STUDY OF VACUUM AND FREEZE DRYING OF BEE HONEY

by

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The aim of this research is to study the drying kinetics of vacuum-dried and freeze-dried bee honey produced from two different varieties: Sunflower honey (Helianthus Annuus L.) and Acacia honey (Robinia pseudo acacia L.). Vacuum drying treatments were carried out with the honey samples' initial temperatures of +25 °C, –20 °C, and –40 °C. Water content, total soluble solids, as well as the water activity of fresh and dried honey samples were determined. Freeze-drying of bee honey with initial sample temperature of –40°C has resulted in shorter drying time (7-9 hours), moisture content (10%-12%), water activity (0.405-0.427 aW) and effective moisture diffusivity coefficient ($8.26 \cdot 10^{-7}$-$9.51 \cdot 10^{-7}$ m^2/s). The high-performance liquid chromatography method was used when analyzing the impact that drying pre-treatments had on honey quality. The application of pre-treatments has led to an increase in hydroxy-methyl-furfural by 39-71%, and a decrease in diastase activity by 17-36%, all compared to fresh honey samples. The solutions of Verma model proved to be the best fit with the experimental results.

Key words: *vacuum drying, freeze drying, bee honey, drying kinetics, quality parameters*

Introduction

Honey can be defined as a natural, sweet substance produced by honey bees (Apismellifera) either through the process of plant nectar transformation, from secretions of plants' living parts or by collecting the excreta of insects that feed by sucking juices of plants' living parts [1]. Furthermore, bees are collecting this substance, processing it and supplementing with their own specific substances, dehydrating and storing into honey-comb cells where the substance matures [1]. Honey represents the food that consumers associate with nature and ecology, appreciate for its complex content and consider a functional food [2, 3]. In the food industry, there are different liquid honey substitutes such as honey powder [4], granulated honey, and honey flakes, all obtained through various drying techniques [5]. Some native honey compounds are thermo-labile and therefore the ultimate quality depends on storage conditions and processing [6]. Thermal processing has significant impact on the antibacterial activity of multifloral honey [7]. Honey is a mixture of many types of sugars, most of which are glucose and fructose, with a glass-transition temperature that prevents spray drying [8-10]. Addition of

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carrier materials can help when drying products such as honey. If water concentration in the product is low, all water remains in an unfrozen state. All the present water is greatly influenced by sugars and organic acid constituents that exist in these food systems and are preventing ice crystallization [11]. In order to reduce the moisture content in honey, a novel method based on passive diffusion and monitoring of subsequent changes in several honey properties was used by [12]. The amount of hydroxyl-methyl-furfural (HMF) and diastasis activity are parameters determing the honey quality and are indicators of its freshness and thermal treatment. Fresh honey has a very low amount of HMF and, on the other hand, high diastase content [13]. The HMF is a cyclic aldehyde formed during the process of sugar dehydration that occurs when warming the honey or storing it for longer time. In very concentrated solutions, with almost no free water, heat supply can influence the redistribution of energy in a way that it becomes the activation energy for potential loss and chemically bound water. Consequently, cyclic aldehydes such as HMF are generated from sugars [14]. Diastase represents an enzyme that is naturally present in honey andthat breaks down during the heating process [15]. According to the EC-Council directive [16] the permitted amount of HMF in general, with the exception of baker's honey, should not exceed 40 mg/kg, while diastase value (Schade scale) should be at least 8 [17]. Within the article [18], an examination of the microwave-vacuum drying (VD) technic for dry honey preparation was performed. Samples were heated and dehydrated in a micro-wave dryer until achieving the moisture content below 2.5%. During the process of drying, the core temperatures of the sample were equal to the surface temperatures. The sample temperature was varied from 30 °C to 50 °C which resulted in a slight change of the content of main sugars (fructose, glucose, maltose, and sucrose) as well as alcohols and esters. In a modern food production industry, the processing of raw material usually requires some sort of drying process [19, 20]. Therefore, research and analysis focused on the drying process are essential when ensuring further improvements. The typical drying process performed at an ambient pressure implies heating the food material bellow 100 °C. This state can be defined as state F (see process A-F on fig.1). In spite of numerous researches conducted in this area, a precise mathematical modeling of the biological material drying process remains complex. Most modeling methods based on the concept of the moisture diffusion through the material describe how the average moisture content of the material is changed over the time [21]. The VD (see process A-E on fig. 1) is the mass transfer operation in which the moisture present in a substance is removed by means of creating a vacuum. State E can be achieved as a result of simultaneous processes E' (vacuuming) and E'' (heating). Drying presents an essential unit operation of moisture removal within the chemical processes preformed in various industries such as food, pharmaceutical, agricultural, textile and paper [22]. The selection of VD temperature has a significant effect on drying kinetics, drying rates, effective moisture diffusivity and quality parameters [23]. The VD is generally used for the purpose of drying substances that are hygroscopic and heat sensitive. Moreover, VD is based on the principle of creating a vacuum in order to decrease the pressure to a value below the water's vapor pressure. Vacuum pumps reduce the pressure of the substance to be dried (approximately 0.0296-0.059 bar), which consequently decreases the boiling temperature of water inside that substance (corresponding boiling temperature of water is 25-30 °C). Accordingly, the evaporation rate is significantly increased. The result is a naturally increased drying rate of the product [24]. During the freeze drying – FD (see process A-B-C-D on fig. 1), the heat of sublimation is supplied by conduction and radiation. After the sublimation process, the temperature of drying chamber increases, and the processes that follow take place purely under the vacuum. The sublimation of pure ice is possible only below the water's triple point (approximately 611.2 Pa, 0.01 °C). However, food contains solid matter and

requires drying at lower temperatures (generally below –10 °C or lower) and at the absolute pressure below 270 Pa [25]. During freezing, depending on the composition of the food product, 65-90% of initial water is in the frozen state, while the rest 10-5% is in the unfrozen (sorbed) state. The rate and amount of ice formation during freezing is dependent on the composition and viscosity of unfrozen fraction [26].

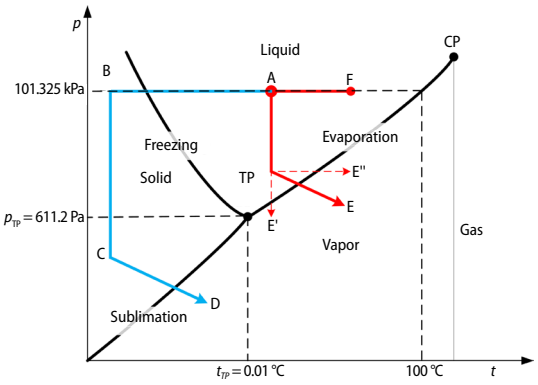

Figure 1. Drying process presented on water *p-T* diagram

Materials and methods

Two honey varieties were analyzed: Sunflower honey (*Heliantusannuus* L.) and Acacia honey (*Robiniapseudoacaia* L.). Sunflower honey was soft crystallized, with distinctive intense yellow color. The odor was medium intense, resembling the vegetal scent and the smell of aged wax. The taste of the sunflower honey was medium sweet, not particularly characteristic and medium persistent. The acacia honey was in a liquid state, with a very light yellow color. The odor was weak and resembled of acacia blossom scent. The taste was very sweet, refreshing and a bit like vanilla. The aroma was weakly persistent.

Experimental apparatus and measurement accuracy

Total soluble solids (TSS) is a measure of dissolved solids in the honey samples. The honey samples with TSS amount higher than 80% can be considered of high grade and highly stable upon storage. On the other hand, honey with less than 80% soluble solids is likely to ferment during storage [27]. The TSS were determined as described by ISO 2173:2003. The amount of solids in a liquid is measured on the Brix scale. Each degree of Brix equals 1 percent sugar. Hand held honey refractometer instrument with ranges of 50° Brix to 90° Brix was first standardized. The instrument was calibrated with deionized water before the measurements (brand name: YIERYI; model: THE01533B; measurement accuracy: 0.1% water; range 10-30% water).

The water content is a limiting factor which determines the ability of honey to retain its quality during storage and its resistance to fermentation. The water content is practically opposite to the Brix value. According to Composition Criteria of Honey [1], the water content should not be higher than 20%. Water content was determined by refractometer, measuring the refractive index (RI) according to harmonized methods of the International Honey Commission (IHC) methods [28], using a standard model Abbetyperefractometer at 20 °C. Water content [%] was then obtained from the Chataway table [29, 30]. The container of honey from which the sample comes was well-mixed. Multiple readings were taken in order to eliminate the possibility of error appearance. Similar approach was retained by article [31].

The water activity in honey is very important since it affects its sensorial quality, microbiological stability, physical characteristics and shelf life [32]. The water activity (aW) represents the amount of water available to microorganisms [33]. According to Composition Criteria of Honey [1], the limit of aW < 0.60 is taken as an indicator of a microbiologically stable food. Testo 650 water activity system (accuracy: ±0.001 aW, range is 0 to +1 aW) was used to provide accurate and repeatable water activity information on chemically non-bonded water.

During the processing and preparation of honey for freeze drying (FD), heating is frequently used for decreasing viscosity or for melting crystallized honey which causes troubles in fractioning and packaging. Heating of honey motivates the loss of thermo-labile, aromatic substances. Losses are proportional to the temperature and heating time. Damages caused by heating can be evidenced by measuring quality control parameters, such as diastase activity and HMF content [28]. These properties are used together as their values are indicative of the heating intensity to which honey has been subjected. According to Composition Criteria of Honey [1], the maximum limit for HMF in honey is 40 mg/kg and the diastase activity number should be not less than 8.

The HMF is determined in a clear, filtered, aqueous honey solution using reverse phase high-performance liquid chromatography (HPLC) equipped with ultraviolet detection. The signal is compared with those from standards of known concentration. Determination of HMF content was measured according to the HPLC method (IHC method, method 5) [28]. Honey samples were diluted up to 5 g in 50 ml of distilled water, filtered on 0.45 lm filter and immediately injected in a HPLC (Waters 1525 Binary HPLC Pump) equipped with a Diode Array Detector (Waters 2487 Dual Absorbance Detector). The HPLC column was a Merck Lichrospher, RP18, 5 lm, 125·4 mm, fitted with a guard cartridge packed with the same stationary phase (Merck). The HPLC conditions were the following: isocratic mobile phase, 90% water at 1% of acetic acid and 10% methanol; flow rate, 0.7 ml per minute; injection volume, 20 µl. All the solvents were HPLC grade (Merck). The wavelength range was 220-660 nm and the chromatograms were monitored at 285 nm. The HMF was identified by splitting the peak in honey with a standard HMF (Alfa Aser LOT 10183841), and by comparison the spectrum of HMF standard with that of honey samples. The amount of HMF was determined using an external calibration curve, measuring the signal at $\lambda = 285$ nm.

Diastase activity was determined by method after Schade [28]. The unit of diastase activity is defined as that amount of enzyme which will convert 0.01 gram of starch to the prescribed end-point in one hour at 40 °C under the conditions of test. The diastase activity is calculated as diastase number (DN). A standard solution of starch, capable of developing, with iodine, a color in a defined range of intensity, is acted upon by the enzyme in the sample under standard conditions. The diminution in the blue color is measured by spectrophotometer (Shimadzu UV-1800) at intervals.

In order to confirm the botanical origin of the honey samples, a melisopalinological analysis was performed according to the method described by [34]. Based on the melisopalinological analysis and the, at least 500, pollen grains counted using the microscope (400× magnification), it was determined that the acacia honey sample contained 31.5% of Robinia acacia grains, 17.0% of Salix, 13.2% of Rosaceae, 11.9% of Fabaceae type, 11.3% of Prunus, 4.3% of Amorphafruticosa, 2% of Gleditsia sp., 2.9% of Brassicaceae, 0.9% of Cornus mas, 0.7% Poaceae and 4.3% were no identified pollen grains. From the microscope slide prepared from sunflower honey different pollen grains were determined: Helianthus annuus (81.2%), Salix (4.1%), Tilia (2.9%), Taraxacumofficinale (5.3%), Rosaceae (4.2%), Ambrosia type (1.2%) and 1.1% were no identified pollen grains. According to the Serbian Rulebook on quality of honey and other bee products [1] for monofloral acacia honey it was prescribed that the minimum proportion of pollen grains in the insoluble part has to be 20% or for monofloral sunflower honey has to be a minimum of 40%. Samples of honey comply with the prescribed minimum for classification into monofloral honey types.

LabconcoFreeZone® 18 FD system was used for laboratory lyophilization procedures. The samples were kept in deep freezer shell at –40 °C before FD process. During the FD,

the collector temperature was maintained at –40 °C and the chamber pressure condition was vacuum less than 0.133 mbar. The mass change of the samples was detected with load cell data acquisition system (accuracy of ±0.1 g) that was placed within vacuum chamber.

Experimental design

Bee honey was stored in glass jars at the ambient temperature of +25 °C until the start of the experiment. When preparing the sample for VD at the ambient temperature, honey was placed in a shallow bowl and then put into the vacuum chamber, fig. 2(a).

The sample size was between 30 g and 50 g per experiment. In case of the FD process, honey was heated up to 40 °C so that it could spill easier on the inner walls of a deep glass jar. Subsequently, the glass jar with honey was placed onto rotating rollers in shell freezer chamber, fig. 2(b), and cooled down to –40 °C for approximately six hours. Immediately after this period of time the sample was moved into the chamber for lyophilization.

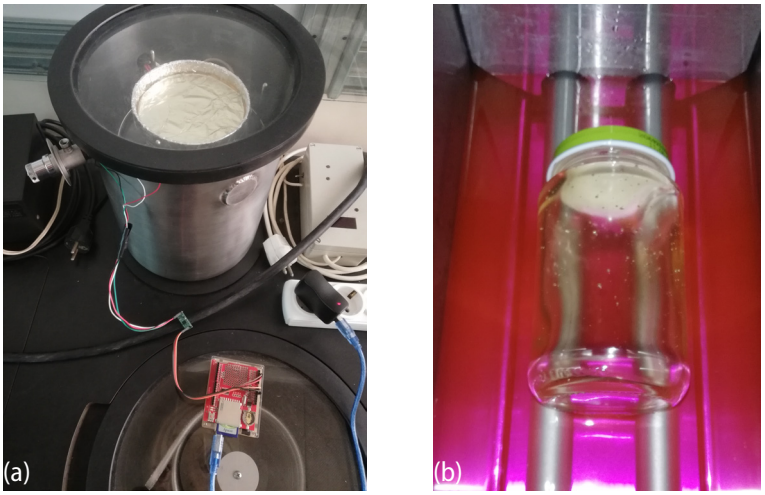

Figure 2. Experimental apparatus: (a) vacuum chamber, (b) shell freezer chamber

Drying models

The estimation of the effective diffusivity coefficient during the drying process of various food materials is very common in literature. The effect of initial sample material temperature for selected thin layer of dried honey on the moisture diffusion was investigated. Fick's Second law of unsteady state diffusion, eq. (1), was used to interpret the drying process since moisture diffusion is one of the main mass transport mechanisms that describe this process:

$$\frac{\partial M}{\partial \tau} = D_{\text{eff}} \nabla^2 M = D_{\text{eff}} \left(\frac{\partial^2 M}{\partial x^2} + \frac{\partial^2 M}{\partial y^2} + \frac{\partial^2 M}{\partial z^2} \right) \tag{1}$$

In the case of symmetric boundary conditions, with neglecting of material shrinkage and with the assumption that water distribution in thin layer material (approximately 2.5 mm) is homogeneous, the moisture ratio (MR) can be determined [35]:

$$MR = \frac{M - M_{\text{e}}}{M_0 - M_{\text{e}}} = \frac{8}{\pi^2} \exp\left(-\frac{\pi^2 D_{\text{eff}} \tau}{4 z^2} \right) \tag{2}$$

Taking into account the change of MR with drying time, eq. (2) can be solved only numerically. In this case, a linear correlation between the natural logarithm of MR and time is obtained and can be used:

$$\ln MR = \ln \frac{8}{\pi^2} + \left(-\frac{\pi^2 D_{\text{eff}}}{4z^2} \right) \tau \tag{3}$$

A plot of gives a straight line with a slope that is used to determine [35] effective moisture diffusivity according to:

$$\text{Slope} = \frac{\pi^2 D_{\text{eff}}}{4z^2} \tag{4}$$

Results and discussion

Heating the honey during processing and preparation, results in certain damages. These deteriorations can be noticed when measuring quality control parameters, tab. 1. Furthermore, tab. 1 also provides the comparison to the composition criteria for honey.

Table 1. Bee honey sample properties

Vacuum drying, Freeze drying (lyophilization)							
Type of honey	State of sample	Hand held honey refractometer		Testo 650 System		IHC Methods, 2009	
		TSS, [°Brix]	Water, [%]	Water activity, [aW]	Temperature, [°C]	HMF, [mgkg^{-1}]	Diastase activity, DN
Composition criteria for honey [1]		Not less than 80	Not more than 20	Not more than 0.60	Not more than 45	Not more than 40	Not less than 8
Sunflower honey	Fresh	80.5	17.9	0.558	23.4	38	19.2
	VD	89.5	10.5	0.405	21.1	–	–
	FD	88.9	10.1	0.425	22.3	65	12.1
Acacia honey	Fresh	82.5	17.5	0.598	23.4	16.5	19.2
	VD	88.3	11.7	0.417	22.4	-	-
	FD	88.5	11.5	0.427	22.6	23	15.9

The TSS content of honey had ranged from 80.5 to 89.5° Brix for both fresh and dried samples. For all the honey samples, TSS were generally above 80% and, thus, they can be considered of high grade and as highly stable upon storage. The highest TSS content was observed in Sunflower honey. The highest value of 89.5° Brix was detected within VD samples and is followed by FD samples that had 88.9° Brix. The average increase of TSS during the drying processes was 9% for VD and 8.8% for FD. Similar results were obtained by [36]. The water content for different honey types varied from 17.5-17.9% for fresh honey, to 10.1-11.7% when observing dried samples. All of the honey samples have fulfilled the composition criteria for honey that limits the water content to a maximum of 20% [1]. Water activity in fresh honey samples was in the range from 0.558 to 0.598. Therefore, it can be concluded that the fresh honey aW value is lower than 0.60 and that it meets composition criteria. However, VD and FD have additionally reduced the water activity by approximately 28%, making the honey microbiologically more stable and with no danger of fermentation. Within VD and FD samples the water activity was in the range from 0.405 to 0.427.

The analysis of HMF and DN change was performed on the initial honey samples as well as after the thermal treatment. Due to the fact that VD is implemented without any thermal

pretreatment, the HMF content after VD will not be changed. On the other hand, the HMF value after performing the FD is implying that a significant destruction has occurred as a result of heating. The HMF content of FD honey samples was 39-71% higher than the one in the fresh sample. The initial high values of HMF of 38 mg/kg for Sunflower honey fresh samples, have led to the composition criteria violation since HMF for FD is 65 mg/kg. On the other hand, HMF content of Acacia honey FD samples was also increased but remained within the allowed limits (below 40 mg/kg) and met the composition criteria. Diastase activity is a honey quality parameter used to determine if honey has been extensively heated during processing. During the thermal pretreatments for FD, DN was reduced by 17-36%. Nevertheless, it had remained significantly above the allowed limit of 8% and met the composition criteria. Similar diastase sensitivity during heat treatment was obtained by [35].

The visual comparison of dried samples shows the difference between VD and FD processes, fig. 3, for Sunflower honey. At the end of the both drying processes, the honey samples achieved the similar density, viscosity and water content. However, due to the creation of water bubbles when boiling water in the vacuum, honey that has undergone the VD had greater mechanical destruction, fig. 3(a). Bubbling is highly undesirable within the later stages of drying process when the honey viscosity gets higher. The honey sample dried with FD process, fig. 3(b), was less disturbed and easier to handle. Similar look of dried product was obtained for Acacia honey.

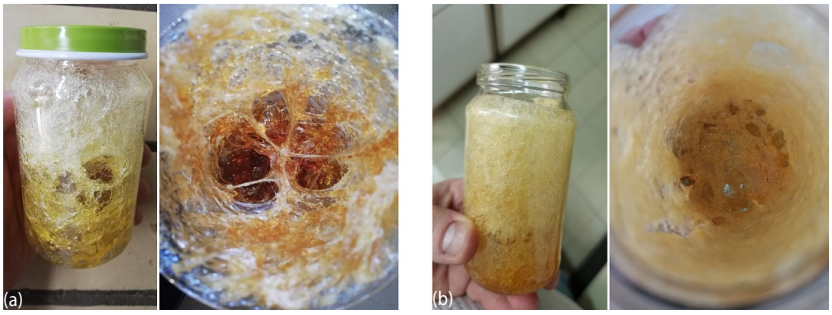

Figure 3. The look of dried Sunflower honey: (a) VD, (b) FD

The initial temperatures of honey samples in experiments were: +25 °C, –20 °C, and –40 °C, fig. 4(a). The drying time of honey samples with initial temperature of +25°C was the longest (approximately 18 hours). Honey samples with initial temperature of –40 °C dried faster (7 to 9 hours). Similar results were obtained for Acacia honey samples, fig. 4(b). Furthermore, similar comparisons of VD and FD method were performed by [37].

At the beginning of drying process the biggest drying rate was detected for the material's initial temperature of +25 °C This occurred as a consequence of material's smaller resistance to inner water diffusion. However, for the honey sample's initial temperature of –40 °C, FD process performed with low intensity (compared with VD), but the overall drying time was shorter. Similar behavior of material can be observed for VD as well as for FD process.

Regardless of the drying method, the drying time of the Sunflower honey was longer, fig. 5. This can be explained through the difference in the structure and variety of honey. Therefore, it can be concluded that Acacia honey is more suitable for drying. For both honey varieties, the first 5 hours of drying had resulted in the highest drying rate. Furthermore, MR values dropped below 0.2 during that period. Thereafter, FD process proved to be more efficient than VD process, and lasted approx. 75% shorter for both honey varieties.

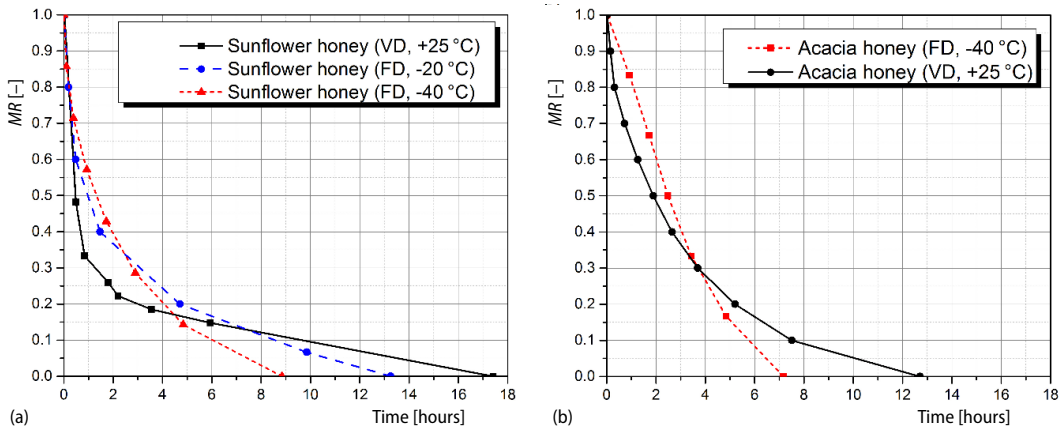

Figure 4. Experimental MR *vs.* drying time: (a) Sunflower, (b) Acacia.

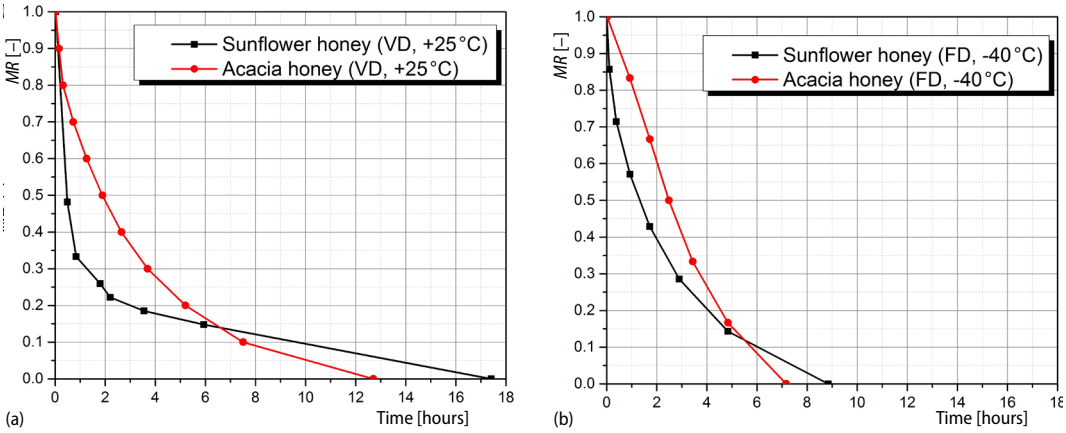

Figure 5. Experimental MR *vs.* drying time: (a) VD, (b) FD

The experimentally obtained results concerning the change of MR in time, during VD and FD process, were compared to the several models commonly used in literature. It was concluded that the solutions of Verma model provide a satisfying match with the results obtained in the experiments. The regression analysis shows that the average value for all experiment's coefficients of determination, tab. 2, calculated for Verma model, is $r^2 = 0.98433$. Since the selected model showed excellent fit with the experimental results there was no need of developing the new model for the MR description within the observed processes [38]. The values

Table 2. Prediction of model coefficients

Experiment		Drying parameters		$MR = a_1\exp(-k_1\tau) + (1 - a_1)\exp(-k_2\tau)$			
				Verma			
No.	Honey	Procedure	T_s, [°C]	a_1	k_1	k_2	r^2
E1	Sunflower	VD	25	0.68856	2.62008	0.14471	0.99192
E2	Sunflower	FD	–40	516.968	0.51774	0.51775	0.95225
E3	Acacia	VD	25	0.85329	0.28417	4.53413	0.99871
E4	Acacia	FD	–40	40.71471	0.64797	0.66301	0.99446

for the bee honey effective moisture diffusivity coefficient, tab. 3, were obtained in the range: D_{eff} =1.05·10^{-7}-2.67·10^{-6} m^2/s for VD, and D_{eff} =2.87·10^{-7}-9.50·10^{-7} m^2/s for FD. The D_{eff} decreases with the increase of FD temperature. Similar results were reported by [39].

Table 3. Effective moisture diffusivity

Experiment		Drying parameters		$D_{eff} \times 10^7$ [m^{2}s^{-1}]	
No.	Honey	Procedure	T_s, [°C]	min	max
E1	Sunflower	VD	25	7.25	26.7
E2	Sunflower	FD	–20	7.52	15.8
E2	Sunflower	FD	–40	8.26	9.50
E3	Acacia	VD	25	1.05	7.06
E4	Acacia	FD	–40	2.87	8.27

Conclusion

Two different bee honey varieties, *i. e.* Sunflower honey (Helianthus Annuus L.) and Acacia honey (Robinia pseudo acacia L.), were experimentally dried using VD and FD methods. Drying kinetics and the efficiency of moisture removal from the material were investigated. Furthermore, the influence of honey processing and preparation for drying were examined by measuring quality control parameters and comparing them to the composition criteria for honey.

The VD treatments were carried out with the honey samples' initial temperatures of +25 °C, –20 °C, and –40 °C. The results confirmed that the freeze-drying process of bee honey with the initial sample temperature of –40 °C has the shortest drying time (7 to 9 hours) despite the reduced intensity of the drying process in the initial period. The Verma model provided to fit the best with the experimental results and had the average value of the coefficients of determination r^2 = 0.98433 for all experiments. The average values of the effective moisture diffusivity were in the range from 8.26·10^{-7} to 9.5·10^{-7} m^2/s. The water content, as well as the content of TSS of fresh and dried honey met the honey composition criteria. The TSS of the dried honeys ranged from 88.3 to 89.5° Brix. Therefore, it can be considered that water content reduction during the drying processes significantly contributes to a higher honey stability upon storage. The VD and freeze-drying reduced the water activity by approximately 28%, making the honey microbiologically more stable and eliminating the possibility of fermentation. The water activity of dried products was in the range from 0.405 to 0.427. The impact that FD pre-treatments had on honey quality was examined using the HPLC method. This was performed by determining the change in HMF (39%-71% increase) and diastase activity (17-36% reduction) compared to the fresh honey samples.

In conclusion, both vacuum and freezedrying processes provided dried product with similar quality parameters and met composition criteria. However, according to the performed observations, a mild preference should be given to the FD. Compared with vacuum dried honey, the freeze-dried honey had well maintained and less damaged structure in the end of the process. Moreover, FD process lasted shorter. On the other hand, thermal preparation of honey prior to the drying process can be a critical point in terms of the unwanted increase of HMF content and diastase activity. Energy efficiency of conducted processes would certainly provide additional information on this matter and will be the subject of future investigations.

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Nomenclature

D_{eff} – effective diffusivity coefficient, [m^2s^{-1}]
MR – moisture ratio, [–]
M – moisture content. [$kgwkg^{-1}d.m.^{-1}$]
r^2 – coefficient of determination
x, y, z – co-ordinates, [m]

Greek symbol

τ – time

Subscripts

e – equilibrium
0 – initial

Acronyms

FD – freeze drying
HMF – hydroxyl-methyl-furfural
HPLC – high-performance liquid chromatography
IHC – international honey commission
RI – refractive index
TSS – total soluble solids
VD – vacuum drying

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