Original scientific paper
Originalni naučni rad

SOME PHYSICO-CHEMICAL CHARACTERISTICS OF OLIVE OILS FROM LIBYA

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Olives in Libya are a basic agricultural commodity. Moreover, olive oil is mostly produced by local producers using a cold-pressing process. To the best of our knowledge, there is no literature data on the quality of olive oils produced in Libya. Therefore, the scope of this research was to analyze the most important parameters for olive oil identification that originated in Libya. Analyses of the oil produced in 5 different geographical regions in Libya, produced by different oil manufacturers, have shown that the oils are not significantly different and are meeting legislated parameters for olive oil quality. It was determined that the iodine value (IV) of investigated oils was in the range of 80 to 87 g/100g, while the refractive index did not vary much, 1.464 to 1.469. Similarly, saponification value (SV) was rather close in values, 188.0-197.0 mgKOH/g, as well as the content of unsaponified matter, 7.7-9.0 g/kg. The oil sample from the Zwit region had the lowest relative density, 0.910 (20^{o}C/water 20^{o}C), while the oil from the El Farok region had the highest value for density, 0.915.

Key words: olive oil, Libya, physico-chemical characteristics of the oil

NEKE FIZIČKO-HEMIJSKE KARAKTERISTIKE MASLINOVOG ULJA IZ LIBIJE

Osnovna uljana kultura u Libiji je maslina, pri čemu ulje proizvode uglavnom individualni proizvođači postupkom hladnog presovanja. Po našim saznanjim u literaturi uopšte nema podataka o kvalitetu maslinovog ulja proizvedenog u Libiji. S toga, u ovom radu su analizirani najvažniji pokazatelji za identifikaciju maslinovog ulja poreklom iz ove zemlje. Analiza osnovnih parametara fizičko-hemijskog kvaliteta pokazala je da se ulja proizvedena u 5 različitih regija - geografskih područja Libije od strane različitih proizvođača međusobno ne razlikuju bitno jedna od drugih, i da ispunjavaju zakonom predviđene okvire. Utvrđeno je da jodni broja ispitivanih ulja ima vrednosti od 80 do 87 g/100g, dok indeks refrakcije varira u uskom intervalu, od 1,464 do 1,469. U rezultatima saponifikacionog broja i sadržaju neosapunjivih materija nije bilo velikih odstupanja između analiziranih ulja, budući da su im vrednosti iznosile, respektivno, 188-197 mgKOH/g i 7,7-9,0 g/kg. Uzorak ulja iz regije Zwit imao je najmanju zapreminsku masu, 0.910, dok je ulje iz regije El Farok imalo najveću vrednost zapreminske mase, 0.915.

Ključne reči: maslinovo ulje, Libija, fizičko-hemijske karakteristike ulja

INTRODUCTION

Unrefined edible oils are an important constituent of the human diet because they provide desirable nutritional properties like taste, aroma and texture of food. Olive oil is one of the most frequently used oils in Europe and some Asian regions. Olive oil is obtained from the fresh and healthy fruits by physical procedures under low thermal conditions (Peri, 2014). The olive oil quality is primarily influenced by the cultivar, environment and agronomic practices that are employed in the olive trees growing, olive harvesting, processing and storage conditions (Salimon and Farhan, 2012; Abu-Reidah et al., 2013; Lopez et al., 2014; Augusto Ballus et al., 2014). Although olive oil is considered to be one of the highest quality oils on the market, its quality is found to be compromised by mixing it with lower quality oils, considering the fact that oil composition declared by manufacturers does not always accurately match the actual chemical composition (McKenzie and Koch, 2004). Therefore, knowledge

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of its physico-chemical properties is indispensable (Kielczynski et al., 2014).

Since olive oil is target for fraud nowadays for economic reasons, the authenticity of it is of great relevance (Angerosa et al., 2006; Frankel, 2010; Garcia et al., 2013). In recent years, one of the most common frauds associated with olive oil is the mixing of olive oil with cheaper oils, like sunflower and soybean oils, where they are not declared. Much attention has been given to analytical method development to be able to test olive oil for its authenticity (Sánchez-Hernández et al., 2011; Chen et al., 2011; Calvano et al., 2012; Garcia et al., 2013).

Olive oil was used for centuries and was always one of the main oil sources for people in Europe. It is one of the best oils for cooking and frying that is also used in other preparations, oil based dressings, etc. Very specific sensory quality along with a high nutritive value are the most probable reasons for its continuous increasing demand on the market (Benabid et al., 2008; Salimon and Farhan, 2012; Lopez et al., 2014; Augusto Ballus et al., 2014). Due to its ever growing popularity, olive oil production has been extended out of the countries in the Mediterranean region (traditionally most important processors and consumers of this oil) to countries such as Argentina, Australia, Azerbaijan, Brazil, China, India, Japan, Mexico, New Zealand, Western and South Africa, USA etc. (Lopez et al., 2014). Therefore it is important to expand the knoweldge and data base of the quality of virgin olive oils that are manufactured in other parts of the world and produced from olive cultivars grown in different countries.

Today, there are about 600 million olive trees planted around the world. These trees occupy the surface of about 7 million ha, and about 95% of them are located in Mediterranean countries (Garcia-Gonzales and Aparicio, 2010). The most recognizable oil producers are from Spain, Italy and Greece. Libya is also one of the large olive oil producers and olive is a basic oil raw material in this country. According to information dated from 1972, there were 3 million olive trees planted in Libya. Today, this number has increased to 8 million. Olive oil in Libya is mostly produced by local producers using a cold-pressing process. According to information from 2010, there were 250 local, independent oil producers. One part of produced olive oil is consumed by these independent producers themselves; one part is for sale and one part is for sale to supply the only factory in the state for oil refining purposes. In the period from 2007/08 to 2012/13, the average volume of produced olive oil was 14.700 tons/year, i.e. 0.5% of the world's olive oil production. The main problem is associated with the low manufacturing output as well as the difference in the quality to be able to meet European standards. Libya has very good natural conditions for increasing olive oil production (Seddiq, 2012; IOOC, 2014). Therefore, the main goal of this research was to analyze the olive oils manufactured in Libya that, to the best of our knowledge, were not of the research subject. Hence there is no published data of olive oil manufactured in this region. Authenticity (identification) of the samples was verified by determining the basic physico-chemical parameters of the oils, such as: iodine value, refractive index, saponification value, unsaponified matter content, as well as relative oil density.

MATERIALS AND METHODS

Material

In this research, 5 samples of virgin olive oil that originated from 5 different regions of Libya were used. The samples were produced using traditional methods of pressing.

Identification of analyzed samples is given in Table 1. Oil samples (1l) were kept in glass bottles under refrigerated conditions (8 ^{o}C) until analyzed.

Table 1. Identification of virgin olive oil samples
Tabela 1. Identifikacija uzoraka maslinovog ulja

Oil sample, No Oznaka uzorak	Production region in Libya Područje proizvodnje u Libiji
1	Aboras
2	Be
3	Zwit
4	El Farok
5	Alati

Methods

Standard methods were used to determine physico-chemical characteristics of the oil samples: iodine value (SRPS ISO 3961:2001), refractive index (SRPS EN ISO 6320:2012), saponification value (SRPS EN ISO 3687:2008), unsaponified matter content (SRPS EN ISO 18609:1012) and relative density (SRPS ISO 6883:2003).

RESULTS AND DISCUSSION

The results of the basic physico-chemical characteristics of oil originated from Lybia are shown in Table 2.

Table 2. Basic physico-chemical characteristics of investigated olive oil samples
Tabela 2. Osnovne fizičko-hemijske karakteristike analiziranih uzoraka maslinovog ulja

Sample Uzorak	IV Jodni broj (g/100g)	Refractive index Indeks refrakcije (n^{20}_{D})	SV Saponi-fikacioni broj (mgKOH/g)	Unsaponifiable matter Neosapunjive materije (g/kg)	Relative density Relativna gustina (20ºC/H_2O 20ºC)
1	82	1.467	197	7.7	0.912
2	87	1.467	188	8.4	0.912
3	81	1.464	192	8.0	0.910
4	80	1.468	192	7.8	0.915
5	84	1.469	197	9.0	0.912

Iodine value is a very important oil characteristic as it indicates its level of saturation (Dosunmu and Ochu, 1995), and can be used to determine the level of impurities in lipids or fatty acids. Iodine value is also important in determining the thermal oxidation of oils during the frying process, as this value is different depending on the oil oxidative state, i.e. was it previously oxidized, polymerized etc. (Dimić i Turkulov, 2000).

Iodine values in olive oil samples that originated in different parts of Libya are shown in Table 2 and were in the range of 80 (sample no. 4 – oil manufactured in El Farok region) to 87 g/100g (sample no. 2, oil manufactured in Be region). These values indicated that these oils are nondrying, highly unsaturated and contain high levels of oleic acid.

Obtained values are in accordance with the Serbian legislation for olive oil quality (Pravilnik, 1999, 2004), as well as published data: 75 to 94 g/100g (Karleskind and Wolff, 1998); 77 to 94 g/100g (Nas et al., 1992); 80 to 88 g/100g (Sonntag, 1979).

Commercial olive oil originated from Tunis (the closest region to Lybia) had an iodine value of 81.23 g/100g (Borchani et al., 2010), while the Turkish olive oil had a wider range: between 79.5 and 92.3 g/100g (Tanilgan et al., 2007). Other authors obtained an iodine value of 86±1.2 g/100g for the Turkish olive oil (Özcan et al., 2013). According to Salimon and Farhan (2012), the iodine value for olive oil originating from Saudi Arabia was 81.4 , while for extra virgin olive oil from the same region this value was 88.4. For 16 samples of olive oil originating in different regions in Algeria, the iodine value was in the range of 77.18 to 87.26 g/100g (Benabid et al., 2008).

According to the standard (Pravilnik, 1999, 2004), the iodine value of all olive oil categories must be between 75 to 94 g/100g. Compared with other edible oils obtained from different oilseeds and different processes, those values are much lower, which indicates better oxidative stability of the oil. For example, refined sunflower seed oil had an IV value of 139-141g/100g, while refined soybean oil had lower IV values (125-133 g/100g). For refined rapeseed oil, this value was reported as 114-116 g/100g (Warner et al., 1989). Cold pressed pumpkin seed oil had an IV value of 122 g/100g (Dimić, 2000); cold-pressed walnut oil of 143.5 g/100g, while the solvent-extracted walnut oil had an IV value of 146.5 g/100g (Rabrenović i Pićurić-Jovanović, 2008)

Refractive index is a specific value for certain types of oils and has a range that can be used to classify unknow oil. It is directly related to the acidic composition of oils (free fatty acid, length of hydrocarbon chains, *cis/trans* fatty acids ratio) and to their status of oxidation (Dimić i Turkulov, 2000; Borchani et al., 2010).

Analyzed oil samples had a refractive index in the range of 1.464 (oil sample no. 3; region Zwit) to 1.469 (oil sample no. 5; region Alati). The differences between the refractive index of oil samples were not correlated to the olive-growing region or oil processing method.

Karleskind and Wolff (1998) pointed out that the refractive index in olive oil was 1.468-1.470, while Sonntag (1979) reported the range of 1.4680-1.4705. Commercial olive oil originating from the Sfax region in Tunis had a refractive index of 1.471±0.001 (Borchani et al., 2010). According to Tanilgan et al. (2007), olive oil manufactured in Turkey had a refractive index of 1.467-1.469, while Özcan et al. (2013) reported this value for pure olive oil made in Turkey as well, as 1.4578±0.0003. According to the standard (Pravilnik, 1999, 2004), the refractive index of olive oil is 1.465 to 1.470.

The refractive index of different types of oils is different. For example, for cold-pressed pumpkin seed oil this value was reported as 1.4725 (Dimić, 2000). For cold pressed walnut oil, it was 1.472, while the solvent-extracted walnut oil had a refractive index of 1.475 (Rabrenović i Pićurić-Jovanović, 2008). In the cold-pressed oil of onion, parsley, cardamom and mullein, the refractive index was 1.4752, 1.4858-1.4862, 1.4666 and 1.4753 (Parry et al., 2006). The same authors (Parry et al., 2005) had previously investiagted cold-pressed oils of red raspberry, marionberry, boysenberry and blueberry, determining refractive indexes of 1.4788, 1.4774, 1.4758 and 1.4783, respectively.

It is well know that the ***saponification value*** is a measure of the total free and combined acids in an oil or fat expressed as the number of milligrams of potassium hydroxide required for the complete saponification of one gram of oil or fat (Dimić i Turkulov, 2000; Salimon and Farhan, 2012). The saponification value is also characteristic for each type of oil and is used for its identification. According to the standard for olive oil (Pravilniku 1999, 2004), the data for the saponification value are between 184 and 196 mg KOH/g.

Investigated oil samples from Libya had a saponification value in the range of 188 mgKOH/g, in sample no. 2 (oil from the Be region), to 197 mgKOH/g in sample no. 1 and 5 (oil from the Aboras and Alati region, respectively) (Table 2). These results indicated that triacilglycerols had fatty acids with longer chains (higher molecular weight due to higher number of carbon atoms).

The values for published data for the saponification value vary according the olive-growing region. Olive oil originating from Tunisia (Borchani et al., 2010), had a much lower saponification value (97.94±0.24 mgKOH/g) than our oil samples (188 to 196 mgKOH/g). Olive oil originating from Algeria had a saponification value ranging from 182.36 to 201.05 mgKOH/g (Benabid et al., 2008), while the olive oil from Turkey (Tanilgan et al., 2007) was in the range of 183.7 to 190.1 mgKOH/g. The saponification value for the olive oil from Saudi Arabia (Salimon and Farhan, 2012) was 189.7 mgKOH/g. Obtained values for the saponification value of the olive oils from Libya investigated in this study were comparable to the values for different plant oils: Aleppo pine seed oil (190 mgKOH/g), raspberry oil (191 mgKOH/g), safflower oil (191.6 mgKOH/g), grape seed oil (192.9 mgKOH/g) (Oomah et al., 2000; Cheikh-Rouhou et al., 2006), walnut oil (188.9 mgKOH/g) (Rabrenović i Pićurić-Jovanović, 2008), cold-pressed pumpkin seed oil (195 mgKOH/g) (Dimić, 2000).

Unsaponifiable matter is a parameter that is used to characterize a type of oil and therefore is a part of the standard (Pravilnik, 2006, 2013). This value measured the content of all matters that are not saponifiable by alkali hydroxides, but are natural components of the lipids (higher aliphatic hydrocarbons, sterols, carotenoids, terpene alcohols, higher esters fatty alcohols, lyposoluble vitamins, tocopherols, phenolics, etc.). These are practically insoluble in water after saponification and are highly soluble in organic solvents (used for oils). Unsaponifiable matter also includes extraneous organic material (mineral oils, for example) that are also solvent soluble but can not evaporate at temperatures of 103±2°C (Dimić i Turkulov, 2000; Salimon and Farhan, 2012). The content of unsaponifiable matter primarily depends on the raw material and oil manufacuring process (Nakić-Nedjeral et al., 2006).

Crude oils usually have unsaponifiable matter content from 0.2 to 2%, and most often at 1% (Dimić i Turkulov, 2000). According to the standard for olive oil (Pravilnik, 2006, 2013), the maximum allowable content of unsaponifiable matter is 15 g/kg, which is much higher than the values obtained in this study. Namely, the unsaponifiable matter content of investigated oils was from 7.7 g/kg (sample no. 1, oil from the Aboras region) to 9.0 g/kg (sample no. 5, oil from the Alati region) (Table 2). The results indicated that the growing region of olives was not a significant factor for this value in oil samples. Sonntag (1979) reported unsaponifiable matter content of 1.8% in olive samples investigated in his study. The average content of unsaponifiable matter in commercial olive oil from Tunisia was 1.5% (Borchani et al., 2010), while this value for the olive oil and extra virgin olive oil from Saudi Arabia was 1.5% and 14%, respectively. The significant differences of the value in these two oils were explained by the minimal modifying process that was applied for pressing the extra virgin olive oil that had much more unsaponifiable matter (antioxidants, tocopherols, sterols, phenolics, etc.) present in the oil. Olive oil from Turkey had an unsaponifiable matter content of 11.9 to 19.1 g/kg (Tanilgan et al., 2007).

Relative density is also one of the parameters used for oil type identification which is regulated by the standard for oil quality. The relative density of the oil samples investigated in this study were according to the standard and fairly uniform, 0.910 in sample no. 3 (oil from the Zwit region), to 0.915, in sample no. 4 (oil from the El Farok region) (Table 2).

The value for relative density depends on the type and fatty acid composition of the oil. For example, cold-pressed oils from different seeds have very different relative densities: red raspberry - 0.929; marionberry - 0.934; boysenberry - 0.948; corn germ - 0.932; onion seeds - 0.923 to 0.930, parsley - 0.981 to 0.985, cardamom - 0.954, mullein - 0.933 and pumpkin - 0.920 (Parry et al., 2005, 2006).

Hopper and Nesbitt (1937) have determined that there was a correlation established between iodine value, refractive index and the saponification number depending on the glycerols and their fatty acids content. However, our results did not confirm any significant correlation between iodine value and refractive index in the oil samples (R^2=0.058; Figure 1).

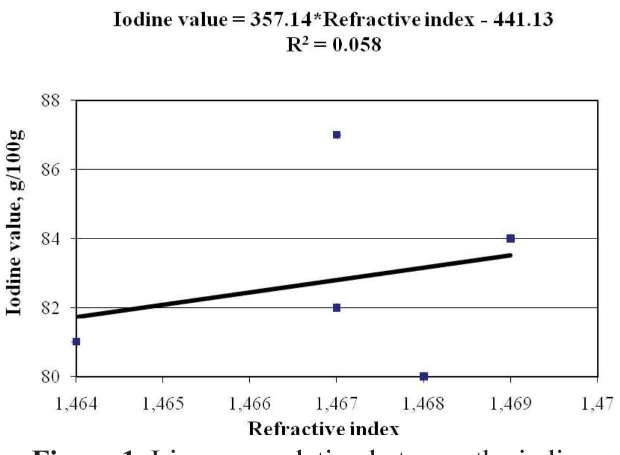

Figure 1. Linear correlation between the iodine value and refractive index of analyzed olive oil samples

Slika 1. Linearna međuzavisnost jodnog broja i indeksa refrakcije analiziranih uzoraka maslinovog ulja

Correlation between the level of saturation, i.e. iodine value and refractive index, was determined as early as 1924, which was conducted on olive oil but also many other oil types: argemone, English mustard, Indian rape, cashew, soya bean, poppy seed (Watson and Sudborough, 1924). The correlation between these parameters in cold-pressed oils is also published in other literature (Hopper and Nesbitt, 1937; Dimić i Turkulov, 2000; Borchani et al., 2010; Zhang et al., 2011).

CONCLUSION

Analyses of the basic physico-chemical parameters of the oil samples originating from Libya have confirmed that these oils are not significantly different, although produced in different geographical regions and by different oil manufacturers. It was determined that the iodine value was in the range of 80 g/100g, in sample no. 4 (oil form the El Farok region), to 87 g/100g in oil sample no. 2 (oil from the Be region). Refractive index was in a narrow range, from 1.464 (sample no. 3, oil from the Zwit region) to 1.469 (sample no. 5, oil from the Alati region). Saponification values and unsaponifiable matter contents were not very different either (188-197 mgKOH/g and 7.7-9 g/kg, respectively). Sample no. 3 had the lowest relative density of 0.910, while sample no. 4 had the highest value, 0.915.

There was no correlation determined in this study between the iodine value and refractive index. Sample no. 5 (Alati region) had the highest refractive index (1.469) and saponification value (197 mgKOH/g), while at the same time the highest unsaponifiable matter content was determined (9.0 g/kg). Sample no.1 (oil form the Aboras region) had the highest saponification value (197 mgKOH/g) and the lowest unsaponifiable matter content (7.7 g/kg).

All investigated olive oil parameters from the oil samples originating from Libya were according to the Serbian standard and indicate the high quality of oil. Therefore, a further study will be conducted to determine the chemical composition and other quality parameters of these oils.

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