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*Research Article*

# Detection of adulteration in Moroccan and Egyptian sesame oil using Isotopic analysis and Infrared spectroscopy

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## Abstract

Sesame oil is frequently targeted for adulteration by blending it with lower-cost oils, compromising its quality and authenticity. This study investigates the efficiency of isotopic analysis ($\delta^{13}C$) and Fourier Transform Infrared Spectroscopy (FTIR) in detecting adulteration in Moroccan and Egyptian sesame oils. The $\delta^{13}C$ values of pure sesame oils varied based on their geographical origin, with Moroccan and Egyptian oils exhibiting values of -28.92‰ and -29.14‰, respectively. Adulteration with paraffin and table oils led to notable isotopic shifts proportional to the blending ratios. Specifically, pure paraffin oil displayed a $\delta^{13}C$ value of -27.32‰, while table oil presented a value of -38.99‰. Mixtures with soybean oil revealed significant decreases in $\delta^{13}C$ values, whereas mixtures with corn oil demonstrated a positive correlation between blending ratios and $\delta^{13}C$ values.The combination of isotopic analysis and FTIR spectroscopy proved highly effective for identifying adulterated blends, offering a robust tool for detecting food fraud. When integrated into routine quality control procedures, these techniques can reliably ensure the authenticity and quality of sesame oils, protect consumer rights, and reinforce trust in the edible oil market.

**Keywords:** Authenticity, Food adulteration, Infrared spectroscopy, Isotopic analysis, Quality control, Sesame oil

## INTRODUCTION

Sesame oil (Sesamum indicum L.) holds a privileged position among edible oils due to its distinctive aroma, stability, and health benefits. It is an essential source of dietary fats in Asian, African, and Middle Eastern countries, where its use as a cooking oil and flavor enhancer is widely established (Rangkadilok *et al*., 2010; Nauta *et al*., 2023). Extensive research has highlighted its antioxidant properties, anti-inflammatory effects, and potential to prevent cardiovascular diseases, hypertension, and aging (Sharma *et al*., 2024).

Sesame oil's millennia-old history—dating back more than 5000 years—establishes it as one of the earliest known oils used by humans. Today, China remains the largest global producer and consumer of sesame oil, while its growing demand has elevated its economic value (Wang *et al*., 2024). However, this high market value also makes sesame oil a frequent target for adulteration, a fraudulent practice involving blending with cheaper oils such as corn, sunflower, or paraffin oils (Zhang *et al*., 2024). Such adulteration compromises product quality and violates fundamental consumer rights, including access to accurate information and value-for-money purchases.

In response to these concerns, modern food authentication techniques have emerged as powerful tools to ensure product traceability and authenticity. Fourier Transform Infrared Spectroscopy (FTIR) has proven to be an effective, rapid, and non-destructive method for detecting oil adulteration. By generating unique spectral fingerprints, FTIR can identify compositional anomalies resulting from adulterants (Strati *et al*., 2024; Sharma *et al*., 2024). Coupling FTIR with advanced chemometric methods has further improved the sensitivity and accuracy of these analyses (Ali *et al*., 2024).

In addition, Isotope Ratio Mass Spectrometry (IRMS) has gained significant attention as a robust technique for verifying geographical and botanical authenticity. By analyzing the $\delta^{13}C$ isotopic signature of oils, IRMS can differentiate between pure and adulterated samples, providing a precise means of traceability (Nasr *et al*., 2022; Wadood *et al*., 2024). Combining IRMS with complementary methods such as LC-MS metabolomics profiling allows researchers to identify unique biomarkers associated with oil adulteration (Sumara *et al*., 2023; Rozali *et al*., 2024).

The primary objective of this study is to evaluate the combined effectiveness of isotopic analysis ($\delta^{13}C$) and FTIR spectroscopy in detecting adulteration in Moroccan and Egyptian sesame oils. Specifically, the study aimed to:

Identify and quantify adulteration with cheaper oils, such as corn and sunflower oil.

Enhance traceability and authenticity using advanced analytical techniques.

Protect consumer rights and ensure the integrity of sesame oil products marketed globally.

## MATERIALS AND METHODS

### Samples and oil extraction

The sesame seeds used in this study were obtained from certified markets that were specialized in authentic seeds in Morocco and Egypt. The seeds were sorted to remove damaged ones and sieved to eliminate foreign matter. The oil was mechanically extracted from sesame seeds using an automatic oil press machine in the laboratory. For each sample, 100 g of seeds were pressed (Savoire et *al*., 2013), and the extraction yields were recorded (Table 1).

### Sample preparation

Two sets of oils for the analyses were prepared:

Authentic Oils: Sesame oils of Moroccan and Egyptian origin, pure table oil (Cheaper priced oil in the Moroccan market), and pure paraffin oil.

Mixed Oils: Several dilutions of mixed oils were prepared by blending Egyptian and Moroccan sesame oils with paraffin oil and table oil (cheaper-priced oil from the market). The sesame oils for the adulteration test were mixed in proportions ranging from 10% to 90% with table oil, and the same procedure was applied to paraffin oil for both Egyptian and Moroccan sesame oils. (*Fig*. 1).

### Isotope Ratio Mass Spectrometry (IRMS) analysis

An isotope ratio mass spectrometer coupled with gas chromatography (EA-IRMS) was used to analyze the stable carbon-13 isotopes to characterize the constitu-

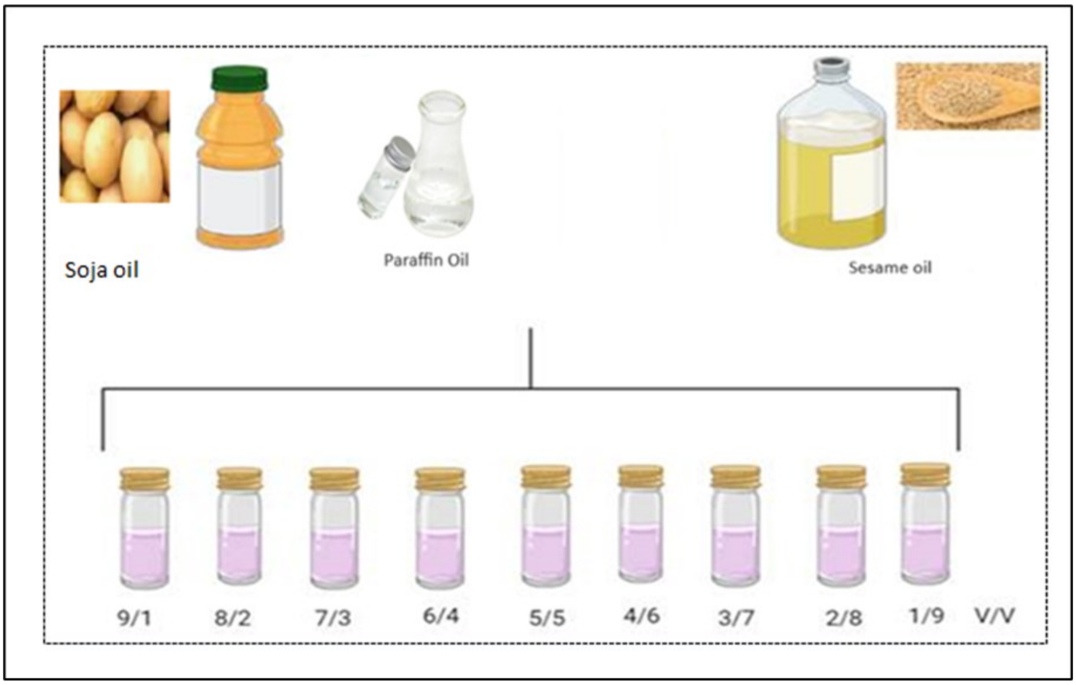

**Fig. 1.** *Preparation of sesame oil mixtures with table and paraffin oils for isotopic analysis.*

**Table 1.** Comparative yields of sesame oil extraction from Egypt and Morocco

| Sample | Oil Mass (g) | Cake Mass (g) | Total Mass (g) | Oil Yield (%) | Cake Yield (%) |
|---|---|---|---|---|---|
| Egypt Oil | 36.909 | 58.632 | 95.541 | 36.909 | 58.632 |
| Morocco Oil | 38.5936 | 58.961 | 97.554 | 38.5936 | 58.961 |

ents of sesame oil. This method provides information on the authenticity of sesame oil by measuring the total carbon content ( Taous *al*.,2020).

### FTIR Spectroscopy analysis

Fourier transform infrared (FTIR) spectra of all samples were collected using a Spectrum Two FT-IR spectrometer (Perkin-Elmer, USA) with an attenuated total reflectance (ATR) crystal. Spectra were obtained in the range of 650-4000 $cm^{-1}$ with 32 co-added scans at a spectral resolution of 4 $cm^{-1}$. Before spectral collection, the adulterated sesame oil samples were shaken to ensure homogeneity (Taous *et al*.,2022). Approximately 10 ml of oil was placed on the ATR surface, and the air background spectrum was subtracted from each obtained spectrum.

### Carbon Isotope Ratio analysis by Isotope Ratio Mass Spectrometry

Carbon analyses in the samples were conducted using isotope ratio mass spectrometry (IRMS) (Isochrom-EA, VG ISOTECH, UK). The samples were mixed, and 0.15 mg of the oil samples were placed in tin capsules. For carbon isotopic analysis, the separated compounds were converted to $CO_2$ by passing the eluted analyte flow through a ceramic oxidation reactor at a gas temperature of 1030 °C. Carbon dioxide gas standards were injected before and after the species of interest to allow for the calculation of $\delta^{13}$C‰ values. Helium was used as the carrier gas. Results are expressed in the conventional per mille notation relative to the Pee Dee Belemnite (PDB) marine carbonate standard. (Brand *et al*., 2014)

### Statistical analyses

Statistical analyses were conducted using SPSS version 21 and Stata version 14. Descriptive analysis was used to calculate means, standard deviations, percentages, and the isotopic ratios' minimum and maximum valus. Pearson's correlation was applied to evaluate the relationships between the different variables studied. A significance threshold of 5% was used for all statistical analyses.

## RESULTS

### Isotopic analysis of sesame oil and table oil mixtures

Isotopic analysis of sesame and table oil mixtures revealed significant trends, highlighting the impact of composition on isotopic ratios. The isotopic ratio δ13C‰, measured at -29.03±0.15 ‰, showed relative stability of the characteristic isotopic signature of pure sesame oil, oscillating within a narrow range of -29.14‰ to -28.92‰.

As the proportion of table oil increased, the dilutions showed a notable trend of decreasing isotopic ratio. At a proportion of 90% sesame oil and 10% table oil (90:10), the $\delta^{13}$C reached -28.93±0.13 ‰, with a range of -29.03‰ to -28.84‰, confirming this negative trend. This decrease continued progressively until it reached -38.99±0.00‰ at 0:100, indicating a strong influence of pure table oil on the isotopic ratio.

The total isotopic ratio value for all dilutions was -27.98±0.53‰, with an extended range from -29.14‰ to -27.18‰. The observed fluctuations in the minimum and maximum values reflected the sensitive response of the isotopic ratio to variations in the percentages of sesame oil and table oil in the mixture, thus highlighting the system's sensitivity to the specific configuration of the blend.

### Isotopic analysis of paraffin oil and table oil mixtures

Isotopic exploration of paraffin oil and table oil combinations revealed distinct trends, highlighting the crucial impact of composition on isotopic ratios. The isotopic ratio δ13C‰, precisely measured at -29.03±0.15 ‰, demonstrated relative stability of the isotopic signature characteristic of pure sesame oil, oscillating within a narrow range of -29.14 ‰ to -28.92 ‰.

As the proportion of paraffin increased in the mixtures, the isotopic ratio also increased, reaching its peak at a proportion of 90% pure sesame oil and 10% paraffin oil (90:10). At this stage, the $\delta^{13}$C‰ rose to -28.510±0.18 ‰, with a range of -28.64‰ to -28.38‰, confirming this positive trend. This increase continued progressively until it reached -27.32±0.00‰ at 0:100, indicating pure paraffin oil's predominant influence on the isotopic ratio.

The total isotopic ratio value for all dilutions was -30.44±2.11 ‰, extending from -38.99‰ to -28.84‰. The observed fluctuations in the minimum and maximum values indicated the sensitivity of the isotopic ratio to variations in the percentages of paraffin oil and table oil in the mixture. The minimum isotopic ratio value was -38.99‰, while the maximum was -28.84‰. These extremes highlight the significant diversity of isotopic ratios in the mixture, underscoring the impact of different oil proportions on the overall isotopic signature (Table 2).

### Correlations between isotopic ratio and dilutions of paraffin oil and table oil

Correlation analysis of the data in Fig. 1(a) revealed significant relationships between the isotopic ratio and

**Table 2.** δ $^{13}$ C‰ value for authentic and adulterated sesame oils

| SP/TO | δ13 C‰ | Min | Max | SP/PO | δ13 C‰ | Min | Max |
|---|---|---|---|---|---|---|---|
| TO (100 :0)[a] | -29.03±0.15 | -29.14 | -28.92 | PO (100 :0)[a] | -29.03±0,15 | -29.14 | -28.92 |
| 90:10 | -28.93±0.13 | -29.03 | -28.84 | 90:10 | -28.510±0,18 | -28.64 | -28.38 |
| 80:20 | -29.27±0.12 | -29.36 | -29.19 | 80:20 | -27,71±0,75 | -28.25 | -27,18 |
| 70:30 | -29.50±0.07 | -29.55 | -29.45 | 70:30 | -28.08±0,13 | -28.18 | -27.99 |
| 60:40 | -29.82±0.09 | -29.89 | -29.75 | 60:40 | -28.14±0,33 | -28,38 | -27.91 |
| 50:50 | -30.10±0.05 | -30.14 | -30.07 | 50:50 | -28.00±0,41 | -28.29 | -27.71 |
| 40:60 | -30.42±0.03 | -30.44 | -30.40 | 40:60 | -27.72±0,43 | -28.03 | -27.42 |
| 30:70 | -30.72±0.09 | -30.79 | -30.65 | 30:70 | -27.88±0,03 | -27.91 | -27,86 |
| 20:80 | -31.07±0.02 | -31.09 | -31.06 | 20:80 | -27.61±0,14 | -27.71 | -27.51 |
| 10:90 | -31.25±0.01 | -31.26 | -31.24 | 10:90 | -27.42±0,16 | -27.54 | -27.31 |
| 0 :100 | -38.99±0.00 | -38.99 | -38.99 | 0 :100 | -27.32±0,00 | -27.32 | -27.32 |
| Total | -30.44±2.11 | -38.99 | -28.84 | Total | -27.98±0.53 | -29.14 | -27.18 |

[a] : Mixing ratio : Egyptian Sesame Oil/Table Oil/Paraffin Oil (100 :0:0); Moroccan Sesame Oil/Table Oil/Paraffin Oil (100:0:0). MSO : Moroccan Sesame Oil ; ESO : Egyptian Sesame Oil. SP/PO : Sample Percentages / Paraffin Oil ; SP/TO : Sample Percentages / Table Oil.

dilutions of paraffin oil as well as table oil. A positive correlation was identified for paraffin oil, indicating a trend of increasing isotopic ratio as the proportion of paraffin in the mixture increases. This correlation was statistically significant ($r=0.750$, $p<0.001$), highlighting the robustness of this trend.

Conversely, a negative correlation was observed between the isotopic ratio and dilution in table oil (Fig. 1 (b)). This means that the isotopic ratio tended to decrease as the proportion of table oil in the mixture increased. This correlation was particularly strong ($r=-0.701$, $p<0.001$), suggesting a very close relationship between the composition of table oil and the isotopic ratio (Fig. 2).

### Influence of paraffin oil and table oil dilutions on isotopic signatures

The analysis of the mean differences in isotopic ratios between pure sesame oil and various paraffin and table oil dilutions revealed significant trends. As the percentage of paraffin oil increased, the mean difference in isotopic ratios showed a negative correlation. This suggests that the isotopic signature of paraffin oil gradually decreased relative to that of pure sesame oil.

In contrast, a positive correlation was observed when comparing the isotopic ratios between pure sesame oil and table oil dilutions. As the proportion of table oil increased, the mean difference in isotopic ratios indicated an increase in the isotopic signature relative to pure sesame oil.

This trend persisted at each dilution level, highlighting a significant influence of the mixture composition on isotopic signatures. These observations reinforced the idea that the specific composition of the mixture played a crucial role in altering the isotopic characteristics of the oils studied (Fig. 3).

The analysis of the results highlighted a significant trend: the use of different dilutions of table oil led to a notable increase in the mean difference of the isotopic ratio for both countries. Conversely, when paraffin oil was used, the mean difference significantly decreased for both countries.

This observation underscored the particular sensitivity of the isotopic ratio to variations in the percentage of table oil in the mixture, with a marked increase in the mean difference. This trend can be attributed to the distinctive nature of table oil, suggesting that dilutions with this type of oil significantly influenced the isotopic signature.

On the other hand, the decrease in the mean difference when paraffin oil was used suggests a lesser influence of this type of oil on the isotopic ratio. This observation may be related to the specific isotopic properties of paraffin oil, which appear less pronounced in the mixture.

Thus, this in-depth analysis highlighted distinct variations in the isotopic response depending on the type of oil used, paving the way for a finer understanding of the influences of each component on the overall isotopic signature (Fig. 4).

## DISCUSSION

The δ$^{13}$C‰ values for authentic sesame oils obtained in this study were -28.92‰ for Moroccan oil and -29.14‰ for Egyptian oil. These values align well with previously reported ranges for sesame oils from other regions: -

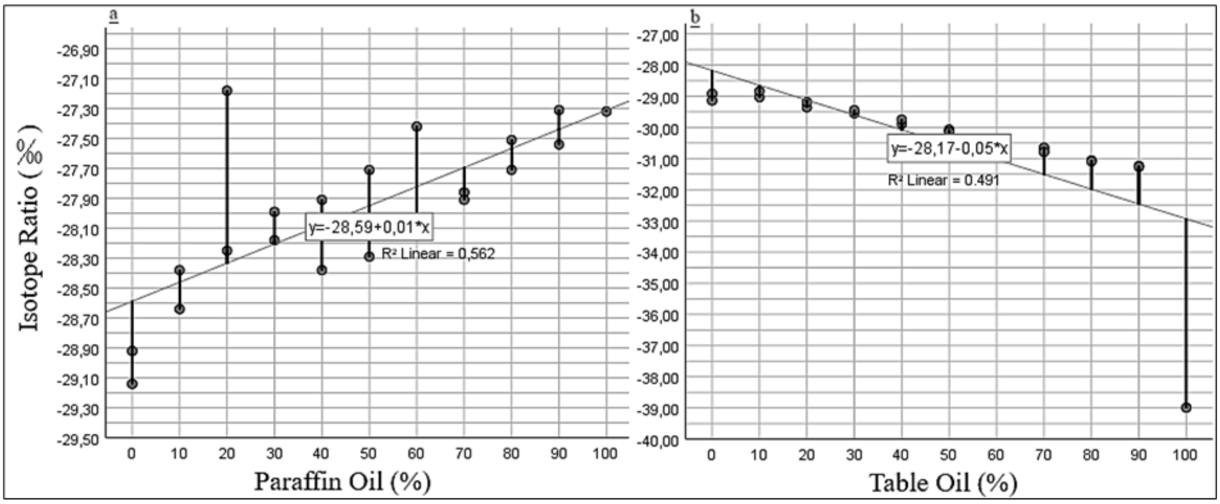

**Fig. *2.*** Correlation between Carbon Isotope Ratio and Concentration of Table Oil (a) and Paraffin Oil (b)

27.68‰ for Sudan, -28.45‰ for China, -28.78‰ for Pakistan, -29.26‰ for Korea, and -28.61‰ for India (Seo *et al*., 2010). The observed geographic variability in carbon isotopic values confirms the utility of isotopic analysis as a robust tool for tracing the geographical origin of edible oils (Nasr *et al*., 2022).

The δ¹³C‰ values for paraffin oil and table oil were measured at -27.32‰ and -38.99‰, respectively. These values are significantly outside the global isotopic range for pure corn oil (typically -14.3‰ to -16.4‰), underscoring the clear isotopic distinction between table oil, corn oil, and the analyzed sesame oils. This result corroborates findings from recent studies employing isotope ratio mass spectrometry (IRMS) to differentiate between authentic and adulterated edible oils (Aslam *et al*., 2024).

A clear downward trend in δ¹³C‰ values was observed when soybean oil was introduced into sesame oil. For a 90:10 mixture (90% sesame oil and 10% soybean oil), δ¹³C‰ decreased to -28.93 ± 0.13‰, ranging from -29.03‰ to -28.84‰. These findings are consistent with previous research by (Kim *al*., 2014), which demonstrated significant reductions in δ¹³C‰ values as soybean oil proportions increased. This trend is explained by the isotopic nature of C3 plants like soybean, which exhibit lower δ¹³C‰ values than other plant oils.

In contrast, adding corn oil (a C4 plant oil) resulted in a positive correlation with the isotopic ratio, with an exceptional correlation coefficient of r² = 0.9994. As observed in previous studies, corn oil's inherently higher δ¹³C‰ values drive this trend (Sharma *et al*., 2024). These results emphasize the effectiveness of δ¹³C‰ analysis in detecting specific adulterants based on their botanical origin (C3 vs. C4 plants).

Furthermore, studies have highlighted the correlation between fatty acid profiles (e.g., palmitic, stearic, oleic, linoleic, and linolenic acids) and the presence of adulterants. Mixing cheaper oils, such as soybean or corn oil, with sesame oil leads to identifiable changes in fatty acid composition (Sumara *et al*., 2023). Combining isotopic analysis with fatty acid profiling could enhance the detection of oil adulteration, providing a multi-faceted

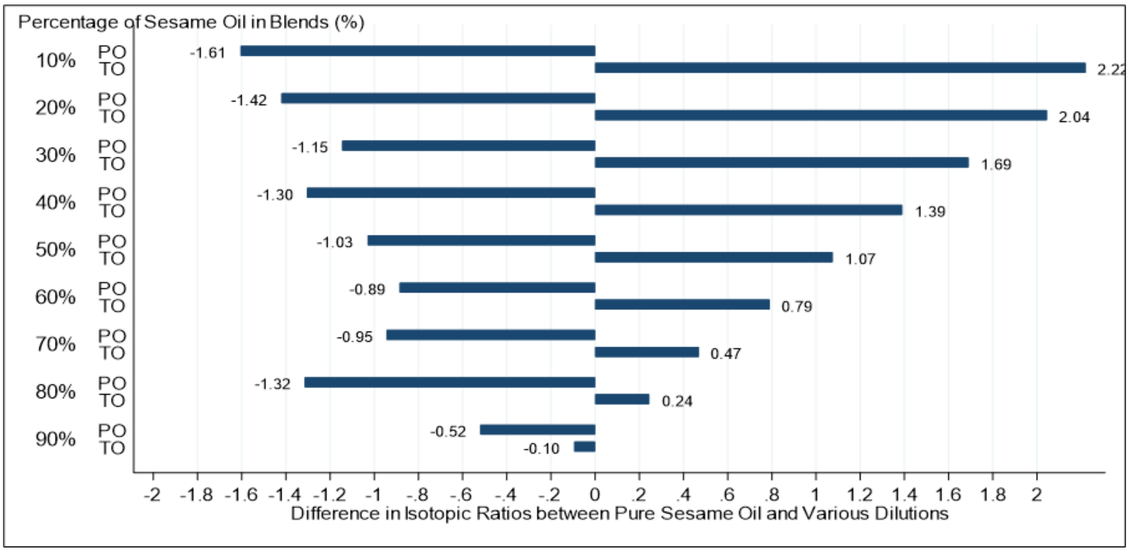

**Fig. 3**.*Percentage of sesame oil in blends (%) as a function of the difference in isotopic ratios between pure sesame oil and various dilutions*

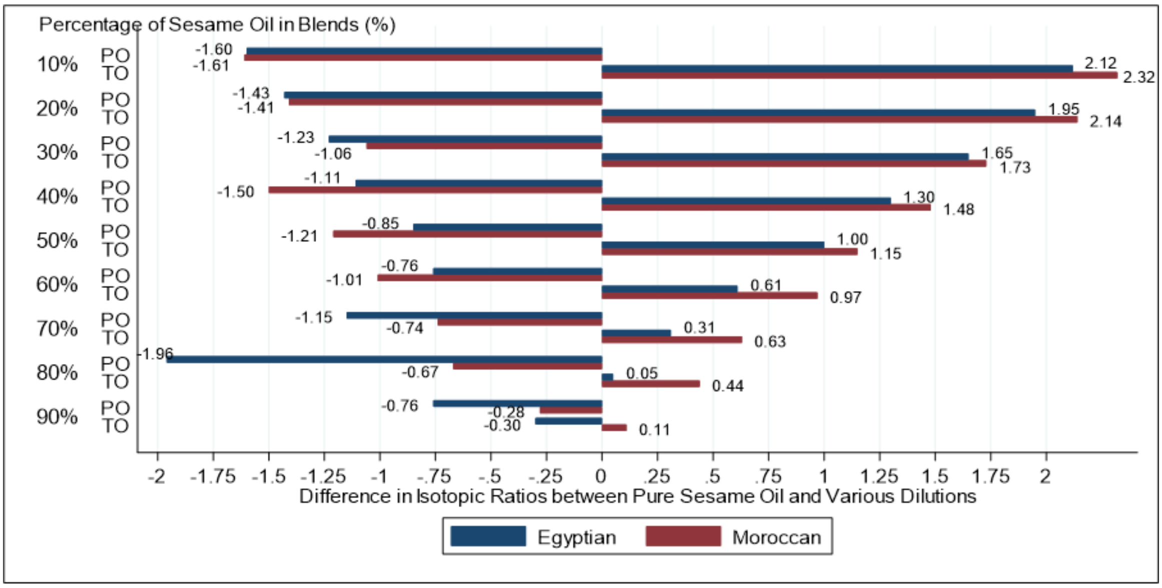

**Fig. 4.** *Percentage of sesame oil in blends (%) as a function of the difference in isotopic ratios between pure sesame oil and various dilutions for both Moroccan and Egyptian sesame*

approach to food fraud detection.

The analysis of paraffin oil mixtures also revealed clear trends. For Moroccan sesame oil, δ¹³C‰ values increased from -28.92‰ (100% sesame oil) to -27.31‰ (10% sesame oil and 90% paraffin oil). A similar trend was observed for Egyptian sesame oil, where δ¹³C‰ increased from -29.14‰ to -27.54‰. These results indicate that increasing proportions of paraffin oil cause a gradual increase in the isotopic ratio, consistent with the findings of (Wadood *al*., 2024).

The sensitivity of δ¹³C‰ values to varying proportions of adulterants, particularly oils of C3 (e.g., soybean) and C4 (e.g., corn) plant origin, demonstrates the robustness of IRMS as an analytical tool. These results align with recent advancements in analytical techniques, such as LC-MS metabolomics and Fourier Transform Infrared Spectroscopy (FTIR), which provide complementary markers for oil authenticity and adulteration (Ali *et al*., 2024; Rozali *et al*., 2024).

### Key points of the analyses

The key findings of this study highlight the precision of isotopic analysis and the efficiency of infrared spectroscopy in detecting adulteration in sesame oils. The δ¹³C values successfully differentiated authentic sesame oils from adulterated mixtures, demonstrating the reliability of this method for traceability and authentication. By leveraging these values, the geographical origin and purity of oils can be effectively determined. Furthermore, Fourier Transform Infrared Spectroscopy (FTIR) proved to be a rapid and accurate technique for identifying adulterants, making it a valuable tool for detecting food fraud in edible oils.

### Weaknesses

Despite the effectiveness of isotopic analysis and FTIR spectroscopy, certain limitations must be acknowledged. The variability of samples, particularly due to geographic differences and production conditions, introduces natural variations in isotopic values. This variability highlights the need for a larger and more comprehensive database to improve the accuracy and reliability of comparisons across different regions. Additionally, while isotopic analysis and FTIR are powerful techniques, they have limitations in certain contexts. They would benefit from being complemented by other advanced analytical methods, such as chromatographic techniques or LC-MS metabolomics, to achieve even greater precision in detecting oil adulteration.

### Practical implications

The practical implications of this study emphasize the importance of integrating advanced analytical techniques into routine quality control practices. Incorporating isotopic analysis and FTIR spectroscopy into standard protocols used by producers and regulators can significantly enhance the detection and prevention of adulteration, ensuring the authenticity and quality of edible oils. These methods also play a critical role in strengthening consumer confidence by providing reliable traceability and assurance that the oils they purchase are free from fraudulent practices. Ensuring product authenticity through these techniques will protect consumer rights and maintain market integrity by deterring adulteration.

## Conclusion

This study demonstrated the effectiveness of isotopic analysis (δ¹³C) and Fourier Transform Infrared Spectroscopy (FTIR) in detecting adulteration in Moroccan and Egyptian sesame oils. The results showed that δ¹³C values varied according to the geographical origin of authentic sesame oils, enabling precise traceability and authentication. The observed isotopic variations in mixtures containing paraffin oil and table oil clearly illustrated the influence of different proportions of adulterants on the overall isotopic signature, confirming the utility of these techniques in identifying fraudulent blends. Producers and regulators can ensure the authenticity and quality of edible oils, strengthen consumer confidence, and protect consumer rights against fraudulent practices by integrating isotopic analysis and FTIR spectroscopy into quality control systems.

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The authors assert that no identifiable conflicting financial interests or personal connections might be perceived as exerting influence on the research presented in this manuscript.

### Data availability

Data will be made available on request.

### Conflict of interest

The authors declare that they have no conflict of interest.

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