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The Influence of Olive Harvest Timing on the Physicochemical Properties of Olive Oil in the Western of Libya

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Abstract:

This study examines the impact of geographical location and harvest month on the chemical composition and quality of Libyan olive oil. Physicochemical properties, including density, water content, refractive index, absorption, acidity value (AV), peroxide number, and heavy metal content (copper and iron), were analyzed for oil samples collected from the Nafusa Mountain (NM) area (Gharyan (GR)& Al-Zintan (ZT)); and the Al-Jafara Plain (AJ) area (Al-Aziziyah (AZ)). Samples were taken at the start (November) and end (February) of the pressing season. Results showed notable variations in AC. At the beginning of the season, AV ranged from 0.22% to 1.6%, with the lowest values in ZT samples and the highest in AZ samples. At the end of the season, AV ranged from 1.62% to 3.03%, and the lowest values were found in GR samples, meeting international standards.

Peroxide number results also showed notable variations. Values ranged from 3.97 to 6.7 mEq/kg at the start of the season, with the lowest values in AZ samples. At the end of the season, peroxide numbers ranged from 5.9 to 13.5 mEq/kg, with the lowest values recorded in GR samples. Despite an increase in peroxide values in ZT samples, they remained within permissible limits.An increase in copper and iron content was observed towards the end of the season. All olive oil samples from the 2022/2023 production year were edible and of high quality. To enhance export competitiveness against neighboring countries, the researchers recommend pressing olives early in the season (October to December).

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Key words: olives oil, harvesting time,physicochemical properties, peroxide value, acidity value.

تأثير موعد قطف ثمار الزيتون على الخصائص الكيميائية والفيزيائية لزيت الزيتون بغرب ليبيا

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الملخص:

تهدف هذه الدراسة إلى فحص تأثير الموقع الجغرافي وموسم حصاد الزيتون على التركيب الكيميائي وجودة زيت الزيتون في ليبيا. تم تحليل الخواص الفيزيوكيميائية، بما في ذلك الكثافة ونسبة الماء ومؤشر الانكسار والامتصاص وقيمة الحموضة (AV) وعدد البيروكسيد ومحتوى المعادن الثقيلة (النحاس والحديد) لعينات زيت الزيتون المتحصل عليها من منطقة جبل نفوسة (NM) (غريان(GR)، الزنتان(ZT)) ومنطقة سهل الجفارة (Aj) (العزيزية (AZ)). تم جمع العينات في بداية موسم عصر الزيتون (نوفمبر) وفي نهاية الموسم (فبراير).

أظهرت النتائج اختلافات ملحوظة في قيمة الحموضة ورقم البيروكسيد بين العينات في بداية موسم العصر ، تراوحت قيمة الحموضة بين 0.22٪ و 1.60٪، مع أدنى قيمة في عينات ZT وأعلى قيمة في عينات AZ. في نهاية الموسم، تراوحت قيمة الحموضة بين 1.62٪ و 3.03٪، وتم العثور على أدنى قيم في عينات GR، والتي تتوافق مع المواصفات القياسية الدولية.

تراوحت قيم البيروكسيد من 3.97 إلى 6.7 مللي مكافئ/كجم في بداية الموسم، مع أدنى قيم في عينات AZ. في نهاية الموسم، تراوحت قيم البيروكسيد من 5.9 إلى 13.5 مللي مكافئ/كجم، وتم تسجيل أدنى قيم في عينات GR. على الرغم من زيادة قيمة البيروكسيد في عينات ZT، إلا أنها لم تتجاوز الحدود المسموح بها وفقًا للمواصفات القياسية الدولية.

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لاحظ الباحثون زيادة في محتوى النحاس والحديد في نهاية الموسم. جميع عينات زيت الزيتون الليبي من إنتاج العام 2023/2022 كانت صالحة للأكل وذات جودة عالية. لتعزيز القدرة التنافسية في التصدير مقابل الدول المجاورة، يوصي الباحثون بعصر الزيتون في بداية الموسم (من أكتوبر إلى ديسمبر).
الكلمات المفتاحية: زيت الزيتون، وقت القطاف، الخواص الفيزيائية والكيميائية، قيمة البيروكسيد، قيمة الحموضة.

Introduction

Olive oil is one of the oldest known vegetable oils, mainly produced in the countries surrounding the Mediterranean Sea. Olive oil has become an important component in the diet of Mediterranean people due to its beneficial effects on human health [1]. Some of these effects are associated with the content of olive oil, such as fatty acids and natural antioxidants (e.g., polyphenols, Vitamin E), which have high value for their positive effect on human health [2]. In addition, it is associated with reduced mortality from cardiovascular disease, neurodegenerative diseases, and a potential role in reducing the potential risk of many types of cancer [3]. The quality of virgin olive oil depends on many factors related to olive tree cultivation, harvesting, storage, processing steps, and time [4], as well as climatic conditions, fertilization, and irrigation systems used for olive trees. Harvest timing can have a significant effect on oil quality, yield, oil stability, and sensory characteristics [5]. In Libya, the number of olive trees reached 10 million in 2020, according to estimates by the Ministry of Agriculture [4-6].

This illustrates the need to determine the quality of olive oil from a range of harvest times and cultivars in order to establish an optimum harvest time. However, few studies have evaluated the physicochemical characteristics of locally cultivated olive oil in Libya. Therefore, the aim of this current study was to assess the effects of olive harvest date and geographical location on the physicochemical characteristics of Libyan olive oil.

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Research Location of the Case Study:

This study was conducted during the crop season of 2022/2023 in two locations: NM (GR-ZT) and AJ (AZ) at two different times. Table (1) presents the abbreviated research locations for the case study.

Table 1. The abbreviated research locations for the case study.

Research Location	Abbreviation
Nafosa Mountain	NM
Garyan City	GR
Alzentan City	ZT
Aljafara	AJ
Alazizia City	AZ

The first sampling was done at the beginning of the harvesting season in October, while the second sampling was done at the end of the harvesting season in February, as shown in Figure 1.

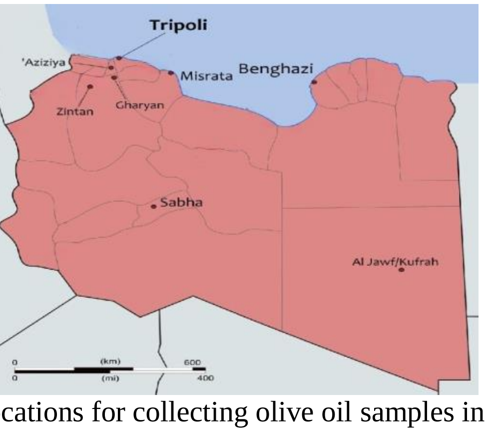

Figure 1. The locations for collecting olive oil samples in the Western of Libya.

Analytical Methods:

In order to study the effects of olive harvest timing and geographical location on the physicochemical properties of olive oil, the olive oil samples were collected immediately after extraction and stored in dark glass bottles at 4°C until analysis. Table (2) presents the instruments that used for each experiment

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Table 2 . The instruments that used for each experiment

Experiment	Instrument Used
Water Content	Centrifuge
Absorbance	Spectrophotometer (Hewlett–Packard, HP 8452 A)
Density Measurement	R.D bottle (capacity 10 ml)

Water Content:
The water content in the samples was determined using a centrifuge instrument.

Absorbance:
Absorbance values were calculated using a spectrophotometer (Hewlett–Packard, HP 8452 A) by measuring the absorption at 323 nm and 270 nm.

Density Measurement:
The densities of the oil samples before and after frying were measured using an R.D bottle with a capacity of 10 ml. The density was calculated using the equation (1):

$$\text{Density} = \frac{\text{mass}}{\text{volum}}\left(\frac{\text{g}}{\text{ml}}\right) \qquad (1)$$

Peroxide Value (PV) Measurement:
The peroxide value (PV) was determined by measuring the iodine released from potassium iodide (KI). A known weight of oil sample was dissolved in acetic acid, followed by the addition of chloroform and a saturated KI mixture. The amount of iodine liberated from KI by the oxidative action of peroxides present in the oil was determined by titration with standard sodium thio-sulphate, using starch solution as an indicator. Iodometric tittration was also performed for blanks. The PV was calculated using the equation (2):

$$\text{PV}\left(\frac{\text{meq}}{\text{kg}}\right) = (\text{S} - \text{B}) \times \text{W} \times \text{N} \qquad (2)$$

Where: B is the volume of sodium thio-sulphate used for the blank, W is the weight of the sample, S is the volume of sodium thio-sulphate consumed by the sample oil, and N is the normality of the standard sodium thio-sulphate [7-8-9].

Acidity Value (AV):

The acidity value was determined using the volumetric titration method. The olive oil was dissolved in (100 mL) of a boilingethanol at 80 C◦ and titrated with NaOH (0.1 N). The AVwas calculated using the equation (3):

$$AV = 0.5 \times 56.1 \times C \times \frac{v}{m} \quad (3)$$

Where: C is the concentration of NaOH (mg/L), v is the volume of NaOH used (mL), 56.**1** (g/mol) is the molar mass of potassium hydroxide, and m is the sample weight (g) [9-10].

Iron and Copper Content:

A 5 g sample was burned in an oven at 550°C for 4 hours. Then, 5 ml of concentrated HNO_3 was added to digest the sample. After filtration, distilled water was added to reach pH 7, and the sample was transferred to a plants instrument.

Results and Discussion:

Acidity Value (AV):

Figure 2 illustrates significant differences in AV among the oils obtained from different regions, namely the NM (GR &ZT) and the AJ (AZ). It was observed that as the elevation above sea level decreased, the AV increased. This finding aligns with previous research [11] that indicates a relationship between elevation above sea level and phenol content, as well as the structure and properties of fruits. Furthermore, the researchers noted that the AV increased proportionally with the degree of fruit maturity towards the end of the harvest season. The ZT region demonstrated the most notable AV at 3.03%, while the AZ region followed closely at 1.91%. On the other hand, the GR area recorded the lowest AV of 1.62%.
Moreover, significant differences were observed between the AV of oils obtained from fruits collected at the beginning and end of the harvest season. The AV for the GR region at the beginning of the season was 0.28%, which increased to 1.62% by the end of the

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season. This increase in AV aligns with previous studies [12-13] that indicate the percentage of free acidity increases as fruit maturity progresses. These findings are also consistent with the results reported by [11-14], which suggest that olive fruits at later stages of maturity yield oils with higher levels of AV due to increased enzymatic activity, particularly by lipolytic enzymes.

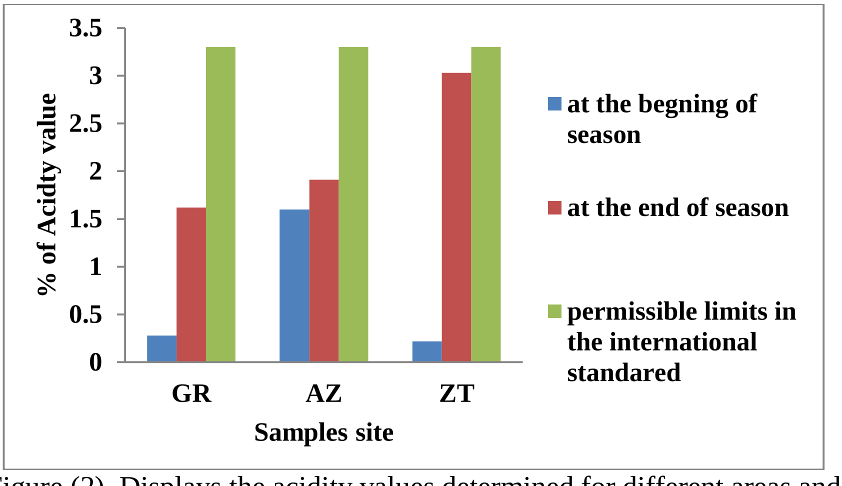

Figure (2). Displays the acidity values determined for different areas and the corresponding time of pressing the olive fruits: Gharyan (GR); Al-azizyah (AZ); Al-Zentan (ZT).

Peroxide Value:

The results shown in Figure (3) revealed that the peroxide values were lower in the mountain areas (GR and ZT) compared to the coastal areas (AZ). The lowest peroxide value of 3.97 mEq/kg was recorded at the beginning of the harvest season in the ZT region, followed by GR with a peroxide value of 4.20 mEq/kg. The highest peroxide value was observed in the AZ region during the harvest shift, reaching 6.70 mEq/kg. These findings support the notion [11] that the peroxide value is influenced by the region's topography and the quality of agricultural soil.

Regarding the harvest timing, the research noted an increase in the peroxide value with delayed harvesting of the olive fruits. The highest value of 13.50 mEq/kg was recorded at the end of the harvest

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season in the ZT region. This finding aligns with the results of [11], which indicated that delaying the harvest and increased fruit maturity lead to higher percentages of free acids and peroxide. Additionally, these results were consistent with the data reported by [13], which found that advanced stages of fruit maturity corresponded to lower peroxide values due to decreased lipoxygenase enzyme activity. The peroxide values obtained in this study were in compliance with the specifications set by the International Olive Council [6], which stipulates that extra virgin olive oil must not exceed 20 mill equivalents of oxygen equivalent per kilogram. Therefore, based on these results, the sampled oil can be classified as extra virgin olive oil suitable for human consumption.

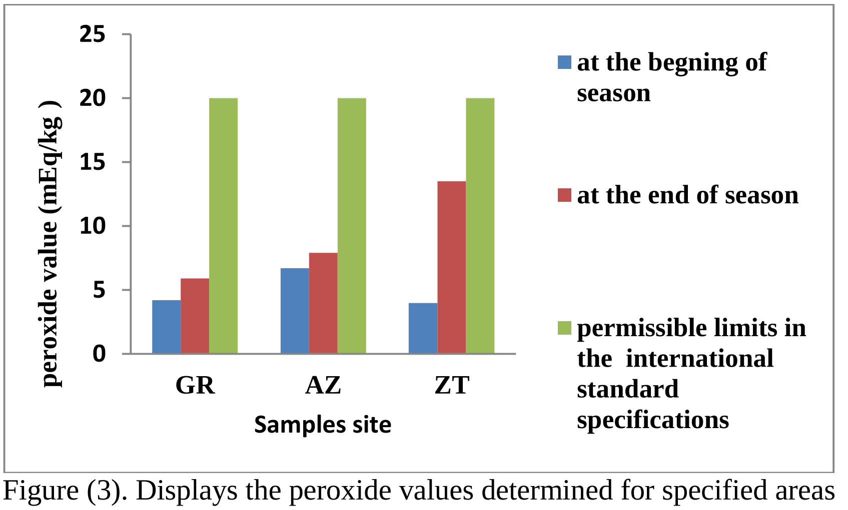

Figure (3). Displays the peroxide values determined for specified areas and the corresponding time of olive fruit pressing; Gharyan (GR); Al-azizyah (AZ); Al-Zentan (ZT).

Refractive Index:

Figure4 illustrates that there were no significant differences in the refractive index among olive oil samples at various stages of maturity and across different regions, including the MN and AJ Plain areas. The refractive index values ranged from 1.4672 to 1.4700 for different ripening timings and regions.In general, the refractive index values obtained from the samples of olive oil fall within the internationally specified range of 1.4677 to 1.4705 for extra virgin olive oil. Consequently, based on these values, the sampled olive oil can be classified as extra virgin olive oil according to international standards.

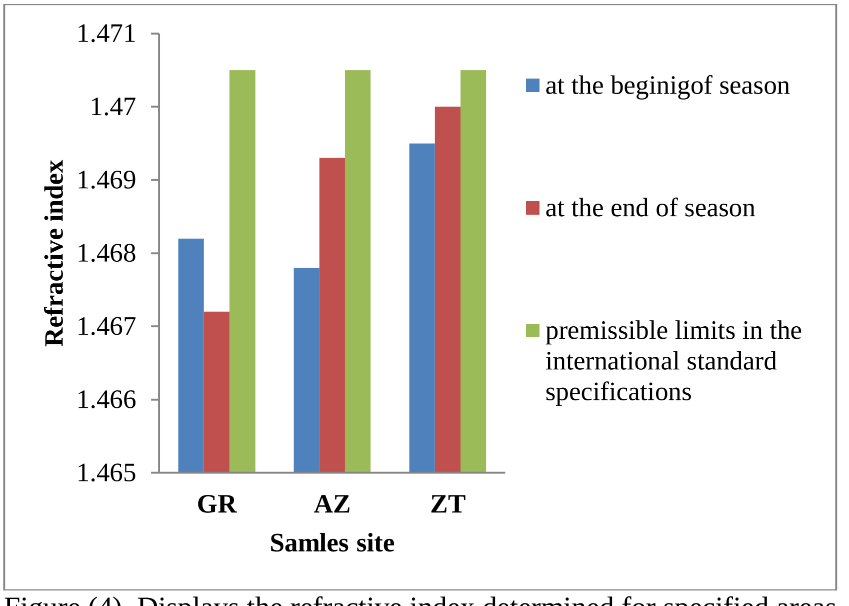

Figure (4). Displays the refractive index determined for specified areas and the corresponding time of olive fruit pressing; Gharyan (GR); Al-azizyah (AZ); Al-Zentan (ZT).

Density:

Figure 5 reveals that there were no significant differences in density between the regions, specifically the areas of NM. and the AJ. The density values ranged from 0.87 to 0.90 kg/m^3 at the beginning of the harvest season. By the end of the season, the density values were consistent across all three regions, ranging from 0.91 to 0.92 kg/m^3. These density values fall within the range specified by international standards for excellent virgin olive oil, indicating that the sampled oil is suitable for human consumption according to international standard specifications.

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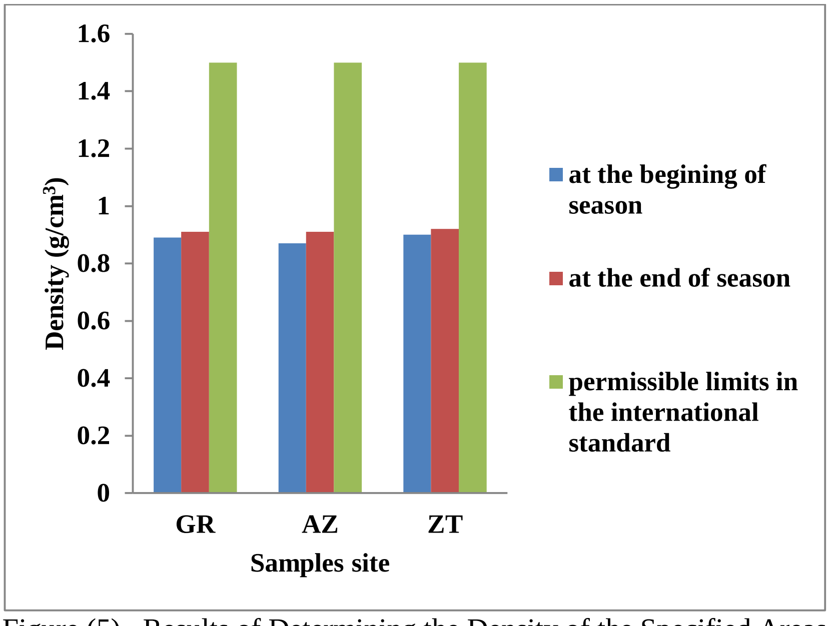

Figure (5). Results of Determining the Density of the Specified Areas and the Time of Pressing Olive Fruits; Gharyan (GR); Al-azizyah (AZ); Al-Zentan (ZT).

Heavy Metals (Iron and Copper):

Table 3 provides insights into the heavy metals content, specifically copper and iron, in the olive oil samples. The researchers observed a slight increase in the content of copper and iron towards the end of the harvest season, but no significant differences were found between the regions. This minor increase can be attributed to the accumulation properties of metals, where their concentration tends to rise as olives ripen. Overall, the percentage of metals remained within the limits and specifications set by international standards. The permitted value for copper is 0.10 %, while for iron, it is 3.00 %. Consequently, the sampled oil is classified as excellent olive oil suitable for human consumption.

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Table 3: Results of Determining the Heavy Metals (Copper and Iron) for the Specified Areas and the Time of Pressing Olive Fruits. Gharyan (GR); Al-azizyah (AZ); Al-Zentan (ZT).

Samples	% of Iron		% of Copper	
	Beginning of the season (October)	End of the season (February)	Beginning of the season (October)	End of the season (February)
GR	0.0	0.15	0.0	0.001
AZ	0.0	0.20	0.0	0.003
ZT	0.0	0.15	0.0	0.001

Absorbance and Wavelength:

Table 4 presents the results of the absorbance at various wavelengths. It is evident that there are no significant differences in the delta (k) values (absorbance at the specific wavelength) among the three mentioned regions. The AZ and ZT regions exhibited a value of 0.001, while the GR region had a slightly higher value of 0.005. Notably, there were no variations in the absorbance values across different ripening dates. The absorbance value remained consistent at the beginning and end of the season for all three regions. Moreover, these values were within the permissible limits defined by international standard specifications, with an absorbance value of less than 0.25

Table 4: Results of Determining the Absorbance (Wavelength) of the Specified Areas and the Time of Pressing Olive Fruits. Gharyan (GR); Al-azizyah (AZ); Al-Zentan (ZT).

Sample name	Beginning of the Season (October)	End of the Season (February)
GR	0.005	0.005
AZ	0.001	0.001
ZT	0.001	0.001

Conclusion:

- Olive oil samples from different olive growing areas in the NM region (GR and ZT) and irrigated olive growing areas (AJ Plain including AZ) were analyzed at the beginning and end of the harvesting season.

- The analysis revealed significant differences in peroxide value and acidity level of the oil.
- These differences can be attributed to factors such as harvest timing, geographic location, and irrigation system.
- All the values obtained from the analysis fall within the permissible limits outlined by international standard specifications.
- To ensure the production of premium extra virgin olive oil, the study recommends early harvesting of olives and immediate pressing.
- The oil samples from the NM areas (GR-ZT) showed high quality and value.
- The results indicate that all olive oil samples from the 2022-2023 production are suitable for consumption.
- Libyan olive oil production is competitive with oils produced in neighboring countries.
- Future policies involving exporting Libyan olive oil to foreign markets may be feasible.

Recommendations:

Future policies involving exporting Libyan olive oil to foreign markets may be feasible.

Despite the possibility of unintentional sample contamination and limitations in relying solely on peroxide value and acidity level as quality indicators, implementing strict protocols to minimize contamination risks and incorporating additional quality parameters, such as sensory evaluation and analysis of specific chemical compounds, can provide a more comprehensive understanding of the oil's characteristics. Moreover, it is important to note that despite the occurrence of these technical problems, the analysis results fall within permissible limits outlined by international standards, suggesting that any encountered technical issues did not significantly affect the study's overall findings and conclusions.

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References

[1]. Kiritsakis, A., Kanavouras, A., &Kiritsakis, K. (2002). Chemical analysis, quality control and packaging issues of olive oil. European Journal of Lipid Science and Technology, 104(9-10), 628- 638.

[2]. Korkmaz, A. (2023). Characterization and Comparison of Extra Virgin Olive Oils of Turkish Olive Cultivars. Molecules, 28(3), 1483.

[3].Usanmaz, S., Öztürkler, F., Helvaci, M., Turgut, A. L. A. S., Kahramanoğlu, I., &Aşkin, M. A. (2018). Effects of periods and altitudes on the phenolic compounds and oil contents of olives, cv. ayvalik. International Journal of Agriculture Forestry and Life Sciences, 2(2), 32-39.

[4]. Elmagassabi, S. A., & Bader, N. R. (2014). Chromatographic Analysis of some Constituents of Libyan Olive Oil (Doctoral dissertation, University of Benghazi).

[5]. Chtourou, F., Valli, E., Ben Mansour, A., Bendini, A., Gallina Toschi, T., & Bouaziz, M. (2021). Characterization of virgin olive oils obtained from minor Tunisian varieties for their valorization. Journal of Food Measurement and Characterization, 15, 5060-5070.

[6]. Abdul Basit Mohammed Mahmoud and Khaled Ramadan Al-Obaidi (2022) . The Sixth International Scientific Conference of the Faculty of Economics Alkoms, (52-63).

[7]. AOAC. (2010). Official Methods of Analysis (15th ed.). Association of Official Analytical Chemists: Washington, DC.

[8]. Zahir, E., Saeed, R., Hameed, M. A., & Yousuf, A. (2017). Study of physicochemical properties of edible oil and evaluation of frying oil quality by Fourier Transform-Infrared (FT-IR) Spectroscopy. Arabian Journal of Chemistry, 10, S3870-S3876.

[9]. Ghreishi Rad, S. A., Jalili, M., Ansari, F., Rashidi Nodeh, H., &Rashidi, L. (2023). Maturity impact on physicochemical composition and polyphenol properties of extra virgin olive oils obtained from Manzanilla, Arbequina, and Koroneiki varieties in Iran. Food Science & Nutrition.

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[10]. ISO 660. (2020). Animal and vegetable fats and oils - Determination of acid value and acidity, 2020. Retrieved fromhttps://www.iso.org

[11]. Mohamed Mousa, Y., Gerasopoulos, D., Metzidakis, I., &Kiritsakis, A. (1996). Effect of altitude on fruit and oil quality characteristics of 'Mastoides' olives. Journal of the Science of Food and Agriculture, 71(3), 345-350.

[12]. Lü, X., Ma, J., Yan, H., Yang, L., Ren, X., Guo, J &Deng, Y. (2021). Effect of degree of ripening on the quality of virgin olive oils produced in Longnan, China. Journal of the American Oil Chemists' Society, 98(11), 1045-1056.

[13]. Jiménez, B., Sánchez-Ortiz, A., Lorenzo, M. L., & Rivas, A. (2013). Influence of fruit ripening on agronomic parameters, quality indices, sensory attributes and phenolic compounds of Picudo olive oils. Food Research International, 54(2), 1860-1867.

[14].Gagour, J., El Ghailassi, K., Ibourki, M., &Gharby, S. (2024). Physicochemical traits of olive fruit and oil from eight Moroccan wild olive (Olea europaea L. Subsp. Oleaster) populations. Biocatalysis and Agricultural Biotechnology, 103021. .