Chromatographic Techniques for the Detection and Identification of Olive Oil Adulteration

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Fecha de publicación: 03.01.2024

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Citar: Khursheed, M, Ahmad, A, Noor, SE, García del Moral, LF, & Martos Núñez, V (2024). Técnicas Cromatográficas para la Detección e Identificación de la Adulteración del Aceite de Oliva. *REIDOCREA, 12*(01), 1-9.
Financiación: The research received financial support from the "SUSTAINABLE" project, which is funded by the European Union's Horizon 2020 Project H2020-MSCA-RISE-2020, under Grant Agreement 101007702.
Área o categoría del conocimiento: Química y Fisiología Vegetal – Docencia

Abstract: Olive oil stands out as one of the most beneficial oils for human health, offering preventive measures against a spectrum of health issues, including cardiovascular problems, cancer, osteoporosis, and various chronic diseases. However, the surging demand for olive oil has led to its susceptibility to adulteration with cheaper oils, such as soybean, peanut, hazelnut, and sunflower oil. Notably, extra virgin olive oil (EVOO), commanding a premium price, is particularly prone to adulteration with lower-cost alternatives. This adulteration poses a significant risk to health, necessitating the development of methods to detect and separate these impurities, thereby ensuring the quality and safety of olive oil. In order to address this concern, chromatographic techniques have emerged as pivotal tools in the purification and detection of adulterants in olive oil. Commonly employed methods include Thin Layer Chromatography (TLC), Gas Chromatography (GC), Liquid Chromatography (LC), and High-Performance Liquid Chromatography (HPLC). These techniques play a crucial role in differentiating between pure and impure olive oil in the market. This article focuses on the most prospective chromatographic methods for the detection and identification adulteration in olive oil. The outcomes of this research have the potential to pave the way for new directions in food security research, contributing to overall sustainability. Moreover, the insights gained from this study can be valuable for young students, serving as a concise reference for analytical methods in this field.

Keyword: Olive oil adulterants

Técnicas Cromatográficas para la Detección e Identificación de la Adulteración del Aceite de Oliva

Resumen: El aceite de oliva destaca como uno de los aceites más beneficiosos para la salud humana, ofreciendo medidas preventivas contra un espectro de problemas de salud, incluyendo problemas cardiovasculares, cáncer, osteoporosis y diversas enfermedades crónicas. Sin embargo, la creciente demanda de aceite de oliva ha llevado a su susceptibilidad a la adulteración con aceites más baratos, como el de soja, cacahuate, avellana y girasol. Es importante destacar que el aceite de oliva virgen extra (AOVE), que tiene un precio premium, es particularmente propenso a la adulteración con alternativas de menor costo. Esta adulteración representa un riesgo significativo para la salud, lo que hace necesario el desarrollo de métodos para detectar y separar estas impurezas, asegurando así la calidad y seguridad del aceite de oliva. Para abordar esta preocupación, las técnicas cromatográficas han surgido como herramientas fundamentales en la purificación y detección de adulterantes en el aceite de oliva. Los métodos comúnmente empleados incluyen la Cromatografía en Capa Fina (TLC), la Cromatografía de Gases (GC), la Cromatografía Líquida (LC) y la Cromatografía Líquida de Alta Resolución (HPLC). Estas técnicas desempeñan un papel crucial en diferenciar entre aceite de oliva puro e impuro en el mercado. Este artículo se centra en los métodos cromatográficos más prometedores para la detección e identificación de adulteraciones en el aceite de oliva. Los resultados de esta investigación tienen el potencial de abrir nuevas direcciones en la investigación de seguridad alimentaria, contribuyendo a la sostenibilidad general. Además, las percepciones obtenidas de este estudio pueden ser valiosas para los estudiantes, sirviendo como una referencia concisa para los métodos analíticos en este campo.

Palabra clave: Adulterantes del aceite de oliva

Introduction

Olive oil, derived from the fruit of the *Olea europaea* tree, is a predominant product in the Mediterranean countries, constituting a major portion of the global supply. The quality of olive oil is discerned by its fragrant and delicate flavor, appreciated by connoisseurs worldwide and cherished by local consumers. Due to variations in fruit quality, a substantial amount of olive oil necessitates refinement. The olive tree, characterized by its small evergreen stature, narrow silvery leaves, and petite white flowers, is renowned for its longevity. The botanical precursor of the olive is believed to be the oleaster, *Olea sylvestris*, a wild olive variant with diminutive, slender fruits, persisting around the Mediterranean and occasionally employed as grafting material. Optimal fruit quality for both table olives and olive oil is achieved through meticulous handpicking. The timing of optimal maturity is subject to fluctuations between years, cultivation regions, individual trees within the same grove, or even among fruits on the same tree. The significant commercial value of olive oil stems from its profound health benefits and desirable effects. Extra Virgin olive oil (EVOO), extracted through mechanical means from sound olive fruits, represents a premium category marketed without additional processing. This method ensures the preservation of the oil's intrinsic qualities, offering a product highly valued in both the culinary and global markets (Aparicio & Harwood, 2013; Kiritsakis & Markakis, 1988).

Triacylglycerol constitutes the primary component of olive oil, while chlorophylls, carotenoids, phenolic compounds, and squalene are present in smaller quantities (Uncu & Ozen, 2020). Composition of fatty acids differ from sample to sample due to environmental factors and place of production (Boskou, 2011). Major constituent (55-83%) of saponifiable fraction is oleic acid while others include palmitic, stearic, linoleic, α-linolenic acid (Calabriso, Scoditti, Pellegrino, & Carluccio, 2015). The chemical composition of oil is influenced by genetic and environmental factors. Stage of ripening also affects oil quality (Fuentes de Mendoza et al., 2013). There are different types of olive oil depending on free acidity (% oleic acid) and their processing methods (Table 1) (Calabriso, Scoditti, Pellegrino, & Annunziata Carluccio, 2015).

Table 1. Types of Olive Oil based on the measure of free fatty acids.

Type	Free acidity (% Oleic acid)
Extra Virgin Olive Oil	≤0.8
Virgin Olive Oil	≤2.0
Ordinary Virgin Olive Oil	≤3.3
Lampante Virgin Olive Oil	>3.3
Refined Virgin Olive	<0.3
Olive Oil	<1.0
Refined Olive Pomace Oil	<0.3
Olive Pomace Oil	<1.0

There is an abundance of health benefits of using olive oil such as prevention of cardiovascular diseases, hypertension, several cancers, and coronary artery disease. It has also antioxidant and anti-bacterial properties (Foscolou et al., 2018). EVOO is the best quality due to its taste and smell. Due to its increased prices, many companies mix other seed oils with it such as maize, sunflower, soy etc., which impose serious health effects (Marcos Lorenzo et al., 2002).

Different qualitative and quantitative techniques have been employed until now to detect different adulterants such as spectroscopic, voltametric, Deoxyribonucleic Acid-based, Differential Scanning Calorimetry (DSC), and digital imaging. Most of these traditional methods are less sensitive, time-consuming, expensive, and often require expertise. Nonetheless, chromatographic techniques, although require expertise in analytical skills, provide more sensitivity and accuracy over other techniques; thereby facilitating industries and government bodies to quickly evaluate the adulterants in olive oil.

Therefore, the aim of this study is to evaluate the currently employed chromatographic techniques for the detection and identification of olive oil adulteration. A summary based on the benefits and limitations of each employed chromatographic technique is presented. The outcomes of this research have the potential to pave the way for new directions in food security research, contributing to overall sustainability. Moreover, the insights gained from this study can be valuable for young students, serving as a concise reference for analytical methods in this field.

Olive oil adulteration

Adulteration typically means the addition of some foreign substance in any pure substance deliberately, whereas adulterants are those substances that are being added (Choudhary et al., 2020). The added ingredients alter the quality and composition of the product that can harm human health. Nevertheless, adulteration may confer advantages to companies by incorporating inexpensive ingredients and marketing the product at elevated prices.

In the case of olive oil, adulteration typically includes addition or substitution of low quality olive oils or edible oils (Frankel et al., 2011). The susceptibility of EVOO to fraudulent practices is heightened due to its exceptionally high quality and considerable cost as compared to other oils (Yang & Irudayaraj, 2001). When edible oil is mixed with EVOO, the resulting oil contains fatty acids and other components of both oils (Ruiz-Samblás et al., 2012). These edible oils are much cheaper than EVOO, consequently resulting in financial gains for fraudulent producer (Vanstone et al., 2018).

Cases of Olive Oil Fraud

A substantial volume of fraudulent occurrences exists, yet only a minority are officially documented. For instance, in Europe, 32 cases of reported olive oil fraud were identified, encompassing 20 incidents of substitution, 16 cases of mislabeling, 11 instances of untrue origins, 5 occurrences of internationally contaminated products, 6 cases of dilution, and 1 incident attributed to theft (Casadei et al., 2021).

Following are few of the documented fraudulent occurrences presented as examples (Casadei et al., 2021):

- In January 2015, four out of six EVOOs were found to be virgin, despite being marketed as extra virgin.
- In December 2015, olive oil that had been mixed with non-European brand oil was fraudulently sold internationally as 100% Italian EVOO. Subsequently, this deceptive practice was exposed.
- On April 10, 2017, an analysis of 35 EVOOs sold in Danish supermarkets revealed that only six were indeed Extra Virgin Olive Oil, while 15 were classified as virgin olive oil, and 12 as lampante olive oil.
- On October 29, 2017, Greek police arrested seven individuals during an investigation into the adulteration of olive oil. The illicit practice involved adding green dye to sunflower oil, which was then sold under different brands in Greece and other European countries.
- On May 24, 2018, Spain's largest olive oil cooperative was found to be importing olive oil from Tunisia and Morocco. They blended this imported oil with local olive oil and sold it in international markets, resulting in a significant fine being imposed on the cooperative.

In light of the aforementioned fraudulent activities, as well as similar instances, a critical imperative arises for the development of robust detection methods for adulterants in olive oil. This is essential for both international and local brands to ascertain and guarantee the quality of their products. The presence of impurities in olive oil raises concerns about potential adverse health effects on consumers.

Analytical methods play a pivotal role in addressing this challenge by offering not only qualitative but also quantitative analyses of these contaminants. Such methods contribute to the safeguarding of consumer health and the maintenance of product quality standards within the olive oil industry. Due to their enhanced accuracy and precision, chromatographic techniques frequently emerge as a preferred method within the array of analytical approaches. Consequently, this research holds significance, particularly for budding scholars, as it provides a succinct point of reference for analytical methods within this domain.

Chromatographic Techniques and Detection of Adulterants

Chromatography is a qualitative and quantitative technique which is used to separate different components present in a mixture (Gupta & Biswas, 2023). This separation of components is based on their difference in interaction between two physical phases, stationary and mobile phase (Hage, 2018). This technique is widely used in laboratories for separation, isolation, and purification of mixtures. Chromatography categorization is contingent upon the nature of solute interaction with the stationary phase, the configuration of the bed, and the physical characteristics of the mobile phase employed.

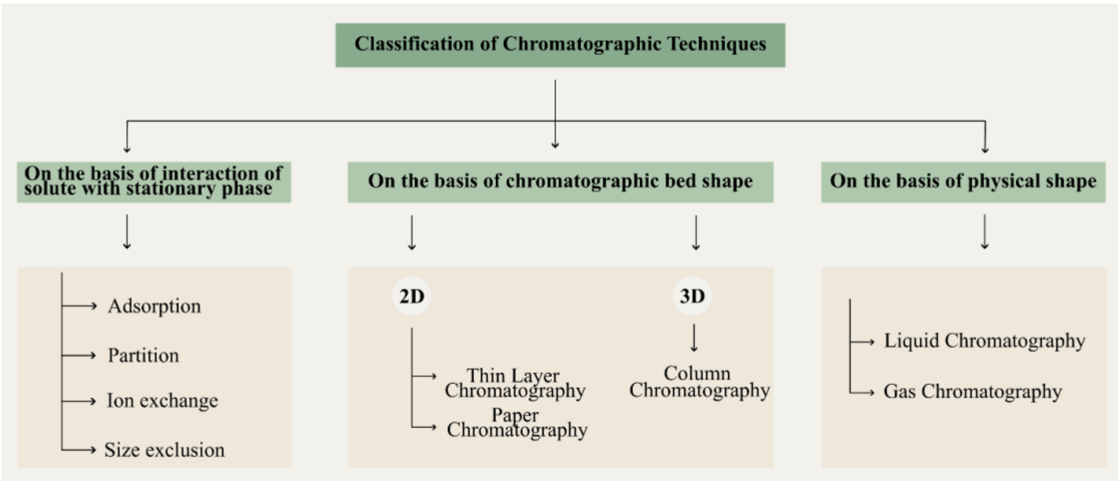

Figure 1. Summary of Chromatographic Techniques employed for olive oil adulteration detection.

Gas Chromatography (GC)

Gas Chromatography (GC) serves as a method for the separation of volatile compounds within a mixture (Al-Bukhaiti et al., 2017). In this technique, inert gases such as Helium and Nitrogen function as mobile phases. The process involves dissolving the sample thoroughly in a solvent, followed by vaporization to separate analytes (Kaur & Sharma, 2018). The stationary phase in GC employs adsorbents such as molecular sieves, aluminum oxides, and silica (Zeeuw & Luong, 2002).

GC finds extensive applications in medicinal and pharmaceutical domains. In forensic science, it proves invaluable for the testing and detection of various analytes, including blood, poison, alcohol, explosive residues, and metal detection (Kaur & Sharma, 2018). This method boasts high efficiency, providing elevated resolution by detecting in parts per million (ppm) and parts per billion (ppb), and typically requires small sample volumes, usually measured in microliters (μL) (McNair et al., 2019). However, a notable limitation of GC lies in its ability to analyze only volatile samples, and proper handling is imperative for sample injection (Patil et al., 2023).

The identification of adulterants in olive oil can be facilitated through the application of GC, as exemplified in the following examples:

- Gas chromatography-olfactometry coupled with mass spectrometry (GC-O-MS) was employed for the detection of adulteration in EVOO. This technique leveraged the olfactory system to facilitate the detection of characteristic odors associated with adulterants (Drira et al., 2021).
- Thermogravimetric Analysis coupled with Gas Chromatography/Mass Spectrometry (TGA-GC/MS) was utilized to identify the presence of soybean oil in olive oil. This method not only separated the two oils but also quantified volatile compounds in olive oil adulterated with soybean oil (Zhou et al., 2021).
- GC was employed for the quantitative detection of the presence of vegetable oil; however, the specific type of adulterant was not confirmed by this method (Meenu et al., 2019).

Liquid Chromatography (LC)

In Liquid Chromatography (LC), a liquid mobile phase is employed, either in a column or plane where a solid stationary phase is packed. This technique is particularly effective when dealing with colored mixtures. LC is categorized into four types: normal phase, reversed phase, ion exchange, and size exclusion chromatography. It is a cost-effective method, involving manual processes such as the flow of the mobile phase and the collection of separated analytes (Broeckhoven & Desmet, 2020; Tanno et al., 2020). LC finds extensive application in various industries, including the food industry, pharmaceuticals, forensics, hospitals, the chemical industry, and environmental analysis. LC could prove valuable for separating adulterants present in olive oil.

Specific instances demonstrating the application of LC include:

- A study reported that a combination of column and Gas Liquid Chromatography efficiently separated refined seed oils mixed with olive oil (Kapoulas & Passaloglou - Emmanouilidou, 1981).
- To expose intentional adulteration in olive oil, sterols were analyzed by using nano-LC which was coupled with UV detector; thereby obtaining quantitative data (Rocco & Fanali, 2009).

High Performance Liquid Chromatography (HPLC)

High-Performance Liquid Chromatography (HPLC) stands out as an efficient technique for both qualitative and quantitative analysis of individual components within a mixture. This method is particularly adept at separating complex mixtures and employs various detectors, including fluorescence, UV, refractive index, radiochemical, and electrochemical detectors (Shockcor, 2017). HPLC finds diverse applications in the separation of sterols, triglycerides, enzymes, antibodies, and other samples relevant to environmental, medicinal, forensic, and food industries. Its significance extends to almost all industries for ensuring product quality through the quantification of adulterants.

Olive oil, containing pigments such as chlorophylls and carotenoids, is indicative of its quality. However, unscrupulous corporations may attempt to deceive consumers by mixing green color into virgin and extra virgin oils. HPLC proves instrumental in detecting such fraudulent practices, as exemplified in the following instances:

- HPLC coupled with High-Resolution Mass Spectrometry (HRMS) was employed to screen and identify biomarkers, enabling the recognition of the presence of Soft Deodorized Olive Oil (SDOO) mixed with Extra Virgin Olive Oil (EVOO) (Navratilova et al., 2022).
- To detect contamination of canola oil with olive oil, reversed phase HPLC was utilized (Salivaras & McCurdy, 1992).
- For the identification of α-tocopherol as a critical parameter in detecting sunflower oil adulteration in olive oil reversed-phase high-pressure liquid chromatography along with a fluorescence detector was used (Bakre et al., 2015).

Thin Layer Chromatography (TLC)

Thin-Layer Chromatography (TLC) is employed for the separation of non-volatile mixtures. In this method, a thin layer of solid adsorbent, typically silica or alumina, is coated on surfaces such as glass, aluminum foil, or plastic to effectuate the separation of mixtures. The differential efficiencies between the stationary and mobile phases contribute to the separation process. This versatile technique finds applications in various fields, including the drug discovery process (Ciura et al., 2017), enantiomeric chiral analysis (Aboul‐Enein et al., 1999), screening fungal extracts (Scott et al., 1970), and environmental and clinical analyses.

Additionally, TLC could also be employed in detecting olive oil adulteration. For example, Solid-Phase Extraction coupled with Thin-Layer Chromatography (SPE-TLC) was employed to determine esterified sterols in olive oil and hazelnut oil. (Cercaci et al., 2003b). This method is particularly useful in discerning fraud when the composition of fatty acids in both oils is suspiciously similar.

Table 2. Practical examples of the application of chromatographic techniques for the identification of adulteration in olive oil.

Country	Adulterant	Method	Marker	Reference
Italy, Spain, Greece, Tunisia, France, Turkey, Switzerland	Palm and grapeseed oils	NP-HPLC	Tocopherols and tocotrienols	(Dionisi et al., 1995)
Spain, Portugal, UK	Crude sunflower and rapeseed oil	GC	Hydrocarbon concentration and composition	(Webster et al., 2000)
Different Mediterranean countries, Italy	Hazelnut oil	GC	Esterified Sterols	(Cercaci et al., 2003a)
UK	Hazelnut oil	RP-HPLC	Polar fractions	(Zabaras & Gordon, 2004)
Greece delicatessen stores	Sunflower, soybean, mustard oil, cotton, corn, peanut oil, almond, walnut, sesame, hazelnut, safflower, canola	GC HPLC	Fatty acids methyl esters, triglycerides	(Christopoulou et al., 2004)
Poland	Virgin olive oil and virgin hazelnut oil	SPME-GC/MS	Volatile compounds	(Mildner-Szkudlarz & Jeleń, 2008)
Spain, Portugal, Italy, Greece, Argentine, Brazil	Soybean Oil	HPLC-APCI-MS/MS	Triacylglycerols	(Fasciotti & Netto, 2010)
Germany, Italy	Hazelnut oil	LC-ESI-Q-ToF-MS	Polar compounds	(Calvano et al., 2010)
Jordan	Sunflower, soybean, corn, cotton seed oil	GC	sterols	(Al-Ismail et al., 2010)

Where: NP-HPLC stands for Normal Phase High Performance Liquid Chromatography; GC: Gas Chromatography; RP: Reverse Phase; SPME-APCI-MS/MS: Solid Phase Microextraction Atmospheric Pressure Chemical Ionization Tandem Mass Spectrometry; LC-ESI-Q-ToF-MS12: Liquid Chromatography Electrospray Ionization Quadrupole Time of Flight Mass Spectrometry.

Challenges and Opportunities

While Chromatographic techniques serve as valuable tools for detecting and quantifying adulterants in olive oil, their efficacy depends on adhering to proper protocols. Understanding the nature of the analyte is essential, and meticulous sample preparation involving multiple solvents is crucial for authentic results. The process is often time-consuming due to the intricate separation of analytes from the mixture.

Chromatographic instruments, though sensitive and precise, pose challenges as they are expensive and consume significant power. Likewise, the presence of similar fatty acids and triglycerols in olive oil and other compounds can impede accurate identification, emphasizing the need for advancements to enhance precision.

To improve accuracy, it is imperative to identify the specific analyte, select an appropriate solvent, and follow pre-sample preparation steps. Artificial Intelligence (AI) has emerged as a transformative force in this domain, enabling the recognition of specific peaks, even with subtle differences. Machine learning aids in optimizing separation conditions, such as choosing the mobile phase, stationary phase, and gradient elution, thereby enhancing sensitivity and resolution.

An advancement in Chromatographic techniques is the application of Ultra High-Performance Liquid Chromatography (UHPLC), which offers higher sensitivity and resolution compared to existing methods. Combining UHPLC with LC and tandem mass spectrometry enhances accuracy and resolution. For instance, hydroxytyrosol fatty acid esters in EVOO can be identified and quantified using UHPLC triple quadrupole tandem mass spectrometry (UHPLC-QqQ-MS/MS) (Medina et al., 2022). Furthermore, innovations and advances that are made in the equipment used in chromatographic techniques might improve accuracy.

Conclusion

Olive oil, constituting a significant share of the global supply, is renowned for its health benefits. The quality of olive oil is characterized by its aromatic and nuanced flavor, esteemed by experts globally and favored by local consumers. However, the increasing demand for olive oil has rendered it vulnerable to adulteration with less expensive alternatives like soybean, peanut, hazelnut, and sunflower oils. EVOO, commanding a premium price, is particularly susceptible to substitution with lower-cost options. Consequently, various analytical methods are employed to detect and identify adulterated olive oil, ensuring food safety and security. Among these methods, chromatographic techniques stand out as the most effective.

Chromatography classification depends on the interaction of solutes with the stationary phase, bed configuration, and the physical properties of the mobile phase. Commonly employed chromatographic techniques for detecting and identifying adulteration in olive oil include GC, LC, HPLC, and TLC.

However, despite the enhanced precision and accuracy of chromatographic techniques, challenges such as high costs, reduced sensitivity, and time consumption persist. Incorporation of AI technologies might present a potential solution to enhance the overall performance of these methods and overcome these challenges.

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