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Thermal loop test to determine structural changes and thermal stability of creamed honey: Rheological characterization

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ABSTRACT

This study was the first attempt to understand if thermal stability of any food product during storage could be determined. In this respect, a novel method, namely, the thermal loop test was used to determine structural changes and thermal stability of creamed honey in this study. The novelty of this method was that thermal stability of a product is tested within a number of thermal cycles over a determined range of temperature. Creamed honey was characterized in terms of physicochemical, thermomechanical and rheological properties. It showed non-Newtonian thixotropic behavior at all temperature levels (10, 25 and 40 °C). Time-dependent flow behavior was successfully defined by Weltman and second order structural models. Hysteresis loop area depended on temperature and decreased with increase in temperature. Creamed honey had liquid-like structure, showing that it had more pronounced viscous nature than elastic nature ($G'' > G'$). Temperature sweep tests were conducted to determine temperature dependency of η_{50}, G' and G'' values using Arrhenius equation. These test results confirmed the thermal stability test results, revealing that thermal loop test can be an accurate method to determine thermal stability of similar food products, as a new information. Relative structural index value (Δ) increased with number of thermal loop, suggesting that creamed honey had low thermal stability and showed a great structural change by the thermal stress applied between 5 °C and 50 °C. These results suggest that crystallized honey be abstained from large temperature fluctuations to avoid from irreversible changes in rheological characters; thus, to maintain spreadability.

1. Introduction

Honey is a natural sweetener produced by honey bees from plant nectars. The predominant compounds found in the honey are carbohydrates mainly composed of fructose and glucose. It also comprises very limited amount of disaccharides, trisaccharides and polysaccharides (Dobre et al., 2012). Proteins, enzymes (invertase, glucose oxidase, catalase, phosphatases, etc.), aminoacids, organic acids (gluconic acid, acetic acid, etc.), lipids, vitamins (ascorbic acid) and phenolic substances (flavonoids and carotenoids) are also compounds present in the honey (Blasa et al., 2006) therefore, consumption of the honey is important for human nutrition.

Some processes such as controlled crystallization are performed to improve sensory and physical properties of the regular (natural) honey such as gaining spreadable character. This product is called as creamed honey. The crystallization process is controlled in order for honey to be spreadable like butter at room temperature (Chen et al., 2009). In other words, honey is crystallized to spread like butter and not to drip. In many countries, this cream form is more preferable than the common liquid form (Chen et al., 2009). Crystallized honey is also named as whipped honey, spun honey, churned honey, honey fondant or creamed honey. Creamed honey includes a large number of small crystals, which, under normal conditions, prevent formation of larger crystals which may form in natural (unprocessed) honey. Storage temperature and fructose/glucose (F/G) ratio are considered as determinants for crystal size formed in the product (Lupano, 1998; Assil et al., 1991).

Rheological properties remarkably influence technological process, involving heating, mixing, filtering, hydraulic transport and bottling, operated to the sample (Yanniotis et al., 2006). Rheological parameters are dependent on some factors such as temperature, shear rate, time, and stress; therefore, these factors are important for designing of the operation. Natural honeys exhibit Newtonian behavior and their rheological properties are strongly influenced by temperature (Yoo, 2004). However, crystallized honeys show Non-Newtonian flow behavior with yield stress and thixotropy (Chen et al., 2009; Smanalieva and Senge, 2009).

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Nomenclature

a_w	water activity	η_{50}	apparent viscosity at 50 s^{-1} (Pa s)
A	area	n'	slope for storage modulus (Pa)
A_0	constant for Arrhenius equation	n''	slope for loss modulus (Pa)
E_a	activation energy (kJ mol^{-1})	n^*	slope for complex modulus (Pa)
f	frequency (Hz)	η^*	complex viscosity (Pa s)
G'	storage modulus (Pa)	R	universal gas constant (kJ mol^{-1} K^{-1})
G''	loss modulus (Pa)	R^2	coefficient of determination
G^*	complex modulus (Pa)	$\tan\delta$	loss tangent (dimensionless)
K	consistency coefficient (Pa s^n)	T	temperature (°C)
K'	intercept for storage modulus (Pa)	$\dot{\gamma}$	shear rate (s^{-1})
K''	intercept for loss modulus (Pa)	η	apparent viscosity (Pa s)
K^*	intercept for complex modulus (Pa)	ω	angular frequency (rad s^{-1})
η_0	initial apparent viscosity at $t = 0$	σ	shear stress (Pa)
n	flow behavior index (dimensionless)		

In storage period, foods are frequently subjected to repeated thermal changes during processing, distribution, handling and storage time even consumption, which affect stability of foods. However, the stability of structure is important for quality of the related product, which will eventually affect consumer acceptance. In the literature (Bengoechea et al., 2010; Smanalieva and Senge, 2009), changes in rheological parameters as a function of temperature have been examined using temperature sweep tests; however, such tests are actually the well-known temperature sweep tests (G' and G'' as a function of time) which cannot provide accurate information on effect of repeated thermal changes, so on thermal stability of foods. Therefore, it is essential to find a way which would have a potential to simulate such thermal fluctuations as well as to accurately determine their effects on rheological properties. Thermal loop test can be proposed to measure thermal stability, in this respect. Thermal loop test is different from classic temperature sweep tests since thermal treatment is applied in more than one thermal cycle. In this test, the samples are subjected to many numbers of thermal cycles from determined initial to final temperature value. Briefly, this test is applied by simulating changes and fluctuations in temperature levels to provide the thermal stress which normally occurs during processing, production, storage and transportation stages.

In the literature, a great number of studies have been reported on steady and dynamic properties of natural honeys; however, no study has appeared so far on overall rheological characterization of creamed honey (steady and dynamic properties along with thixotropy and temperature sweep tests). Furthermore, to the best of our knowledge, no study has attempted so far to determine thermal stability of honey, even that of other food systems using a peltier system rheometer. However, thermal stability is very important for quality of creamed honey in terms of consumer acceptance, so it is essential to find a way by which changes and fluctuations in temperature levels should be simulated to provide the thermal stress which normally occurs during processing, production, storage and transportation stages. In the present study, it was aimed (1) to observe thermal stability of creamed honey using a novel method, namely the thermal loop test that was performed based on repeated thermal cycles, (2) to investigate steady, dynamic and thixotropic properties of creamed honey in detail at different temperature levels.

2. Material and methods

2.1. Materials

Creamed honey samples were procured from a local market in Konya, Turkey.

2.2. Physicochemical analyses

Color was analyzed by using an automatic colorimeter (Konica Minolta brand spectrophotometer model CM-5, Mississauga, ON, Canada). Turbidity was measured using a turbidimeter (HACH, 2100 N, USA) and the results were expressed as NTU (Nephelometric Turbidity Unit). pH was measured with a pH meter (WTW-Inolab, Weilheim, Germany) in a solution of 10% (w/v) honey in distilled water at 25 °C. An Aqualab water activity (a_w) meter (Decagon, Pullman, WA) was used for determination of water activity of the samples at 20 °C. Brix value was determined using an automatic refractometer (Reichert AR 700, USA) at 20 °C. Dry matter content was measured by conventional drying method as described in AOAC method (AOAC, 2000). Ash content was determined by incinerating the samples at 625 °C in a muffle oven (Protherm, Ankara, Turkey). Protein content of the sample was determined using an automatic nitrogen analyzer (Leco FP 528, USA) based on Dumas method. Major sugar (fructose, glucose and sucrose) composition of creamed honey was determined according to the method described by Jahanbin et al. (2012) using HPLC (Agilent 1100, USA) equipped with a refractive index detector (RID).

2.3. Rheological analysis

Steady, dynamic shear and thermal loop measurements for creamed honey samples were performed using stress and strain controlled oscillatory and rotational rheometer (Anton Paar, MCR 302, Austria) equipped with a peltier heating system.

2.3.1. Steady shear properties

Shear rate was controlled in measurements that were performed in the shear rate range of 1–70 s^{-1} at 10 °C and 1–100 s^{-1} at, 25 and 40 °C using a parallel plate configuration (diameter 50 mm, gap 0.5 mm). In this study, at 10 °C, torque capacity of the rheometer was not sufficient to measure stress values at shear rate values higher than 70 s^{-1}; therefore, only at 25 and 40 °C, the shear stress values were measured at the shear rate values up to 100 s^{-1}. Each measurement was repeated three times. The apparent viscosity of the sample was determined as a function of shear rate. The relation between shear rate and shear stress was explained by the Ostwald de Waele model based on the following equation:

$$\sigma = K\dot{\gamma}^n \quad (1)$$

where σ is the shear stress (Pa), K is the consistency coefficient (Pa s^n), $\dot{\gamma}$ is the shear rate (s^{-1}) and n is the flow behavior index (dimensionless).