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### Physicochemical characterization of olive oil from Aljouf area of Saudi Arabia

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**Abstract:** The aim of this study is to characterize the extra virgin olive oil obtained from the most abundant olive cultivar grown in Aljouf area of the northern part of Saudi Arabia which is Sorani. Twenty samples were collected from each orchards in Sakaka, Dumat Aljendal, and Qurriyat during two seasons (2015 and 2016) and were examined for the main International Olive Council chemical quality parameters (acidity, peroxide value, and UV absorption (K232 and K270)). In addition, the fatty acid profile of olive oil samples was also analyzed, including the more commonly present fatty acids (oleic, palmitic, linoleic, stearic, linolenic and palmitoleic acids) together with the total polyphenolic content (from 157 to 287 mg /kg), the iodine value and the refractive index. The acidity of the samples analyzed were (0.64-0.88), peroxide value were (5.5-10.1) and iodine value were (76.8-91.7). The variation of the obtained results may be attributed to the different geographical and climatic reasons as well as other conditions such as the production process, harvesting, storage and extraction method.

**Key words :** Olive oil, physical and chemical properties, Aljouf, Saudi Arabia.

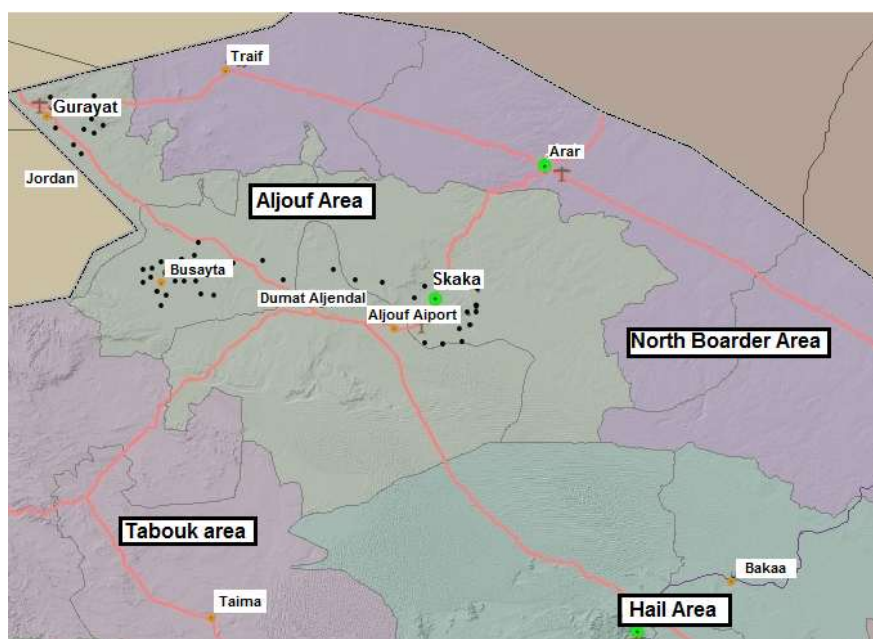
#### Introduction

Olive tree is one the most important trees in the world due to the economical and beneficial healthy effects of the oil obtained from its fruit (olive oil). The olive tree originally comes from the Mediterranean basin due to climate requirements, and CA. 75% of the production of olive oil in the world are from this zone, being Spain, Italy, Greece, Turkey, Morocco and Syria the main producers. Virgin olive oil (VOO) is an important nutritional fruit juice and is defined as the oil obtained only by mechanical extraction or other physical means of the olive that do not lead to alteration of their natural composition. It exhibits the highest edible oil quality among other vegetable oils because of the high level of monounsaturated fatty acids and balanced amount of other fatty acids, together with a high content of phenolic compounds, which provides important healthy properties. It is frequently associated with reduced incidence of diseases, such as cardiovascular, cancer, metabolic syndrome and diabetes<sup>1-3</sup>. In this sense, linoleic and linolenic acids represent the main essential fatty acids that lower the risk of coronary heart disease and cancers.

The chemical characterization of the composition of extra virgin olive oils provides information that may allow their classification according to its quality, thus improving the economical impact of olive oil,

especially in the Mediterranean area<sup>4-8</sup>. It is known that the quality of extra virgin olive oil depends on many factors, including cultivation and harvesting conditions, storage and processing steps. Another important factor is an olive cultivar (variety) and climate conditions<sup>9</sup>. The most important fatty acid is oleic acid, which is a monounsaturated fatty acid that controls cholesterol levels and has an important role in oil stability. Olive oil is also the source of many minor components such as phenols or tocopherols with remarkable antioxidant capacity, phytosterols and natural pigments.

During the last twenty years, agriculture of olive trees had been developed and spread in the northern part of Saudi Arabia. The Aljouf area exhibits a high potential for olive production due to climate and geographic factors, which made this area the main producer in Saudi Arabia<sup>10,11</sup>. According to governmental statistics, over 13 million olive trees have been planted in Aljouf, which represent more than 90% of Saudi Arabia overall<sup>12</sup>. The rest being distributed mainly in Tabouk and Hail. The main successful cultivars in Aljouf area are Sorani, Nabali, K18 and Picual. However, to the best of our knowledge, no previous studies have been conducted to characterize the VOO obtained from this region. The aim of this study is to investigate the main physical and chemical properties of extra VOO produced from the main cultivar; (Sorani) which is grown in different geographical sites from Aljouf area. The locations of olive tree farms, from which samples were collected are shown in (Figure 1). The characterization accomplished includes most of the International Olive Council quality control parameters such as acidity, iodine value, peroxide value, UV absorption, fatty acid profile, together with refractive index and total polyphenolic content. From these data, the quality of olive oil produced from different cultivated has been evaluated and discussed.



**Fig. 1** Locations of different sample collection points in Aljouf area\*.

\*From ministry of interior-saudi arabia web site.

## Experimental

### Olive oil samples:

Olive oil samples were obtained from different fruit varieties cultivated from three regions from Aljouf area: Sakaka, Dumat Aljandal and Qurayat samples coded as S,D and Q respectively, in the ripening stage of october and november (2015, 2016 seasons) VOO samples were obtained from the industrial olive mills that use two and three phases-continuous olive oil extraction systems, that covered different geographic positions in the area. A total of twenty samples was collected from each selected orcheds, with a total of sixty samples, the samples were stored in sterilized bottles.

### Physicochemical properties:

Refractive index, and K232 and K270 (extinction coefficients calculated from absorption at 232 and 270 nm respectively), were determined according to the standard methods described in the European Union regulation 2568/91. Refractive index measurements were measured according to method<sup>13</sup>, using (ATAGO9)<sup>(R)</sup> Japan Refractive index. Absorbance measurements were carried out using a double beam UV-VIS Labomed 3200 spectrophotometer. All parameters were determined in triplicate for each sample. pH measurements were obtained using a pH meter (Hanna HI112, Mauritius), calibrated using two standard buffers at pH 4.00 and pH 7.00. Chemical analysis (free acidity, peroxide value, iodine value) was performed according to the AOAC Wijs method<sup>14</sup>.

Free oil acidity was measured by weighing a known amount of olive oil, then dissolved in diethyl ether/ethanol (1:1 v/v), the mixture was shaken, then titrated with 0.05 M KOH in methanol using phenolphthaleine as an indicator. For Peroxide value, about 5 g of olive oil was dissolved in a mixture of acetic acid/chloroform (3:2 v/v), then 1.0 ml of a saturated solution of KI was added. The liberated iodine was then titrated with 0.05 M sodium thiosulfate in the presence of starch.

### Analysis of fatty acids

Analysis of fatty acids was performed according to the official method of the European Commission Regulation<sup>13</sup>. (EEC/2568/91, 1991); olive oil samples were esterified with methanol solution of 2N KOH for 30 minutes at 50° C. Gas chromatography separation was done using Thermo GC (model; Trace 1300) with Flame Ionization Detector. The column was a fused silica capillary SE30, diameter 0.25 µm and a length of 25 meters. Helium was used as a carrier (6ml/min). The column temperature program was: initially isotherm at 140 °C for 10 minutes, then an initial programmed rate of 1 °C / min up to 160 °C, then a second rate of 2°C/min up to 220 °C and a final isotherm for 15 min. Samples were injected into the split mode. The GC peaks were identified as the corresponding fatty acid methyl esters according to the retention time compared to those ones of pure standards.

### Total phenolic content:

The total phenolic contents were determined according to the methods described by Tsimidou<sup>15</sup> (Tsimidou Z. 1999). 10 g of oil was extracted three times with 50 ml methanol:water 60:40 (v:v) made up to a final volume of 50 mL and one aliquot of 5 ml is analyzed for the total phenolic contents in the oil extract by the Folin - Ciocalteu method: a 5.0 ml of extract or phenolic standards (aliquots of gallic acid solutions to give a final concentration for calibration line from 1 to 9 mg/l) was mixed with 2.5 ml of Folin-Ciocalteu and 4.0 ml of 1M aqueous Na<sub>2</sub>CO<sub>3</sub>, and maintained at room temperature for 60 min. The measurements were carried out at 765 nm using UV-Vis spectrophotometer. Results were expressed as mg of Gallic acid equivalent per Kg oil.

## Results and Discussion

### Physical and chemical properties:

The Physical and Chemical characteristics of olive oil sample were shown in (Table 1). There were little differences found in chemical values (free fatty acid and peroxide value) among olive oil samples. These properties, especially, depend on the initial quality of the olives fruit samples. In (Table1), Acidity (% oleic acid) was in the range (0.67 – 0.88 %). According to the International Olive Council, extra virgin olive oils should have acidity (% as oleic acid) ≤ 0.8%, and the acidity contents of almost all samples were not higher than limited: only samples S1, D3 and D4 showed a slightly (in practice not significant) higher values. As can be seen, there were no big significant differences among samples (Figure 2). On the other hand, the results obtained for peroxide value, indicate that the values of this quality parameter were not higher than the limit established by the International Olive Council for extra virgin olive oil (≤ 20 meq O/Kg oil) and ranged between 5.51 meq O/kg and 10.10 meq O/kg. Therefore, from the point of view of these two quality indexes, the quality of the olive oils produced in the studied region of Aljouf is the highest one (corresponding to extra virgin olive oil). Nevertheless, the obtained values for UV absorption (K232 and K270) are higher than those ones established by the International Olive Council for extra virgin olive oil. Finally, the iodine value and

refractive index are both in all cases within the range established for this type of olive oil (75-94 and 1.4677-1.4705, respectively) by Codex Alimentarius<sup>16</sup> (Codex Stan 33, 1981).

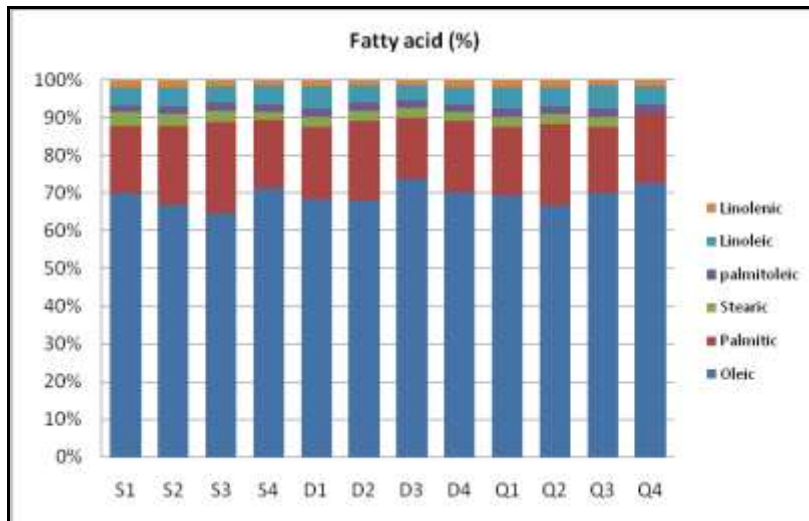
**Table 1. Physicochemical properties (free fatty acid, peroxide value, K232 and K270, Refractive index and iodine value) of olive oil samples (crop seasons, 2015,2016).**

Sample code	FFA (% Oliec acid)	Peroxide value (meq O <sub>2</sub> /kg)	K232	K270	Refractive Index	Iodine value
S1	0.85± 0.03	5.51 ± 0.4	3.13 ± 0.03	0.38 ± 0.05	1.4702	87.86 ± 0.08
S2	0.67± 0.06	6.81 ± 0.07	3.16b± 0.01	0.35 ± 0.08	1.4732	83.76 ± 0.04
S3	0.76± 0.03	9.14 ± 0.04	3.43d± 0.04	0.49± 0.07	1.4692	84.73 ± 0.04
S4	0.72± 0.04	10.10 ± 0.08	3.27 ± 0.05	0.57 ± 0.06	1.4687	89.588 ± 0.05
D1	0.74± 0.06	7.50 ± 0.02	3.18± 0.02	0.32 ± 0.11	1.4700	91.73 ± 0.06
D2	0.67± 0.04	7.47 ± 0.07	3.26 ± 0.06	0.38 ± 0.11	1.4767	84.87 ± 0.04
D3	0.82± 0.08	6.58 ± 0.03	3.17± 0.03	0.66 ± 0.08	1.4689	79.59 ± 0.07
D4	0.88± 0.05	7.38 ± 0.02	3.24 ± 0.02	0.44 ± 0.13	1.4683	88.84 ± 0.03
Q1	0.68± 0.03	8.39 ± 0.03	2.88± 0.02	0.39± 0.06	1.4704	76.85 ± 0.04
Q2	0.72± 0.04	6.09 ± 0.07	3.49 ± 0.03	0.33 ± 0.08	1.4698	78.93 ± 0.05
Q3	0.69 ± 0.03	7.39 ± 0.03	2.86 ± 0.02	0.37 ± 0.06	1.4679	81.58 ± 0.07
Q4	0.73 ± 0.04	8.09 ± 0.07	3.48± 0.03	0.36 ± 0.08	1.4683	86.94 ± 0.06

In all measured values, mean values were calculated for samples from the area, so, standard deviation gives idea about the variability of olive oils in term of composition, but not about the repeatability of the analytical methods used.

#### Fatty acid profile analysis:

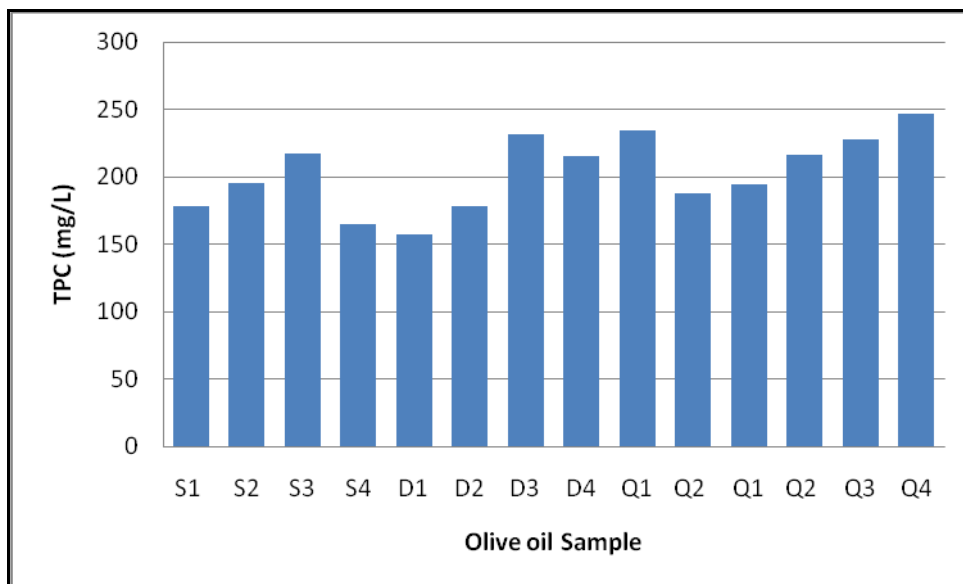
The fatty acid composition of the sixty olive oil samples was determined using gas chromatography and the results are shown in (Figure 2). When examining the fatty acid composition, differences among the samples were observed. It is clear, as usual, that oleic acid was present in the highest concentration; the values were ranged between 51.73% and 72.84%. It was followed by palmitic acid (14.65%- 20.61%). Sample coded **Q4** contained the highest concentration of oleic acid (72.84%) while sample coded **S3** has the lowest percentage of the same fatty acid (51.73%). The results showed that palmitoleic acid was detected in two out of five olive oil samples. It is appeared that the total unsaturated fatty acid contents such as palmitoleic, oleic, linoleic and linolenic acids were at high levels. Unsaturated fatty acid values were between 58 % and 82%. Higher level of unsaturated fatty acids is obtained from Qurayat samples which is attributed to the higher level of rainfall. Differences in these values can be due to species, genetics, variety, etc. and the highest value of oleic acid was obtained from Sorani type olive. To our knowledge, there is still limited information about the fatty acid composition of our local olive oils (Olive oils produced in Aljouf area) and there is no research carried out to determine this chemical parameter. The results found in this study were in agreement with those reported by several authors for different geographical origins<sup>17-19</sup>.



**Fig. 2 Fatty acid content for studied samples.**

### Total phenolic content:

The results of total phenolic contents of olive oils samples are shown in (Figure 3). Total phenol contents expressed as gallic acid values ranged from 157 to 287 mg /kg. Total phenol content as gallic acid equivalent in sample coded **Q4** was the highest (247 mg/kg) which was obtained from Picual variety, but sample coded **D1** has a lowest percentage of these components (157 mg/kg). These results showed a difference in total phenol contents of olive oil samples. These differences may be due to maturation state and nature of cultivar. The phenol contents of olive oils were found higher than reported by (Ibrahim et al. 2013). These authors determined that the contents of the total phenol of olive oils from five olive varieties ranged between 22.5-197.1 mg/kg as gallic acid equivalent.



**Fig. 3 Total phenol content (TPC): mg gallic acid/L**

### Conclusion

For the first time, the main physical and chemical characteristics of olive oil produced in different Aljouf areas of Saudi Arabia (Sakaka, Dumat Aljendal, and Qurryat ) have been investigated. Interestingly, considerable differences were found in some parameters amongst the different samples tested. The fatty acid composition was useful for distinguishing the monovarietal olive oils belonging to particular cultivars which is

Sorani. Our results showed that samples of olive oils extracted from Gurayat region has the best properties of both fatty acid profile and total phenolic content. Finally, in order to confirm characteristic differences among the olive oils examined in this preliminary study, further studies are to be conducted to cover other regions of the northern part of Saudi Arabia and other new varieties/cultivars of olives.

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