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Influence of extraction systems on oil yield, wastes and olive oil properties

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Abstract

The effect of extraction systems (Continuous Process Three-Phases and press process) on quality properties of olive oil extracted from picual variety was studied. The oil yield, flow rate, acid value, peroxide value, refractive index, saponification value, moisture and volatile matter, fatty acid composition and specific coefficients of extinctions (K232, K270 and ΔK) were determined. The CP3P extraction system produced a high olive oil yield. According to the results that obtained, it could be observed that there were no marked differences between both extraction systems in view of oil values such as acid value, peroxide value and refractive index which showed almost the same results. Meanwhile, also the results showed a slight difference in fatty acids composition, relying on the extraction systems were used. the olive oil produced from the CP3P has high quality than the olive oil that produced from the PP extraction system. Olive pomace produced from PP extraction methods contains high natural antioxidants.

Keywords: Olive oil; Extraction systems; Press process and Three-phase; Fatty acids; specific coefficients of extinctions.

Introduction

Olive trees (*Olea europaea L.*) are mainly grow in different zone of the world, the Mediterranean region is considered the main habitat for cultivating olive trees and the major crop production part, this region's olive production is 98% of world production (**Di Giovacchino et al., 2002**).

There are several factors that affecting on olive oil chemical composition could be divided into four groups: technological factors (post-harvest storage, extraction system, oil storage conditions), cultivation (harvesting, ripeness), agronomic (irrigation, fertilization), and environmental (soil, climate) , These four factors actually affect the physicochemical quality, the organoleptic properties and the chemical composition of the extracted extra virgin olive oils **(El-Gharbi et al., 2018**).

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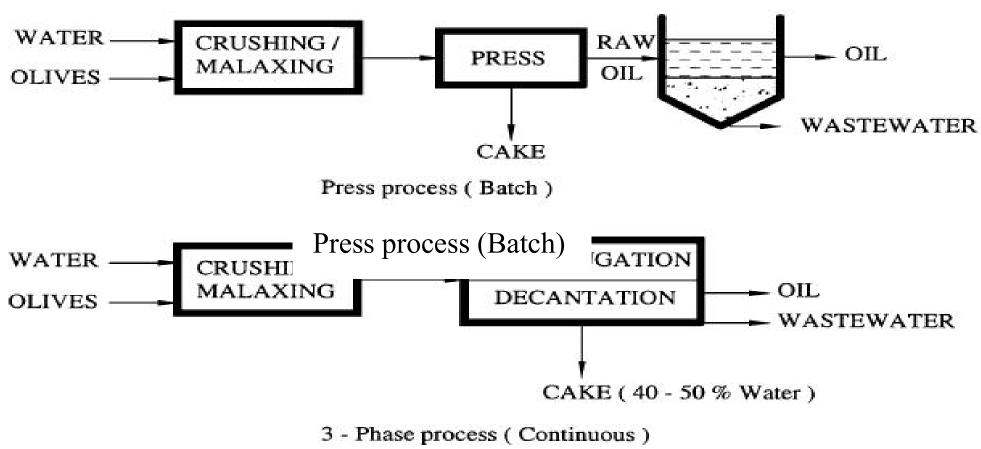
The virgin olive oil (VOO) was produced from ripening, healthy and fresh olive fruits (*Olea europaea L.*) utilizing by physical (Continues system) a **et al.,** nd mechanical (Discontinues system) processes (Criado-Navarro et al., 2021). Olive oil, its major derivative product, has experienced a rise in its popularity because of its organoleptic properties and its related to the beneficial health effects (**Oliveras-López et al., 2014**).

Olive oil is defined as the oil produced from olive fruit, and its production operations involved many steps as illustrated in (**Fig. 1**). The extraction methods of olive oil are realized through the traditional discontinuous system (pressing) or continuous system (centrifuging) processes in traditional (press) mills or in modern units (decanter) (**Dermeche et al., 2013**).

The mechanical oil extraction affects formation of volatile compounds and the release of phenolic antioxidants, which greatly influence the quality of virgin olive oil. During mechanical extraction of virgin olive oil, the olive paste for**et al.,**med after the fruit has been crushed, is mixed in a process called malaxation (**Kalua et al., 2006**).

the olive oil could be extracted by oldest extraction method such as press process to obtain the olive oil, but this method has disadvantages like discontinuous, high-cost labor and oil contamination diaphragms. this technique does not require addition water to olive paste. Meanwhile, the three phase continuous needs to adding water to dilute the water-soluble components. That made the separation olive paste into three phases: wastewater, pomace and oil (**Abd El-Hamied et al., 2019**)

The main aim of this study is to investigate the influence of extraction methods on quality characteristics of extra virgin olive oil.



Three phase process (Continuous)

Figure 1. The different olive oil extraction systems

Materials and Methods

Olive samples:

This investigation was conducted on olive trees (Olea europaea) of Picual cultivar harvested at season 2019-2020. Olive fruit samples were purchased from local market (Fayoum governorate, Egypt).

Reagents and Chemicals:

Sodium hydroxide, sodium carbonate, sodium thiosulphate, potassium hydroxide, potassium iodide, iodine, thiobarbituric acid (TBA) were purchased from El-Gomhoria Company, Egypt.

All solvents used in this study (methanol, ethanol, hexane, petroleum ether, glacial acetic acid and chloroform) were obtained from El-Gomhoria Company, Egypt.

Determination of flow rate:

The flow time of the studied olive oils were measured according to the method reported by (**Joslyn, 1950**). Flow time was measured as the time in second required to discharge 5 ml oil at a temperature (40°C).

Determination of peroxide value:

Peroxide value (Recommended Practice Ca 5a-40) of olive oil were determined according to (AOCS, 2017c).

Saponification value:

The Saponification value for olive oil samples were determined according to the method described by (**A.O.A.C., 2000**)

Refractive index:

A Carl Zeiss refractometer was used for measuring the refractive index of olive oil and the results were standardized to 25ºC (AOCS, 2017d**).**

Moisture and volatile content:

The moisture content and the volatile content for olive oil were determined according to the method described by (**AOCS 2017b**).

Insoluble impurities:

The insoluble impurities is measured by using a light petroleum according to method that described by **AOCS, 2017c and ISO, 2017.**

The K and ΔK measurements:

The approximately 0.1 g of olive oil were dissolved in 25 ml iso-octane. UV absorbance was measured at 232, 266, 270 and 274 nm. ΔK values were calculated by using the following equation: $\Delta K = K270 - [(K266 + K274) / 2]$ according to the standard method of (IOC, 2019).

Gas chromatography–mass spectrometry analysis (GC-MS) for FAME:

Fifteen mg of olive oil samples were mixed with One mL of n-hexane then vortexed for 30 sec. after that 1 mL of sodium methoxide was added (0.4 mol). The mixtures have been vortexed for 30 sec. and were permitted to settle for fifteen minutes. The top layer phase have the fatty acids methyl esters (FAMEs) was recovered and analyzed by (GC-MS).

The GC-MS system (Agilent Technologies) was equipped with gas chromatograph (7890B) and mass spectrometer detector (5977A) at Central Laboratories Network, National Research Centre, Cairo, Egypt. The GC was equipped with DB-WAX column (30 m x 250 µm internal diameter and 0.25 μm film thickness). Analyses were carried out using hydrogen as the carrier gas at a flow rate of 1.9 ml/min at a split 1:20, injection volume of 1 µl and the following temperature program: 50 °C for 1 min; rising at 25 °C /min to 200 °C and held for 5 min; rising at 3 °C/min to 220 °C and held for 10 min ; rising at 5 °C/min to 240 °C and held for 8 min. The injector and detector were held at 250 °C and 290 °C, respectively. Mass spectra were obtained by electron ionization (EI) at 70 eV and using a spectral range of m/z 60-400 and solvent delay 6 min. Identification of different constituents was determined by comparing the spectrum fragmentation pattern with those stored in Wiley and NIST Mass Spectral Library data.

Results and Discussion

Effect of different extraction methods on Olive oil yields and mill wastes:

According to the results shown in **Table 1**, the amount of olive oil produced from CP3P extraction method was higher than that of PP extraction method. Where amount of olive oil was 13.35±0.15 and 11.75±0.25 for CP3P and PP methods respectively.

The results obtained in **Table 1** also indicated that the recovery percent of olive oil was 81.25 ±0.00 and 78.12±0.00 for CP3P and PP methods respectively. We can conclude that the CP3P method is better than the PP method because CP3P contains a malaxation step which increase the oil yield. The malaxation is an important step in olive oil extraction methods.

Table 1 : The percentage of oil, olive mill wastes and oil recovery from olive fruits by using different extraction methods.

Parameters (%)	Continuous Process Three-Phases [CP3P]	Press Process [PP]
Oil yield	13.35±0.15	11.75±0.25
olive oil mill wastewaters (OMWW)	54.45±0.15	53.25±0.45
Cake	32.20±0.00	35.00±0.20
Olive oil recovery	81.25 ±0.00	78.12±0.00

Each value represents mean ± S.D of three repeated

The percentage of OMWW as shown in **Table 1** were 54.45±0.15 and 53.25±0.45 for CP3P and PP methods respectively. The high percent of CP3P method may be due to the large amount of water used during production in a comparison with PP method. In spite of, the CP3P method produces a high oil yield it is producing a large amount of OMWW which should be treated before drawn to the sewers.

In the opposite direction, the amount of olive pomace for PP method is higher than that obtained by the CP3P method. It may be due to that the high content oil and water in pomace which produced by PP method.

Physiochemical properties of olive oil:

The flow rate of olive oil:

The results found in **Table 2** show that the flow rate of olive oil was 4.709±0.78s and 4.688±.035s for CP3P and PP methods respectively. The outcomes obtained in our investigation are comparable to that reported by (**Siddiqui and Ahmed, 2013**).

Acid value of olive oils:

The acid value (AV) is an indicator of hydrolytic rancidity, which may occur enzymatically or non-enzymatically at high temperatures, producing free fatty acids.

The results in **Table 2** showed that the acid value and acidity (percent as oleic acid) were (0.56±0.005, 0.283±0.002) and (0.60±0.01, 0.302±0.005) for CP3P and PP methods respectively. The oil produced by two methods considered as extra virgin olive oil because the acidity percent is ≤0.80 (Stan & Stan, 2015).

Table 2 : The effect of different extraction methods on some physicochemical properties of olive oils.

Parameters	Methods	
	PP	CP3P
Flow rate	4.688±.035	4.709±0.78
Acid value (A.V)	0.60±0.01	0.56±0.005
Acidity (Oleic acid)	0.302±0.005	0.283±0.002
Peroxide value	nil	nil
Refractive Index (RI) (25 °C)	1.470±0.00	1.470±0.00
Saponification value	191.47±12.79	197±2.86
%Non-soluble matter	1.402±.064	2.239±0.477
Moisture and volatile matter	0.2 ±0.05	0.1 ±0.02

Each value represents mean ± S.D of three repeated.

Peroxide value of olive oil samples:

The peroxide value (PV) is one of the most important chemical values as it can be used to determine degree of deterioration of oils. (**O'Keefe & Pike, 2010**). Both oils produced from CP3P and PP methods are free from the peroxide as shown in **Table 2**.

Results in **Table 2** shows there is no significant differences in peroxide value between different production methods. Our results are an agreement with that mentioned by Egyptian standard for olive oil stated that the maximum peroxide values for extra-virgin olive oil ≤20 meq O_2/kg^{-1}.

Refractive index of olive oil samples:

The refractive index of aqueous solutions and oil is played a fundamental role and crucial importance in detection of purity and adulteration of oils **(Yunus, et al., 2009)**.

Table 2 illustrated that the average outcomes of the refractive index have been obtained by refractometer, the RI values for PP and CP3P were given the same value (1.470±0.00) at 25 °C. Similar results were found by (**Bahti, 2015**).

Saponification value of olive oil samples:

The saponification values are the evaluation of the mean molecular weight of the triacylglycerol in an olive oil samples **(Lotfy, et al., 2015)**.

The saponification numbers of olive oil samples were given in **Table 2** The results demonstrated that the saponification value of olive oil produced from CP3P extraction method was **(**197±2.86 mg KOH g^{-1}) and olive oil produced from PP extraction method was **(**191.47±12.79 mg KOH g^{-1}). There are no marked differences in saponification values for olive oil produced from both extraction methods. Our results are agreement with that mentioned by **(Parthasarathy, et al., 2015)** who reported that the saponification value of the olive oil is 187-196.

Insoluble impurities values of olive oil samples:

The definition of insoluble impurities content is a quite popular investigation implement on virgin olive oils that is generally requested together with the calculation of the moisture content of the olive oil sample, the olive oil includes high levels of insoluble impurities and moisture, the value of olive oil paid is decreased correspondingly (**Gila, et al., 2020**).

The insoluble impurities of olive oil samples measured, and the obtained results presented in **Table 2**. The Insoluble impurities are diverse with the extraction methods of olive oil, for CP3P method shown the high value of insoluble impurities with value of 2.239±0.477, while for the PP method was 1.402±.064. The conclusion of our results ensures the produced olive oil from both contains insoluble impurities higher than that reported by Egyptian standard for olive oil and IOC for extra virgin olive oil. The oil produced by two methods should be decanter before send to the markets.

Moisture and volatile matter

Results stated in **Table 2** indicated that olive oil produced from both extraction methods has moisture and volatile matter less than 0.2. The results agree with that reported by IOC for extra virgin olive oil. The results also indicated that olive oil produced from CP3P extraction method contains 0.1% moisture and volatile matter on the other hand the olive oil produced from the PP extraction method has 0.2 % moisture and volatile matter. We can conclude that the olive oil produced from the PP extraction method may be contains a high amount of moisture and this may be due to there is no efficient system for removing water from the oil.

These results is in agreement with that reported by IOC (**IOC, 2019**) who stated that the moisture and volatile matter for extra virgin olive oil should not exceed ≤0.2%.

Quality characteristics of olive oil samples:

The quality of the olive oil evaluated by measuring the characteristics of the absorption bands between 200 and 300 nm. These are frequencies related to conjugated diene and triene systems. A low absorption in this region is indicative of a high-quality extra virgin olive oil, whereas adulterated/refined oils show a greater level of absorption in this region (**Houshia *et al.*, 2014**).

Table 3 : Effect of extraction methods on quality characteristics of olive oil

Extraction methods	Parameters		
	K 232	K270	ΔK
Olive oil from CP3P	1.66 ±0.15	0.13 ±0.01	0.004 ±0.00
Olive oil from PP	1.90 ±0.17	0.15 ±0.02	0.004 ±0.00

Each value represents mean ± S.D of three repeated.

K 232 K 270 and ΔK for olive oil produced from both extraction methods were determined and illustrated in **Table 3**. The quality parameters for olive oil produced from two extraction methods less that that stated by IOC for extra virgin olive oil (**IOC, 2021**).

There is a small difference in K 232 and K 270 between the oil produced from both methods. Olive oil produced from CP3P extraction method has low value K 232 and K 270 this indicate that CP3P extraction method produced olive oil with high quality characteristics in comparison with that produced by PP extraction method.

Evaluation of antioxidant activity (DPPH˙) radical-scavenging activity of olive pomace:

The outcomes of scavenging properties of the OPPM and OPCM extracts are illustrated in **Fig. 2**. It could be observed that the increasing of concentrations of all tested extracts cause increasing inhibition ratio. The 62.5 $\mu L/ml^{-1}$ concentration caused inhibition% 25.31% for OPCM extract and 35.49% for OPPM extract. The OPPM extract showed relatively high scavenging activity than OPCM, that is mean pomace produced from PP extraction methods contains more natural antioxidant than the pomace produced from CP3P extraction method.

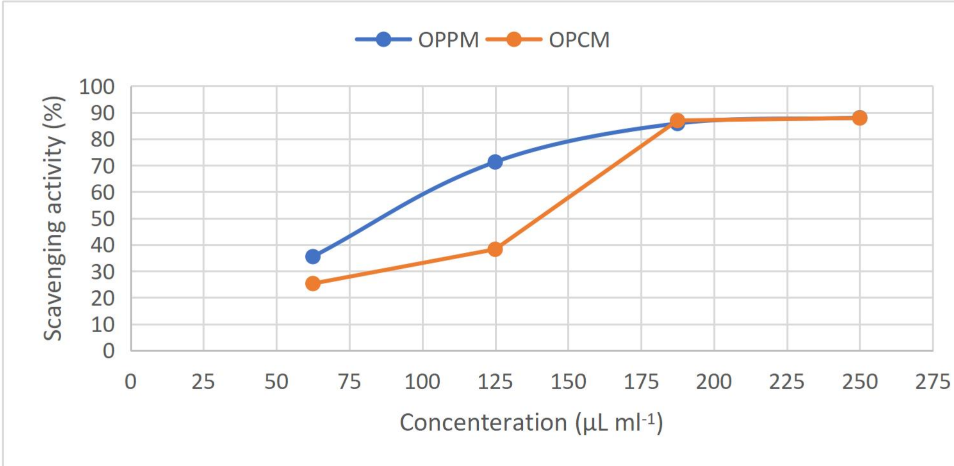


Fig. 2: Scavenging activity of OPPM and OPCM against DPPH radicals at different concentrations.

As shown in **Table 4** and **Fig. 2**, The results showed that OPPM has a high scavenging activity and recorded IC_{50} values of 0.83 µg/µL DPPH$^{\bullet}$, at the same time it has antiradical efficiency values of 1.2048. The outcomes also showed that OPCM extract was the lower scavenging antioxidant activity with IC_{50} value of 1.30µg/µL DPPH$^{\bullet}$. The strong scavenging activity can be connected to the high content of the antioxidant compounds in OPPM (Leouifoudi et al., 2014).

Table (4): The influence of different extracts from olive pomace on IC_{50} and Antiradical efficiency by DPPH*.

Sample	IC_{50} µg/µl DPPH$^{\bullet}$	Antiradical efficiency
OPCM	1.30	0.7692
OPPM	0.83	1.2048

OPC = pomace from CP3P, OPP = pomace from PP, OL = olive leaf For USAE and CE extraction systems. While (M) stands for a methanol solvent (80%).

Influence of different extraction methods on fatty acid composition of olive oils:

The major composition of edible vegetable oils is triacylglycerol, which consists of different fatty acids. The extra virgin olive oil has high content of monounsaturated fatty acids, mainly oleic acid, which ranges from 56% to 84%. The high percentage of oleic acid is an important component of the nutritional profile of olive oils, thus showing great importance, particularly in relation to its effect on cardiovascular system health.

The outcomes of fatty acid composition have listed in **Table 5**. The fatty acid composition affected by the extraction method. The results indicated that the main fatty acid composition of olive oil extracted by CP3P and PP extraction methods was oleic acid (74.11-79.19%), palmitic (13.9-9.56%), linoleic (6.94-8.30%), stearic (1.56-2.36%), and palmitoleic (0.75-1.66%), respectively. The olive oil extracted by CP3P is lower in oleic content (74.11%) than the olive oil extracted by PP (79.19%). These outcomes are in agreement with that reported by **(El-Gharbi et al., 2018; Abd El-Hamied W.A., et al., 2019)**. On the other hand, olive oil extracted by CP3P contains a high percent of palmitic acid (13.9%) in comparison with that extracted by the PP method (9.56%).

There are many factors affecting the fatty acid composition of olive oil such as the olive species, location, climatic conditions, irrigation management, and ripeness stage of the fruit (**Bozdogan Konuskan & Canbas, 2014; Bozdogan Konuskan & Mungan, 2016**).

Table (5): Effect of different extraction methods on fatty acid composition of virgin olive oil*.

Compounds	P3CP	PP	Allowable range 2022((IOC%
C Palmitic 16	13.9	9.56	20.00-7.50
C Palmitoleic 16:1	1.66	0.75	3.50- 0.30
C Stearic 18	2.36	1.56	5.00-0.50
C cis Oleic 9n 18:19	74.11	79.19	83.00-55.00
cisLinoleic 6n 18:2 C	6.94	8.30	21.00- 2.50
C Linolenic 3n 18:3	0.37	0.50	1 ≥
arachidic C20	0.3	nd	-
Eicosatetraenoic 5,8,11,14-n 20: 4	nd	0.14	-

* The cultivar is Picual

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he extraction methods used in this study produced virgin olive oil depending on the acidity which mentioned above and the IOC regulations **(IOC, 2021**).

Conclusion

Vegetable oils are the main source of dietary fat and have important functional and sensory roles in foods. It could be concluded that the olive oil produced from the CP3P has high quality than the olive oil that produced from the PP extraction method, wherefrom high chemical properties and shelf life. The extract of olive pomace produced from the PP extraction method could be used as safe natural antioxidants in food applications.

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تأثير طرق الأستخلاص على كمية الزيت والمخلفات وخصائص زيت الزيتون

محمد محمود شعبان حسن، عادل عبد الرازق عبد العظيم مهدلي، نادي عبد العزيز عبد العظيم النعيري، عوض عبد التواب محمود عوض

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تمت دراسة تأثير طرق الاستخلاص (طريقة الأستخلاص المستمرة ثلاث مراحل و طريقة الأستخلاص التقليدية بالعصر) على خصائص جودة زيت الزيتون المستخرج من الصنف البكوال المنتج في محافظة الفيوم. تم تقدير كمية الزيت الناتجة ، معدل الأنسياب كدلالة للزوجة ، رقم الحموضة، رقم البيروكسيد، معامل الانكسار، رقم التصبن، الرطوبة والمواد المتطايرة وتركيب الأحماض الدهنية الموجودة في عينات الزيت الناتج وقياسات الجودة النوعيةK270 ، K232) وΔK). أنتجت طريقة الأستخلاص المستمرة ثلاث مراحل كمية زيت اكبر من الناتجة من الطريقة التقليدية. وبناءً على النتائج التي تم الحصول عليها ، يمكن ملاحظة عدم وجود فروق بين طريقتي الاستخلاص في الثوابت الطبيعية والكيميائية للزيت. وفي الوقت نفسه، أظهرت النتائج اختلافًا طفيفًا في تركيب الأحماض الدهنية للزيت الناتج من طريقتي الاستخلاص. زيت الزيتون المنتج من بطريقة الأستخلاص المستمرة ثلاث مراحل له جودة عالية عن زيت الزيتون المنتج من طريقة استخلاص التقليدية بالعصر بناءً علي قياسات الجودة النوعية. يحتوي تفل الزيتون المنتج من طريقة استخلاص التقليدية على مضادات أكسدة طبيعية عالية عن الناتج من طريقة الأستخلاص المستمرة ثلاث مراحل.