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Traditional vs. Modern Olive Oil Extraction in Libya: A Comparative Study of Fatty Acids and Heavy Metal Contamination

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استخلاص زيت الزيتون التقليدي مقابل الحديث في ليبيا: دراسة مقارنة للتلوث بالأحماض الدهنية والمعادن الثقيلة

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Abstract:
Olive oil is a vital agricultural product in west of Libya and a core component of the Mediterranean diet. Its quality largely depends on the extraction method. This study aims to compare traditional (manual pressing) and modern (centrifugation) extraction methods by analyzing the physicochemical properties, fatty acid profiles, and heavy metal content in both olive oil and olive mill wastewater (OMWW). Samples were collected from traditional and modern oil mills in Libya. Fatty acid composition was determined using GC-FID, and heavy metals (Pb, Cd, As, Hg) were analyzed using ICP-OES. Results showed that olive oil from modern extraction had a higher oleic acid content (59.01%) than traditional oil (57.03%), indicating better oxidative stability. The saponification value was also higher in modern oil, reflecting improved purity. All oil and OMWW samples had cadmium levels below 0.1 ppm, while lead, mercury, and arsenic were undetected, suggesting good safety standards. OMWW samples from traditional extraction showed higher turbidity and coloration, potentially indicating greater organic and metal retention. These findings support the environmental and nutritional advantages of modern extraction techniques. The study recommends encouraging modern methods in Libyan olive oil production, improving waste treatment in traditional mills, and regularly monitoring product safety.

Keywords: Olive oil, Libya, Extraction method, Fatty acids, Heavy metals, OMWW.

الملخص
يُعتبر زيت الزيتون من المنتجات الزراعية الحيوية في غرب ليبيا ومن المكونات الأساسية في النظام الغذائي المتوسطي، حيث تعتمد جودته على طريقة الاستخلاص المُستخدمة. تهدف هذه الدراسة إلى مقارنة طريقتين شائعتين في استخلاص زيت الزيتون: الطريقة التقليدية (الضغط اليدوي أو الشوامي) والطريقة الحديثة (الطرد المركزي)، من خلال تقييم الخصائص الفيزيائية والكيميائية، تركيب الأحماض الدهنية، ومحتوى المعادن الثقيلة في كل من الزيت والمياه الناتجة عن العصر) مياه المرجين أو (OMWW). تم جمع عينات من معاصر تقليدية وحديثة في ليبيا، وتم تحليلها باستخدام تقنيات قياسية مثل ICP-OES لتحليل المعادن الثقيلة، و GC-FIDلتحليل الأحماض الدهنية. أظهرت النتائج أن الزيت الناتج عن المعاصر الحديثة يحتوي على نسبة أعلى من حمض الأوليك (59.01%) مقارنة بالتقليدي (57.03%)، وهو مؤشر

على الجودة العالية والثبات التأكسدي. كما كانت قيمة التصبن أعلى في الزيت الحديث، مما يشير إلى نقاوة أفضل. لوحظ أيضًا أن جميع العينات تحتوي على نسب غير مكتشفة من الرصاص، الزئبق، والزرنيخ، في حين كان الكادميوم أقل من 0.1 جزء في المليون. من جهة أخرى، أظهرت عينات مياه المرجين الناتجة عن المعاصر التقليدية عكارة ولونًا أعلى، مما يشير إلى احتمال احتوائها على مواد عضوية ومعادن أكثر. تشير النتائج إلى أن الطرق الحديثة توفر جودة زيت أفضل وأثر بيئي أقل، وتدعو إلى تعزيز استخدام هذه التقنية وتحسين ممارسات المعالجة في المعاصر التقليدية.

الكلمات المفتاحية: زيت الزيتون، ليبيا، طريقة الاستخلاص، الأحماض الدهنية، المعادن الثقيلة، مياه المرجين.

Introduction

Olive oil, often referred to as “liquid gold,” is a central pillar of the Mediterranean diet and holds a significant socio-economic and cultural role in many Mediterranean countries, including Libya. Beyond its culinary value, olive oil is renowned for its high nutritional and therapeutic properties, primarily due to its favorable fatty acid composition, especially its high content of monounsaturated oleic acid, and the presence of antioxidant compounds such as polyphenols, tocopherols, and phytosterols. These bioactive compounds contribute to numerous health benefits, including cardiovascular protection, anti-inflammatory effects, and cancer risk reduction.

In Libya, olive cultivation and oil production are longstanding traditions, especially in rural and semi-arid regions where olive trees are well adapted to the environment. However, the technological approaches used in oil extraction vary widely. Traditional methods, such as stone mills and manual pressing, remain prevalent in many communities due to their simplicity and low operational costs. In contrast, modern mechanical and centrifugal extraction techniques are increasingly adopted for their ability to increase yield, maintain hygiene, and preserve the biochemical integrity of the oil.

The choice of extraction method significantly affects the final quality of olive oil. Traditional processes often expose the oil to air and water for prolonged periods, potentially leading to oxidation and hydrolytic degradation. Modern systems, especially the two-phase and three-phase decanter centrifugation techniques, aim to minimize such deterioration by optimizing temperature control and reducing exposure to contaminants. However, the environmental implications of these processes must also be considered, particularly regarding the generation and management of olive mill wastewater (OMWW) and solid waste (grignon). These by-products are characterized by high organic loads, acidic pH, and the presence of phytotoxic and recalcitrant compounds such as phenolics and long-chain fatty acids, posing serious environmental challenges if improperly managed.

From a safety standpoint, recent studies have raised concerns about the possible contamination of olive oil and its by-products with heavy metals such as lead (Pb), cadmium (Cd), arsenic (As), and mercury (Hg), which can originate from soil, water, or processing equipment. Advanced analytical techniques such as Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) have enabled more precise detection of these contaminants, supporting quality control efforts and regulatory compliance.

Given these complexities, there is a growing need to evaluate olive oil production comprehensively, not only in terms of yield and sensory properties but also considering chemical safety and environmental sustainability. This study seeks to fill this gap by conducting a comparative analysis of olive oil produced using traditional and modern extraction techniques in Libya. The evaluation will focus on key physicochemical parameters, fatty acid composition, and the presence of heavy metal residues in both the oil and its effluents. This integrated approach will contribute to a better understanding of how extraction technologies impact oil quality and environmental health, ultimately guiding producers and policymakers toward more sustainable and safe practices.

Problem Statement

Despite the widespread adoption of modern extraction technologies, many Libyan producers still rely on traditional presses. However, there is a lack of comparative data on the impact of these methods on oil quality and environmental safety. Specifically, the accumulation of toxic metals in the oil and its by-products may differ between systems, raising concerns for public health and sustainability.

Significance of the Study

This study will provide critical insights into the nutritional quality and safety of olive oil from different processing techniques. Moreover, it will inform producers, consumers, and regulators about the environmental implications of wastewater produced by both methods, supporting sustainable olive oil production practices in Libya.

Objectives

- To compare the fatty acid composition of olive oil extracted by traditional and modern methods.
- To determine heavy metal concentrations (Pb, Cd, As, Hg) in olive oil and OMWW.
- To assess the environmental and health implications of each method.
- To propose recommendations for improving safety and quality in Libyan olive oil production.

Literature Review

Numerous studies have examined the impact of extraction techniques on the physicochemical and nutritional properties of olive oil and their environmental consequences. For instance, Baccouri et al. (2008) emphasized the significance of the oleic/linoleic acid ratio as a crucial indicator of oxidative stability in olive oil, which varies notably with processing methods. Similarly, Servili et al. (2014) investigated the influence of different extraction systems, finding that modern centrifugation methods tend to preserve higher concentrations of phenolic compounds and improve fatty acid profiles compared to traditional press systems.

The chemical composition of olive oil produced by various pressing techniques was further explored by Gómez-Caravaca et al. (2016), who demonstrated clear variations in the lipid and minor component profiles between traditional and modern extractions. In parallel, Benitez-Sánchez et al. (2003) focused on heavy metal accumulation in virgin olive oils, revealing that the presence of metals such as lead and cadmium can be linked to environmental and technological factors.

Environmental aspects of olive oil production have also been a subject of concern. Rico et al. (2007) and Achak et al. (2009) assessed the environmental pollution potential of olive mill wastewater (OMWW), reporting high concentrations of organic load, phenolic compounds, and metals. These findings were supported by Tarchouna et al. (2010), who reported residual contamination in soils and water bodies near olive oil processing plants.

In terms of regional data, Zarrouk et al. (2012) examined Tunisian olive oils and detected varying levels of heavy metals, influenced by both geographic origin and processing methods. A more recent study by Nouioua et al. (2022) confirmed the accumulation of heavy metals in olive mill environments, suggesting the need for routine environmental monitoring.

Finally, García et al. (2015) analyzed the environmental performance of two-phase versus three-phase extraction systems and found that the former generated lower amounts of wastewater and reduced the environmental burden. Together, these studies underscore the need for integrated evaluations that combine quality assessment of olive oil with environmental safety considerations.

Materials and Methods

- Study Area and Sample Collection

This study was conducted in the northwestern region of Libya, where two olive oil extraction facilities were selected to represent both traditional and modern extraction techniques. The **traditional facility** used manual or mechanical pressing without temperature control or centrifugation, while the modern facility employed a three-phase centrifugation system with controlled conditions. Samples of olive oil and olive mill wastewater (OMWW) were collected during the 2023–2024 olive harvesting season. From each facility, three replicate samples were taken for both oil and wastewater to ensure statistical reliability. Olive oil samples were collected in sterilized dark-glass bottles and stored at 4 °C until analysis to prevent oxidation. OMWW samples were collected in polyethylene containers, acidified to pH < 2 using nitric acid for metal preservation, and stored at 4 °C.

- Fatty Acid Composition Analysis

The fatty acid profile of olive oil samples was determined using Gas Chromatography (GC) following the International Olive Council (IOC) method COI/T.20/Doc. No. 33/Rev.2 (2017).

- Preparation of methyl esters (FAMEs): Fatty acids were transesterified to methyl esters using methanolic potassium hydroxide and n-heptane.

Instrument: A gas chromatograph equipped with a flame ionization detector (FID) and a capillary column (e.g., 30 m × 0.25 mm × 0.25 µm) was used.

- Temperature program: Initial oven temperature was set at 180 °C, held for 10 minutes, then raised to 220 °C at a rate of 3 °C/min.
- Carrier gas**:** Nitrogen was used at a constant flow rate of 1 mL/min. Identification and quantification were based on retention times of certified fatty acid methyl ester standards.

Fatty acids analyzed included myristic acid (C14:0), palmitic acid (C16:0), palmitoleic acid (C16:1), stearic acid (C18:0), oleic acid (C18:1), linoleic acid (C18:2), linolenic acid (C18:3), arachidic acid (C20:0), behenic acid (C22:0), and others.

- Heavy Metal Analysis

The concentrations of selected heavy metals (Pb, Cd, As, and Hg) in both olive oil and OMWW were determined using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), in accordance with AOAC Official Method 2015.01 and ISO 11885:2007.

- Sample preparation for OMWW: Samples were filtered, acidified with HNO_3, and digested using microwave-assisted acid digestion (HNO_3 and H_2O_2 mixture).
- Sample preparation for olive oil: Samples were digested using a mixture of nitric acid and hydrogen peroxide, followed by dilution in ultrapure water.

- Instrument parameters: The ICP-OES system was calibrated using multi-element standard solutions, and quality control was ensured with certified reference materials (CRMs). The limits of detection (LOD) were <0.01 ppm for all metals analyzed.

- Data Analysis

All experiments were conducted in triplicate. Data were expressed as means ± standard deviations. Statistical analysis was performed using SPSS v26.0, with one-way ANOVA used to assess differences between extraction methods. A significance level of $p < 0.05$ was considered statistically significant.

Results

Fatty Acid Composition of Olive Oil

The fatty acid profile of olive oil extracted using modern methods revealed a predominance of oleic acid (C18:1) at 57.76%, a value indicative of high oxidative stability and nutritional quality. Palmitic acid (C16:0) was present at 19.07%, slightly higher than optimal values but still within acceptable limits for virgin olive oils. Linoleic acid (C18:2), an essential polyunsaturated fatty acid (PUFA), constituted 16.41%, contributing to the nutritional balance of the oil.

Minor components such as palmitoleic acid (C16:1) (2.46%), stearic acid (C18:0) (2.77%), and linolenic acid (C18:3) (0.83%) were also detected. Other long-chain saturated and monounsaturated fatty acids, including arachidic (C20:0), gadoleic (C20:1), behenic (C22:0), erucic (C22:1), and lignoceric acid (C24:0), were found in trace amounts (<0.5%), consistent with typical olive oil profiles.
Overall, the monounsaturated fatty acid (MUFA) content dominated, primarily due to oleic acid, while saturated fatty acids (SFA) remained at a low and acceptable level, indicating good oil quality from the modern extraction process. The following (Table 1 and Figure 1) show the distribution of saturated, monounsaturated, and polyunsaturated fatty acids in olive oil samples extracted using modern and traditional extraction techniques.

Table 1: Comparison of Fatty Acid Profiles in Olive Oil Extracted by Modern and Traditional Methods.

Fatty Acid	Type	Modern (%)	Traditional (%)
Myristic acid (C14:0)	Saturated (SFA)	0.001	0.02
Palmitic acid (C16:0)	Saturated (SFA)	20.32	16.15
Heptadecanoic acid (C17:0)	Saturated (SFA)	0.03	0.04
Stearic acid (C18:0)	Saturated (SFA)	2.50	3.00
Arachidic acid (C20:0)	Saturated (SFA)	0.27	0.00
Lignoceric acid (C24:0)	Saturated (SFA)	0.01	0.05
Palmitoleic acid (C16:1)	Monounsaturated (MUFA)	2.00	1.50
Heptadecenoic acid (C17:1)	Monounsaturated (MUFA)	0.008	0.05
Oleic acid (C18:1)	Monounsaturated (MUFA)	59.01	57.03
Gadoleic acid (C20:1)	Monounsaturated (MUFA)	0.11	0.47
Linoleic acid (C18:2)	Polyunsaturated (PUFA)	16.72	19.45
Linolenic acid (C18:3)	Polyunsaturated (PUFA)	0.43	0.24

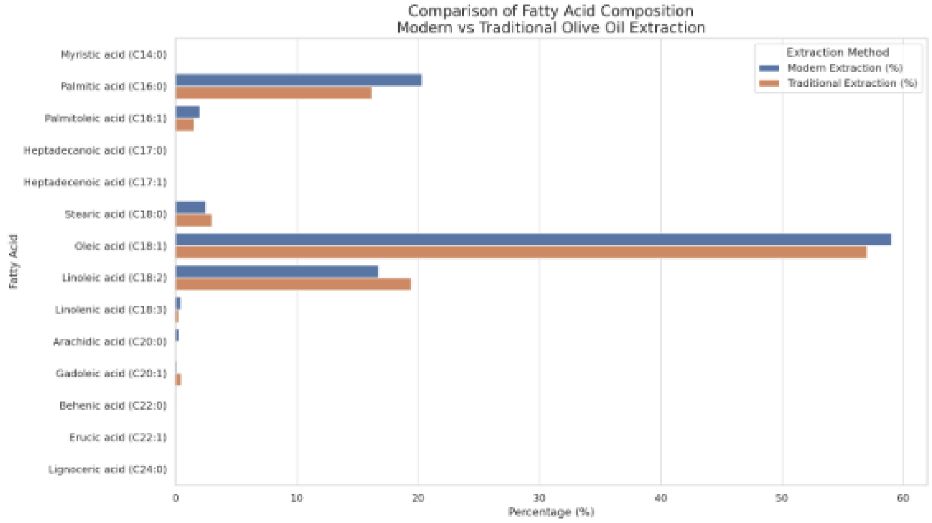

Figure 1: Comparison of Fatty Acid Profiles in Olive Oil Extracted by Modern and Traditional Methods.

Heavy Metal Content in Olive Oil

The results of ICP-OES analysis showed that cadmium (Cd) was below the detection limit in all olive oil samples (<0.1 ppm). Lead (Pb), arsenic (As), and mercury (Hg) were either not detected or were below the method's quantification threshold. These results comply with international standards for edible oils, confirming that the olive oil samples are safe for human consumption and free from heavy metal contamination **(**Table 2 and Figure 2).

Table 2. Heavy Metal Concentrations in Olive Oil Samples Extracted by Modern and Traditional Methods (ppm).

Sample Type	Cadmium (Cd) [ppm]	Lead (Pb) [ppm]	Arsenic (As) [ppm]	Mercury (Hg) [ppm]
Olive Oil (Modern)	<0.1	<LOD	<LOD	<LOD
Olive Oil (Traditional)	<0.1	<LOD	<LOD	<LOD
OMWW (Modern)	<0.1	<LOD	<LOD	<LOD
OMWW (Traditional)	<0.1	<LOD	<LOD	<LOD

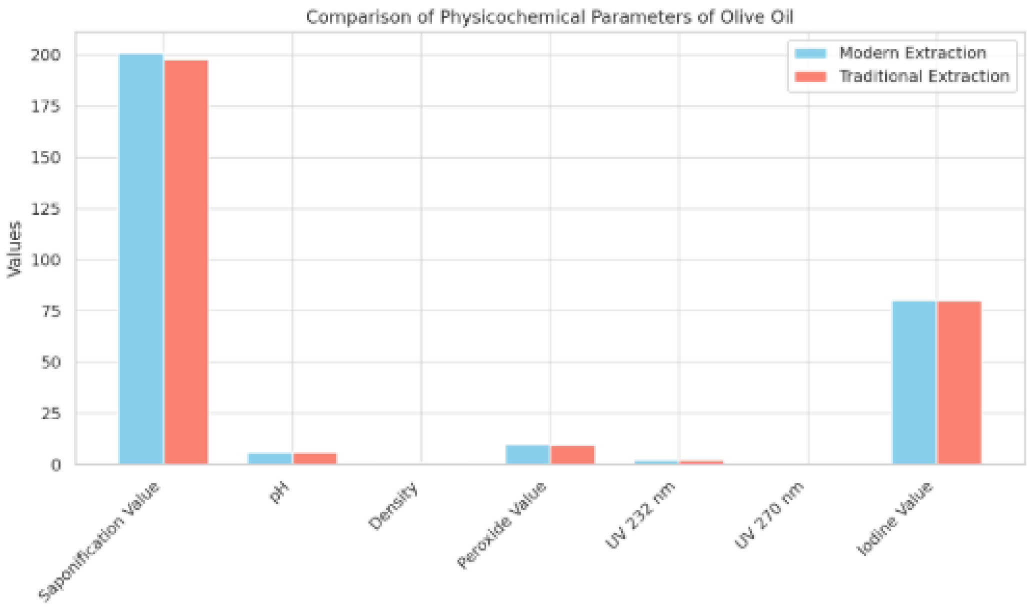

Figure 2. Heavy Metal Concentrations in Olive Oil Samples Extracted by Modern and Traditional Methods (ppm).

Heavy Metal Content in Olive Mill Wastewater (OMWW)

Analysis of OMWW (also referred to as grignon water) revealed similar trends in metal content. Cadmium was found at levels <0.1 ppm, while lead, arsenic, and mercury were not detected in the tested samples. However, wastewater samples from traditional extraction methods showed slightly higher turbidity and coloration, potentially indicating higher organic load and metal retention (Table 3)**.** This suggests that modern extraction may offer environmental advantages by reducing potential pollution from effluents.

Table 3. Heavy Metal Concentrations in Olive Mill Wastewater (OMWW) from Traditional and Modern Extraction Methods (ppm).

Heavy Metal	Traditional Extraction	Modern Extraction	Detection Limit
Cadmium (Cd)	<0.1	<0.1	0.1
Lead (Pb)	Not Detected (ND)	ND	—
Arsenic (As)	ND	ND	—
Mercury (Hg)	ND	ND	—
Turbidity / Visual Color	Slightly Higher	Lower	Qualitative

Discussion

The physicochemical parameters and heavy metal analysis of olive oil and olive mill wastewater (OMWW) obtained from both traditional and modern extraction methods in Libya provide insights into the relative advantages of each technique in terms of oil quality and environmental safety.

- Physicochemical Properties

The saponification value was slightly higher in modern olive oil (201) compared to traditional oil (197.7), indicating a higher proportion of short-chain fatty acids and better ester composition, which aligns with findings by *Gómez-Caravaca et al. (2016)*, who reported superior chemical characteristics in oils obtained using modern techniques.

pH values were also marginally higher in the modern sample (6.03 vs. 5.92), reflecting lower acidity and better oil stability. This trend is consistent with the observations by *Servili et al. (2014)*, who noted that modern centrifugation systems generally yield oil with lower free acidity due to reduced exposure to water and oxygen.

The density of both samples remained within the acceptable range for extra virgin olive oil, although slightly higher in the modern method (0.918 g/cm³ vs. 0.913 g/cm³), indicating a denser lipid profile, potentially due to higher unsaturated fatty acid content.

The peroxide values, which measure the extent of primary oxidation, were nearly identical (9.83 vs. 9.71 meq O_2/kg), suggesting comparable freshness and shelf stability. These values fall within acceptable international limits, supporting the findings by *Baccouri et al. (2008)* that peroxide levels are not solely dependent on extraction method but also on olive variety and post-harvest handling.

UV absorbance measurements at 232 nm and 270 nm were slightly higher in oils from the modern process (2.3 and 0.21) compared to the traditional method (2.28 and 0.19). Although the difference is minor, it may reflect better oxidative resistance and phenolic content in the modern oil, consistent with *Servili et al. (2014)* who demonstrated improved preservation of minor bioactive components in modern extraction systems.

Iodine values, indicative of unsaturation, were nearly equal in both samples (80.3 vs. 80.0), reflecting similar fatty acid unsaturation, as also noted in the fatty acid profiles. This is aligned with *Zarrouk et al. (2012)*, who found that unsaturation levels tend to be more cultivar-dependent than method-dependent.

- Fatty Acid Profile

The dominant fatty acid in both oils was oleic acid (C18:1), accounting for 59.01% in modern and 57.03% in traditional oils. This confirms the nutritional quality and oxidative stability of both oils, as emphasized by *Baccouri et al. (2008)*, who associated a higher oleic/linoleic ratio with improved oil shelf life.

Palmitic acid (C16:0) levels were higher in the modern sample (20.32%) compared to traditional (16.15%), while linoleic acid (C18:2) was slightly lower in modern oil (16.72%) than in traditional (19.45%). This profile suggests a better oxidative stability for the modern oil, again supporting the oleic/linoleic balance discussed in *Benitez-Sánchez et al. (2003)* and *Nouioua et al. (2022)*.
Overall, the profile revealed slightly higher monounsaturated fatty acids (MUFA) in modern oil, supporting its superior nutritional and shelf-life characteristics, as also observed by *García et al. (2015)* in comparative studies of extraction techniques.

- Heavy Metal Content

The heavy metal analysis revealed that cadmium (Cd) was below the detection limit (<0.1 ppm) in all oil and OMWW samples. Lead (Pb), arsenic (As), and mercury (Hg) were not detected in any sample. These results are consistent with *Zarrouk et al. (2012)* and *Nouioua et al. (2022)*, who reported low levels or non-detectable concentrations of heavy metals in olive oils from the Mediterranean region, suggesting good agricultural and processing practices.

However, traditional OMWW samples showed slightly higher turbidity and visual coloration, suggesting a higher potential for metal or organic compound accumulation in the waste matrix, as previously indicated by *Tarchouna et al. (2010)* and *Achak et al. (2009)*. Although metal concentrations remained under detection limits, the environmental load from organic matter may still be of concern and warrants further analysis.

Conclusion

This study provides a comparative evaluation of olive oil samples and olive mill wastewater (OMWW) derived from traditional (press-based) and modern (centrifugation-based) extraction methods used in Libya. The results reveal that olive oil produced by modern extraction methods demonstrates slightly superior physicochemical characteristics, including higher saponification values, more favorable fatty acid profiles (especially higher oleic acid and lower linoleic acid content), and marginally better oxidative stability based on UV absorbance indices. Both extraction methods yielded oils within international quality standards for extra virgin olive oil, and no significant contamination by heavy metals (Pb, Cd, As, Hg) was detected in either the oil or OMWW samples. However, traditional OMWW appeared to have slightly greater turbidity and color, potentially indicating a higher environmental burden in terms of organic and residual metal content, although within permissible limits. These findings underscore the advantages of modern extraction technologies not only for enhancing oil quality but also for reducing the potential environmental impact of olive mill effluents. Nevertheless, the traditional method remains widely used and still produces oil of acceptable quality.

Recommendations

- Promote modern extraction methods to improve oil quality and reduce environmental impact.
- Implement routine monitoring of oil and OMWW for safety and compliance.
- Develop effective OMWW treatment strategies, especially for traditional mills.
- Provide training for producers on hygiene, processing, and waste management.
- Support small-scale producers in adopting improved technologies.
- Encourage further research on olive oil quality and extraction efficiency.

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