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Lipid composition and investigation of the physicochemical characteristics of high-oleic sunflower oil at different temperatures

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High-oleic sunflower oil is characterized by high stability, and hence high quality. The aim of the present work is to determine the chemical composition of the lipid fraction (fatty acids, unsaponifiable matter, tocopherols and sterols) and some physicochemical parameters (density, surface tension, dynamic and kinematic viscosity) of high-oleic sunflower oil. The main fatty acids in the investigated oil were: oleic (77.9%), linoleic (11.5%), palmitic (5.0%), and stearic (3.3%). *ß*-Sitosterol (79.7%) and *α*-tocopherol (100%) were the major components in the sterol and tocopherol fractions of the oil. The variation of the physicochemical parameters was followed at seven temperatures (20, 30, 40, 50, 60, 70, and 80 °C). With an increase in temperature, all parameters decreased and a good linear dependence was observed – density (from 0.868 g/ml to 0.847 g/ml), surface tension (from 29.775 mN/m to 12.461 mN/m), dynamic viscosity (from 44.401 mPa/s to 40.067 mPa/s), and kinematic viscosity (from 51.148 mm^2/s to 47.268 mm^2/s). The present results are a basis for future research on the incorporation of high-oleic sunflower oil into food products with improved functional characteristics.

Keywords: high oleic sunflower oil, fatty acids, density, surface tension

INTRODUCTION

Sunflower oil is obtained by extraction or pressing of the sunflower seeds (*Helianthus annuus* L., family Asteraceae). It is an easily mobile liquid with a light yellow color and a specific taste and odor. The main fatty acids were found to be oleic (20-40%) and linoleic acid (46-70%). The oil is mainly used in the food industry and infrequently in cosmetics, medicine and technology [1].

In recent years, through selection, new sunflower hybrids have been developed and the obtained oil is with a high content of oleic acid (60-80%) and low in linoleic acid (18-40%) [1-3]. This oil can be used in various food products [4-6] as well as for the production of biolubricants and biodiesel [4].

In the case of vegetable oils, the determination of their physicochemical parameters, such as density, viscosity and surface tension, measured at different temperatures, are important both for their production and application [7, 8].

Vegetable oils are usually used for frying and cooking of different food products. Elhefian *et al*. [9] established the physicochemical properties such as density, viscosity and acid value of four edible vegetable oils, determined at room temperature before and after frying of potatoes, repeated five times.

The chemical, thermal and viscous characteristics of high-oleic sunflower and olive oils were determined by García-Zapateiro *et al*. [2] using different acid-catalyzed synthesis and reaction times. Maximum viscosity values were obtained for oils prepared using the sulfuric acid-catalyzed method. The temperature dependence of viscosity for all studied high-oleic oils was significantly stronger than for the original oils.

Vegetable oils are promising alternatives to mineral-based lubricants. High-oleic sunflower oil was used to develop new environmentally friendly lubricant formulations. It was blended with polymeric additives, such as ethylene vinyl acetate and styrene-butadiene-styrene copolymers at different concentrations (0.5-5% w/w). Dynamic viscosity and density measurements were performed. The viscosity of high-oleic sunflower oil increased with an increase in polymer concentration [10].

Some vegetable oils such as soybean, olive,

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castor, sunflower and coconut oil were investigated and it was established that the compounds present in them stimulated biosurfactant production. The results showed a decrease in surface tension of the culture medium without oil from 64.54 mN/m to 29.57 mN/m, with a critical micelle dilution CMD(-1) and CMD(-2) from 41.77 mN/m and 68.92 mN/m, respectively. Sunflower oil (with 60% content of linoleic acid) gave the best results (29.75 mN/m) with a CMD(-1) and CMD(-2) of 36.69 mN/m and 51.41 mN/m, respectively. The addition of linoleic acid decreased the surface tension from 53.70 mN/m to 28.39 mN/m, with a CMD(-1) of 29.72 mN/m and CMD(-2) of 37.97 mN/m, which suggested that the linoleic acid is responsible for the increase in biosurfactant production [11].

Density, surface tension and viscosity of five vegetable oils were also experimentally measured. The measurements were performed from 23 ± 1 °C to their respective smoke point at intervals of 20 °C. The density and surface tension linearly decreased with the increasing of temperature, whereas the viscosity decreased exponentially. It was reported that the type of oil influenced the density and viscosity, but did not affect the surface tension [8].

The aim of this study is to determine the lipid composition of sunflower oil with a high content of oleic acid and to monitor the changes in some physicochemical parameters at different temperatures.

MATERIALS AND METHODS

Sunflower oil was purchased from the commercial network of the city Plovdiv, in 2023.

Lipid composition of the oil was determined by the following analysis:

Composition of fatty acids: Gas chromatography (GC) was used for determination of the fatty acid composition of the oil. Briefly, the vegetable oil was pre-esterified with methanol in the presence of sulfuric acid in order to obtain fatty acid methyl esters (FAMEs) [12]. Determination of FAMEs was carried out on Agilent 8860 gas chromatograph (Santa Clara, CA 95051, US) equipped with a capillary column Supelco (SP™ – 2380, Fused Silica (Bellefonte, PA, USA), 30 m × 0.25 mm × 0.25 μm (film thickness)) and a flame ionization detector (FID). For the identification of the FAMEs a standard mixture Supelco, USA (FAME mix 37 components, Supelco, Bellefonte, PA USA) was used.

Content of sterols: unsaponifiables were determined according to ISO standard [13]. The sterols were isolated from the unsaponifiable matter by thin-layer chromatography (TLC) [14] and their total content was determined spectrophotometrically at a wavelength of 597 nm. Individual sterol composition was determined on a HP 5890 gas chromatograph (Santa Clara, CA 95051, US) equipped with DB–5 capillary column (25 m × 0.25 mm) (Santa Clara, CA 95051, US) and FID. Identification was performed by comparing the retention times with those of a standard sterols mixture (Acros Organics, New Jersey, USA) [15].

Content of tocopherols: individual tocopherols were determined by high performance liquid chromatography (HPLC) on a Merck-Hitachi (Merck, Darmstadt, Germany) instrument. The column was Nucleosil Si 50-5 (250 mm × 4 mm). Fluorescence detection was used (excitement at 290 nm and emission at 330 nm). The mobile phase used was *n*-hexane:dioxane, 96:4 (v/v) and the flow rate was set at 1 ml/min [16].

The physicochemical parameters of the oil were determined at seven temperatures (20, 30, 40, 50, 60, 70, and 80 °C). Their selection was according to a diversity of technological regimes in which it's included in various food products.

The investigated parameters were as follows:

Surface tension [17] by equation (1):

$$\gamma = \frac{rg}{2}(\Delta H \rho_0 - h\rho) \ , \qquad (1)$$

where r – radius of the capillary, m; g = 9.8 m/s^2 – the acceleration of gravity; ΔH – the maximum difference in the two gauges of the gauge, m; h – liquid level, m; ρ_0, ρ – the density of the water and the oil, g/ml.

Dynamic viscosity, by equation (2):

$$\eta = \frac{2}{9}\frac{(\rho_l - \rho_b)gr^2}{v} \text{ (vertical fall down)}, \qquad (2)$$

where g – acceleration of gravity, m/s^2; ρ_l – density of the liquid, kg/m^3; ρ_b – density of the ball, g/ml; r – radius of the ball, mm; v – speed with uniform movement determined by the road per unit time.

Kinematic viscosity, by equation (3):

$$\nu = \frac{\eta}{\rho}, \qquad (3)$$

where η – dynamic viscosity, Pa.s; ρ – density, g/ml.

Density, by equation (4):

$$\rho = \frac{m_1 - m}{V}, \qquad (4)$$

where ρ – density of the oil, g/ml; m – mass of the pycnometer, g; m_1 – mass of the pycnometer with liquid, g; V – volume, ml.

All measurements were performed in triplicate and the results were presented as the mean value of

the individual measurements with the corresponding standard deviation (SD), using Microsoft Excel.

RESULTS AND DISCUSSION

Data about fatty acid composition of the studied oil is presented in Table 1.

Table 1. Fatty acid composition of high-oleic sunflower oil.

Fatty acids		Content, %
Capric	$C_{10:0}$	0.2 ± 0.0
Lauric	$C_{12:0}$	0.1 ± 0.0
Myristic	$C_{14:0}$	0.1 ± 0.0
Palmitic	$C_{16:0}$	5.0 ± 0.01
Palmitoleic	$C_{16:1}$	0.2 ± 0.0
Stearic	$C_{18:0}$	3.3 ± 0.01
Oleic	$C_{18:1}$ (n-9)	77.9 ± 0.60
Linoleic	$C_{18:2}$ (n-6)	11.5 ± 0.09
α-Linolenic	$C_{18:3}$ (n-3)	0.1 ± 0.0
Arachidic	$C_{20:0}$	0.3 ± 0.0
Eicosenoic	$C_{20:1}$	0.2 ± 0.0
Behenic	$C_{22:0}$	0.8 ± 0.0
Lignoceric	$C_{24:0}$	0.3 ± 0.0
Saturated fatty acids		10.1
Unsaturated fatty acids		89.9
Monounsaturated fatty acids		78.3
Polyunsaturated fatty acids		11.6

The main fatty acids among all thirteen identified (100% of total composition) were found to be: oleic (77.9%), linoleic (11.5%), palmitic (5.0%), and stearic acid (3.3%). The results about the lipid composition were similar to the data published in the literature. The comparative analysis showed that there is variation in the amounts of some fatty acids – the content of oleic acid is lower than the data of García-Zapateiro *et al*. (81.50%) [2] and Roman *et al*. (82.33%) [3], for linoleic acid it is higher than reported by García-Zapateiro *et al*. (6.85%) [2] and Roman *et al.* (8.79%) [3], and for palmitic acid it is higher than the results of Roman *et al.* (3.59%) [3]. The differences can be explained by the different origin of the plants, which is a common tendency found for comparisons between other oils [1].

The contents of saturated, unsaturated, mono- and polyunsaturated fatty acids are given in Table 1. Sunflower oil was abundant in unsaturated fatty acids which are almost 90% of the fatty acid profile of the examined vegetable oil. Among them, monounsaturated fatty acids (78.3% of the fatty acid composition) represent about 87.1% of the content of unsaturated acids, while the amount of polyunsaturated ones was substantially low. The results are in agreement with those reported by Roman *et al*. [3] where the content of monounsaturated fatty acids of high oleic sunflower oil is 82.41%, while the levels of polyunsaturated ones are 8.86%.

Data about the content of the main biologically active components in the investigated oil is given in Table 2.

Table 2. Content of unsaponifiable matter, sterols, and tocopherols in high-oleic sunflower oil.

Biologically active components	Content
Unsaponifiable matter, % of the oil	1.7 ± 0.01
Tocopherols, mg/kg in the oil	219 ± 2.00
α–Tocopherol, % from total tocopherols	100 ± 0.0
Sterols, % of the oil	0.6 ± 0.0
Sterol composition, % of the sterol fraction	
Cholesterol	0.7 ± 0.0
Brassicasterol	3.4 ± 0.03
Campesterol	0.9 ± 0.0
Stigmasterol	11.1 ± 0.10
Δ^7-Campesterol	4.2 ± 0.04
β-Sitosterol	79.7 ± 0.70

Unsaponifiable matter of the oil mainly consists of different compounds as terpenic (sterols, tocopherols, tocotrienols, carotenoids, *etc.*) and aliphatic (fatty alcohols, saturated and unsaturated hydrocarbons) [18]. The content of unsaponifiable matter in the oil is close to that published in the literature for sunflower (1.5%), but it is lower than that for rapeseed (2.0%), maize (2.8%), and grapeseed oil (2.0%) [19].

The major part of the unsaponifiable matter in the oil consists of sterols [8]. β-Sitosterol was the main component in the sterol composition of the studied oil (79.7%) followed by stigmasterol (11.1%), Δ^7-campesterol (4.2%), and brassicasterol (3.4%), while the rest of the identified sterols was below 1%.

In the tocopherol fraction α-tocopherol predominates, which is in agreement with data for sunflower oil [1–3].

The physicochemical parameters of high-oleic sunflower oil – density, surface tension, dynamic and kinematic viscosity were experimentally determined. All measurements were performed in the temperature range between 20 and 80°C.

Surface tension of the high-oleic sunflower oil was measured by the maximum bubble pressure method. The values of the surface tension were between 29.775 ± 0.103 mN/m and 12.461 ± 0.126 mN/m at different temperatures. In earlier studies

there were no experimentally determined results for the surface tension of high-oleic sunflower oil. According to the authors of [11] the addition of linoleic acid to the oil lowers the surface tension from 53.70 mN/m to 28.39 mN/m at room temperature (25°C). Good linear dependence was observed between surface tension and temperature according to the authors of [20]. Temperature dependence of surface tension was also observed in the investigated range of this work. With an increase in the temperature, the surface tension decreased. The results are presented in Fig. 1. After regression analysis, the linear equation was obtained ($y = 33.778 – 0.283*x$). High correlation coefficient $R^2 = 0.964$ indicates a good dependence between the two parameters.

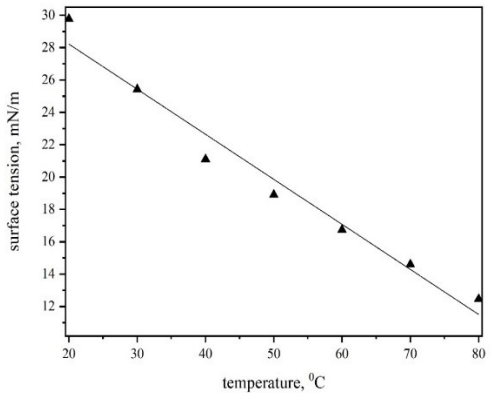

Fig. 1. Dependence between surface tension and temperature.

Experimental density was determined. Similarly to the data in the literature, a good correlation between temperature and density was observed over the same temperature range. The results are presented in Table 3. The data show that with an increase in temperature the density decreases, established by other authors [20, 21] as well.

Dynamic viscosity was determined after performing the experiment in a temperature range between 20 to 80 °C. The results are presented in Table 3. The viscosity decreases with the increase in temperature. The observed values are between 44.401 mPa.s at 20 °C and 40.067 mPa.s at 80 °C. According to other researches, the viscosity of pure sunflower oil is 39.55 mPa.s [9] and this value is comparable to the results obtained in the present study.

According to equation (3) there is a correlation between dynamic and kinematic viscosity. The kinematic viscosity is obtained by calculating while the dynamic viscosity is divided by the density (Table 3). The kinematic viscosity has values between 51.148 mm^2/s and 47.268 mm^2/s. The values obtained in this study do not differ from those published by Jamil *et al*. [22] who investigated pure sunflower oil and found the dynamic viscosity to be 45.38 mPa.s and the kinematic viscosity – 49.56 mm^2/s.

Table 3. Density, dynamiac and kinematic viscosity of high-oleic sunflower oil.

Temperature, ˚C	Density, g/ml	Dynamic viscosity, mPa.s	Kinematic viscosity, mm^2/s
20	0.868 ± 0.004	44.401 ± 0.221	51.148 ± 0.156
30	0.864 ± 0.001	43.682 ± 0.162	50.519 ± 0.229
40	0.861 ± 0.007	42.963 ± 0.135	49.883 ± 0.187
50	0.857 ± 0.004	42.241 ± 0.094	49.240 ± 0.135
60	0.854 ± 0.005	41.518 ± 0.108	48.592 ± 0.242
70	0.851 ± 0.001	40.449 ± 0.184	47.527 ± 0.117
80	0.847 ± 0.007	40.067 ± 0.137	47.268 ± 0.148

CONCLUSION

The main fatty acids of the high-oleic sunflower oil were oleic, linoleic, palmitic, and stearic acid. β-Sitosterol and α-tocopherol were the major lipid – soluble biologically active components in the whole lipid fraction. For the first time some physicochemical parameters of investigated oil were calculated at seven different temperatures (20, 30, 40, 50, 60, 70, and 80 °C). The obtained values for the composition of the lipid fraction and physicochemical parameters are important for future research on the incorporation of high-oleic sunflower oil into different food products.

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