Control of olive oil adulterated by soybean oil in a population where low olive oil consumption is an issue. A case study in Brazil

Controle de azeite adulterado com óleo de soja em uma população onde o baixo consumo de azeite é um problema. Um estudo de caso no Brasil

Control del aceite de oliva adulterado con aceite de soja en una población donde el bajo consumo de aceite de oliva es un problema. Un estudio de caso en Brasil

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Abstract
The objectives of this work were to evaluate the ability of spectroscopic techniques to distinguish unadulterated and adulterated olive oil (OL) and to investigate consumers perception of adulterated OL samples by sensory tests in a population with low OL consumption. The adulterated OL samples were prepared with the addition of soybean oil (SO) in varying concentrations of 5 to 50% (v v-1). Samples were characterized using Fourier transform infrared (FTIR) spectroscopy and ultraviolet-visible (UV–visible) absorption spectroscopy. The capability of spectroscopy techniques to distinguish unadulterated and adulterated OL was evaluated by using a chemometric tool, principal component analysis (PCA). A questionnaire about the frequency of OL consumption was used. Multiple comparison and hedonic tests were used to discriminate and evaluate the acceptance of the adulterated OL samples. FTIR and UV–visible spectroscopy coupled with PCA proved to be good instruments to be used to distinguish OL samples intentionally adulterated with the addition of 5, 10, 15, 20, 30, 40, and 50% SO, and have great potential for classification of adulteration involving OL. The results confirmed that this group of Brazilian consumers has low OL consumption. Consequently, there was low discrimination of adulterated OL and they had the same acceptance as unadulterated and adulterated OL.
Keywords: Correspondence analysis; Multiple comparison test; Principal component analysis; Sensory analysis; Spectroscopy techniques.

Resumo
Os objetivos deste trabalho foram avaliar a capacidade das técnicas espectroscópicas de distinguir o azeite não adulterado e adulterado e investigar a percepção de amostras de azeite adulteradas por testes sensoriais em uma população com baixo consumo de azeite. As amostras de azeite adulterado foram preparadas com a adição de óleo de soja em concentrações variadas de 5 a 50% (v v-1). Amostras não adulteradas e adulteradas foram caracterizadas usando espectroscopia de infravermelho com transformada de Fourier (FTIR) e espectroscopia de absorção na região do ultravioleta-visível (UV-visível). A capacidade das técnicas de espectroscopia para distinguir o azeite não adulterado e o adulterado foi avaliada usando a ferramenta quimiométrica, análise de componentes principais (ACP). Um questionário sobre a frequência do consumo de azeite foi aplicado. Os testes de comparação múltipla e hedônico foram empregados para avaliar a discriminação e aceitação das amostras de azeite adulteradas. A espectroscopia FTIR e UV-visível acoplada a ACP demonstrou ser uma boa ferramenta para distinguir amostras de azeite intencionalmente adulteradas com a adição de 5, 10, 15, 20, 30, 40 e 50% de óleo de soja e apresentou um grande potencial na classificação de adulteração envolvendo azeite. Os resultados confirmaram o baixo consumo de azeites pelos brasileiros. O que por consequência levou à uma reduzida discriminação dos azeites adulterados e à mesma aceitação de amostras adulteradas e não adulteradas. Em uma população com baixo consumo de azeite, mais vulnerável a

fraudes, o estudo do uso de técnicas sensíveis e rápidas para a detecção de fraudes surge como uma estratégia importante.

Palavras-chave: Análise de correspondência; Teste de comparação múltipla; Análise de componentes principais; Análise sensorial; Técnicas espectroscópicas.

Resumen

Los objetivos de este trabajo fueron evaluar la capacidad de las técnicas espectroscópicas para distinguir el aceite de oliva adulterado y sin adulterar e investigar la percepción de muestras de aceite de oliva adulterado mediante pruebas sensoriales en una población con bajo consumo de aceite de oliva. Se prepararon muestras de aceite de oliva adulterado con la adición de aceite de soja en concentraciones variables (5-50% (v v-1)). Las muestras adulteradas y sin adulterar se caracterizaron mediante espectroscopia infrarroja transformada de Fourier (FTIR) y espectroscopia de absorción ultravioleta-visible (UV-visible). La capacidad de las técnicas de espectroscopia para distinguir el aceite de oliva adulterado y sin adulterar se evaluó utilizando la herramienta quimiométrica, análisis de componentes principales (PCA). Se aplicó un cuestionario sobre la frecuencia de consumo de aceite de oliva. Se utilizaron pruebas hedónicas y de comparación múltiple para evaluar la discriminación y aceptación de muestras de aceite de oliva adulterado. La espectroscopía FTIR y UV-visible junto con ACP demostraron ser una buena herramienta para distinguir muestras de aceite adulteradas intencionalmente con la adición de 5, 10, 15, 20, 30, 40 y 50 % de aceite de soya y mostraron un gran potencial en la clasificación de adulteraciones que involucran aceite de oliva. Los resultados confirmaron el bajo consumo de aceite de oliva por parte de los brasileños. En consecuencia, esto condujo a una discriminación reducida de aceites adulterados y la misma aceptación de muestras adulteradas y no adulteradas. En una población con bajo consumo de aceite de oliva, más vulnerable al fraude, el estudio del uso de técnicas sensibles y rápidas para la detección del fraude surge como una estrategia importante.

Palabras clave: Análisis de componentes principales; Análisis de correspondencia; Análisis sensorial; Prueba de comparación múltiple; Técnicas de espectroscopia.

1. Introduction

Olive oil (OL), a highly valued vegetable oil, is the product of the extraction of olive fruits (Gonçalves et al., 2022; Minuceli et al., 2021). According to Dais e Hatzakis (2013), it is one of the best sources of fatty acids and natural antioxidants, e.g. polyphenols, tocopherols, among other important minor components, such as sterols, hydrocarbons, and terpenic acids, resulting in a complex composition.

The high nutritional value and pleasant sensory characteristics are important advantages of this vegetable oil compared to seed oils (such as rapeseed oil, soybean oil, and corn oil) (Guzmán et al., 2015; Milanez et al., 2017). These characteristics offer high quality and high commercial value to the product, making it a frequent target of adulteration by the addition of vegetable oils or OL of lower quality (Johnson, 2014; Yan et al., 2020).

In Brazil, the government recently announced fraud in more than 79 market brands in the last two years. And this condition is especially worrying for a population with low OL consumption, reducing the chance of sensory identification of fraudulent samples. According to Aued-Pimentel et al. (2017) consumption per capita in Brazil is about 0.3 kg/habitant year, very low when compared with countries such as Spain and Italy, where consumption is about 20 kg/habitant year. And as is well known, the consumption of adulterated OL leads to health and safety issues for consumers, primarily if it is being purchased for its nutritional and health-promoting benefits (Meenu et al., 2019). Internationally, the regulation and supervision of OL are carried out by the European Union Commission (EEC, 1991), Codex Committee on Fats and Oils (CCFO, 2017), and the International Olive Council (IOC, 2015). In Brazil, OL quality parameters are established and enforced by Ministério da Agricultura, Pecuária e Abastecimento (MAPA) and by Agência Nacional de Vigilância Sanitária (ANVISA). Studies have reported success in detecting adulteration using spectroscopic and chemical techniques, both quantitatively and qualitatively (da Silveira et al., 2017; Dais & Hatzakis, 2013; Jiménez-Carvelo et al., 2017; Li et al., 2018; Milanez et al., 2017). However, it is worth noting the lack of studies that explore OL adulteration results by spectroscopic techniques correlated with discriminatory sensory tests by untrained panel.

Sensory analysis is recognized as one of the most important instruments for determining the quality of OL (Fernandes

et al., 2018) and has been applied especially to assess the OL profile by trained panels (Delgado & Guinard, 2012; Lukić et al., 2018; Óǧüçü & Yilmaz, 2009; Quintanilla-Casas et al., 2020), and experts (Delgado & Guinard, 2012). To the best of our knowledge, no literature data are available to explore the consumer`s or untrained panel`s perception of adulterated olive oil.

In this sense, the objectives of this study were: (i) to evaluate the ability of spectroscopic techniques, Fourier transform infrared (FTIR) and UV-visible absorption associated with chemometric tools to detect OL adulterated with soybean oil (SO); (ii) investigate consumer`s perception of adulterated OL samples by sensory tests in a population with low OL consumption.

2. Methodology

2.1 Oil Samples

Ten oil samples (SO, corn oil (CO), sunflower oil (SU), canola oil (CA), and OL) from different brands were acquired in a local market in Pato Branco - PR, Brazil. For each sample, two brands were analyzed, acquired on the same date and at the same commercial establishment. The oil samples (unadulterated oils) consisted of 2 SO, 2 CO, 2 SU, 2 CA, and 2 OL (Table 1).

Table 1. Identification of the vegetable oil samples (unadulterated and adulterated) analyzed in the study.

Samples (unadulterated oil)	Vegetable Oils (%)	Identification Oil
1	Soybean (100%)	SO
2	Soybean (100%)	SO
3	Sunflower (100%)	SU
4	Sunflower (100%)	SU
5	Canola (100%)	CA
6	Canola (100%)	CA
7	Corn (100%)	CO
8	Corn (100%)	CO
9	Olive (100%)	OL
10	Olive (100%)	OL
Samples (adulterated oil)		
11	Olive (95%) + Soybean (5%)	OL5
12	Olive (90%) + Soybean (10%)	OL10
13	Olive (85%) + Soybean (15%)	OL15
14	Olive (80%) + Soybean (20%)	OL20
15	Olive (70%) + Soybean (30%)	OL30
16	Olive (60%) + Soybean (40%)	OL40
17	Olive (50%) + Soybean (50%)	OL50

Source: Authors (2022).

2.2 Adulteration of OL with SO

The OL were intentionally adulterated with the addition of SO. The percentage of the OL varied among 95 and 50% (v v^{-1}), and the percentage of SO varied among 5 and 50% (v v^{-1}) in the oil blend samples. According to the percentage of SO in the OL, samples were identified as OL5, OL10, OL15, OL20, OL30, OL40, and OL50 (Table 1).

This study was focused on the evaluation of the addition of SO to OL that is a common problem of OL bottled in Brazil, a major SO producer (Fasciotti & Pereira Netto, 2010). In addition to that, the frequent use of SO is also due to the cost of this vegetable oil (a cheap oil) compared to the others (SU, CO, and CA).

All samples were preserved in their original containers under refrigeration (6 to 10 °C) and sheltered from light.

2.3 Spectroscopy Characterization

FTIR spectra were acquired in a Frontier Spectrophotometer (Perkin Elmer, USA) equipped with an accessory of attenuated total reflectance (ATR). For measurement, a drop of oil (approximately 0,1 mL) was deposited on the surface of the crystal (ZnSe). The infrared spectra were recorded in the range from 4000 to 400 cm^{-1}, at 4 cm^{-1} resolutions and 32 scans. Two replicates of each sample were measured in identical conditions.

UV–visible absorption spectra were obtained in the range of 300 to 600 nm using a UV-visible spectrophotometer model lambda 40 (Perkin Elmer, USA). The cells are rectangular quartz cuvettes with a path length of 1 cm.

The SO, CO, SU, and adulterated OL were diluted 1:50 (oil samples: hexane) (v v^{-1}), and the OL was diluted 1:10 (oil sample: hexane) (v v^{-1}).

Three replicates of each sample were measured in identical conditions at 25 ºC.

2.4 Sensory analysis

Fifty-two assessors (20-50 years old) participated of the Multiple Comparison Test for discrimination and hedonic evaluations of oil samples. The participants also answered a questionnaire on OL consumption. The study was approved by an appropriate research ethics review committee number CAAE 02040818.8.0000.5547.

2.4.1 Multiple Comparison Test (CM) for discrimination of oil samples

In the multiple comparison test, the differences were evaluated for a specific attribute and the degree of difference of three or more samples in relation to a control sample (ISO, 2016). For this study, the evaluators were provided with a standard sample (OL) and five more coded test samples of adulterated OL (5, 10, 15, 20, 30%). The evaluators were asked to evaluate each test sample in relation to the standard sample according to flavor attribute, using the 9-point difference degree scale, ranging from "Extremely less intense than the standard" to "Extremely more intense than the standard".

2.4.2 Hedonic evaluation of the oil samples and frequency of OL consumption

The hedonic test was applied using a facial 'smiley' scale with seven points. Each consumer was asked to mark with an X over the emoji that identified their level of global acceptance of the tested product (Deubler et al., 2020). "How frequently do you consume OL?" was the question referring to OL consumption and was measured on a 7-point scale: (7) Daily- (6) several times a week- (5) weekly- (4) several times a month- (3) monthly- (2) less than monthly- (1) never.

2.5 Statistical analysis

The principal component analysis (PCA) was developed using Pirouette software (version 4.0, Infometrix Inc., Woodville, WA, USA) to obtain a global view of the main variation in the data set.

In this study, the areas between 4000 and 3060 cm^{-1}, 2788 and 2414 cm^{-1}, and 2298 and 1790 cm^{-1} from FTIR spectra for all oil samples were excluded from the PCA. These regions were not included in the analysis for showing a low signal to noise ratio. PCA score graph was carried out using the UV-visible spectral data (300-600 nm) from all oil samples.

Before the analysis, the FTIR and UV-visible spectral data were subjected to data pre-processing, mean-centered, and first derivative, to eliminate the baseline drift and variability associated with the intensity.

PCA was selected to provide an overview of the capacity to distinguish between vegetable oils and unadulterated and adulterated OL based on spectral data obtained by FTIR and UV-visible spectroscopy. According to Gok et al. (2015) and

Amit et al. (2020), PCA is considered an approach for complex data sets collected by the spectroscopic technique to see the differences and relationships between samples.

The PCA was carried out using three spectra of each oil sample. This makes it possible to predict the composition uniformity of oils, from the same brand and from different brands.

On Multiple Comparison data, Analysis of Variance (ANOVA) analysis was used. To compare the averages of the test samples with the average of the control sample, the Dunnet test was applied.

The acceptance index was calculated from the obtained average, with the maximum value of the scale (7) representing 100%. Tukey test was applied to evaluate the significance among means.

Correspondence analysis was applied to verify the association between the frequency of OL consumption and the hedonic test using Statistic 12.7 (Mitterer Daltoé et al., 2017).

3. Results

3.1 Spectroscopy techniques

Figure 1 shows the FTIR spectra from SO, CO, SU, CA, and OL of all brands.

Overall, the FTIR spectra exhibit a similar spectroscopic signature. In general, the oils (SO, CO, SU, CA, and OL) showed signals around 3004; 2922; 2854; 1744; 1464; 1378; 1236, and 1162 cm^{-1} (Figure 1). This confirms the similar spectroscopic characteristics of selected oils. This section may be also divided by subheadings. It should provide a concise and accurate description of the experimental results, their interpretation as well as the experimental conclusion that can be drawn.

Figure 1. FTIR spectra of five different types of vegetable oil samples: SO, CO, SU, CA, and OL.
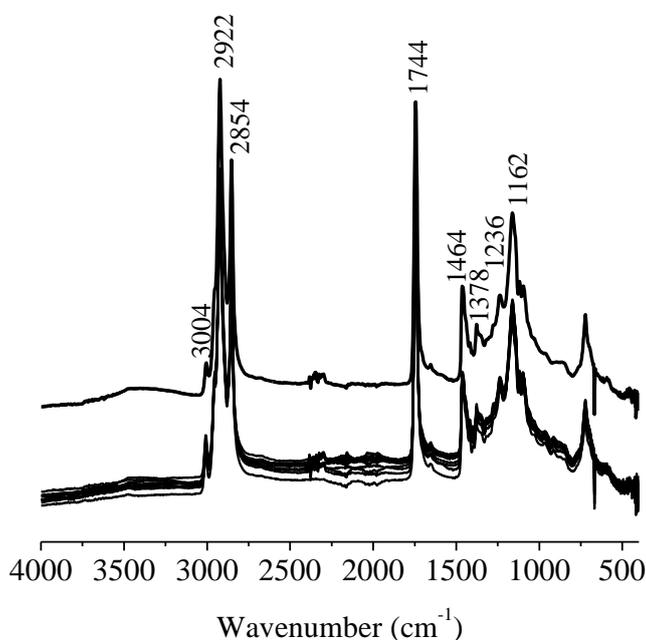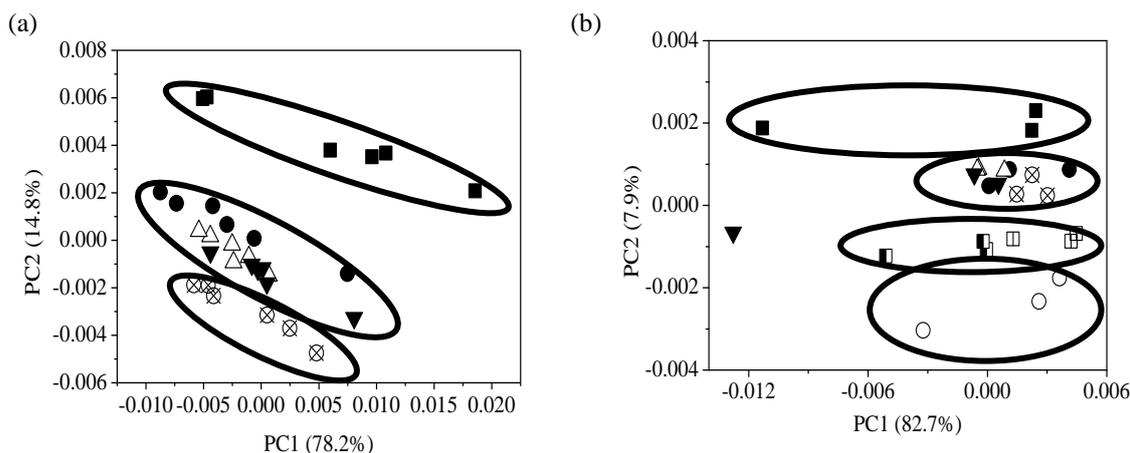

Source: Authors (2022).

For all the oils, the strong FTIR intensity signs were observed around 2922 and 1744 cm^{-1}, due to -C–H (-CH$_2$) and –C=O (ester group) stretching, respectively (Figure 1). In oils spectra signals were also observed around 3004, 2854, 1464, and 1378 cm^{-1} due to –C–H stretching, around 1236 cm^{-1} due to –C–O stretching, and around 1162 cm^{-1} which corresponds to –C–O–CH$_2$– stretching/ banding (Figure 1) (Amit et al., 2020; Naranjo & Baliga, 2012).

PCA score graph was carried out on all FTIR spectral data of oils (Figure 2). The PCA (Figure 2a) showed the correlation between the oil samples (OL, CA, SU, CO, and SO). The PCA (Figure 2b) showed the to distinguish amongst the OL (unadulterated OL, and OL intentionally adulterated). In the present study, PCA was applied for extracting principal components (PC), and for the selection of the most informative variables (wavenumber) for analysis (Amit et al., 2020).

Figure 2. PCA scores plot of FTIR data from (a) (■) OL, (●) CA, (Δ) SU, (▼) CO and (⊗) SO, and (b) (■) unadulterated OL, and OL intentionally adulterated with the addition of SO (●) 5%, (Δ) 10%, (▼) 15%, (⊗) 20%, (⊞) 30%, (◨) 40%, and (○) 50%.

Source: Authors (2022).

PCA is considered an approach for complex data sets collected by the spectroscopic technique to see the differences and relationships among samples (Gok et al., 2015).

The PCA score graph obtained with all FTIR spectral data of oils shows that the PC1 and PC2 demonstrated that 93.0% of the variations and differences in the spectral data were revealed (Figure 2a). As observed in Figure 2a, the PC2 (14.8%) provides better visualization of the separation amongst the samples and suggests the formation of three clusters. The first group, formed by OL samples has a high positive score in the PC2. The second group, formed by CA, SU, and CO samples has intermediate scores in the PC2, around zero. In the end, the third group had a negative score in the PC2 and is formed by SO samples.

The score graph confirms the differences in the structural composition of the oils analyzed. The OL presents oleic and linoleic acids in the composition. The oleic acid, chemical formula $CH_3(CH_2)_7CH=CH(CH_2)_7COOH$, is the dominant component (55–83%). The percentage of linoleic acid, chemical formula $CH_3(CH_2)_4CH=CHCH_2CH=CH(CH_2)_7COOH$, is between 3.5 and 21% (Beltrán et al., 2004). In contrast, seed oils (SU, CO, and SO) contain higher concentrations of linoleic acid (Bellaloui & Gillen, 2010; Zhang et al., 2011).

In Figure 2a the OL are clearly separated from the others according to PC2, and the largest variations occur between OL and SO. The results suggest that FTIR combined with multivariate chemometrics tools (PCA) can help in detecting adulteration in OL with SO.

The FTIR spectra from OL intentionally adulterated with the addition of 5, 10, 15, 20, 30, 40, and 50% SO (data not shown) exhibited a spectroscopic signature similar to other oils and to unadulterated OL (Figure 1). This similarity between the FTIR spectra suggests the need for statistical tools to assist in the interpretation of data.

In Figure 2b the PCA score graph of OL intentionally adulterated with the addition of SO (5, 10, 15, 20, 30, 40, and 50%) and unadulterated OL samples are shown. It is believed that the PCA provides an overview of the capacity to distinguish among unadulterated and adulterated OL based on spectral data obtained by FTIR spectroscopy.

In Figure 2b, the PC1 and PC2 can explain 91.9% of the total variance in the OL samples. The PCA score graph showed that the adulterated and unadulterated OL samples can be clustered into four groups. The first group formed by unadulterated OL samples has a high positive score in the PC2. The second group, formed by OL samples intentionally adulterated with the addition of 5, 10, 15, and 20% SO have intermediate scores in the PC2, around zero. The third group has negative score in the PC2 (Figure 2b) and is formed by OL samples intentionally adulterated with the addition of 30 and 40% SO.

At least the present intentionally adulterated OL samples by the addition of 50% SO. (Figure 2b). PCA results suggest that it is possible to discriminate between unadulterated and unadulterated OL (5, 10, 15, 20, 30, 40, and 50%).

The results obtained show that the presence of SO (as adulterant) in OL can be detected by FTIR spectroscopy and statistical tools (PCA) (Figure 2b). The PCA can be used to successfully distinguish pure and adulterated OL samples with an addition of 5 to 50% SO.

The UV-visible absorption measurements allowed spectroscopic information concerning the pigments of the oil samples to be obtained. SO, CO, SU, CA, and OL, and different mixtures of OL intentionally adulterated with the addition of SO (5, 10, 15, 20, 30, 40, and 50% SO) were analyzed. Figure 3 shows the UV-visible absorption spectra from SO, CO, SU, CA, and OL.

Figure 3. UV-visible absorption spectra of the vegetable oil samples: (a) SO and CA, (b) CO, (c) SU, and (d) OL.
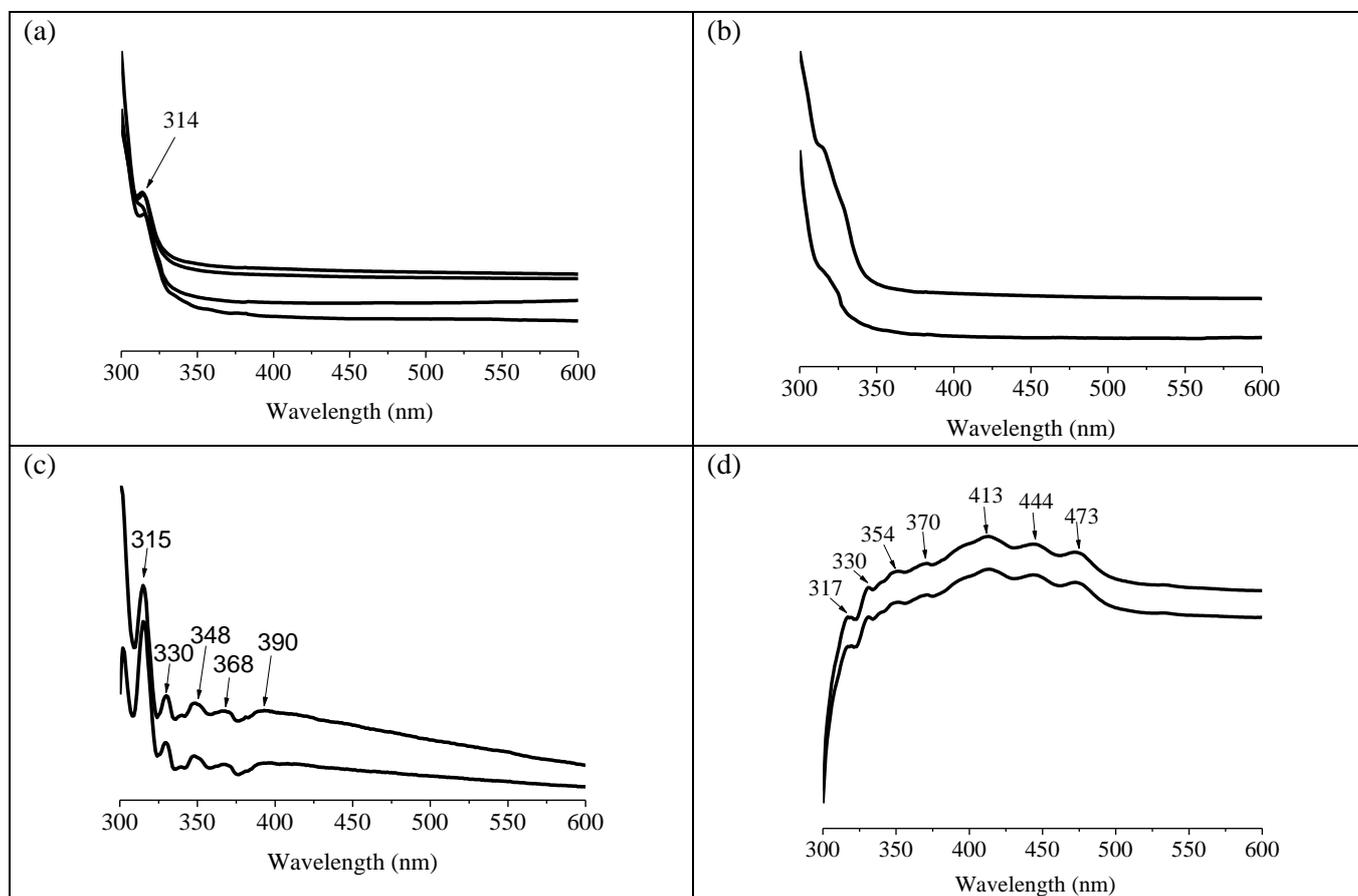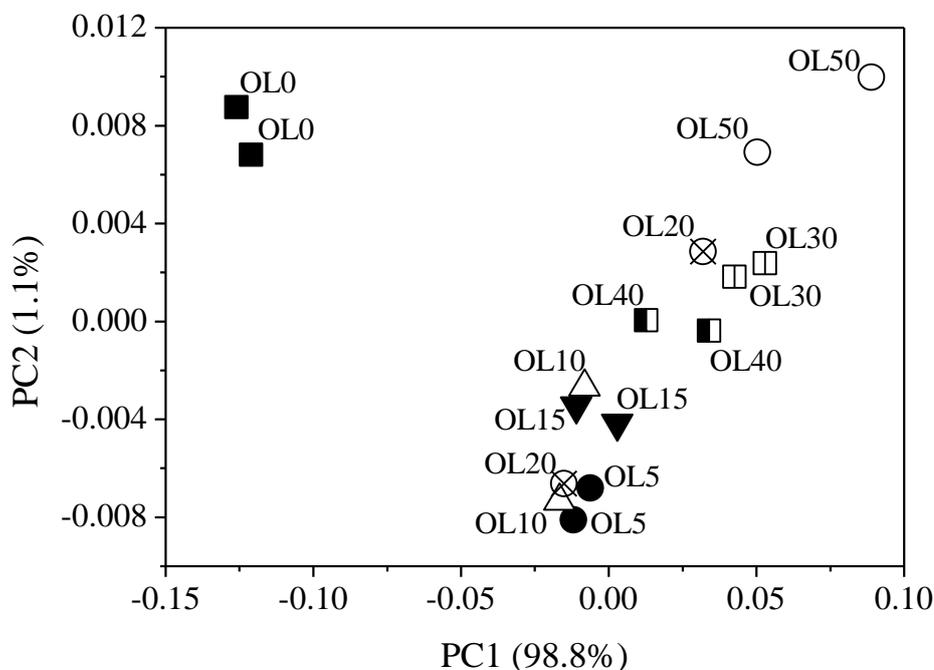

Source: Authors (2022).

In Figure 3, the oils analyzed showed differences in the UV-visible absorption bands. In SO and CA spectra 314 nm signals were observed (Figure 3a). The SU presented an absorption band of around 315, 330, 348, 368, and 390 nm (Figure 3c), and OL of around 317, 330, 354, 370, 413, 444, and 473 nm (Figure 3d). The differences can be associated with the vegetable (CO, SO, CA, and SU), and OL from which the oil was extracted, and suggest this spectroscopy as a technique able to distinguish between the oil samples.

OL spectra have absorption bands in the region between 400 and 550 nm that corresponds to the absorption of carotenoid and chlorophyll pigments (Giacomelli et al., 2006). The main carotenoid pigments in OL are β-carotene and lutein, while pheophytin is the main chlorophyll pigment (Moyano et al., 2010).

In Figure 4 is presented the PCA score graph of all UV-visible spectral data of OL unadulterated and OL intentionally adulterated with the addition of SO (5, 10, 15, 20, 30, 40, and 50%). This study with unadulterated OL samples was carried out to provide an overview of the ability to distinguish among unadulterated and adulterated OL based on spectral data obtained by UV-visible spectroscopy.

In Figure 4, the PCA score graph plotted with UV-visible spectral data showed that the PC1 and PC2 could explain 99.9% of the total variance in the OL samples. The PCA score graph showed that the adulterated and unadulterated OL samples can be clearly separated according to PC1 (98.8%). The unadulterated OL (OL0) samples have negative values in the PC1 and the intentionally adulterated OL samples with the addition of 5 (OL5), 10 (OL10), 15 (OL15), 20 (OL20), 30 (OL30), 40 (OL40), and 50% (OL50) SO have positive values in the PC1 (Figure 4).

Figure 4. PCA scores plot of UV-visible data from (■) OL unadulterated, and OL intentionally adulterated with the addition of SO (●) 5%, (□) 10%, (▼) 15%, (⊗) 20%, (⧈) 30%, (◧) 40%, and (○) 50%.

Source: Authors (2022).

The OL samples intentionally adulterated with the addition of SO can be distinguished according to PC2 (1.1%). The PC2 provides better visualization of separation among the adulterated samples, and suggests the formation of two clusters. The

first group is formed of adulterated OL samples (OL30, OL40, and OL50) has a high positive score in the PC2. The second group is formed of adulterated OL samples (OL5, OL10, OL15, and OL20) and has negative scores in the PC2, around zero. The score plot confirms the differences in the structural composition of the OL analyzed and suggests that it is possible to discriminate between adulterated OL using UV-visible spectral data and chemometrics tools.

3.2 Sensory Analysis

In order to verify at what level of adulteration consumers are able to perceive differences in flavor, the multiple comparison test was applied. Table 2 presents the results of the Analysis of Variance (ANOVA) and Dunnet test. The results indicated a difference between the flavour of the standard (OL) and the test samples (p=0.012217). The signal of difference between the standard and any of the adulterated samples by ANOVA, Dunnet test was applied. It was noted that the difference between the standard appeared in the sample adulterated with 20 % SO. Therefore, it can be confirmed that this group of consumers does not perceive adulteration of OL until there is a concentration of 15% SO. In other words, adulteration of up to 15% can occur without sensory perception.

This behavior can be better understood analyzing the data from OL consumption. On a scale of 7 points, the consumers that took part in the study presented a mean of 4.6, corresponding to OL consumption between several times a month and weekly. This result, not only possibly justifies the low perception of adulterated OL but also highlights the low consumption of OL by this group of Brazilians. The recommended consumption of OL is a half tablespoon per day (Guasch-Ferré et al., 2020). This daily consumption is supported by the evidence that OL consumption is beneficial for human health, particularly for the prevention of cardiovascular diseases, breast cancer, and type 2 diabetes mellitus (Foscolou et al., 2018).

Table 2. ANOVA results and Dunnet test between standard sample and each test sample.

ANOVA					
Effect	Sum of squares	Degree of freedom	Mean squares	F value	P value
Intercept	7463.705	1	7463.705	1546.830	<0.0001
Sample	71.795	5	14.359	2.976	0.012217
Error	1476.500	306	4.825	-	-
Dunnet test					
Sample			p value		
Standard			-		
5%			0.999892		
10%			0.563677		
15%			0.977349		
20%			0.025854		
30%			0.037490		

Source: Authors (2022).

Until now, this group of Brazilian consumers had a low discrimination of adulterated OL, which can be justified by low OL consumption. To reinforce this lack of habit, Table 3 presents the results of hedonic tests. As can be seen, all samples showed a low acceptance index, of around 60 %, with non-significant differences by Tukey test. It means that for the consumers the OL sample (standard) had the same hedonic perception as the OL sample adulterated with 30% SO.

Table 3. Acceptance of oil samples by hedonic test.

| Samples	Mean	Acceptance index (%)
OL (standard)	4.25[a]	60.7
5%	4.17[a]	59.6
10%	4.31[a]	61.6
15%	4.41[a]	63.0
20%	4.35[a]	62.1
30%	4.31[a]	61.6

Equals letters represent non-significant differences by Tukey test $p \geq 0.05$. Source: Authors (2022).

It is important to note that the fact that they accept at the same intensity does not mean that they are not able to discriminate differences, as occurred in the multiple comparison test. However, these results can have negative consequences for this group of consumers, since they are more vulnerable to adulterated products, mainly if they are being consumed due to the nutritional appeal that olive oil offers.

To better explore the relationship between hedonic perception of OL (standard) and OL consumption, correspondence analysis (CA) was applied. CA is a technique of interdependence whose main benefit is the ability to represent rows and columns on a percentual map (Hair et al., 2009).

Figure 5 presents the results of CA and reveals that the first two dimensions explained 67.92% of the association between frequency of OL consumption and emoji hedonic scale groups. Analysis of the perceptual map reveals that the group of consumers who least accepted pure OL was the group of consumers who least consumed OL. These results draw attention to the lack of habitual OL consumption and the consequences that this can have, not only with regard to health but also in the ease of adulteration of OL.

Figure 5. Correspondence Analysis between frequency of OL consumption and hedonic perception.
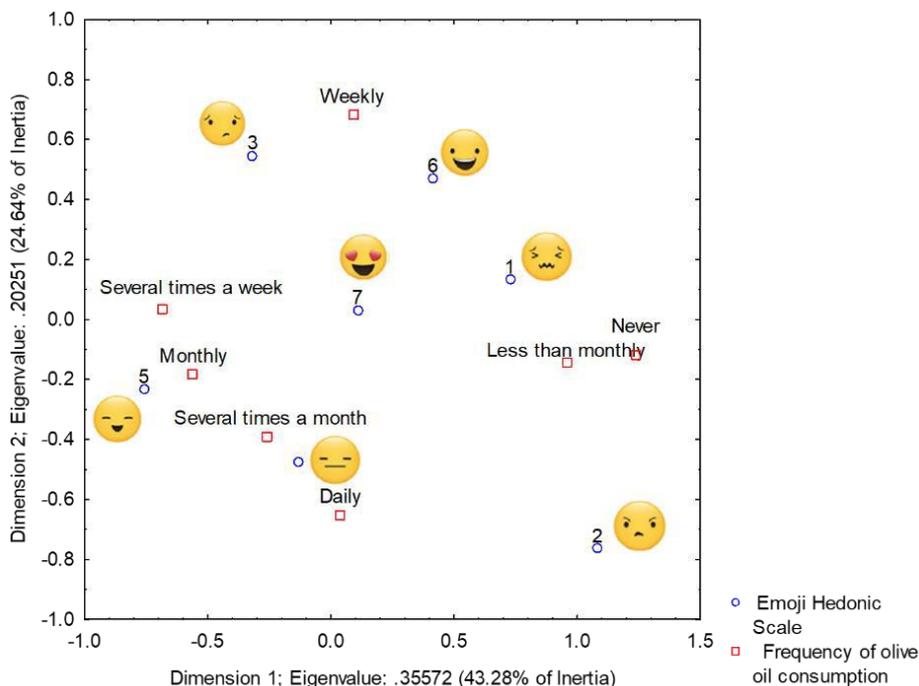
Source: Authors (2022).

4. Discussion

OL adulteration incidents in many countries around the world have been frequently reported (da Silveira et al., 2017; Johnson, 2014; Tfouni et al., 2017; Tibola et al., 2018; Yan et al., 2020).

In Brazil, OL adulteration is frequently reported and inspected by MAPA. However, the OL samples bottled in Brazil had a higher incidence of adulteration than OL bottled in other countries (Aued-Pimentel et al., 2017). The MAPA conducts periodic inspections however, this does not inhibit this practice and the number of frauds is still significant.

This fact is worrying due to the high fraud vulnerability in a population with low olive oil consumption as observed in the sensory tests. The results showed that this group of Brazilian consumers had low discrimination of adulterated OL, had low OL consumption, and accepted at the same intensity pure OL and adulterated with 30% SO.

Thus, it appears that in a population with low consumption of OL, they are not able to unconsciously protect themselves through sensory perception, which makes them more vulnerable to the consumption of products with low quality and low food safety.

In this sense, the inspection using fast, sensitive (detect small percentages of SO in the OL composition), and accurate techniques such as spectroscopy techniques associated with chemometrics tools can be useful in the detection of frauds and is important to ensure consumer safety (Guimet et al., 2005). It is worth mentioning that as important as the study and application of these techniques are in the defense of consumers are the campaigns aimed at creating the custom of consuming OL in Brazil. Not only with the aim of greater awareness to defend against fraud, but also because it is a food highly recommended for daily consumption, as it is directly related to benefit human health. In addition, these campaigns should be aimed at younger people, as acquiring a habit requires time and has a slow-to-change memory trace that ensures this learning is retained in the future (Wood & Neal, 2009). Besides the nutritional appeal, price is another issue that deserves discussion. The high prices charged for olive oil in Brazil possibly are an important cause of its low consumption. Public policies aimed at reducing the price of olive oil could be a strategic alternative to promote the habit of olive oil consumption.

FTIR spectroscopy associated with chemometrics tools (PCA) allows rapid and non-destructive detection of OL adulteration with SO starting at 5% (v v^{-1}). This result suggests that this spectroscopic technique is useful to detect OL adulteration with SO (the most common). The novelty of the work is in the levels of adulteration evaluated (5, 10, 15, 20, 30, 40, and 50%).

Similar results were obtained by using UV-visible absorption spectroscopy. In general, the UV-visible spectra of SO (signal at 315 nm) presents different qualitative characteristics than OL (signals at 317, 330, 354, 370, 413, 444, and 473 nm). These qualitative differences between the spectra, and the advantages of this technique when compared to other spectroscopy techniques, have stimulated the use of this spectroscopy technique in the detection of fraud in OL (Alves et al., 2019). However, the interpretation of the spectral data requires the help of the chemometrics tool. UV-visible absorption spectroscopy coupled to PCA allowed rapid and non-destructive detection of OL adulteration with SO starting at 5% (v v^{-1}), and suggests the use of the UV-visible absorption spectroscopy in the detection of OL adulteration.

Amit et al. (2020), showed the potential of FTIR spectroscopy combined with multivariate chemometrics tools to detect argemone oil adulteration in mustard oil. Amit et al. (2020) using FTIR spectroscopy and chemometrics tools developed a methodology for classification and quantification of virgin coconut oil from its adulterant paraffin oil in proportions of 1–18% (v v^{-1}). Karunathilaka et al. (2016) showed that the FT-NIR spectroscopy in combination with univariate and multivariate data analysis methods can be a rapid tool for evaluating authenticity of extra virgin OL.

Philippidis et al. (2017) evaluated using Raman and visible spectroscopy of OL adulterated with SU and the results indicate that both techniques are suitable for OL quality control.

From this research, the spectroscopy techniques FTIR and UV-visible coupled with PCA proved to be successful tools to be used to differentiate between adulterated OL samples. The use of these techniques in the characterization of oil samples was motivated by the advantages, such as simplicity, rapidity, sensitivity, and accuracy when compared to high performance liquid chromatography, nuclear magnetic resonance spectroscopy, Raman spectroscopy (Dais & Hatzakis, 2013; Jiménez-Carvelo et al., 2017; Li et al., 2018; Rohman & Man, 2010; Tay et al., 2002), and conventional methods. The conventional methods commonly used to analyze these oils are destructive and time-consuming (Tay et al., 2002), which also occurs with some of the spectroscopy methods, such as high performance liquid chromatography, nuclear magnetic resonance spectroscopy, Raman spectroscopy, that are characterized by being time consuming, expensive, generally destructive of the sample material, and require a high degree of technical knowledge when interpreting the data (Rohman & Man, 2010).

PCA is essentially a descriptive method used to visualize samples present in the n-dimensional space of a starting set of variables into a smaller number of dimensions, called PC, that represent sources of successively maximized variances of data (Borato et al., 2006; Jackson, 1991; Kemsley, 1996). In the literature, PCA has frequently been applied to the classification of adulterated samples (Amit et al., 2020; Drira et al., 2020; Jiménez-Carvelo et al., 2017; Shen et al., 2021). According to Vázquez-León et al. (2017), PCA represents the most appropriate statistical approach when the goal is to establish the relative importance of individual variables in determining the data structure.

Mendes et al. (2015) applied PCA to the data obtained by gas chromatography with a flame ionization detector, to evaluate the chemical similarity between ten oil samples (OL and SO). PCA and mass spectrometry were also applied to compare nine oil samples, that included OL and of a blend of OL and SO (Fasciotti & Pereira Netto, 2010). From this research, similar PCA scores graphs were obtained by both techniques and suggest that the FTIR and UV-visible spectroscopy can assist in the classification of OL.

However, it is important to point out that PCA cannot be used to detect and predict adulterations in OL. Other chemometric tools are suggested for this, such as partial least square, principal component regression, and analyses of more samples of unadulterated and adulterated OL.

It is worth noting that this study was limited to one sample of OL and one sample of SO since the focus was the level of SO and the comparison with other methods (UV-visible spectroscopy and sensorial analysis). Thus, this method is suggested to assess the use of other OL and vegetable oils and other levels.

5. Conclusion

From this research FTIR spectroscopy combined with PCA proved to be a successful tool to characterize SO, CO, SU, CA, and OL from different brands. More than FTIR and UV–visible spectroscopy characterization, both when combined with chemometric tools such as PCA, proved to be good tools to be used to distinguish OL samples intentionally adulterated with the addition of 5, 10, 15, 20, 30, 40, and 50% SO. For both spectroscopy techniques, the results indicated that the presence of SO (as adulterant) from 5% (v v^{-1}) in OL can be successfully allied in clusters with statistical tools (PCA). Successful separation of unadulterated and adulterated OL (OL5, OL10, OL15, OL20, OL30, OL40, and OL50) was achieved from PCA.

The present work shows the vulnerability to OL fraud in a population that presents low consumption of this food. For this, the study of the use of sensitive and quick techniques for the detection of fraud emerges as an important strategy. his section is not mandatory but can be added to the manuscript if the discussion is unusually long or complex.

Acknowledgments

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