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Texture characteristics of raw rapeseed honey after storage at room temperature or freezing and heating up to 50°C

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A b s t r a c t. The effects of storing honey for 18 months at room temperature or in a freezer as well as the impact of heating it to different temperatures (30, 40 and 50°C) were studied. The rheological parameters of fresh rapeseed honey were compared to those of the non-stored and unheated samples (control). The storage temperature significantly influenced the rheological behaviour of the honey (except adhesiveness). Higher values of all textural parameters were found in frozen honey in comparison with control and room temperature samples. However, in comparison with the control samples, higher values (around twice) of the dynamic viscosity, firmness, work and adhesive force were produced by honey stored at room temperature. Thermal treatment of the honey heated at 30, 40 and 50°C influenced a continuous decrease in all of the textural properties (with the exception of the cohesive force) compared to the control samples (20°C). The average change in the texture-temperature coefficient for dynamic viscosity, firmness, work, adhesiveness and adhesive force from 20 to 50°C was similar (between 6.62 and 4.09%). Significant, very close and positive Spearmen's rank correlations ($0.894 \leq r_S \leq 0.987$) were observed for dynamic viscosity, firmness, work, adhesiveness and adhesive force. Lower correlations ($0.524 \leq r_S \leq 0.684$) were found between the cohesive force and all of the other textural properties.

K e y w o r d s: rapeseed honey, storage, temperature, rheology, texture

INTRODUCTION

In 2014, the total number of beehives in Poland was estimated at 1.52 million with a production capacity of 12.8 t of natural honey (FAO, 2017). Rape (*Brassica napus* L.) as well as other species and varieties are cultivated in Europe mainly to obtain seed oil. This plant is very alluring to bees both for its pollen and nectar. Moreover, it represents a very important spring source of nourishment for the bees in Central and Eastern Europe, giving rise to large amounts of very pure monofloral honey (Persano Oddo *et al*., 2004). Honey is a natural condensed aqueous solution of sugars. After harvesting, it passes through successive stages including natural crystallization, as well as storage time until consumption. Crystallization occurs spontaneously and results in significant changes, mainly to the honey's texture. Most importantly, it does not reduce the quality of the product. The granulation of honey is a two-stage process, the first stage is the formation of crystals and the second one is the gradual growth of their dimensions. The rate of change depends on the composition of the honey and the storage temperature (Lupano, 1997). Controlled crystallization can be used to make desirable products, such as creamed honey, which may be spread as easily as butter at room temperature and does not drip (Chen *et al*., 2009). The domestic conditions of honey storage at room temperatures are dependent on geographical localization, the time of year and household heating arrangements.

Although the freezing process does not occur in honey (Sopade *et al*., 2002), low temperatures gradually minimize (or halt) the process of crystallization, inhibit fermentation, and reduce the viscosity of the honey (Horn, 2008), with no negative effects on its quality (Bakier, 2011). The storage

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of honey in a freezer thus seems to result in the maintenance of high honey quality similar to raw honey in the long term (Horn, 2008). Additionally, creamed honey has a fine crystalline structure and specific rheological properties (Bakier, 2011).

Turhan *et al*. (2008) revealed that overheating honey is superfluous for its conditioning and packaging. Indeed, mild temperatures (a heating level of 40 to 50°C) are sufficient to minimize viscosity and inhibit crystallization. According to Bakier (2006), the effective process of honey liquefaction requires heating to between 52 and 55°C, but for not less than 10 minutes.

According to Bourne (2002) the term texture includes physical properties which are connected with the food structure. In general, texture refers to solid food products and viscosity relates to fluid ones. However, many solid products exhibit liquid properties and many liquids exhibit the properties of solid foods (Bourne, 2002). The influence of temperature and chemical composition on the parameters of the texture profile analysis (TPA) of honey was previously evaluated by Oroian *et al*. (2016). Furthermore, over the past 10 years consumers have expressed a preference for more monofloral honeys than polyfloral ones, which are much cheaper than single floral honeys (Belay *et al*., 2017; Oroian *et al*., 2017).

The aims of the study were: (1) to assess the effect of the storage temperature for 18 months (room vs. freezing); and (2) to evaluate the impact of different heating temperatures (30, 40 and 50°C) on the textural properties of Polish raw rapeseed honey.

MATERIALS AND METHODS

The study was carried out using seven samples of raw (unprocessed) rape honey purchased at a local market directly from beekeepers. The apiaries were located in the Lublin region. The characterization of honey sample collection and storage was described in detail in an earlier paper (Kędzierska-Matysek *et al*., 2016a). Briefly, honey samples were collected in May/June 2014. Analyses undertaken using fresh honey samples in the first trial were performed until 15th July 2014, details of their heating treatment were given in the paper of Kędzierska-Matysek *et al*. (2016b). Briefly, samples of honey were submerged in a water bath and heated to a specified temperature (30, 40 or 50°C) for 15 min, before being cooled at room temperature and analysed. Untreated (a non-stored and unheated) samples of fresh honey (control) were analysed at 20°C. In the second trial the experimental groups were stored for 18 months, the first batch in the freezer at –20°C (FRO), and the second one was stored in darkness at room temperature (RT), *i.e.* between 20 and 26°C, within the range in which honey is usually stored.

Textural tests were conducted with a Zwick/Roell universal testing machine Proline BDO-FB0.5TS (Zwick GmbH and Co, Ulm, Germany) using a back extrusion cell and a disc plunger (5 mm height and 45 mm diameter). In the first test, the piston pushed the honey over 20 mm with a speed of 100 mm min^{-1}, causing the extrusion of the sample up and around the edge of the disc, before finally returning to its original position. The parameters measured were recorded as firmness (g), work (mJ), and adhesiveness (mJ) (Fig. 1a). In the second test the probe was pushed into- the honey sample (holding time = 5 s) in order to achieve a satisfactory bond between the two surfaces, and then withdrawn until the sample completely separated. In the case where honey residue remained on the probe at the end of the test, the force exerted never return to zero. The parameters measured were recorded as the adhesive force (or stickiness) (g) and cohesive force (g) (Fig. 1b). In both trials, all parameters were determined using testXpert II software.

Dynamic viscosity was measured in line with the procedure published previously (Kędzierska-Matysek *et al*., 2016a; Kędzierska-Matysek *et al*., 2016b) using the Zwick/Roell Proline BDO-FB0.5TS universal testing machine (Zwick GmbH and Co, Ulm, Germany) with the back extrusion rig and the testXpert II software.

In order to evaluate the influence of temperature on the texture measurements between 20 and 50°C, the texture-temperature coefficient (T-TC) was calculated (Bourne, 1982) and expressed in terms of a percentage texture parameter change per degree temperature change.

The analyses were performed using Statistica ver. 13 (Dell 2016) and the Nonparametric Statistics package. The tables present the mean, standard deviation, and minimum and maximum values for each dependent variable. The influence of different storage conditions (CON, RT, FRO) and heating treatments (20°C *vs*. 30, 40 and 50°C) on the texture properties was verified by the Kruskal-Wallis test (comparison of many independent groups). The differences between the means at the 95 and 99% ($p \leq 0.05$ and $p \leq 0.01$) confidence levels were considered to be statistically significant. The relationships between the rheological properties of honey were calculated using Spearman's rank correlation coefficients (rS).

RESULTS AND DISCUSSION

In comparison to the CON samples, all of the parameters significantly ($p \leq 0.05$ or $p \leq 0.01$) increased for FRO honey, *i.e.* for the cohesive force from more than three times to almost nine times for dynamic viscosity (Table 1). Only in the case of adhesiveness, such a difference was not observed possibly due to the considerable variability of the frozen honey samples. In turn, for honey stored at RT changes in such properties as the granulation effect were observed for dynamic viscosity, firmness, work and adhesive force (but to a lesser and insignificant extent). It

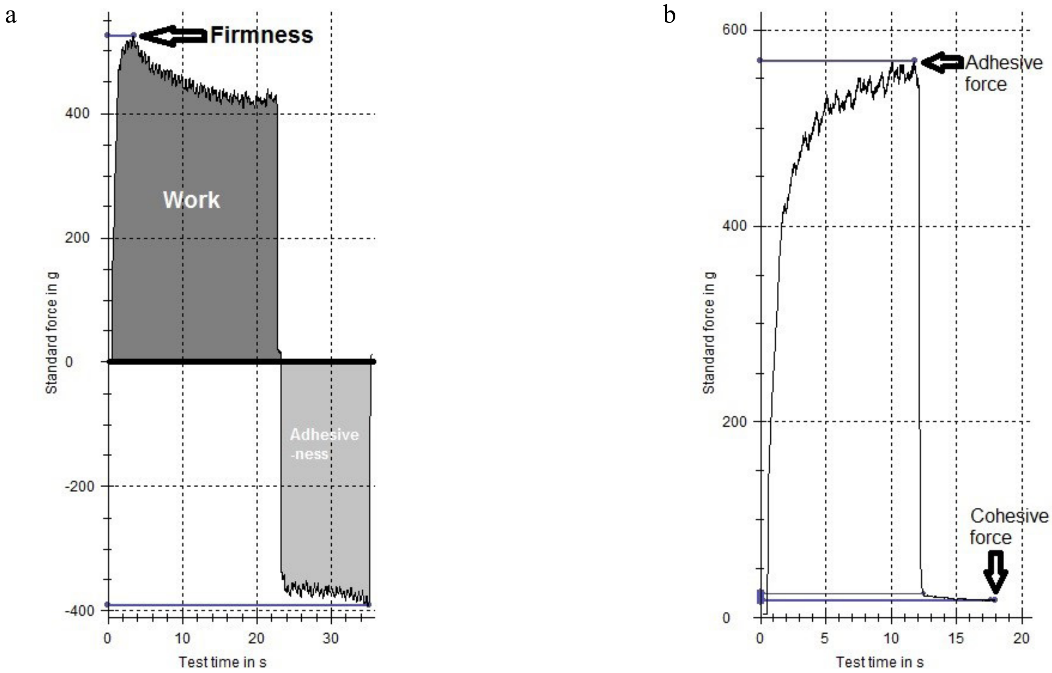

Fig. 1. Back extrusion test results (a), adhesion test results (b).

Table 1. Descriptive statistics of texture parameters of raw rapeseed honey depending on storage conditions (mean value ± s.d., min-max)

Specification	CON n = 7	RT n = 7	FRO n = 7
Dynamic viscosity (Pa s)	33.57^{A} ± 18.63 13.5-67.7	78.36^{AB} ± 13.72 59.7-98.5	288.43^{B} ± 91.24 186.0-403.5
Firmness (g)	402.14^{a} ± 220.96 198.0-830.0	713.43^{a} ± 95.95 521.0-815.0	3022.14^{b} ± 1398.09 945.0-4740.0
Work (mJ)	60.52^{a} ± 46.08 18.3-149.4	128.65^{ab} ± 25.07 94.6-163.2	381.42^{b} ± 221.13 119.4-694.8
Adhesiveness (mJ)	54.59 ± 34.99 27.7-125.2	50.18 ± 26.00 29.0-85.3	100.03 ± 79.36 24.8-268.2
Adhesive force (g)	367.29^{a} ± 222.36 160.0-799.0	564.00^{a} ± 43.31 487.0-615.0	1988.57^{b} ± 775.14 1210.0-3600.0
Cohesive force (g)	16.33^{a} ± 3.18 13.7-21.7	18.67^{a} ± 2.45 14.1-21.4	55.70^{b} ± 19.36 36.7-83.6

Means denoted by different letters in rows differ significantly: $^{a, b}$ $p \leq 0.05$; $^{A, B}$ $p \leq 0.01$. CON – fresh honey, RT – honey stored at room temperature (20-26°C) for 18 months, FRO – honey stored in the freezer (at -20°C) for 18 months.

should be noted that all of the textural properties of honey (except for adhesiveness) stored at RT were significantly ($p \leq 0.05$) lower than those for frozen samples (FRO). Soria *et al*. (2004) observed a wide range of textural properties for 46 artisanal Spanish honey samples. The firmness and adherence (stickiness) ranged from 24 to 213 g and from 23 to 264 g, respectively. The given ranges for firmness and adherence are lower than those obtained in the present study; however, the temperature at which the analysis was conducted is unknown. Lazaridou *et al.* (2004) reported that the apparent viscosity of thirty types of Greek honeys at 20°C ranged widely *i.e.* from 9.9 to 200.0 Pa s. Although these differences resulted from natural variations in individual sugar and water contents, all of the honeys showed Newtonian behaviour. Polish honeys are usually reported to be Newtonian liquids (Juszczak and Fortuna, 2006). In contrast, Dobre *et al*. (2012) reported non-Newtonian flow for rapeseed honey from Romania, which was additionally correlated with a high concentration of carbohydrates (78.3%). Similarly, other authors reported a typical crystalline structure for rape honey from Germany and Lithuania and, simultaneously, a non-Newtonian semi-solid behaviour (Smanalieva and Senge, 2009; Stelmakienė *et al*., 2012). Bakier (2006) reported dynamic viscosities ranging between 11.4 and 16.9 Pa s for Polish rape honey measured between 19.85 and 21.85°C. In the present study, the range of this parameter for honey samples stored at room temperature was higher, *i.e.* between 13.5 and 67.7 Pa s. It should also be noted that the samples of rape honey measured by Bakier (2006) at 1.85°C showed that the viscosity ranged from 261.6 to 317.7 Pa s, and that this range was within the values obtained in this study for FRO samples (186.0-403.5 Pa s). In turn, the apparent viscosity of Persian honeys at 10°C ranged from 120 to 150 Pa, while at 20°C they dropped between 20 and 25 Pa s (Tavakolipour and Kalbasi, 2010). According to Bakier (2017), temperature changes in the lower ranges had a greater influence over the rheological properties of honey. It is interesting that for a temperature below 0°C, all types of honey show a high viscosity exceeding 1000 Pa s.

The essential characteristic which distinguishes rapeseed honey from other types is the predominance of a number of small, oval crystals with maximum diameter <10 μm (Bakier *et al*., 2016). Therefore, it undergoes granulation relatively rapidly. Furthermore, it is in this context that significantly high concentrations of glucose and reducing sugars (approx. 40.5 and 80%, respectively) occur along with an almost equal glucose/fructose ratio (Devillers *et al*., 2004). Oroian *et al.* (2016) found that the textural parameters were negatively influenced by the sum of the fructose and glucose and the ratio of these two compounds. According to White (1974), honey granulation is enhanced at temperatures from 13 to 15.5°C, but this process is retarded at temperatures below 0°C. However, Conforti *et al*. (2006) point out that factors, which regulate the crystallization of honey at room temperature conditions produce a different result during refrigerated storage. Lupano (1997) reported lower values of crystal melting enthalpies for honey in a frozen state at -20°C compared to samples stored at 20°C. This implies different values of enthalpy in diverse crystals. On the one hand, coarse crystals formed in honey kept at 20°C had melting temperatures (Tm) from 45 to 65°C and on the other hand, fine-grained particles present in the frozen honey at -20°C had Tm ranging from 25 to 45°C. Therefore, samples of FRO honey demonstrated higher values of viscosity and other textural parameters determined by fine-grained crystals.

There is usually an inverse dependence between viscosity and temperature. As expected, the dynamic viscosity decreases substantially with an increase in temperature (Table 2). The mean value of the dynamic viscosity at 20°C (33.57 Pa s) was substantially altered by temperature increases up to 30°C (8.24 Pa s) at 40 and 50°C (2.49 and 1.44 Pa s, respectively). The influence of temperature was very noticeable up to 30°C, and was not significant above this level. The mean values of firmness, work, adhesiveness, adhesive force and cohesive force of the unheated (at 20°C) rape honey samples (CON) obtained in the present study were 402.14 g, 60.52 mJ, 54.59 mJ, 367.29 g, and 16.33 g, respectively (Table 2). The thermal treatment of honey heated at 30, 40 and 50°C significantly influenced ($p \leq 0.05$ or $p \leq 0.01$) a continuous decrease in all textural properties (with the exception of the cohesive force) at a given temperature compared with CON samples. However, the effect of heating on textural properties was observed only for temperatures up to 30°C, which was observed in the viscosity values. Above this level, the differences were insignificant.

The apparent or dynamic viscosity of honey significantly depends on several factors including its water content, temperature and chemical composition (Sopade *et al*., 2002; Yanniotis *et al.*, 2006). The 1% change in water content has the same effect on the viscosity of honey as a 3.5°C temperature change (Juszczak and Fortuna, 2006). Apart from water content and its effects on composition, temperature

Table 2. Textural properties of raw rapeseed honey depending on temperature (mean value ± s.d., min-max)

Specification	Temperature			
	20°C/CON	30°C	40°C	50°C
Dynamic viscosity (Pa s)	$33.57^{c} \pm 18.63$ 13.5-67.7	$8.24^{bc} \pm 3.68$ 4.4-14.2	$2.49^{ab} \pm 1.17$ 1.5-4.4	$1.44^{a} \pm 0.51$ 1.1-2.3
Firmness (g)	$402.14^{c} \pm 220.96$ 198.0-830.0	$146.94^{bc} \pm 59.50$ 90.4-254.0	$94.19^{ab} \pm 19.69$ 75.1-131.0	$64.74^{a} \pm 16.45$ 49.9-93.0
Work (mJ)	$60.52^{B} \pm 46.08$ 18.3-149.4	$1.52^{AB} \pm 0.61$ 0.8-2.4	$0.85^{A} \pm 0.21$ 0.6-1.3	$0.67^{A} \pm 0.26$ 0.4-1.0
Adhesiveness (mJ)	$54.59^{B} \pm 34.99$ 27.7-125.2	$15.00^{AB} \pm 7.41$ 8.7-28.9	$5.65^{A} \pm 3.04$ 1.63-10.4	$1.77^{A} \pm 1.09$ 0.7-3.4
Adhesive force (g)	$367.29^{c} \pm 222.36$ 160.0-799.0	$100.80^{bc} \pm 48.37$ 54.9-188.0	$45.19^{ab} \pm 18.77$ 17.6-75.0	$26.51^{a} \pm 6.15$ 19.9-37.2
Cohesive force (g)	16.33±3.18 13.7-21.7	14.94±2.22 13.1-19.4	14.93±0.86 14.1-16.4	14.16±0.31 13.8-14.7

Means denoted by different letters in rows differ significantly: $^{a, b, c}$ $p \leq 0.05$; $^{A, B}$ $p \leq 0.01$.

has a major role in changing the viscosity of honey (Tavakolipour and Kalbasi, 2009). Increasing the temperature of the honey from 298 to 308 K caused a decrease in viscosity from 12.95 to 5.52 Pa s, which is a decrease of over 57% (Bakier, 2017). For rapeseed honey, which contained 18% moisture Juszczak and Fortuna (2006) reported a decrease in viscosity from 20.9 to 1.8 Pa s with temperature increases from 20 to 40°C. In addition, the presented viscosity results are comparable with those obtained by Dobre *et al*. (2012) at 10-40°C, Tavakolipour and Kalbasi (2009) at 20-30°C, Belay *et al*. (2017) at 30-40°C, Yanniotis *et al*. (2006) at 25-45°C, and Oroian (2013) at 20-50°C for different honey types. In contrast, Indian honeys exhibited lower viscosity values at 20°C (between 4.93 and 23.00 Pa s) and 30°C (between 1.31 and 5.15 Pa s) (Saravana Kumar and Mandal, 2009) compared to the present results and those cited above. The greatest diversity in viscosity was observed for the samples measured at 20°C, whereas at higher temperatures, the variability became gradually smaller, and the lowest values were observed at 50°C. Similarly, Belay *et al*. (2017) studied nine Ethiopian monofloral honeys and observed the highest viscosity variability at 25°C. The measurement results in a dynamic rheological test of liquid honeys are similar to the rotational measurements. The values of the complex viscosity of the analysed media are similar to the values of the dynamic viscosity for instance, the relative differences between the average values of dynamic viscosity and complex viscosity for rape honey was 3.2% (Bakier *et al*., 2016).

Stelmakienė *et al*. (2012) reported significant differences in the rheological properties (thixotropy and consistency index) of Lithuanian commercial rapeseed honey, between the temperatures of 30 and 40°C, as well as between 30 and 50°C, contrary to present results. Interestingly, on the one hand, Lithuanian rapeseed honey exhibited at 30°C the highest consistency coefficient compared to other honey varieties, and it also showed elastic properties. On the other hand, at temperatures of 40 and 50°C it demonstrated pseudoplastic substance properties (Stelmakienė *et al*., 2012). At the highest temperature (50°C) used in the present study, the average values of the investigated textural properties were 64.74 g, 0.67 mJ, 26.51 g, and 14.16 g, respectively. These results are in agreement with those reported by Oroian (2013), who stated that temperature (at 20-50°C) negatively influenced the rheological characteristics of honey such as density, viscosity, surface tension and ultrasonic velocity due to decreasing intermolecular forces in the honey.

The temperature influence on the texture measurements between 20 and 50°C as expressed by the texture-temperature coefficient (T-TC, %/°C) is shown in Fig. 2a-f. It assumes linearity between the percentage texture changes per degree temperature change. For instance, the T–TC value is equal to -2.5%/°C for water at 20°C, which means that the viscosity at 21°C is 2.5% less than that at 20°C. For foods in which this relationship is not linear, this definition may still be used if the temperature range is narrowed to an approximately linear segment and the temperature range over which T-TC applies is specified (Bourne, 1982). For all texture parameters (with the exception of the cohesive force, Fig. 2e), the T-TC values decrease as the temperature increases. The minus sign means that T-TC is inversely related to the temperature level at which the measurement was made. The average change in T-TC for dynamic viscosity, firmness, work, adhesiveness and adhesive force (Fig. 2a, b, c, d and e, respectively) between 20, and 50°C was similar and varied from -6.62% to -4.09%. The biggest relative change in T-TC ranged from -9.67% (for work, Fig. 2c) to -6.01% (for firmness, Fig. 2b) and was noted to lie between 20 and 30°C. It gradually decreased at higher temperature ranges i.e. from -6.90% (for dynamic viscosity, Fig. 2a) to -3.15% (for firmness, Fig. 2b) between 30 and 40°C and from -6.66% (for adhesiveness, Fig. 2d) to -2.26% (for work, Fig. 2c) between 40 and 50°C. The obtained results are in agreement with the literature data indicating that the rheological behaviour of honey is closely linked to the crystalline structure and accelerating process of honey crystals melting at higher temperatures (Chen *et al*., 2009; Smanalieva and Senge, 2008). Only in the case of cohesive force (Fig. 2f) was the T-TC value substantially different (averaging -0.35%, overall range from 0.18% to -0.74%) when compared to other textural parameters.

For all of the studied honey samples (n = 42), significant ($p \leq 0.01$) and positive Spearman's rank correlation coefficients were obtained (Table 3). The highest coefficients ($r_s \geq 0.916$) were found between dynamic viscosity, firmness, work, adhesiveness and adhesive force. Lower correlations ($0.524 \leq r_s \leq 0.684$) were observed between cohesive force and all of the other properties.

Stelmakienė *et al*. (2012) assessed the rheological characteristics of Lithuanian honey and found very close and significant correlations between thixotropy and the index of flow consistency (direct), and between thixotropy and the flow behaviour index (inverse), however, no detailed data were provided by authors. Oroian *et al*. (2016) reported that all studied typical TPA textural properties (hardness, adhesion, cohesiveness, springiness, gumminess, chewiness) and the viscosity of Romanian honeys were substantially positively influenced by one another ($p < 0.001$), and that the correlation coefficients were between 0.815 and 0.999. Similar and very close relationships for viscosity and other TPA parameters were also reported by Oroian *et al*. (2017) in subsequent investigations. Admittedly, these values were higher in comparison with correlations obtained in the presented study, which may be caused by the application of different methodologies of textural measurements (TPA, back extrusion) and testing devices. In contrast to Oroian *et al.* (2016) and as a result of the present study, Conforti *et al*. (2006) reported for Argentinian honey a linear inverse dependence ($R^2 = 0.94$) between adhesivity and firmness.

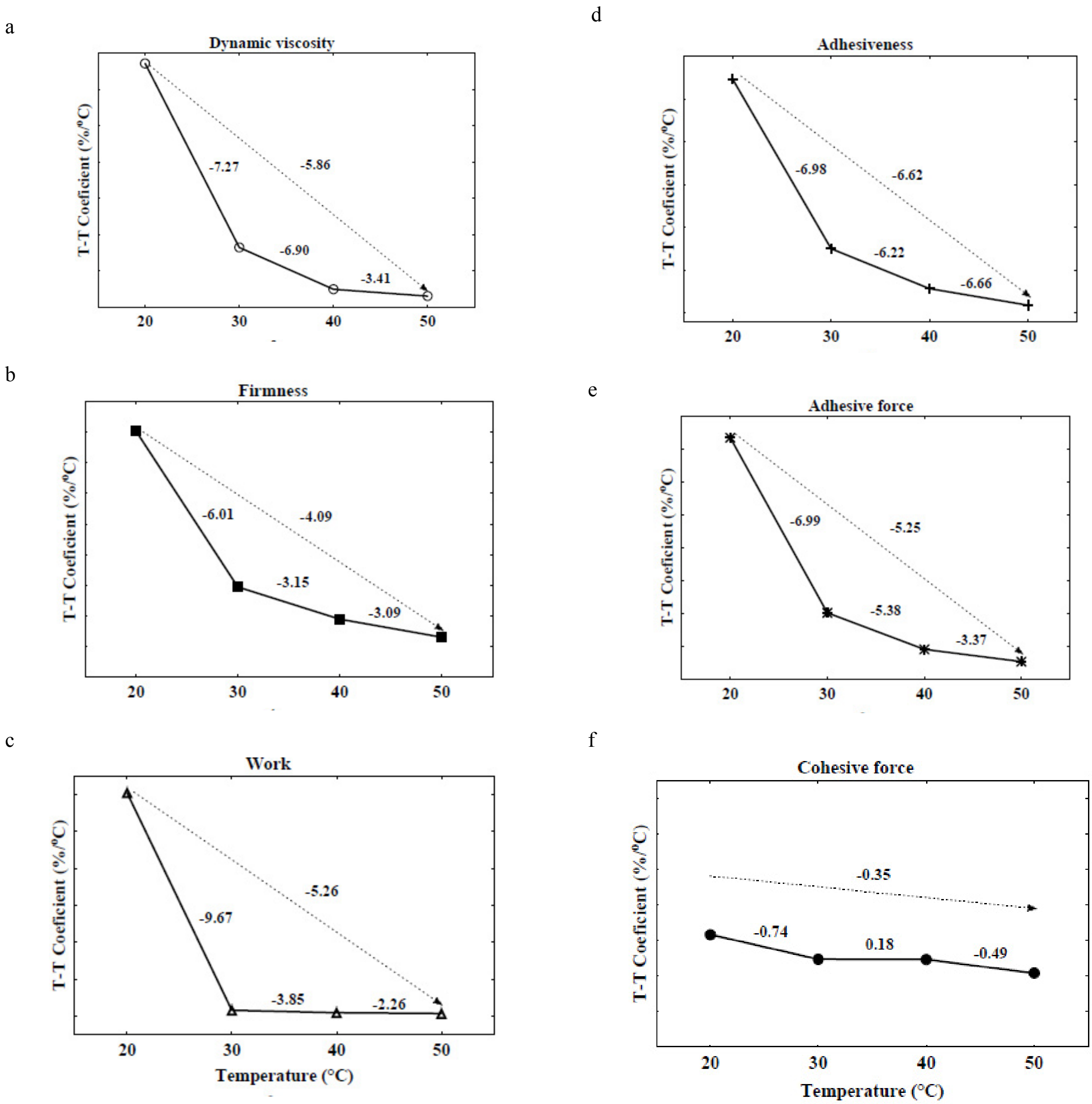

Fig. 2. Texture-temperature coefficient (T-TC) for dynamic viscosity (a), firmness (b), work (c), adhesiveness (d), adhesive force (e) and cohesive force (f) of rapeseed honey samples at 20, 30, 40 and 50°C.

Table 3. Spearman's rank correlation coefficients (r_S) of dynamic viscosity and textural properties of rapeseed honey (n = 42)

Parameter	Firmness	Work	Adhesiveness	Adhesive force	Cohesive force
Dynamic viscosity	0.975	0.952	0.916	0.980	0.668
Firmness	–	0.968	0.921	0.987	0.684
Work		–	0.894	0.954	0.643
Adhesiveness			–	0.936	0.524
Adhesive force				–	0.667

All correlation coefficients significant at $p \leq 0.01$.

CONCLUSIONS

In the present study, raw rapeseed honey was investigated for textural behaviour after storage at different temperatures and heating.

1. After freezing the honey had a significantly and sometimes several times higher dynamic viscosity, firmness, work, adhesiveness, adhesive force and cohesive force values in comparison to fresh samples and those stored at room temperature. Honey kept at room temperature also had higher (nearly twice) the dynamic viscosity values, firmness, work and adhesive force in comparison with fresh samples.

2. The effect of heating (between 20 and 50°C) worsened all of the textural properties of the honey (with the exception of the cohesive force), there were significant changes up to 30°C.

3. The results obtained with use of a back extrusion rig confirmed that the dynamic viscosity used to describe the rheological behaviour of the investigated rapeseed honey (determined by the back extrusion rig) had similar values to those cited in the literature, which referred to viscosity verified by rheometric measurements. Furthermore, the viscosity was positively and significantly influenced by all of the examined textural traits of the honey. Additionally, a greater effectiveness and reliability could be anticipated by optimizing the experimental conditions. However, supplementary investigations concerning honey texture parameters are required.

Conflict of interest: The Authors declare they have no conflict of interest.

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