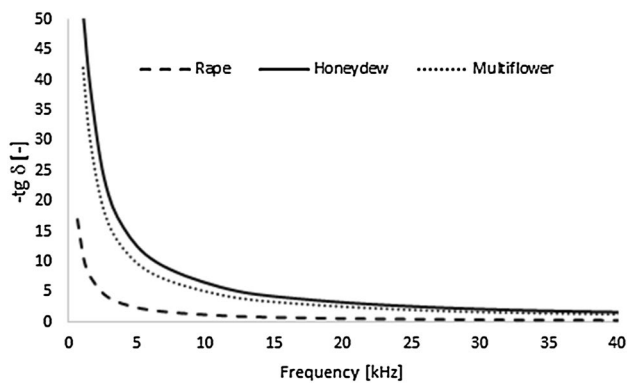


Fig. 4 The frequency dependence of the relative dielectric loss coefficient for various honey types at a temperature of 20 °C

temperature and both relative permittivity and dielectric loss coefficient (measured at frequency 1.6 and 11 kHz).

Since the methods of extracting the relative contribution of input variables in a neural model can be ineffective when inputs are interdependent, before building the ANN-based prediction models, interdependent inputs have to be excluded. In Table 2, Pearson's correlation coefficients between the explanatory variables are presented.

The data presented in Table 2 show that the correlation coefficients between input model parameters are of very low values. The highest correlation coefficient (-0.45) is observed between pH and HMF content. Thus, no explanatory variables were excluded from the model.

The architectures of neural network models used for the determination of the relative contribution of input parameters are presented in Table 3. The model quality assessment was based on Pearson's correlation coefficients between output values expected and calculated by a model as well as mean square error (MSE), both for the validation data set. The MSE value was calculated for normalized data.

The results detailed in Table 3 show that ANN architectures used for quantifying importance of input variables were constructed with the wide range of the number of neurons in the hidden layer. Nevertheless, all ANNs are of the high R value and of the low MSE value calculated for the validation data set which means a high quality of models and their high generalization ability. The results of input variables contribution are presented in Figs. 5, 6, 7.

It can be concluded that both methods of determination of the relative contribution of input variables produced comparable results. However, some differences in results can be noticed and this phenomenon was also reported by other authors. Even if the general results obtained using various methods are comparable, the percentage influence of input variables on output variables can be different for each method [39–41]. The results presented by Gevrey et al. [42] show that more than one method should be used to analyze the contribution of the inputs and results should then be compared because, for each method, they are not always the same.

The data presented in Figs. 5, 6 show that temperature and pH are of the highest influence on the relative permittivity (about 30% in each case) measured at both frequencies

Table 2 Correlation coefficients between explanatory variables [13]

	Glucose content/fructose content ratio [%]	Water activity of liquid honey	HMF content [mg/kg]	pH	Diastase	Temperature [°C]
Glucose content/fructose content ratio [%]	1.00	-0.13	0.26	-0.09	-0.13	0.02
Water activity of liquid honey	-0.13	1.00	0.12	-0.06	0.12	0.00
HMF content [mg/kg]	0.26	0.12	1.00	-0.45	-0.06	0.01
pH	-0.09	-0.06	-0.45	1.00	0.09	-0.01
Diastase	-0.13	0.12	-0.06	0.09	1.00	0.00
Temperature [°C]	0.02	0.00	0.01	-0.01	0.00	1.00

Table 3 Statistics of ANN architectures used for quantifying variable importance

The explained variable	The range of number of neurons in hidden layer	The range of R for validation data set	The range of MSE for validation data set
Relative permittivity ($f=1.6$ kHz)	12–22	0.885–0.911	0.003–0.005
Dielectric loss coefficient ($f=1.6$ kHz)	12–22	0.833–0.892	0.002–0.006
Relative permittivity ($f=11$ kHz)	11–20	0.798–0.859	0.002–0.003
Dielectric loss coefficient ($f=11$ kHz)	12–22	0.935–0.959	0.001–0.003

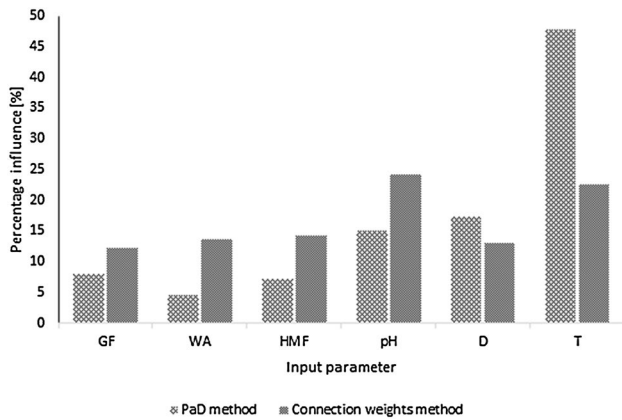


Fig. 5 The contribution of variables used in ANN model of the relative permittivity measured at a frequency of 1.6 kHz

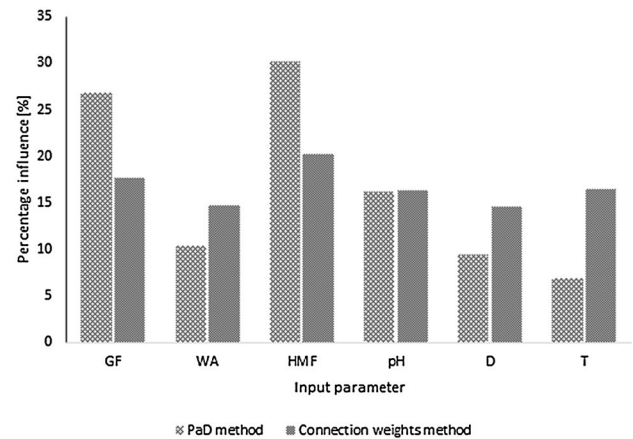


Fig. 8 The contribution of variables used in ANN model of the dielectric loss coefficient measured at a frequency of 11 kHz

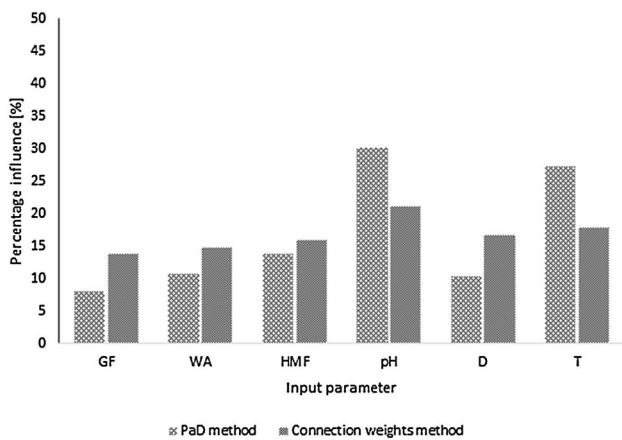


Fig. 6 The contribution of variables used in ANN model of the relative permittivity measured at a frequency of 11 kHz

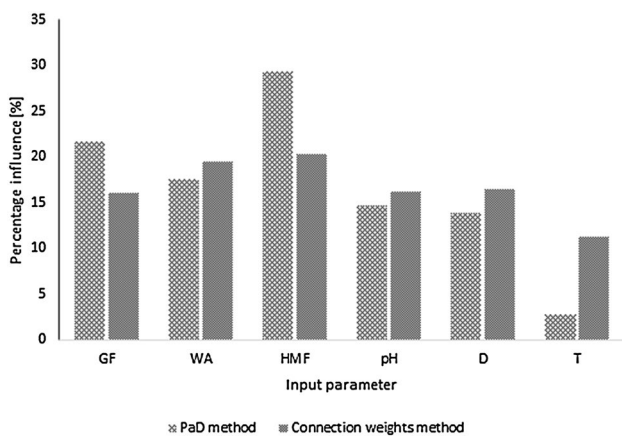


Fig. 7 The contribution of variables used in ANN model of the dielectric loss coefficient measured at a frequency of 1.6 kHz

(1.6 and 11 kHz). The influence of other parameters is lower, about 15%. As it was stated above, the results obtained by PaD and connection weights methods are similar. However, in the case of the relative permittivity measured at a frequency of 1.6 kHz, the influence of diastase activity is comparable with pH according to PaD method. This effect is not visible for relative permittivity measured at a frequency of 11 kHz. In the case of the dielectric loss coefficient (Figs. 7, 8), the highest percentage influence is noticed for HMF content. Generally, for the frequency 1.6 kHz, the glucose/fructose content ratio, pH, water activity of liquid honey, and diastase activity are slightly less important. According to connection weights method, the similar influence of water activity and HMF was calculated. The difference between an impact of HMF and impact of other parameters is more significant in the case of PaD method. For the frequency 11 kHz, the influence of water activity of liquid honey, pH, and diastase activity is comparable and lower than the influence of HMF content and glucose/fructose content ratio, especially according to PaD method. In the case of connection weights method, differences in the influences of specific parameters are less meaningful. For both frequencies, the influence of temperature on dielectric loss coefficient is lower than the impact of other parameters. This effect is more significant in the case of PaD method. Taking into account the applicability of certain chemical parameters for honey quality assessment proven in prior literature, the potential usability of dielectric parameters in this regard can be determined. Honey pH values influence the texture, stability, and shelf life [11], therefore, relative permittivity which is significantly affected by pH can be considered as applicable for possible microbial contamination detection. The level of HMF depends on temperature and time of heating as well as storage conditions [6, 43]. Since the value of dielectric loss coefficient is affected the most by

HMF content, this parameter is promising for honey freshness assessment. It is in agreement with results presented by Łuczycska et al. [44] who demonstrated that the dielectric loss coefficient changed significantly as a result of honey overheating. In our previous work, the other electrical honey parameter, complex impedance, was found as potentially useful for honey quality deterioration detection (freshness assessment, micro-organisms growth, and overheating detection) [13].

The frequencies of dielectric parameters measurement (1.6 and 11 kHz) used in this analysis are in the frequency range where significant differences in values of relative permittivity and dielectric loss coefficient are observed. The differences depend on both, the temperature and honey type. Since no significant differences in results of the relative contribution of input variables are found depending on frequency, it can be stated that dielectric parameters measured at frequencies in a relatively wide range (especially in the case of relative permittivity) can be used in practice for honey deterioration detection.

Conclusions

Dielectric honey parameters measured at low frequencies can be considered as potentially useful parameters for honey adulteration detection. These parameters are easy to measure, therefore, methods based on dielectric parameters determination can be easier, not time-consuming, and cheaper than the most common methods involving chemical indicators measurement. It is known from literature review that pH and water activity can be used as indicators of possible microbial growth. The results produced by chemometric methods (ANNs) show that relative permittivity is significantly affected by pH, however, the influence of water activity on this parameter is rather small. It can be stated that relative permittivity seems to be a promising parameter for possible microbial contamination detection but it should be the subject of further analysis with the use of larger number of honey samples. In addition, the high influence of temperature on relative permittivity was observed. Hence, during the development of the methodology of honey adulteration detection based on this dielectric parameter, not only frequency, but also temperature must be taken into account. The high impact of HMF content on dielectric loss coefficient leads to the conclusion that this parameter is potentially useful for honey freshness assessment.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

Research involving human participants and/or animals During research no human participants and animals were involved.

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