Detection and quantification of olive oil adulteration using fluorescence spectroscopy and chemometric tools

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Abstract. The increasing incidence of fraud in the olive oil market, particularly through adulteration with cheaper oils, poses a major challenge for the industry. This study examines the effectiveness of fluorescence spectroscopy combined with chemometric tools as a method to detect and quantify olive oil adulteration. Chemical analyses were used to measure parameters such as specific absorption coefficients K232 and K270, peroxide value, and acidity, in accordance with International Olive Council (IOC) standards. These measurements were conducted on samples of olive oil blended with varying percentages of adulterating oils. 3D fluorescence spectra were analyzed to examine the changes induced by adulteration. The results show that free acidity and peroxide value decrease with increasing adulteration degree, while the parameters K232 and K270 increase with the degree of adulteration. Principal component analysis (PCA) was effectively used to differentiate samples based on the percentage of adulterant. A partial least squares (PLS) regression model was developed, achieving a correlation coefficient of 0.999 for predicting the percentage of adulterant in olive oil. This PLS model also proved effective in predicting oil quality parameters such as free acidity and K232 and K270 indices. The results of this study demonstrate the significant potential of fluorescence spectroscopy as a rapid and non-destructive method for olive oil authentication. PCA emerges as a powerful tool for characterizing pure oils without requiring sample preparation or destruction. Furthermore, PLS models provide accurate means to predict both the presence of adulterants and olive oil quality parameters. This approach offers a promising solution to combat fraud in the olive oil industry, ensuring product integrity and quality.
Keywords : *adulteration, fluorescence spectroscopy, free acidity, peroxide value, virgin olive oil*.

1 Introduction

Olive oil, derived from the fruit of the olive tree through cold pressing, stands out as a prominent vegetable oil. Abundant in unsaturated fatty acids, particularly oleic acid, it boasts a treasure trove of beneficial bioactive compounds such as squalene, tocopherols, polyphenols (including oleuropein, hydroxytyrosol, and tyrosol), β-carotene, and sterols [1]. Apart from its culinary allure in enhancing flavors, olive oil has garnered attention for its potential health benefits, including the prevention of cardiovascular diseases, reduction of blood lipids, and overall positive impact on human health [2-5].

Because of its superior nutritional profile, olive oil commands a higher price compared to many other edible oils. However, this economic incentive has led to fraudulent practices driven by profit motives and imbalances in supply and demand. Among these deceitful practices, the most common involves blending less expensive vegetable oils such as palm oil or soybean oil with olive oil [6-7]. This fraudulent activity not only violates consumer rights but also jeopardizes public health, breaches legal regulations, disrupts market integrity, and casts doubts on the efficacy of olive oil quality control measures.

A variety of techniques and methodologies have been explored to identify fraudulent practices in olive oil, ranging from sensory analysis to classical chemistry, chromatography, spectroscopy, electronic nose, electronic tongue, and DNA analysis. However, sensory evaluation is susceptible to external influences like inspector bias, environmental conditions, and subjective evaluation standards, leading to low precision, reproducibility, and high labor costs, thus hindering its effectiveness in detecting olive oil adulteration. Classical chemical analysis and chromatography methods primarily focus on quantifying specific chemical constituents (such as acidity index, peroxide index, fatty acids, and tocopherols), which may not offer a comprehensive assessment of oil samples. Despite advancements, electronic nose and electronic tongue technologies are constrained by limited selectivity, sensitivity, and susceptibility to signal interference. Conversely, DNA technology has emerged as a promising tool for detecting olive oil falsification by providing insights into the botanical characteristics, particularly the varieties, of sampled oils [8].

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Currently, spectroscopy methods offer a promising solution to overcome the limitations of traditional analytical techniques for detecting olive oil falsification. Unlike conventional methods that involve complex sample preparations and the use of toxic solvents, spectroscopy techniques provide several advantages including low operational costs, rapid analysis, high sensitivity and precision, non-destructiveness, and elimination of sample preparation processes [9, 7, 10, 11, 12]. Spectral techniques can offer comprehensive chemical information about olive oil by relying on spectral fingerprints rather than targeting specific components. When combined with chemometrics, these methods enable the analysis of large amounts of spectral data through mathematical separation. Consequently, spectroscopy has gained popularity in detecting olive oil falsification, particularly through techniques such as near-infrared spectroscopy (NIR), mid-infrared spectroscopy (MIR), and fluorescence spectroscopy. Moreover, three-dimensional fluorescence spectroscopy (also referred to as excitation-emission matrix spectroscopy (EEM)), when coupled with chemometrics, can effectively process complex fluorescence signals and swiftly provide comprehensive insights into olive oil fluorescence, thus proving to be successful in detecting oil falsification [10-11].

In order to assess the performance of fluorescence as a detection technique for olive oil adulteration, this study aimed to investigate the effects of olive oil adulteration when mixed at different proportions with a table oil.

2 Material and methods

2.1 Sampling

Edible oil samples, which included extra virgin olive oil (sourced from the Tanger-Tetouan-Al Hoceima region) and soybean oil, were acquired from local supermarkets in the Tanger province of Morocco. These samples were securely stored away from light and kept at room temperature until the day of analysis. Soybean oil served as the adulterating agent. Adulterated oil samples were crafted by introducing varying proportions of soybean oil (5 %, 10 %, 15 %, 20 %, 25 %, 30 %, 35 %, 40 %, 45%, 50 %,55%, 60%, 65%, 70%, 75%, 80%, 85%, 90%, 95% and 100 % p/p) into pure olive oil samples. Consequently, 19 adulterated oil samples were generated. Each of these adulterated oil samples underwent a 3-minute vortex mixing process at room temperature.

2.2 Reference chemical analysis

For each sample, Peroxide Value (PV), acidity, and specific UV absorption at 270 nm (K_{270}) and 232 nm (K_{232}) are assessed using the official method outlined by the International Olive Council (IOC), as detailed in documents COI/T.20/Doc. No. 34, COI/T.20/Doc. No. 35, and COI/T.20/Doc. No. 19, respectively. This standardized methodology enables the categorization of olive oil into different grades according to the criteria established by the IOC.

2.3 Sample analysis by fluorescence spectroscopy

Fluorescence spectra were obtained using a JASCO FP-8300 spectrofluorometer at a temperature of 20°C. To minimize the effects of reflected light, scattered radiation, and depolarization phenomena, the excitation radiation was set at an angle of incidence of 60 degrees. Each sample of edible oil, measuring 3 mL, was placed into a cuvette, and fluorescence spectra were recorded. Emission spectra corresponding to tocopherols (305-450 nm), polyphenols (290-450 nm), and chlorophylls (450-800 nm) were captured with excitation wavelengths of 270 nm, 290 nm, and 430 nm, respectively.

2.4 Statistical analysis

Hierarchical Cluster Analysis (HCA) (using Euclidean distance as the metric and average linkage criteria) and Principal Component Analysis (PCA) were conducted utilizing the data analysis tool "XLSTAT 2016". Furthermore, Partial Least Squares Regression (PLS) analysis was carried out using the software "The Unscrambler X". These analytical techniques were applied to the fluorescence measurements of the 3D spectrum as well as the assay results for absorption coefficients K_{232} and K_{270}, peroxide value, and free acidity, individually for each oil sample.

3 Results and discussion

3.1 Fluorescence spectra of olive oil samples

Fluorescence is a phenomenon where molecules absorb energy through photons, leading to the release of fluorescent photons that have a longer wavelength. This process typically requires the presence of substances with a stable conjugated system. However, such molecules are relatively scarce in nature, which means not all substances can exhibit fluorescence. Key contributors to fluorescence in edible oils include elements like vitamin E, phenolic compounds, and various pigments.

Figure 1 shows representative EEM spectra of pure and adulterated olive oil samples and illustrates the changes observed between 100% olive oil (left), 50% and 95% olive oil. Two fluorescent zones were detected in samples of pure and adulterated olive oil.

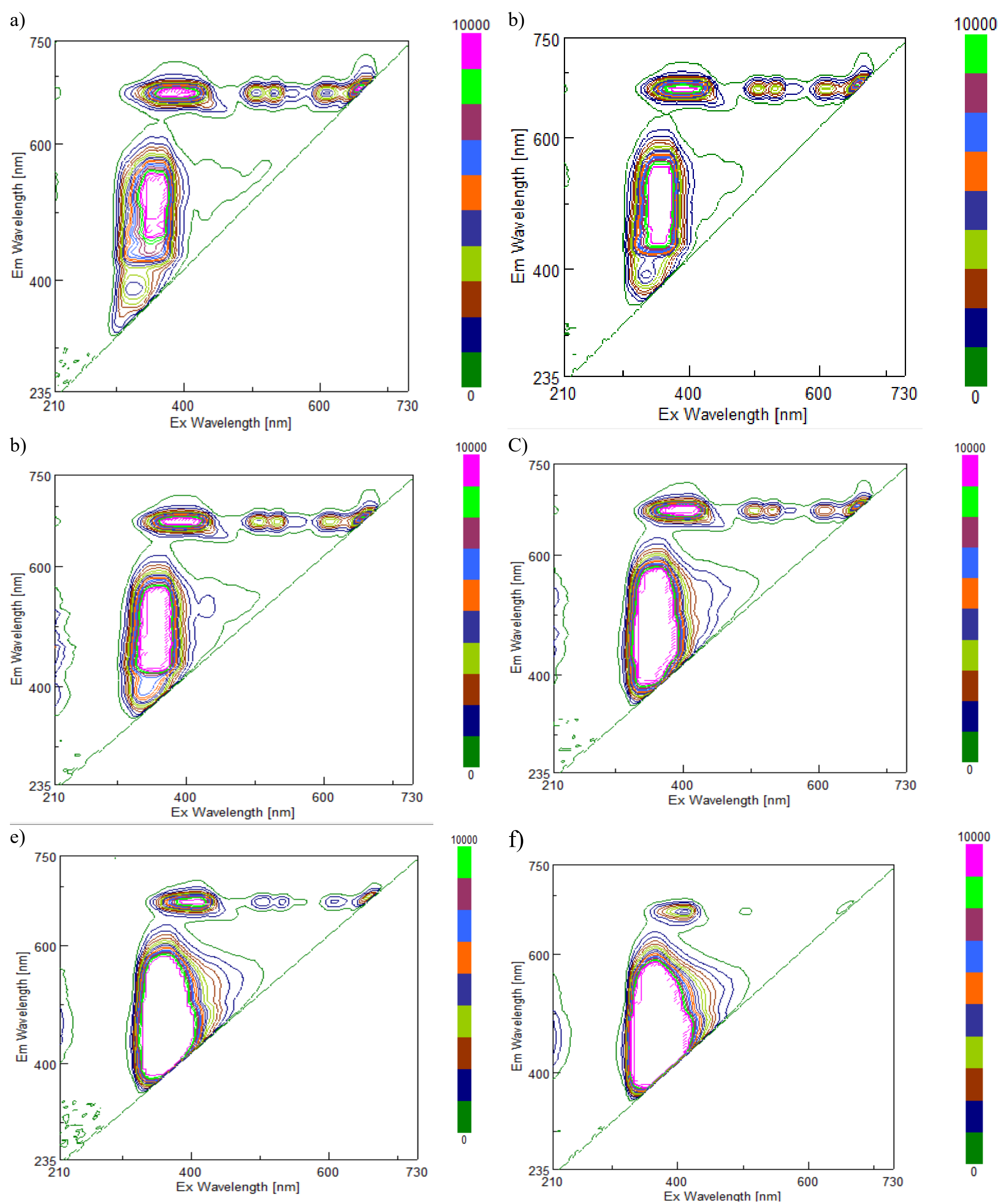


Fig. 1. Raw fluorescence excitation-emission matrices of adulterated olive oil samples a) 100% b) 15% c) 25% d) 50% e) 75% f) 95%.

In the initial area, two fluorescent absorption peaks are observed within the excitation wavelength spectrum of 300-400 nm, and emission occurs within the range of 340-630 nm. Moving to the subsequent area, there are five primary peaks of fluorescent absorption occurring within the excitation wavelength range of 300-700 nm, with emission wavelengths falling within the span of 650-720 nm.

In this investigation, we identified distinct regions of fluorescence associated with different compounds. The short-wavelength area, characterized by excitation wavelengths between 270 and 330 nm and emission wavelengths between 280 and 330 nm, was found to be correlated with phenolic compounds [10]. A prominent characteristic band was observed, attributed to tocopherol and tocotrienol, with excitation ranging from 300 to 360 nm and emission between 350 and 400 nm [15].

Furthermore, we noted the presence of low-intensity doublet bands with excitation occurring between 368 and 420 nm and emission falling within the range of 430 to 470 nm. These bands are indicative of conjugated hydroperoxides and oxidation products of unsaturated fatty acids, as previously reported [14]. Given the high concentration of polyunsaturated fatty acids in soybean oil, the formation of conjugated hydroperoxides through oxidation is facilitated. Consequently, as the level of adulterating soybean oil increases, there is a corresponding rise in EEM fluorescence intensity within this region.

Additionally, a distinct band emerged around 350 nm in excitation and 520 nm in emission, potentially associated with absorptions from tocopherol and β-carotene [16-17].

In the second spectral region, olive oil displays five faint bands, characterized by excitation wavelengths ranging from 320 to 680 nm and emission wavelengths between 650 and 700 nm. These bands are attributed to pheophytin and chlorophyll derivatives [9].

To sum up, as the concentration of adulterating soybean oil increases, there is an escalation in the levels of oxidation products of fatty acids and a decrease in chlorophyll content. Notably, there is a noticeable increase in band intensity within the excitation range of 330-440 nm and the emission range of 660-700 nm. Conversely, a decrease in intensity is observed within the range of excitation at 320-680 nm and emission at 650-700 nm.

Considering that soybean oil and olive oil share similar chemical constituents, adulterated olive oil and pure olive oil samples exhibit analogous spectral patterns, posing challenges for visual differentiation. Therefore, employing chemometric methods to analyze spectral discrepancies becomes imperative for detecting olive oil adulteration.

3.2 Discrimination of the adulterated oils samples based on PCA

Initially, the PCA model was constructed utilizing spectral datasets derived from samples. Subsequently, cluster analysis was conducted, concentrating on distinguishing pure olive oil samples from those adulterated with soybean oil. Preprocessing of the raw spectral data is essential prior to model development in chemometrics, particularly when the measured spectra are influenced by substantial noise, baselines, and other undesirable factors.

In this study, we performed preprocessing by eliminating regions with minimal fluorescence and retained the most pertinent fluorescence information to the fullest extent possible. The spectral range selected for fluorescence data analysis spans from 210 to 730 nm for excitation wavelength and from 235 to 750 nm for emission wavelength.

the fluorescence detection was employed to analyze adulterated olive oil samples, resulting in the generation of two-dimensional datasets. Principal Component Analysis (PCA) was subsequently utilized to analyze these spectral datasets. Most of the variations observed in the spectral measurements of the pure blends can be explained by two principal components, referred to as PC1 and PC2. Together, these two components account for 95.02% of the total variability, with PC1 alone explaining 78.90%.

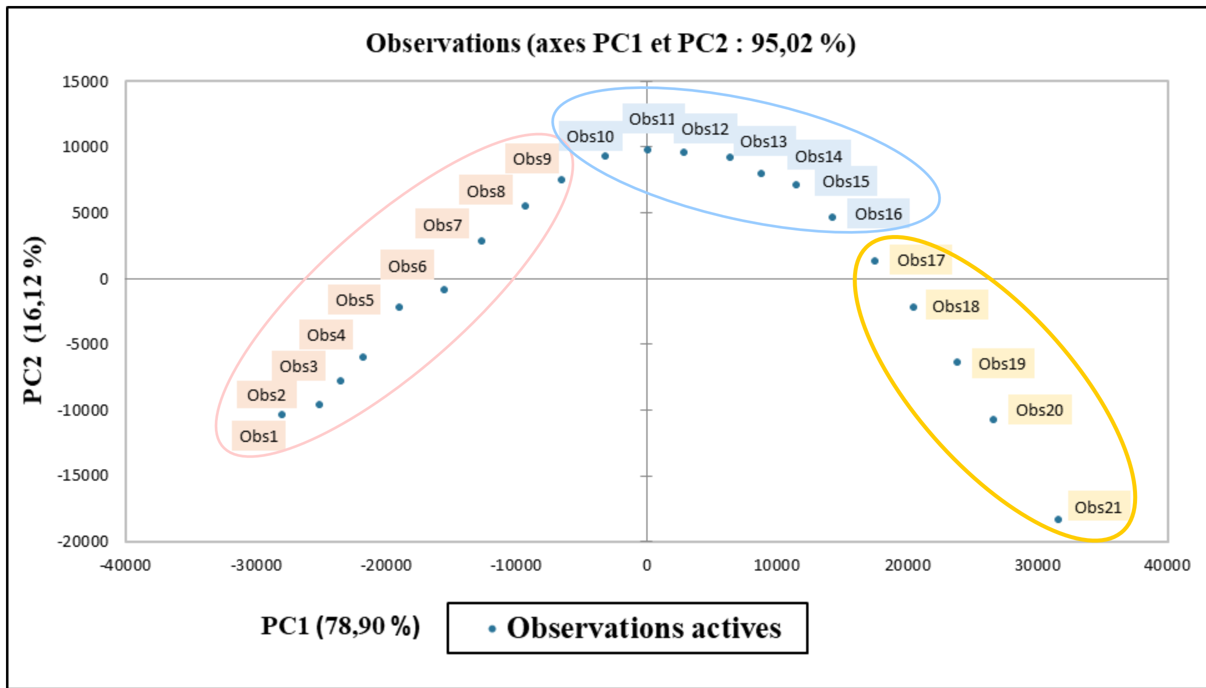


Fig. 2. Distribution through PCA of pure blends based on the variation in fluorescence measurements according to the percentage of adulteration.

The results of the PCA presented in Fig. 2 show that the samples are evenly distributed in a two-dimensional space defined by PC1 and PC2. The samples appear closely positioned to each other, yet there remains a noticeable clustering pattern among olive oil samples with differing degrees of adulteration.

Analyzing this dispersion through Hierarchical Agglomerative Clustering (HAC) allows the classification of samples into three closely related groups (Cluster 1 to 3). The dispersion is organized based on the percentage of olive oil adulteration; Cluster 1 includes non-adulterated olive oil as well as adulterated samples from 5% to 40%, Cluster 2 consists of adulterated samples from 45% to 75%, and Cluster 3 comprises adulterated samples from 80% to 100%.

3.3 Prediction of the adulterated oils samples based on PLS

The analysis of this correlation was performed using the PLS method with processed spectral data. Figure 3 illustrates the correlation between spectral measurements and the percentage of adulterant added to olive oil.

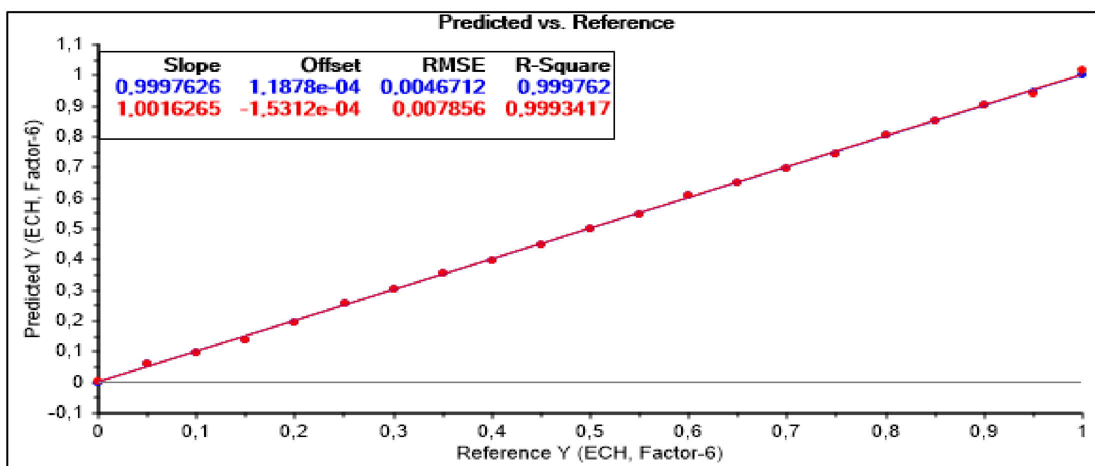


Fig. 3. Correlation between the percentage of adulterant and spectral measurements added to olive oil.

After analyzing the 3D spectral measurements of pure blends using PLS, a model was established to predict the percentage of adulterant. This model exhibits a high correlation coefficient of 0.999, confirming the validity of the applied PLS model. These findings imply that fluorescence spectroscopy can serve as a valuable technique for enhancing the

comprehension and surveillance of adulteration processes. Moreover, they align with the conclusions reported by Ali et al. (2018) [18] and Poulli et al. (2005) [19].

3.4 Physicochemical analysis results of adulterated oils

Figure 4 and table 1 represents the variation of results obtained from acidity (A%), peroxide value (PV), K_{232}, and K_{272} of adulterated oil according to the percentage of adulterant.

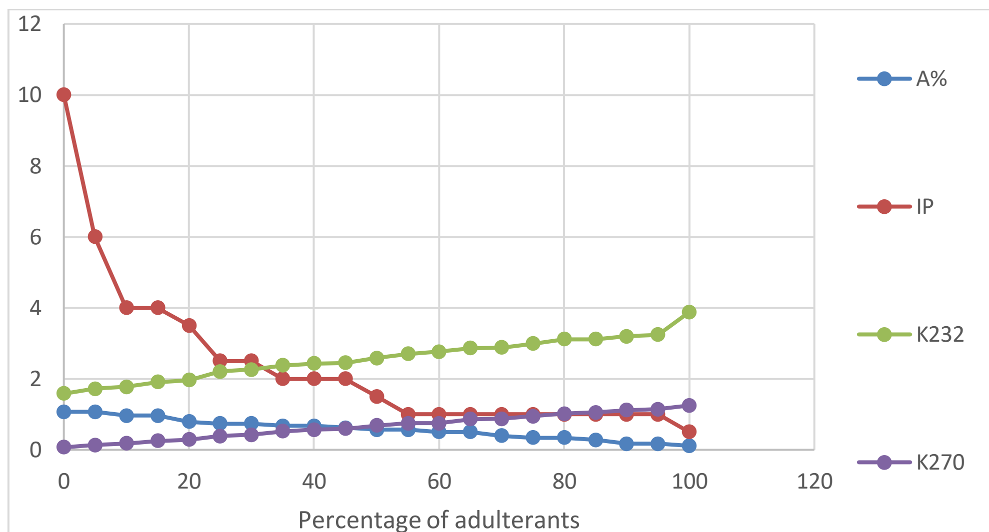


Fig. 4. Variation of results obtained from acidity (A%), peroxide value (PV), K_{232}, and K_{270} of adulterated oil according to the percentage of adulterant.

Table 1. Results obtained from acidity (A%), peroxide value (PV), K232, and K270 of adulterated oil according to the percentage of adulterant.

Le pourcentage ajoutée (%)	A%	IP	K_{232}	K_{270}
0	1,0716	10	1,58087	0,0675991
5	1,0716	6	1,72356	0,13025
10	0,9588	4	1,77628	0,177054
15	0,9588	4	1,91246	0,25353
20	0,7896	3,5	1,96037	0,288674
25	0,7332	2,5	2,20307	0,388045
30	0,7332	2,5	2,26152	0,4236
35	0,6768	2	2,38023	0,51794
40	0,6768	2	2,43278	0,568592
45	0,6204	2	2,44742	0,590337
50	0,564	1,5	2,58235	0,679272
55	0,564	1	2,70344	0,743654
60	0,5076	1	2,76676	0,747828
65	0,5076	1	2,86787	0,86801
70	0,3948	1	2,87964	0,871204
75	0,3384	1	2,99088	0,941106
80	0,3384	1	3,11275	1,01655
85	0,282	1	3,11378	1,05491

90	0,1692	1	3,1978	1,11364
95	0,1692	1	3,24506	1,14063
100	0,1128	0,5	3,88048	1,24884

The results obtained in this study highlight the significant impact of adulterating olive oil with table oils on key physicochemical parameters, such as free acidity and peroxide value. A notable decrease in these parameters was observed with the increase in the percentage of table oil, indicating a complex interaction between the components of the two types of oils.

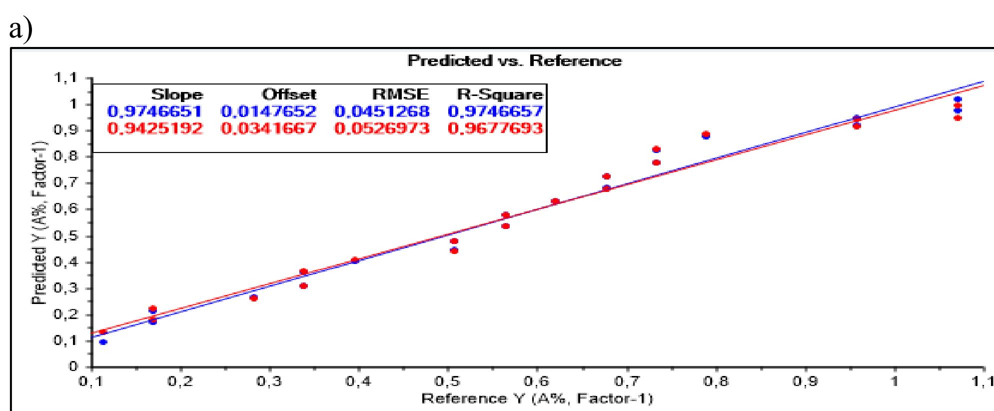
The differences in fatty acid composition between olive oil and table oils appear to play a crucial role in diluting acidity. Table oils, often derived from advanced refining processes, contain a lower amount of free fatty acids, thus contributing to the reduction of the overall acidity of the mixture. This observation suggests that adulteration alters the lipid profile of olive oil, potentially affecting its nutritional and organoleptic qualities. The impact of adulteration on the peroxide value reveals the significance of the refining treatments undergone by table oils. These treatments, aimed at improving the stability and neutrality of the oils, seem to confer protection against oxidation when mixed with olive oil. This could indicate that the integration of refined table oils may artificially improve the oxidative stability indicators of adulterated olive oil.

Conversely, the values of the absorption coefficients K_{232} and K_{270} increase proportionally with the increase in the percentage of table oil. This elevation signals an increased absorption of light at these specific wavelengths in the adulterated oil. The absorption coefficients K_{232} and K_{270} are commonly used as indicators of olive oil quality, and their increase suggests changes in the oil composition due to the incorporation of table oil.

3.5 Analysis of the correlation between spectral measurements and chemical analysis

This analysis is carried out using PLS. For this purpose, two data matrices were constructed: the first contained the spectral data, and the second grouped the results of the chemical criteria (peroxide value, free acidity, extinction coefficients K_{232} and K_{270}).

a)



b)

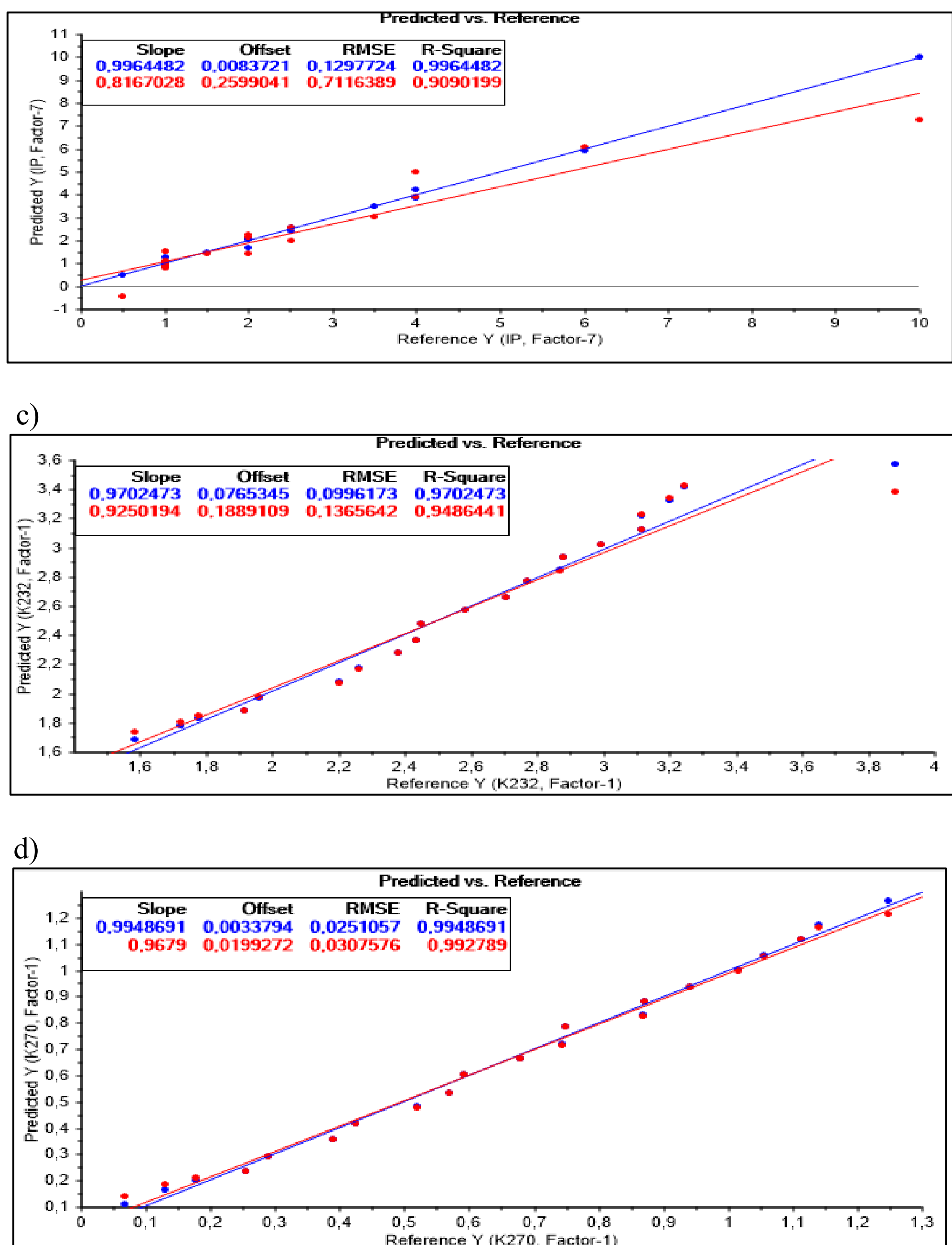


Fig. 5. Correlation between the different values obtained for a) free acidity b) peroxide value c) K_{232} d) K_{270} of adulterated olive oil at various percentages against the values predicted by the acidity model, the peroxide value model, and the K_{232} and K_{270} mod, respectively.

The analysis revealed a strong correlation between the measured values of free acidity and those predicted by the model, with a correlation coefficient of 0.96, confirming the validity of the mathematical model used (PLS). A moderate correlation was observed between the measured peroxide index values and the model predictions, with a correlation coefficient of 0.9. This suggests that the prediction model was not able to provide highly accurate results, especially for high doses of table oil. It is possible that the official method used in the laboratory for peroxide analysis could introduce errors, as it may be heavily influenced by external factors. This influence could explain the high calibration error values observed for this parameter, consistent with findings by Guimet et al. (2005) [20], who also used 3D fluorescence to construct a peroxide index calibration model. Furthermore, a strong correlation was found between various measured values of the extinction coefficients K_{232} and K_{270}, with correlation coefficients of 0.94 and 0.99, respectively,

demonstrating the validity of the applied mathematical models (PLS). The most promising outcomes were achieved in predicting K270, enabling the detection of degraded olive oils.

4 Conclusion

In this study, we explored the use of fluorescence spectroscopy in conjunction with chemometric tools for assessing the adulteration of olive oil with soybean oil. Through 3D spectrofluorometric analyses, we established a significant correlation between the degree of olive oil adulteration and variations in spectral measurements, analyzed using advanced chemometric techniques. Principal Compo

nent Analysis (PCA) proved effective in discriminating between pure olive oil samples and those adulterated with soybean oil, while Partial Least Squares (PLS) regression accurately quantified the percentage of adulteration.

The study also highlighted the impact of adulteration on various physicochemical quality parameters of olive oil, including peroxide value, acidity, and UV absorbance indices K_{232} and K_{270}. A correlation between these quality criteria and spectral data was established, underscoring the significance of these measurements in evaluating the integrity of olive oil.

In conclusion, our findings confirm the potential of the spectrofluorometric method combined with chemometric analyses as an advanced inspection technique for the characterization of olive oils. This approach offers a rapid, non-destructive, and reliable method for identifying olive oil adulteration and assessing the degree of falsification, this marks a notable advancement in endeavors aimed at ensuring the quality and authenticity of food products available in the market.

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