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SENSORY AND SOME CHEMICAL CHARACTERISTICS OF OLIVE OILS PRODUCED IN LIBYA

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Libya is one of the considerable olive oil producers in North Africa. The consumption of olive oil in this country is part of the cultural heritage and a large number of individual manufacturers produce oil applying the process of cold pressing. The aim of this work was to determine the sensory profile and some main chemical characteristics, as free fatty acids, peroxide value, total phenols content and induction period of virgin olive oils. Oil samples were extracted from a blend of three olive cultivars typical for Libya: Roghiani, Hammudi and Endori, harvested in five different regions of northern Libya in the crop year 2015. Obtained results have shown that investigated olive oil samples were characterized by different sensory profile and chemical characteristics, due to peculiar effect of harvest areas of olive fruit. On the basis of investigated attributes, some oils were of excellent quality, but other were under. The free fatty acid contents varied between 0.73% to 2.78 % of oleic acid, peroxide values ranged from 6.40 mml/kg to 13.65 mmol/kg and induction period from 4.89 h to 18.33 h. The content of total phenols, as especially important compound for health benefit of olive oil, was also significantly various, from 56.0 to 238.3 mg GAE/kg of oil. It can be concluded that the oil from Gharyan production region was quite different from the other Libyans' olive oils. This oil had the best sensory profile, basic chemical quality, oxidative stability and the highest phenolic content.

Keywords: olive oil, sensory quality, acidity, peroxide value, oxidative stability

INTRODUCTION

From the points of view of both commercial value and health benefits, the authenticity of the various categories of olive oil is of great importance. Olive oil has gained in popularity in many countries and there is a growing demand, particularly in countries where the standard of living is rising. The increased popularity of olive oil is due to its unique and distinguish sensory characteristics and its health benefits (1, 2). The health benefit of olive oil is no longer just „alleged", but well proven (3). The olive oil supplemented diet is effective in the prevention of coronary atherosclerosis, the principal cause of death in western civilization (4). Olive oil is one of the most important features of the Mediterranean diet, replacing the saturated fats that are common in other diets by healthier monounsaturated oil. Evidence shows that olive oil consumption improves risk factors for a multitude of diseases like: type 2 diabetes, Alzheimer, breast cancer, neuropsychiatric disorders, multiple cerebral disorders (5).

Growing olive trees (*Olea europaea* L.) is a widespread activity throughout the Mediterranean Basen, as well as other regions. Olive oil is produced from fresh and healthy olive fruits by cold pressing or by other physical processes using centrifugation and filtration systems under low thermal conditions that do not lead to alteration in the oil (1, 6). Olive oils command a higher price than other vegetable oils, due to their popularity and

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high costs of production, so there is a great temptation for the purveyors of fraud to produce and market sophisticated olive oils. Thus, it is important to increase studies of the characterization of virgin olive oils produced from different olive cultivars growing in different countries and in different olive growing areas to increase the database on varietal virgin olive oils characteristics.

Quality standards of virgin olive oil can be divided into two main groups: chemical and sensory standards. The chemical standards have to be evaluated for classifying the characteristics of quality levels of oils. However, quality standards are useful also to verify hydrolitic and oxidative degradation that can take place in the olives and the oil during processing and storage (7).

Official quality parameters comprise acidity, peroxide value, halogenated solvents, ultraviolet adsorption and sensory assessment. In addition to these official parameters important roles are attributed to the content of biomolecules like phenolic antioxidants, as well as to the correlated induction time value (1). The evaluation of the sensory profile is the basic step for defining the sensory style of an extra-virgin olive oil - EVOO. In addition to the compilance with quality standards fixed by products specifications, EVOO shows singular and distinctive composition and sensory profiles (8).

Libya is one of the considerable olive oil producers in North Africa. The consumption of olive oil in Libya is a part of the culture heritage and a large number of individual manufacturers produce oil applying the process of cold pressing (9). The average quantity of olive oil manufacturing in Libya is up to 0.5% of the world's olive oil production (10). Anyway, Libya has good natural conditions for the further development of olive growing and for enhancing of olive oil production.

Sensory profile, basic chemical parameters, phenolic compounds characterization, as well as shelf life of olive oils are very relevant to their commercialization and increase the product value. Since these characteristics of oil depend on cultivar and growing areas (geographical origin, climate conditions etc.), the aim of this study was concerned with assessment these quality parameters of olive oil samples originated from five different olive-growing regions throughout northern Libya produced from olive cultivars: *Roghiani*, *Hammudi* and *Endori.* To the best of our knowledge, up today there is very few published data on the characteristics of the olive oils processed in Libya and the lack of such data is evident.

EXPERIMENTAL

Material

The investigation was carried out on olive oil (*Olea europea* L.) samples obtained from fruits of olive cultivar *Roghiani*, *Hammudi* and *Endori* harvested from five different geographic regions in Libya. Olive fruit samples were hand-picked and collected at the same time - beginning of January 2015. The cultivars were growing in the north of Libya in following harvest regions: Gharyan, Tarhuna, Msallata, Tripoli and Q. B. Ghashir. The mean rain precipitation registered in these regions was about 383 mm/year, with a mean temperature of approximately 27^{o}C. Olive fruit samples were collected from regions specified above and processed within 3 days after harvesting. Fruits were washed, milled and olive pastes were malaxed with a mixer for 40 min at 35-40^{o}C. After separation at speed of 3000 rpm by centrifugal separation process (Rapanelli, Foligno, Italy), extracted olive oil samples were decanted and filtered through filter press. Oil samples were stored in the refrigerator at 8 ^{o}C in dark glass bottles until further analysis. Samples were tempered at room temperature for 24h before analysis (11).

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Methods

Sensory analysis. The most widely used method in sensory analysis of olive oil is Descriptive Sensory Analysis (DSA). Sensory characteristics were evaluated by a panel of five skilled members, according to European Union Commission regulations and other literature data (12, 13). Coded oil samples were presented in covered glasses at 28 ± 2°C. For the sensory analysis, each sample of 15 ml, after removing the cover, was smelled and then tested by the panelist to judge it for its flavor, according to the sensory profile sheet (Figure 1). Results were expressed as the mean intensity of the sensory perceptions of the tasters.

Intensity of perception of positive atributes	
Fruity (green, mature)	
Bitter	
Pungent	
Intensity of perception of defects	
Fusty/muddy sediment	
Musty/humidearthy	
Winey/vinegary	
Rancid	
Frozen/woody	
Name of taster: Sample code: Date:	

Figure 1. Descriptive sensory profil sheet of the olive oil samples (14)

Total phenolic content. The content of total phenolic compounds (TPC) was estimated according to the spectrophotometric method described by Haiyan et. al. with Folin-Ciocalteu reagent (15). The absorbance at 725 nm was measured against a reagent blank on a UV/VIS spectrophotometer (model T80+, PG Instruments Limited, London). Calibration was performed using gallic acid and a good calibration curve was obtained (R^2 = 0.999 in the range 0 to 100 µg/10 mL). TPC was expressed as gallic acid equivalents (GAE) in mg/kg of oil.

Free acidity (FFA), ***peroxide value*** (PV) and ***iodine value*** (IV) were determined according to the Commission Regulation ECC 2568/91 (12) and subsequent amendments.

Anisidine value. The *p*-anisidine value (AnV) is determined by standard ISO method (16).

Induction period. The oxidative stability, measured as Induction Period (IP), was determined as described by Metrohm Application Bulletin 204/2e. Rancimat apparatus, model 743 (Metrohm, Switzerland) at temperature of 110°C and air flow of 20 l/h was used. Portions of oil (2.5 g) were weighted into each reaction vessel and analyzed simultaneously. The IP was determined automatically by the device and expressed as hours.

Statistical analysis. All results are presented as a mean value ± standard deviation (n = 3). One way an analysis of variance (ANOVA) with a Tukey's test was used to determine statistically significant differences among data ($P \leq 0.05$).

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RESULTS AND DISCUSSION

Sensory profile of olive oil samples

The sensory profile of oils is presented as spider plot (Figure 2) and it can be seen that the samples are rather different subject to different harvest region.

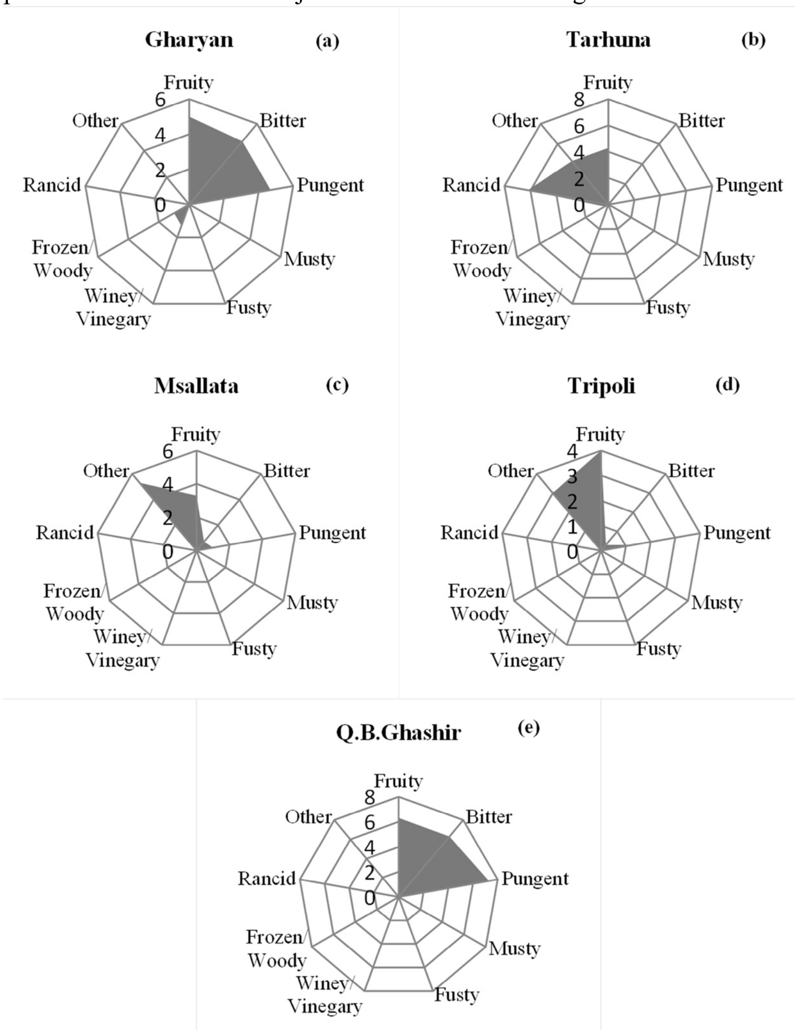

Figure 2. Sensory profile of investigated olive oil samples

Concerned with sensory characteristics, especially the prominent attributes like fruity, bitterness and pungency, the best rated oils are from Gharyan and Q.B. Ghashir areas. Regarding the EC Council Regulation (12) with respect to the flavour intensity, oil from Q.B. Ghashir region can be classified as extra virgin olive oil (EVOO), and oil from Gharyan production region as virgin olive oil (VOO). Oils from other olive production

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areas had poorer sensory properties, mainly with weak fruity flavour and with a slightly pronounced pungency, so they belong to the category of ordinary VOO. In the flavour of oil from Tarhuna region was observed even rancidity. The unpleasant odor of virgin olive oils of poorer categories can derive from saturated and monounsaturated aldehydes (C5-C9), some dienals, other aldehydes and some C8 ketones developed durig the process of unsaturated fatty acid oxidation (1).

The demand of high-quality VOO can be attributed not only to its health benefits but also to reports of its flavour and aroma compounds. Volatiles are responsible for oil's aroma while phenolic compounds are related to its taste. The presence of these compounds gives rise to the particular VOO flavor characterized by a unique balance of green, fruity, bitter and pungent attributes that makes it a distinctive edible oil (6). Extra-virgin olive oil quality should be considered in relation to the capacity of the oil to modify the sensory properties of a dish and the enhance the acceptability of the food to which it is combined (17, 18). Sensory profile in connection with olive cultivar, climate condition, ripeness stage, etc. is well known. By Vinchi et al. (19), despite the proximity between Catalan PDOs (Protected Designations of Origin) oils, the unique pedoclimatic conditions and traditional olive cultivars contribute to produce extra virgin olive oils with singular and distinctive composition and sensory profiles.

Basic chemical characteristics of olive oils

The introduction into the market of different olive oil types with different characteristics and chemical quality is of a big interest today, especially for the so called fraud (adulteration) which is unfortunately very common, even in developed countries. Namely, it is well known that according to the International Olive Oil Council (20), olive oils are by their quality and characteristics classified into several categories. Basic categories of such oil are so called virgin oil or refined olive oil, i.e. the mixture of them. Depending on the free fatty acids content, virgin olive oil is further classified as: a) **virgin olive oil – extra**, b) **virgin olive oil** and c) **virgin olive oil – ordinary**.

The basic chemical characteristics of the analyzed oil samples was examined by the determination of FFA and total phenols content, as well as iodine value (Table 1). Especially, the FFA content is very important parameter as it indicates the acidity of the oil, which is in connection with the quality and possible damages of olive fruit before pressing (e.g., olive fly attacks or improper systems of harvesting, transport and storage of olives) (21).

Table 1. Some basic chemical characteristics of olive oil samples

Region	**FFA (% ol. acid)**	**TPC (mgGAE/kg)**	**Iodine value (g/100g)**
Gharyan	0.80 ± 0.14^{a}	238.3 ± 16.26^{a}	82.98 ± 0.39^{a}
Tarhuna	2.23 ± 0.21^{c}	144.2 ± 29.69^{c}	88.18 ± 0.11^{b}
Msallata	2.78 ± 0.07^{d}	139.4 ± 7.70^{c}	85.66 ± 0.12^{c}
Tripoli	2.38 ± 0.07^{b}	56.0 ± 21.49^{b}	89.18 ± 0.02^{d}
Q. B. Ghashir	0.73 ± 0.07^{a}	136.8 ± 0.07^{c}	88.97 ± 0.11^{d}

Values are means ± standard deviations (n = 3).
Different letters in the same column indicate significantly different values ($p \leq 0.05$).

The values of free acidity proved the different chemical quality of the analyzed oils, since they ranged from 0.73 ± 0.07 % to 2.78 ± 0.07 % of oleic acid. The FFA content was only in two samples (Gharyan and Q.B. Ghashir regions) lower than 1%, so these oils belong to category of EVOO. Oils from other regions has much higher acidity, over 2%, so they belong to the category of ordinary VOO. Iodine number of all samples had

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the typical values ranging from 82.98±0.39 to 88.97±0.11 g/100g, which confirms that the oleic acid content in oils was at least 55%, which is in accordance with legal regulations. Namely, by Regulations of quality and other requirements for edible olive oil (22) iodine value ranges from 80 to 92 g/100g, and oleic acid content from 55 to 83wt%.

The total phenol content ranged from 56 ± 21.49 to 238.3 ± 16.26 mg GAE/kg. There is a significant difference between Gharyan VOO and VOO from other regions, while no significant difference ($p \leq 0.05$) between VOO from Tarhuna, Msallata and Q. B. Ghashir regions. Similar results were obtained for oils of olive variety "*Roghani*" from the same harvest regions (11), as well as with some others Lybian VOO (varieties: *Gargashi, Nab-Elgamal and Oscolana*) (23).

Oxidative stability of olive oils

Although the olive oil is well known for its good oxidative stability, the values of parameters, shown in Table 2, confirm the great statistical differences ($p \leq 0.05$) in the shelf life of investigated oil samples from different harvest regions. The best, that is, the lowest peroxide value (6.40 ± 0.14mmol/kg) had the oil from Gharyan region, while in all other samples the peroxide value was very high, even above 10 mmol/kg. However, according to our previous studies, PV of virgin olive oils originated from Libya ranged from 0.96 mmol/kg (oil from the Zwit region) to 2.40 mmol/kg (oil from the Alati region) (24).

Table 2. Oxidative stability of olive oil samples

Region	PV (mmol/kg)	AnV ($100A^{1\%}_{350nm}$)	$IP_{110\,^oC}$ (h)
Gharyan	6.40 ± 0.14^a	8.34 ± 1.27^a	18.33 ± 0.10^b
Tarhuna	11.70 ± 0.01^b	3.84 ± 0.23^b	5.58 ± 0.16^a
Msallata	9.20 ± 0.00^c	3.64 ± 0.00^b	6.80 ± 0.08^a
Tripoli	13.65 ± 0.35^d	3.10 ± 0.15^b	4.89 ± 0.01^a
Q. B. Ghashir	11.00 ± 0.14^b	11.94 ± 6.30^a	9.87 ± 0.11^c

Values are means ± standard deviations ($n = 3$).
Different letters in the same column indicate significantly different values ($p \leq 0.05$).

The induction period (18.33 ± 0.10h) of oil from Gharyan region was the highest and it was about two or three times longer compared to IP of other oil samples. The oxidative stability of edible oils, pronounced as IP, is closely connected with iodine value and some other parameters. From Table 1 and Table 2 it is obvious that the Gharyan oil sample had the lowest iodine value (82.98 g/100g) and peroxide value (6.40 mmol/kg), which finally resulted the highest IP. On the other hand, the very good oxidative stability of Gharyan oil may be, also, due to the significantly higher content of total phenolic compounds (238.3 mgGAE/kg) with respect to other samples (Table 1). The phenol content and the related induction period, defined as the delay in the commencement (initiation) of oxidation in oil, merits particular comment: the longer the IP, the better the oil (1). Resistance to oxidation depends on both chemical factors (accessibility to oxigen, degree of unsaturation, metal compounds, antioxidants such as phenols) and physical factors (temperature and light). It is obvious that the physical factors depend greatly on technology and household practices.

As a chemical characteristic, the *p*-anisidine value also indicates the oxidative deterioration of oil (it means the secondary products - mainly unvolatile aldehydes) which may be result of lipoxygenase activity. As it can be noticed, there is a significant difference in AnV between Gharyan VOO and oils from other regions, while no significant difference ($p \leq 0.05$) between VOO from Tarhuna, Msallata and Q. B. Ghashir regions.

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CONCLUSIONS

The results of our investigations indicate that oils originated from Libya from various olive growing regions vary greatly in all tested parameters. It can be concluded that the oil from Gharyan production region was quite different from the other oils. Namely, this oil had the best sensory profile, good basic chemical quality (FFA - 0.80±0.14 % ol. acid, PV- 6.40± 0.14 mmol/kg), very good oxidative stability (IP_{110^oC} - 18.33 ± 0.10 h) and the highest total phenolic content (238.3 ± 16.26 mgGAE/kg). These considerations indicate the notable impact of Gharyan harvest region on these characteristics of VOO, probably due to the higher altitude. However, authentication of VOO from this point of view is a quite difficult issue which requires more studies along conducted during several harvest years. Our presented results are only preliminary results and further studies are needed.

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