

INFLUENCE OF TEMPERATURE AND WATER CONTENT ON THE RHEOLOGICAL PROPERTIES OF POLISH HONEYS*Sławomir Bakier**Department of Food Industry Machines and Appliances, Technical University, Białystok*

Key words: Arrhenius model, honey, Newtonian fluid, rheological properties, viscosity, Wiliam-Landel-Ferry model

The study was aimed at investigating rheological properties of Polish honeys. In particular, the influence of water content and temperature on honey viscosity was analysed. The measurements were conducted within a wide temperature range from 260 K to 330 K. The water mass fraction in all the investigated honeys was 0.146-0.20 g/g. The rheological measurements were carried out using both cone and plate viscometer and the Searle-type system. The shear rates used were in the range of $\dot{\gamma} \in \langle 0.1667; 437.4 \rangle \text{ s}^{-1}$.

In the liquid state all the investigated honeys manifested the properties of a Newtonian fluid. It was found out that the temperature was the parameter that had the most significant effect on honey viscosity. An exponential dependence between honey viscosity and temperature and water content was determined. Making use of multiple regression it was possible to create a mathematical model to describe honey viscosity in the function of temperature and water content. The model took the following form: $\mu = 1.27 \cdot 10^{22} \cdot \exp(-38.363W - 0.1398T)$. The obtained dependence was verified based on the data found in literature on the subject.

ABBREVIATIONS

A, B – constant in VTF model (-); C_p, C_2 – coefficients of WLF equation (-); D – constant in prediction model (-); E_a – activation energy ($\text{kJ} \cdot \text{mol}^{-1}$); r – correlation coefficient (-); R – universal gas constant ($8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$); R^2 – determination coefficient (-); t – temperature ($^{\circ}\text{C}$); T – temperature (K); T_g – glass transition temperature (K); w – water content (%); W – water content (g/g); $\dot{\gamma}$ – shear rate (s^{-1}); μ – viscosity (Pa.s); μ_e – experimental viscosity (Pa.s); μ_g – viscosity at glass transition temperature (Pa.s); μ_p – predicted viscosity (Pa.s); μ_T – viscosity at T temperature (Pa.s); μ_0 – constant in Arrhenius model (Pa.s); τ – shear stress (Pa).

INTRODUCTION

Honey is a sticky and aromatic product made by bees from vegetable raw materials such as nectar or honeydew. It is a concentrated water solution of carbohydrates whose chemical composition varies depending on climatic conditions and geographical origin [Anklam, 1998]. Honey is mostly composed of monosaccharides, such as glucose and fructose, whose quantitative share may vary between 80% to 95% of the total mass of all carbohydrates [Cavia *et al.*, 2002]. Honey moisture content generally ranges from 13 to 20% [White, 1978; Mendes *et al.*, 1998]. Honey may also contain some considerable additions of about 25 other oligosaccharides [Anklam, 1998]. There are reports confirming melezitose content of up to 11% [Lazaridou *et al.*, 2004], maltose 9.8% [Terrab *et*

al., 2002], sucrose up to 6.13% [Popek, 2002], erlose 1.55% [Devillers *et al.*, 2004], arabinose 0.9% [de Rodriguez *et al.*, 2004]. Apart from the above, honey also contains a number of other ingredients; they include acids, proline, enzymes and minerals [Anklam, 1998; Terrab *et al.*, 2002]. However, carbohydrates and water are the determinants of such physico-chemical properties of honey as its viscosity, hygroscopicity and granulation [Cavia *et al.*, 2002].

The rheological properties of honey are important qualities that influence the sensory quality of the product and also affect a number of technological operations, such as honey heating, mixing, filtering, hydraulic transport and bottling [Yanniotis *et al.*, 2006]. Honey viscosity also plays a significant role in the crystallization process [Rüegg & Blanc, 1981]. Due to the above, the preoccupation with the rheological properties of honey seems to be well grounded [Bhandari *et al.*, 1999; Lazaridou *et al.*, 2004; Yoo, 2004; Yanniotis *et al.*, 2006]. Besides, there is a general agreement that honey, in its liquid state, behaves like a Newtonian fluid whose viscosity is mainly dependent on temperature [Sopade *et al.*, 2002] and water content [Zaitoun *et al.*, 2001; Yanniotis *et al.*, 2006]. However, there have also been reports indicating a possibility of non-Newtonian behavior of some honeys *e.g.* occurrence of thixotropic effect [Sopade *et al.*, 2004]. At room temperature honey viscosity is equal to 9.9 Pas with water content of 18.9% and up to 61.1 Pas with 13.9% of water [Lazaridou *et al.*, 2004]. It should be noted that the presence of a trisaccharide – melezitose – considerably increases honey viscosity, for instance, honey containing 10.5% of melezitose and 13%

of water content reaches 200 Pas at 20°C [Lazaridou *et al.*, 2004]. A temperature drop to 0°C will cause further increase of honey viscosity – as high as 450-2400 Pas [Bhandari *et al.*, 1999; Bakier, 2006]; still manifesting the properties of a Newtonian fluid. At temperatures between minus 40 and minus 46°C honey undergoes glass transition reaching the viscosity of the order of 7×10^{10} Pa s [Sopade *et al.*, 2002]. Like temperature, water content has a significant influence on honey viscosity – its increase results in an exponential drop of viscosity [Zaitoun *et al.*, 2001; Yanniotis *et al.*, 2006].

THEORETICAL BACKGROUND

There are a number of mathematical models used to describe the rheological properties of honey [Sopade *et al.*, 2002]. They focus generally on the description of viscosity as dependent on temperature. As a rule, Arrhenius equation is used for the purpose and it takes the following shape [Zaitoun *et al.*, 2001; Yanniotis *et al.*, 2006]:

$$\mu = \mu_0 \exp\left(\frac{E_a}{RT}\right) \quad (1)$$

The dependence is said to describe honey viscosity with a relatively small error, not greater than 4.41% [Bhandari *et al.*, 1999]. However, some papers have shown the above model to be unsatisfactory to describe honey viscosity with respect to fructose and glucose solutions [Ollet & Parker, 1990].

In relation to temperature honey viscosity is often described by WLF (William-Landel-Ferry) equation [Williams *et al.*, 1955]. The model makes use of such parameters as glass transition temperature and glass state viscosity. However, in literature on the subject, there are also a number of papers that define the WLF dependence in different ways. For example, Roos [1992] defines the dependence as:

$$\ln\left(\frac{\mu}{\mu_g}\right) = \frac{-17.44 + (T - T_g)}{51.6(T - T_g)} \quad (2)$$

Mossel *et al.* [2000] on the other hand, present the WLF equation in a considerably changed way:

$$\ln\left(\frac{\mu}{\mu_g}\right) = \frac{-17.44(T - T_g)}{51.6 + (T - T_g)} \quad (3)$$

Sopade *et al.* [2002] define the WLF dependence in somewhat generalized way and use it to describe the viscosity of various types of honey:

$$\ln\left(\frac{\mu}{\mu_g}\right) = \frac{-C_1(T - T_g)}{C_2 + (T - T_g)} \quad (4)$$

Lazaridou *et al.* [2004] present the WLF model in yet another form:

$$\ln\left(\frac{\mu}{\mu_g}\right) = \frac{-C_1(T_g - T)}{C_2 + (T_g - T)} \quad (5)$$

It is pointed out however that the WLF dependence gives the most exact description of honey viscosity in the func-

tion of temperature [Sopade *et al.*, 2002]. Nevertheless it is stressed that the model is also sensitive to changes of the medium composition.

Another mathematical relation describing the rheological properties of liquid honey in the function of temperature including the glass transition temperature is the VTF (Vogel-Tamman-Fulcher) equation that takes the following form [Sopade *et al.*, 2002]:

$$\mu = A \exp\left(\frac{B}{T - T_g}\right) \quad (6)$$

Disregarding the mathematical inaccuracies connected with the interpretation of the WLF model, it should be noted here that the main drawback of the above dependencies is that they describe honey viscosity in relation to temperature only. They fail to account for water content though. Additionally, using the models requires determining some troublesome quantities such as glass transition temperature and viscosity at glass state (WLF model) or the activation energy (Arrhenius model). Determination of the quantitative values of these parameters constitutes a problem *per se* and creates more difficulties than the measurements set to determine honey viscosity at a given temperature. As a result, the use of the described models in practice is rather limited.

There are a number of dependencies that include, apart from temperature, the influence of water content on honey viscosity. As early as in 1939 Oppen & Schuett determined a linear correlation between the viscosity logarithm and water temperature [Mossel *et al.*, 2000]. The correlation is expressed in the form of the following equation:

$$W = (62500 - 156.7T)[T(\log\mu_r + 1) - 2287(313 - T)] \quad (7)$$

Junzheng & Changying [1998] published a relatively simple empirical dependence in the form of the equation:

$$\mu = 14.2 \cdot 10^3 \cdot \exp(-0.31w - 0.085t) \quad (8)$$

Analogously, the equation was used to describe the viscosity of Spanish honeys [Gómez-Díaz *et al.*, 2005]:

$$\mu = 19.2 \cdot 10^3 \cdot \exp(-0.31w - 0.087t) \quad (9)$$

It should be noted, however, that equations (8) and (9) were used for a relatively high water content ranging from 17.07 up to 34.06%. In spite of this, the equations illustrate a comparatively easy way of describing honey viscosity including both temperature as well as water content.

Summing up the above analysis, it is possible to demonstrate by using simple mathematical transformations that nearly all the dependences presented above can be reduced to the following simplified mathematical form:

$$\mu = a \exp(-bT - cW) \quad (10)$$

As it can be seen, honey viscosity decreases exponentially with an increase in temperature and water content. The relation has a relatively convenient form that makes it possible to

determine the viscosity of any given type of honey by taking a sample measurement of water content using fast and cheap refractometric instruments.

The present paper formulates the thesis stating that the viscosity of all Polish honeys within a wide range of their temperature, *i.e.* from 260 K to 330 K, and independently of their origin can be both successfully and accurately described by equation (10). The only condition imposed was that the investigated honeys complied with Polish honey standards and the amount of water remained within the 14 to 20% limit.

MATERIALS AND METHODS

The measurement data on honey viscosity had been systematically gathered since 1995. As a result more than several dozen samples of different water content and temperature values were analysed to finally obtain a set of honey viscosity data consisting of 255 different values. Thus it was possible to verify dependence (10) by making use of both Statistica 7.1 software and multiple regression [Statistica, 2005]. However, due to the program requirements, the dependence (10) was linearized by performing logarithming into the following form:

$$\ln\mu = \ln a - bT - cW \quad (11)$$

After substituting $\ln a = D$ into relation (11), it was possible to obtain a linear dependence $\ln\mu$ dependent on two variables – temperature T and water content W :

$$\ln\mu = D - bT - cW \quad (12)$$

The equation obtained as a result of regression was additionally verified using literature data on honey viscosity published by Junzheng & Changying [1998], Lazardou *et al.* [2004], and Sopade *et al.* [2002].

Sample collection and preparation. In the investigations honey samples acquired from all regions of Poland were used. They had been acquired directly from the producers through the years 1995 and 2005 and their origin was determined on the basis of both producers' certificates and sensory verification. Due to the main aim of the paper, the investigations did not include a verification of the honey origin by performing pollen analysis. The samples were systematically collected and stored in the summer periods. They were stored in a dark room at temperatures below 18°C, bottled and kept in tightly sealed containers. The viscosity measurements were carried out after heating the honey up to 55°C. Then the samples were filtered in order to remove all the unwanted fibre crystals found in the honey after liquification [Bakier, 2004]. On cooling the refractometric method was used to determine the water content by measuring the refractive index at 20°C [Abu-Jdayil *et al.*, 2002]. To this end, an Abbe refractometer produced by Carl Zeiss Jena was used. Prior to this, the refractometer was calibrated using immersion oil with a known value of the light refractive index.

Rheological properties. The rheological measurements were carried out with the help of a rotational viscometers:

Searls-type systems and the cone and plate apparatus. Independent use of both rheometers made it possible to avoid biases. At the same time it enabled simultaneous verification of the results. For the purpose, use was made of both Rheotest 2 MLW and Brookfield rheometer series DVII+. Sample thermostating during the measurements above ambient temperatures was performed by using ultrathermostats with a heating element. To obtain honey samples at temperature below ambient temperature a thermostat with an inbuilt cooling system was used. The device made it possible to determine honey viscosity ranging from 260.15 K to 373.15 K. Viscosity values were obtained by the analysis of regression of the empirical flow curve $\tau = f(\dot{\gamma})$ to constitutive equation (Newtonian fluid): $\tau = \mu \cdot \dot{\gamma}$ [Ferguson & Kembłowski, 1991].

RESULTS AND DISCUSSION

Figure 1 shows some exemplary results of rheological measurements of a liquid polyfloral honey sample of water content $W=0.176$ and at the temperature of 293.15 K. The regression equation obtained after the approximation of the measurement results took a shape of a straight line $\tau = 16.42 \cdot \dot{\gamma}$ with the determination coefficient $R^2 = 0.995$. All the measurements points are located within the confidence interval amounting to 0.95 and the dynamic viscosity value of the investigated sample, according to the Newtonian law is equal to 16.42 Pas. Analogous measurements were conducted for 254 honey samples of defined water content but at different temperature. Only the range of shear rates varied. This was due to the measurement ranges of the rotational viscometers. The shear rate values were within the range of $\dot{\gamma} \in (0.1667; 437.4) \text{ s}^{-1}$.

Figure 2 shows the dependence of the viscosity of different honey samples – both water content $W=0.12$ and temperature range $T \in (266.15; 295.15) \text{ K}$. The distribution of measurement points with respect to the equation resulting from the regression and the determination coefficient $R^2=0.995$, shows in an unequivocal way that the dependence of honey viscosity on temperature has a strictly exponential character.

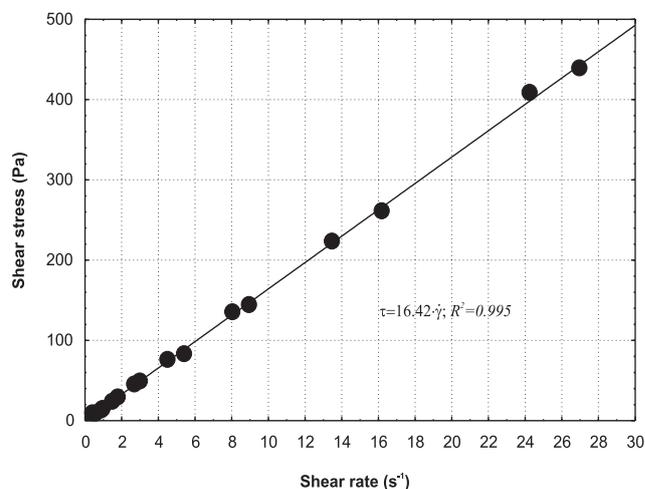


FIGURE 1. Flow curve of polyfloral honey at 293.15 K and water content $W=0.176$.

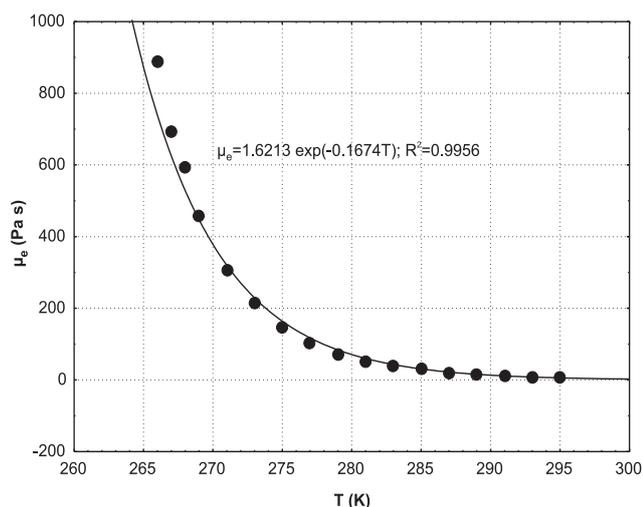


FIGURE 2. Dependence of viscosity on temperature for polyfloral honey samples of water content $W=0.19$.

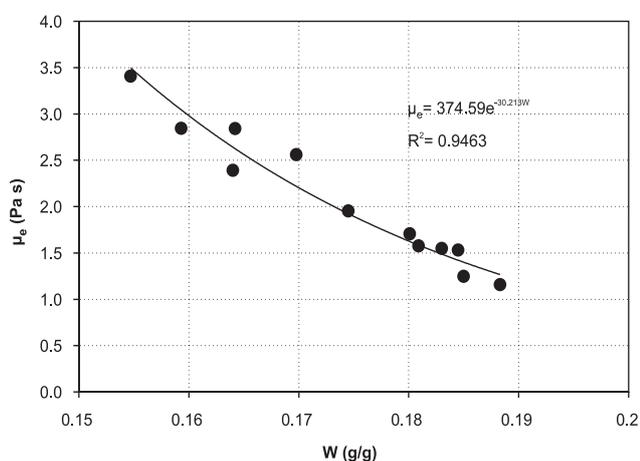


FIGURE 3. Influence of water content W on the viscosity of different honey types at temperature 313.15 K.

The received result is analogous to findings of other authors [Mossel *et al.*, 2000; Bhandari *et al.*, 1999] However, at low temperature it is possible to observe that the viscosity values arrived at empirically are higher than the values predicted using the equation obtained as a result of regression. The effect is clearly seen below 213.15 K (0°C).

The influence of water content on honey viscosity at 313.15K is shown in Figure 3. The regression equation obtained as a result of the approximation of measurement results also takes the form of an exponential curve analogous to that observed by other authors [Gómez-Díaz *et al.*, 2005; Lazaridou *et al.*, 2004; Yanniotis *et al.*, 2005; Zaitoun *et al.*, 2001]. A high value of determination coefficient $R^2=0.9463$ confirms the right choice of the adopted mathematical model. At lower water content higher scattering of viscosity values is obtained. It is assumed that greater scattering of measurement values is caused by the changeability of the chemical composition of honey samples.

An overall presentation of all measurements is shown in Figure 4 in a form of a 3D graph. The temperature range of the conducted measurements was between 260.15 K up to

330.15 K and the water content W varied in the investigated honeys from 0.15 to 0.20. The lower limit of temperature measurements was determined by the reading limits of rotational viscometers.

The analysis of the curve in Figure 4 evidently shows that the increase of both water content and temperature gives a non-linear drop of honey viscosity. The viscosity of all investigated samples reaches the value of about 1000 Pa s below 273 K. At lower temperature values the curve gets even steeper demonstrating a growing influence of water content on honey viscosity.

As a result of the approximation process using multiple regression it was possible to obtain in Statistica 7.1 the numerical values of their coefficients from equation (12). Table 1 presents the obtained data with their standard deviation and other basic statistical parameters. Special attention should be paid to a relatively high value of the determination coefficient $R^2=0.975$. Standard deviations of the determined parameters of equation (12) do not exceed 5% of their values, which proves that the mathematical model adopted here is correct and provides a good description of honey viscosity in the investigated variability interval of parameter T and W . Figure 5 shows all the empirically obtained values with regard to the predicted ones. The points lying above the straight line indicate the cases where the observable values are higher than the predicted ones and *vice versa*. In the middle part of the chart there is a slight overestimation of the viscosity value in the model, whereas there is a reverse tendency on the boundaries of the region. The experimental viscosity values exceed the

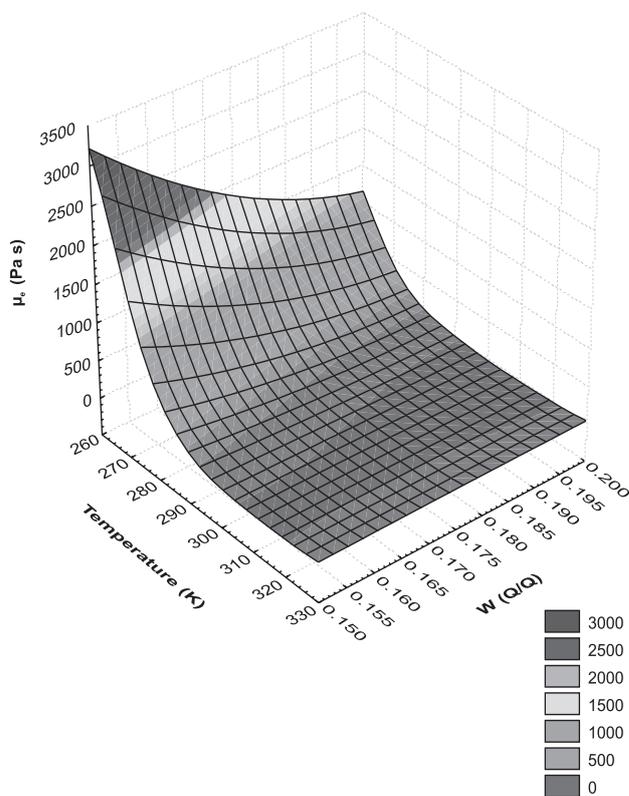


FIGURE 4. Graphical representation of honey viscosity measurements within water content $W \in (0.15; 0.20)$ and temperature range of $T \in (260.15; 330.15)$ K.

TABLE 1. Results of multiple regression of dependent variable $\ln\mu$ with respect to independent variables W and T .

Value of determination coefficient: $R^2=0.975$. Corrected value of determination coefficient: $R^2=0.974$.			
Parameter of equation (12)	Value resulting from regression	Standard deviation	S.D (% of value)
D	50.895	0.594	1.166
b	38.363	1.903	4.959
c	0.1396	0.00142	1.014

predicted ones there. In these intervals all the measurement points are located above the straight prediction line. By limiting the interval of temperature changes it is possible to obtain much better results of matching the experimental results to the analysed model. Such an action will probably improve the regression quality, but unfortunately it would produce a dependence of merely local character. It seems that the regression analysis yields satisfactory results.

Substituting the parameters resulting from the regression analysis into equation (12) we obtain the following dependence:

$$\ln\mu = 50.895 - 38.363 - 0.1398T \quad (13)$$

or after transformation we get the form analogous to the one obtained by Junzheng & Changying [1998]:

$$\mu = 1.27 \cdot 10^{22} \cdot \exp(-38.363 - 0.1398T) \quad (14)$$

Comparing the obtained equation with the analogous ones [Junzheng & Changying, 1998; Gómez-Díaz *et al.*, 2005] it is worth noting a relatively high convergence of the number parameter accompanying water content W . It should be remembered that in equations (8) and (9) water content was expressed in percentages whereas this paper expresses water content in honey by the mass fraction. The use of temperature on the absolute scale considerably modified the value of the constant found with the

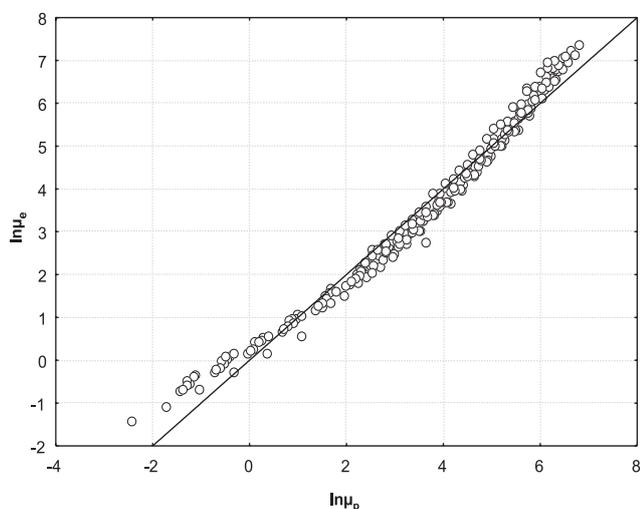


FIGURE 5. Comparison of the experimental results the observed with respect to the predicted values.

temperature. However the greatest difference can be observed in constant value preceding the exponent. The value of this constant has the viscosity value and comes close to the μ_g coefficient found in the WLF model [Sopade *et al.*, 2002].

Figure 6 shows the verification of equation (13) obtained on the basis of the bibliographical data. Making use of the viscosity values of Australian [Sopade *et al.*, 2002], Chinese [Junzheng & Changying, 1998] and Greek [Lazaridou *et al.*, 2004] honeys, the vertical axis displays the viscosity values from the bibliographical data whereas the horizontal axis shows the viscosity predicted on the basis of equation (12). Most points in Figure 6 are located just below the straight line, which means that there is an overestimation of the viscosity value according equation (12). Similarly to Polish honeys the overestimation occurs only in the middle part of analyzed parameter. Nevertheless there exists a numerous set of honey viscosity values that fulfill the equation.

Comparing Figure 5 for Polish honeys with Figure 6 for honeys from other countries, it is possible to state that at the same water content and temperature the former possess slightly higher viscosity values than the latter. This could be directly related to the origin of the honey. The distribution of points on the diagrams in Figures 5 and Figure 6 shows that the dependence is not perfect, nevertheless it gives a very close and valuable approximation for the determination of honey viscosity.

CONCLUSIONS

As a result of the analysis of literature sources on honey viscosity it was possible to build a mathematical model that was subsequently subjected to empirical verification. It was based on the description of honey viscosity using two parameters: absolute temperature and water content in terms of mass fractions. A large number of measurements of honey viscosity taken systematically for many years have made it possible to verify the reliability of the adopted model on the honeys originating from all parts of Poland.

The results of the research based on the multiple regression analysis enabled determining the empirical dependence

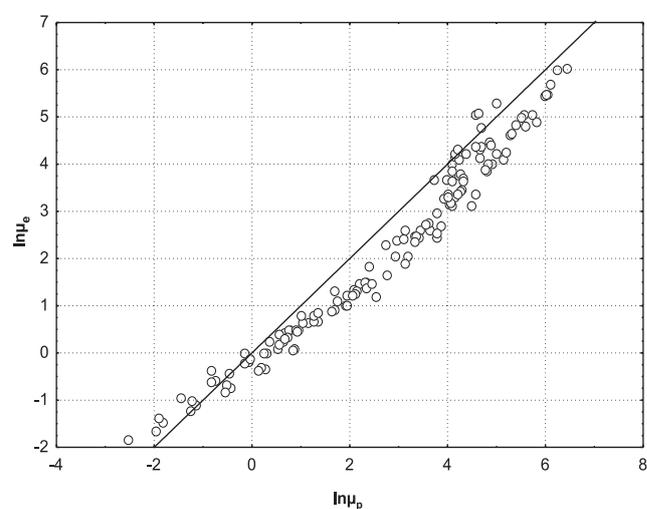


FIGURE 6. Comparison of honey viscosity values from bibliography and the values obtained on the basis of prediction from equation (13).

in the form of equations $\ln\mu = 50.895 - 38.363 - 0.1398T$ or $\mu = 1.27 \cdot 10^{22} \cdot \exp(-38.363 - 0.1398T)$. The main advantage of the dependencies is the possibility of determining honey viscosity in a relatively wide range by a simple measurement of its water content. In this way we can obtain an approximated value of honey viscosity paying no heed to honey origin or chemical composition. These two factors, however, can significantly affect honey viscosity values, for example, the presence of melezitose [Lazaridou *et al.*, 2004] and have an effect on uneven distribution of the observed and forecasted viscosity values. In spite of all the above drawbacks of the model presented, it makes it possible to satisfactorily (relative error not exceeding a dozen per cent) determine the viscosity of Polish honeys. This is particularly valuable for technological purposes. It is extremely critical in such processes as honey mixing, heating, hydraulic transport and bottling.

The verification of the dependence on the basis of literature on the subject made it possible to state that Polish honeys possess slightly higher viscosity values. This property could be related to the origin and chemical composition of the honeys investigated.

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WPLYW TEMPERATURY I ZAWARTOŚCI WODY NA WŁAŚCIWOŚCI REOLOGICZNE POLSKICH MIODÓW*Sławomir Bakier**Katedra Maszyn i Urządzeń Przemysłu Spożywczego, Politechnika Białostocka, Białystok*

Badano właściwości reologiczne wybranych polskich miodów pszczelich. Analizowano wpływ zawartości wody i temperatury na ich lepkość. W trakcie pomiarów stosowano szeroki przedział temperatury od 266 do 333 K. Udział masowy wody w badanych miodach wynosił od 0.146 do 0.20 g/g. Pomiarów reologicznych prowadzono w wykorzystaniu układu pomiarowego stożek-platek oraz w przepływie Sarrisa. Stosowano szybkości ścinania zawarte w przedziale $\dot{\gamma} \in (0.1667; 437.4) \text{ s}^{-1}$. Wszystkie badane miody w stanie płynnym w zakresie badanych parametrów wykazywały właściwości płynu newtonowskiego i krzywe płynięcia analogiczne jak na rys.1. Stwierdzono, że temperatura jest parametrem wywierającym największy wpływ na lepkość miodów. Występuje przy tym wykładnicza zależność pomiędzy temperaturą i zawartością wody a lepkością miodu – rys. 2, rys. 3 i rys 4. Wykorzystując regresję wielokrotną – wyniki zamieszczone w tabeli 1, wyznaczono model matematyczny opisujący lepkość w funkcji temperatury i zawartości wody w postaci równania: $\mu = 1.27 \cdot 10^{22} \cdot \exp(-38.363W - 0.1398T)$. Porównanie przewidywanych wyników wartości lepkości miodu za pomocą uzyskanego modelu z wartościami uzyskanymi na drodze doświadczalnej przedstawiono na rys. 5. Na rys. 6 przedstawiono również efekt porównania wartości lepkości uzyskiwanej na podstawie powyższego równania z wartościami zaczerpniętymi z bibliografii.