Application of IR and UV–VIS spectroscopies and multivariate analysis for the classification of waste vegetable oils

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ABSTRACT

Due to the ever-increasing worldwide interest in the exploitation of waste vegetable oils, the development of analytical tools able to detect their adulteration with edible oils, is considered a priority for the scientific and industrial community. In this work, edible and waste vegetable oils have been analysed by Fourier Transform-InfraRed (FT-IR) and Ultraviolet-Visible (UV–VIS) spectroscopies and the corresponding spectral data subjected to statistical multivariate analysis for classification purposes. In particular, Principal Component Analysis (PCA) and Partial Least Square Discriminant Analysis (PLS-DA) were performed in order to develop an analytical tool which is able to distinguish between edible and waste vegetable oil. Qualitative analysis of the spectra suggested FT-IR and UV–VIS as the more suitable techniques to distinguish between wastes and edible samples. Also, statistical multivariate analysis revealed that FT-IR-based methodology is more adequate for the target, even if the elevated sensibility of the method produces an undesired distinction between edible oils of the same type. Finally, further attempts on UV–VIS data obtained in reflection mode allowed to produce a good dataset which after statistical treatment gave a clear differentiation between edible and waste oil samples.

1. Introduction

The adoption of circular economy models for the development of new processes, as well as for the improvement of existing productions represents one of the main targets of the current academic and industrial research activity (Corona et al., 2019; Rodriguez-Anton et al., 2019; Schroeder et al., 2018). Furthermore, in the European Union (EU) significant political exposure has recently been given to the development of circularity as a core activity (Circular Economy Action Plan, 2020) (Available at https://ec.europa.eu/environment/circular-economy/, accessed on 10/09/2021). In this context, in order to ensure the circularity of processes and materials, the exploitation of wastes as valuable raw materials is of primary importance (Cardenas et al., 2021).

Among the wastes suitable for potential highly efficient circular productions, Used Vegetable Oil (UVO) stands out as one of the cheapest, available and easy to process (Mannu et al., 2020; Vlahopoulou et al., 2017). Also, due to its specific chemical composition, it can be converted in many valuable products (Borrello et al., 2017; Mannu et al., 2019; Nogueira and Nunes, 2018). Used Vegetable Oils (UVOs) represent a specific category of municipal wastes, classified in the European

waste catalog under the code 19 08 09 (edible oils and fats) (Available at http://ec.europa.eu/environment/waste/framework/list.htm, accessed on 10/09/2021). Looking at the origin of this material, it is composed of almost 90% cooking oil (most frying oil), and oils used for preservation of food. UVO is mainly produced by kitchens and catering industries (Choe et al., 2007) in very high amounts on a global scale. Only EU produces about 190 million of metric tons of UVO per year (Shashidhara et al., 2010; Zhang et al., 2003).

Recycling of UVOs is mandatory in many Countries and in some cases legislative endorsement has been pursued by important Institutions. EU stated in the RED II directive that the waste vegetable oils employed for biodiesel production can count twice in the calculation of the shares of renewable energies (double counting rule) (Ibanez et al., 2020). In this context, used vegetable oil reaches a market value even higher than some edible oils such as some seed oils and in particular palm oil. Therefore, the possibility to counterfeit UVOs with edible oils represent a risk to be faced (Alcock et al., 2021; van Grinsven et al., 2020).

The issue is not easy to solve as no control on the international supply chain of UVO is currently possible. Some researchers presented preliminary studies related to the determination of palm oil concentration

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Table 1
Relevant data and nomenclature for sample oils.

ID Sample	Origin	Name[a]
S1	commercial	tuttifritti
S2	cooking oil	seadas
S3	cooking oil	breaded chicken
S4	cooking oil	breaded chicken 2
S5	cooking oil	aubergines
S6	commercial	olive
S7	used oil	tuna (olive)
S8	commercial	sunflower
S9	commercial	tuttifritti 2

[a] The name used for each type of oil was related to the food which has been in contact with (for cooking and used oils) or to the trademark.

in samples of waste vegetable oils. Lim and coworkers (Lim et al., 2018), as well as Alexa et al. (2009), propose the employment of FT-IR spectroscopy for this purpose. Also, UV–VIS spectroscopy in combination with NMR relaxometry (Ok et al., 2017) or fluorescence (Uncu et al., 2019) have been proposed as analytical tools for detecting adulteration of olive oils. Finally, Raman spectroscopy has been effective in distinguishing between edible and waste cooking oils when combined with multivariate statistics (Huang et al., 2019; Jin et al., 2021).

Nevertheless, the necessity to develop an analytical tool which is able to be easily used in operating conditions for the detection of waste vegetable oil adulteration, is still urgent. Recently, it has been reported the possibility to classify edible and waste vegetable oils by GC–MS analysis combined with statistical multivariate analysis (Mannu et al., 2019). These preliminary results opened up the possibility to develop a proper combination between a specific analytical methodology and a statistical processing which would be suitable to be implemented in a

portable device for the quality assessment of UVOs (Wang et al., 2021). In the present paper, an exploratory study on the applicability of FT-IR and UV–VIS spectroscopy for the classification of waste vegetable oils is reported. In particular, the best combination between optical spectroscopy and multivariate analysis for the discrimination between waste and edible oils is described. Four edible oils and five different waste UVOs were subjected to FT-IR and UV–VIS (in absorbance and reflectance mode) analyses, and the resulting data were processed with multivariate statistics in order to develop the target analytical tool.

2. Materials and methods

2.1. Samples preparation

n-Hexan (95%, CARLO ERBA) was used as solvent to solubilize the oils with a V/V ratio of 200:1, n-Hexan:Oil. In particular, 20 μL of oil were dissolved in 4 mL of hexane. More concentrated solutions revealed low homogeneity and spectroscopic measurements were affected by a relevant error (out of range data). Before analysis, each sample was filtered on a woven wire mesh sieves - Ø 200 mm / 203 mm (Verder Scientific, Germany).

Samples of waste cooking oils (S2-S5) were obtained after three frying cycles, apart from breaded chicken which was obtained from just one frying cycle. Edible oils S1, S6, S8, and S9 were purchased from local markets. Fried oils and used tuna oil have been recovered from the specific containers after 24 h at room temperature. This procedure allowed the complete setting of the solid by-products upon frying process. For each type of oil (Table 1), three different samples were collected and each sample analysed in triplicate. The average value of the three measurements was taken as suitable for multivariate analysis.
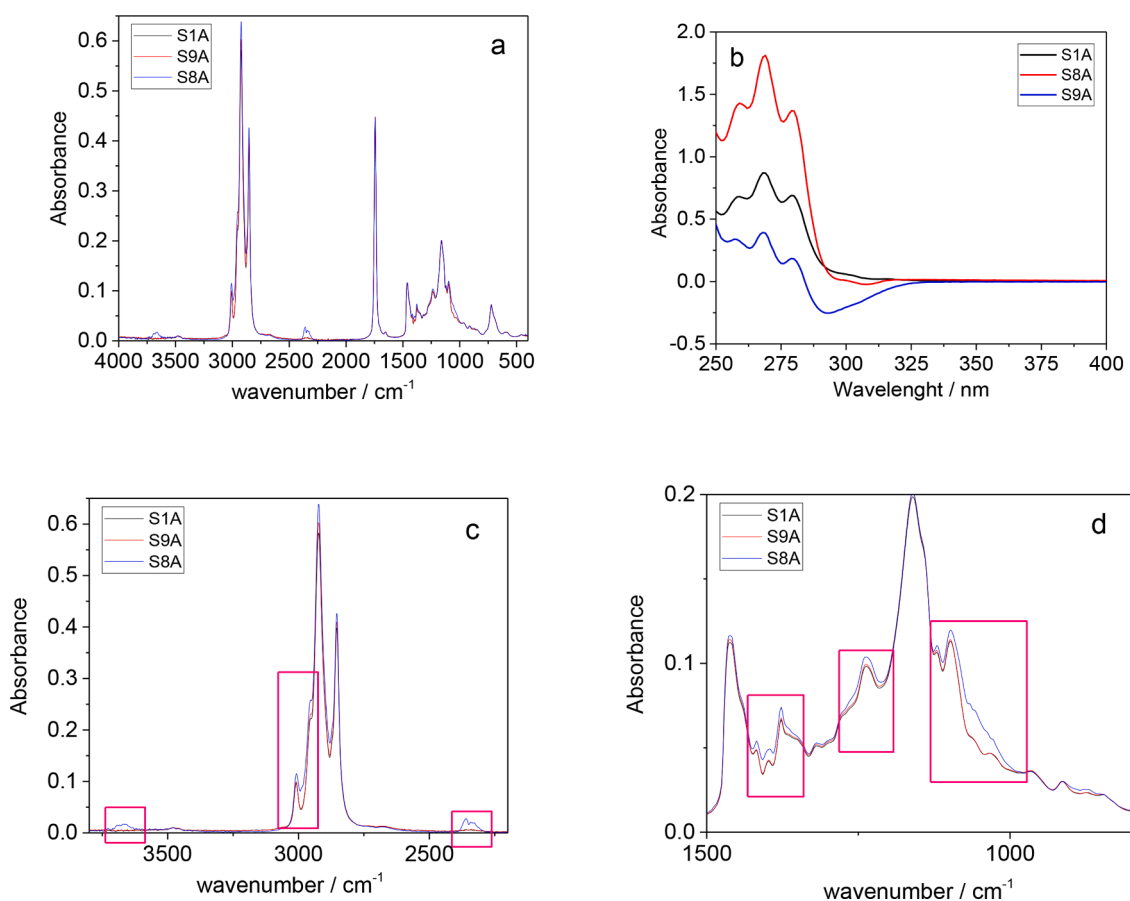

Fig. 1. FT-IR (**a**) and UV–VIS (**b**) spectra of edible oils S1, S8, and S9. The main differences between FTIR spectra are more obvious in the red boxes introduced into the images placed at the boom side.
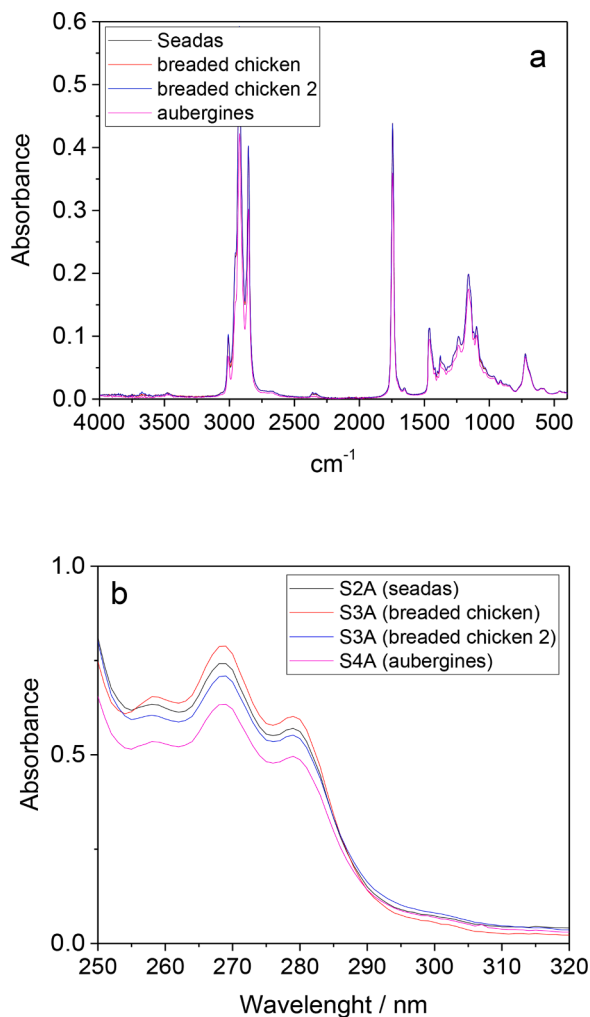
Fig. 2. FT-IR (**a**) and UV–VIS (**b**) spectra of samples S2A (seadas), S3A (breaded chicken), S4A (breaded chicken 2), and S5A (aubergines).

2.2. Spectroscopic analyses

Ultraviolet−visible (UV−vis) spectra of the oils solubilized in n-Hexane were measured in absorbance mode from 200 to 600 nm by using a "Nicolet Evolution 300″ UV−vis spectrophotometer (Thermo Fisher).

Fourier-transform infrared (FTIR) analysis of the oils was carried out by a "Vertex 70″ interferometer (Bruker) in the range of $4000-400$ cm^{-1} with a 4 cm^{-1} resolution (32 scans) equipped with an A 225/Q (Platinum ATR diamonds F) with a diamond crystal of single reflectance and a DigiTect DLaTGS detector with integrated preamplifier, with KBr window detector. Attenuated total reflectance (ATR) mode was used to directly analyze the oils.

2.3. Multivariate analysis

Multivariate analysis was performed with the online software metaboanalyst 5.0 (Available online at https://www.metaboanalyst. ca/, accessed on 10/09/2021). The PLS-DA regression was performed using the *plsr* function provided by *R pls* package (Wehrens et al., 2007).

3. Results and discussion

In order to reproduce the current situation of disposal and collection of the waste vegetable oil, nine within edible and waste typology of samples were considered. Their origin and corresponding nomenclature

are reported in Table 1.

Several kinds of wastes and edible oils were considered: samples S1, S2, S4, and S5 represent the typical waste cooking oil, while S1, S8, and S9 are representative of commercial edible oils. Sample S3 was derived from S1 which has been used for a fast frying (just one fast cycle) and it should represent an intermediate composition between samples S1 and S4. Finally, S7 is an olive oil used for preservation of tuna (waste arising not from cooking), and S6 is a generic edible olive oil. The consideration of edible olive oil within the studied samples is related to its usual presence in the batches of used vegetable oils that are collected and delivered to the recyclers.

From each oil batch, three samples were collected and subjected to FT-IR and UV–VIS analyses. At first, qualitative analysis can be performed on the basis of the visual inspection of the spectra obtained.

In Fig. 1, a comparison between the edible oils S1, S8, and S9 is reported.

As emerged from FT-IR and at UV–VIS measurements, samples of seed oils, namely S1A, S8A, and S9A, present small differences. In the first case, as expected, the spectra of samples S1A and S9A (same brand, different bottles) are superposable, while oil S8A (different brand) shows some differences at 1000 cm^{-1}, 2700 cm^{-1}, 300 cm^{-1}, and between 3500 and 3700 cm^{-1}. Looking at the UV–VIS spectra it is possible to notice a relevant shoulder between 280 and 290 nm relative to sample S9A.

As commented in the introduction part, waste vegetable oils are constituted by a mixture of cooking (mainly frying) oils and other used vegetable oils, as for instance, edible oils used for preservation and storage of the food products. It is reasonable to expect that the variation in composition of these oils due to the contact with food under different conditions could produce a significant variation of the of their properties. In the case of cooking oils, high temperature-promoted processes occur (i.e. oxidation, polymerization, hydrolysis), while in the case of other alimentary oils, leaching from food and cooking tools represents the main origin of contamination (Saguy et al., 2003; Ziaiifar et al., 2008). The main composition of waste cooking oil is represented by fatty acid methyl esters and a small fraction of free fatty acids (Di Pietro et al., 2020; Lin et al., 2013). Minor constituents are a large number of volatile compounds (Mannu et al., 2019), pigments, sterols and phenolics, which are responsible for the typical smell of waste vegetable oils.

In Fig. 2, FT-IR and UV–VIS spectra relative to samples S2A (seadas), S3A (breaded chicken), S4A (breaded chicken 2), and S5A (aubergines), are compared.

Looking at the FT-IR plots in Fig. 2a, some small differences can be detected in the intensities of the signals relative to waste oils used for frying different food. In particular, the FT-IR pattern of S5A (aubergines) results slightly different from the one obtained with seadas, breaded chicken and breaded chicken 2 samples. From a qualitative analysis, no differences can be highlighted between oils S3A (breaded chicken) and S4A (breaded chicken 2), which differ just for the number of frying cycles (figure S1, Supporting Information).

On the contrary, it is interesting to notice that UV–VIS analyses (Fig. 2b) allowed to distinguish between all the samples, even within S2A and S3A which represent the same kind of waste and differ just for the degree of contamination.

Finally, the UV–VIS and FT-IR analyses of samples of edible olive oil S6A and olive tuna oil S7A confirmed the highest sensitivity of UV–VIS spectroscopy (Figure S2, Supporting Information).

The specific interest in studying the applicability of analytical techniques to the classification of wastes, is mostly related to the opportunity to distinguish between edible and waste oils. As many edible oils are produced and sold, and many waste oils are daily disposed worldwide, the more important parameter in selecting a technique for the quality assessment of wastes would be the ability to detect the edible oils as similar and the waste ones as different. From the preliminary analysis based on the visual analysis of the spectra registered, UV–VIS represents the more promising technique of analysis.
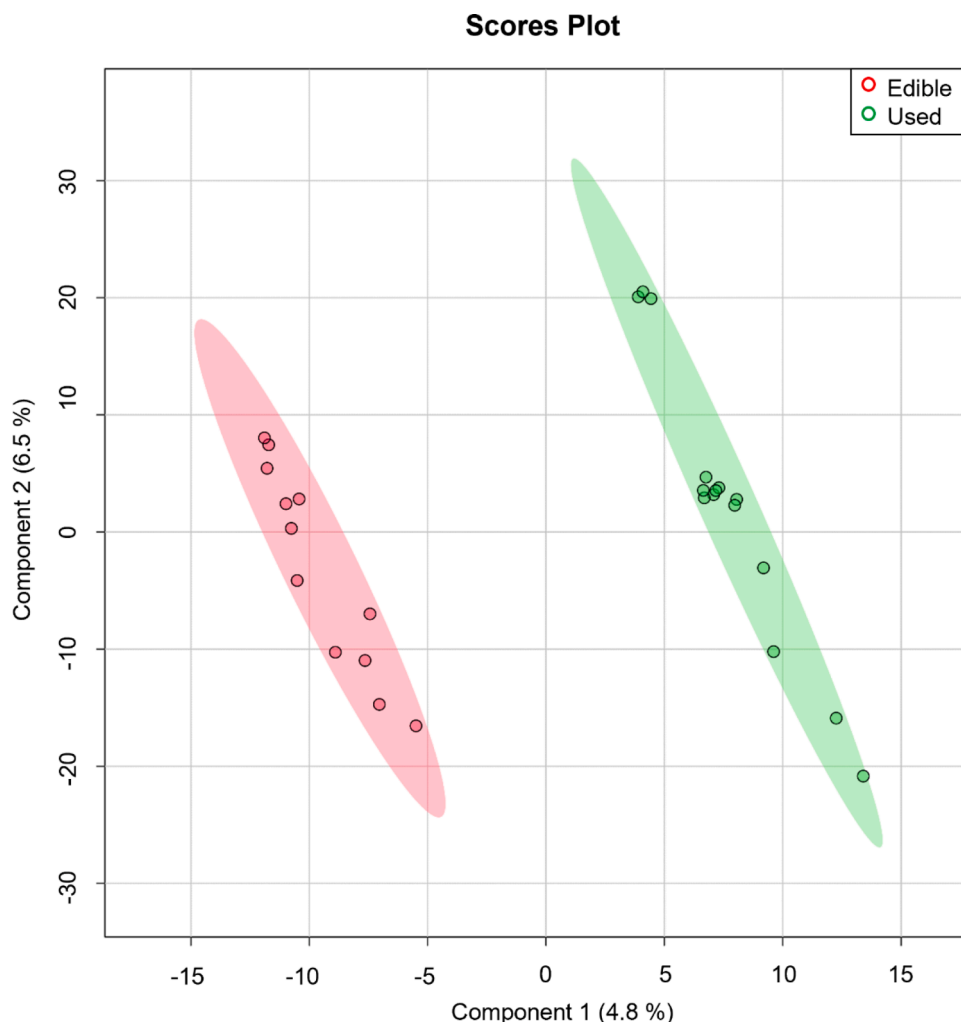
Scores Plot

Fig. 3. PLS-DA of samples S1A/B/C-S9A/B/C from FT-IR data.

In order to confirm this hypothesis, and to develop an analytical tool for quantitative analysis, all the oils listed in Table 1 were analysed by FT-IR and UV–VIS and the crude spectral data subjected to multivariate analysis. For this purpose, the entire spectrum was considered without selecting any specific spectral window, or selecting specific metabolites by employing the tool metaboanalyst (Chong et al., 2018). This approach, previously used for the classification of wines from ^{13}C NMR data (Mannu et al., 2020), and for the distinguishing of celluloses from X-ray data (Ferro et al., 2020), was already successful in detecting waste and edible oils starting from MASS spectrometry data. Considering the raw data relative to the entire spectrum and by performing an ANalysis Of the Variance (ANOVA), Principal Component Analysis (PCA), and Partial Least Square Discriminant Analysis (PLS-DA), it is then possible to determine a specific chemical fingerprint for each category of sample considered. The fingerprint is representative of the overall composition of the sample and if properly customized for the corresponding category, it can be used to discriminate between wastes. ANOVA results are reported in the Supporting Information.

3.1. FT-IR-based chemical fingerprint

From batches S1-S9 a total number of 27 samples were prepared (S1A/B/C – S9A/B/C) and analysed in triplicate by FT-IR. The relative values of raw integrals resulting from the average value of the 3 analyses conducted for each sample were subjected to multivariate analysis. The overall samples were divided into two main groups, namely edible vs

waste oil. The specific treatment of the data (filtering, normalization, data scaling, ANOVA results) is described in the supporting information file. Principal component analysis based on FT-IR data was not able to distinguish edible from waste oil, making this statistical tool not suitable for our purposes.

Supervised PLS-DA tool was used in data analysis (Fig. 3). PLS regression is usually useful in such cases when the number of variables is higher than the number of observations (Brereton et al., 2014; Ramadan et al., 2006; Westerhuis et al., 2008).

The supervised PLS-DA analysis can be used to distinguish edible and waste oils without any superposition and thus it can be employed as statistical tool for build-up a classification protocol.

3.2. UV–VIS-based chemical fingerprint

The preliminary qualitative analysis of UV–VIS spectra revealed a good potential due to the ability of the technique to distinguish between waste and not-waste oils and the poor capacity to distinguish between edible frying oils (S1, S8, S9). A first attempt to classify quantitatively the samples by using the PCA analysis was unsuccessful (figure S5, Supporting Information).

As in the previous case, unsupervised PCA method was not able to distinguish clearly between waste and not-waste oils. Therefore, samples S1-S9 were subjected to supervised PLS-DA treatment. Nevertheless, the simple discriminant analysis was not suitable for obtain a good separation of the two categories considered, target that was reached through
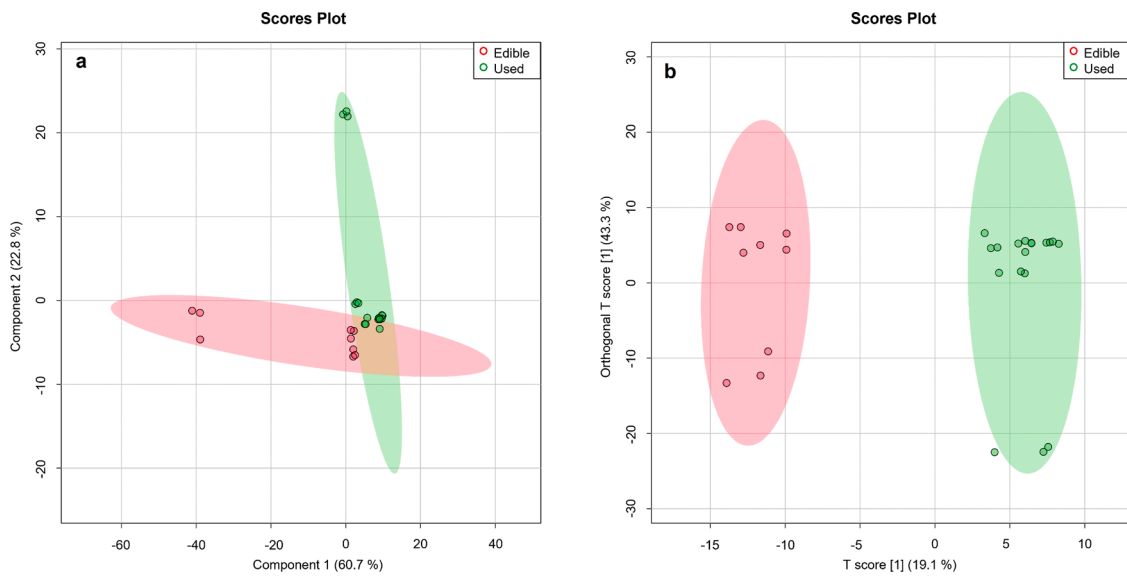
Fig. 4. PLS-DA (**a**) and o-PLS-DA (**b**) of samples S1A/B/C-S9A/B/C from UV–VIS data.
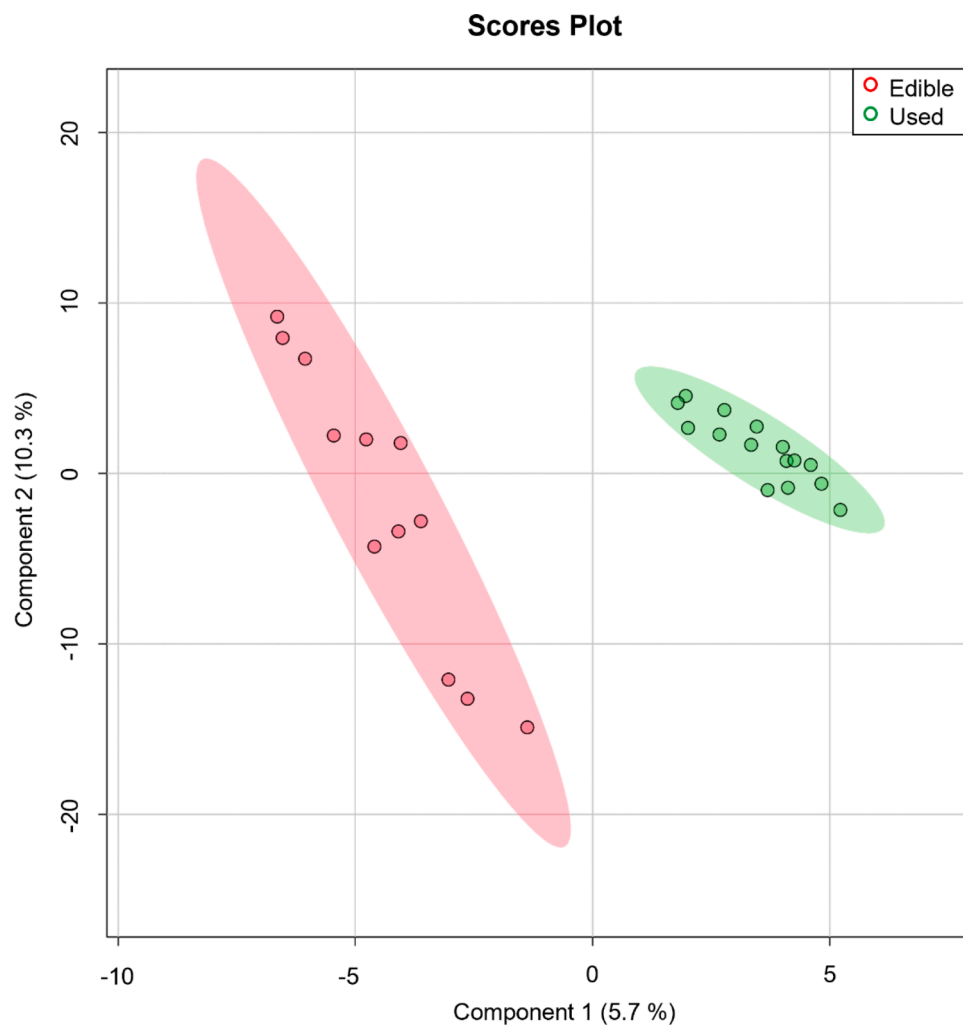

Fig. 5. PLS-DA 2D-plot derived from UV–VIS data (reflectance mode) relative to samples S1A/B/C-S9A/B/C.

the orthogonal projection o-PLS-DA, as showed in the plots reported in Fig. 4.

Despite the significant improvement in the general classification

when o-PLS-DA is employed, the absorbance data obtained by UV–VIS analysis, do not allow to reach a very good classification. In fact, there is a large variability within each group indicating important differences
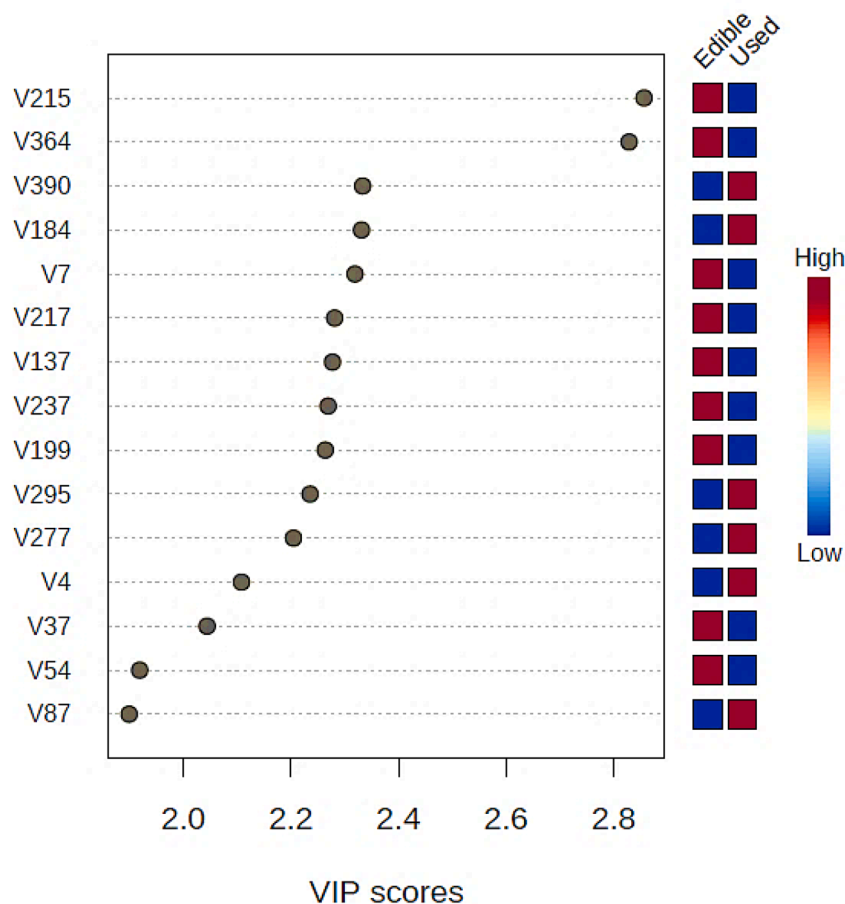
Fig. 6. Important features identified by PLS-DA.

inside each of the two considered groups.

Nevertheless, with the aim to check all the potential of this technique, all the 27 samples were analysed also by UV–VIS in reflectance mode. The interest versus this specific proceeding is strongly related with the possibility to develop a tool which can be used in spectroscopically-based portable devices, which are usually configured in reflectance mode (Sarikas et al., 2017; Wang et al., 2021). PCA analysis pf the raw reflectance data resulted ineffective as in the case of the absorbance UV–VIS analyses (Figure S9, Supporting Information). On the other side, simple PLS-DA allowed a good separation within samples as showed in the Fig. 5.

Looking at the distribution of the samples in each group, it is possible to observe a reduced variable of the samples within each group with respect to the distribution obtained from the UV–VIS data in absorbance mode. As a result of the current study, raw UV–vis data obtained in reflectance mode and evaluated with PLS-DA indicated chemical fingerprint characteristic of edible and waste oils. A *variable importance in projection* plot was derived (VIP plot) (Farrés et al., al.,2015) as reported in Fig. 6.

The VIP plot reported in Figure 6 takes in consideration the 15 most representative factors determining the variance. To each one of the considered relevant factors is attributed a corresponding intensity which is related to the impact it has on the overall variance (Mannu et al., 2020). The combination between the most representative factors and their intensity both for edible and waste oil, represent a unique code related to the specific chemical composition of the two considered categories and thus it can be used as chemical fingerprint.

4. Conclusions

With the aim to develop a tool for the classification of the waste vegetable oil, the potential of FT-IR and UV–VIS techniques (in absorbance and reflectance mode) combined with multivariate analysis, was explored. A total of 27 samples of edible and waste vegetable oils were considered for this exploratory study. In particular, after a qualitative visual inspection of the spectra and a quantitative analysis of the spectra by Principal Component Analysis (PCA) and Partial Least Square Discriminant Analysis (PLS-DA), a characteristic chemical fingerprint was determined for each category (edible and waste) analysed. At the end, both FT-IR and UV–VIS (reflectance mode) analyses produced spectral raw data with evaluation using partial least square discriminant analysis, that allowed to reach a differentiation between waste and edible oils. In particular, the results obtained from UV–VIS analysis in reflectance mode, are of particular interest as the tool developed and herein presented can potentially be installed in a portable device, providing an important practical application in the field of waste vegetable oils management. The analyses and procedures herein reported represent a preliminary study which should be extended to a larger number of samples in order to improve the statistical model.

CRediT authorship contribution statement

Alberto Mannu: Conceptualization, Software, Validation, Formal analysis, Data curation, Writing – original draft, Writing – review & editing, Visualization, Supervision. **Matteo Poddighe:** Methodology, Formal analysis, Investigation. **Sebastiano Garroni:** Conceptualization, Writing – original draft, Writing – review & editing, Visualization, Project administration, Funding acquisition. **Luca Malfatti:**

Conceptualization, Methodology, Resources, Project administration, Funding acquisition.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.resconrec.2021.106088.

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