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3D-Fluorescence Spectroscopy Analysis of Olive Oils from Three Algerian Varieties extracted with three different systems

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Abstract

In this paper, the 3D-fluorescence spectroscopy technique was used for the determination of fluorescent components and olive oil discrimination. Three different systems (two-phase centrifugation, pressure system, and traditional process) were used to extract olive oils from three Algerian varieties: Chemlal, Azeradj, and Sigoise. The fluorescence ranges from 300 to 700 nm. The raw 3D fluorescence spectra of olive oils were analyzed using component analysis (ICA) to obtain independent spectra with excitations ranging from 284 to 460 nm and emission wavelengths. According to the results, high-quality olive oils with lower concentrations of oxydation products and higher concentrations of polyphenols, chlorophylls, and pheophytins are produced by two- phase centrifugation. Olive oils from the Sigoise variety had low levels of pigments and natural antioxidants (polyphenols and tocopherols).

Keywords : Olive oil, Azeradj, Chemlal, Sigoise, pressure system, Two-phase centrifugation, Traditional process, 3D-fluorescence spectroscopy, ICA-by-Blocks, Independent component analysis (ICA)

1. Introduction

The olive tree (Olea europaea L.), has long been a significant source of oil. Olive is a member of the Oleaceae family, which has 600 species and about 30 genera (Cronquist, 1981). There are over thirty species in the genus Olea L., dispersed over Africa, Asia, Oceania, and Europe; only Olea europaea L. is grown (Green, 2002 ; Fogher et al., 2010). Virgin olive oil is extracted from olives using physical or mechanical methods which don't change the oil; it has not been subjected to any other processing save washing, decantation, centrifugation, or filtration; this excludes oils extracted using solvents or re-esterification procedures, as well as any blends with other types of oils (Mariotti, 2014). Because of its many health benefits, people have come to value olive oil, a traditional food that has been consumed for thousands of years. Its rich antioxidant composition, absence of trans fatty acids, and presence of monounsaturated fatty acids make it a special nutritional source (Portilla and al., 2014). According to Cichelli and Pertesana (2004), there are

significant variations in the chemical composition of olive oils based on factors such as fruit variety, ripeness level, growing region, processing and storage methods, and environmental conditions.

In Algeria, olives are the most widely grown fruit crop.It is among the Mediterranean basin nations whose climates are suitable for olive tree cultivation (Chikhi and Djelloul, 2022).However, various factors can impact the quality of olive oil produced, including Algeria's large surface area and distinct bioclimatic stages linked to a wide range of cultivated varieties. Algeria has a large number of olive tree cultivars; the ITAFV has homologated 36 of them (Touati et al., 2022). However, a number of factors, including Algeria's large surface area and distinct bioclimatic stages linked to a variety of grown varietals, might impact the quality of olive oil produced. Algeria boasts an abundance of olive tree cultivars, of which 36 have been homologated by ITAFV (Touati et al., 2022). The most significant olive cultivar, Chemlal, makes up 40% of Algeria's olive orchard and is grown primarily for the extraction of olive oil. Growing in the west of Algeria, the Sigoise cultivar is highly productive and has a little alternate production season. It accounts for 25% of the country's oliviculture area and produces two distinct uses: table olives and olive oil.). Its characteristics include medium-intense, early flowering, a high pulp/stone ratio (6.44%), quick pulp and stone separation, and medium and alternating productivity.

This type is found across Algerian area. Azeradj: Located in the east central Kabylie region, it makes up 10% of the country's olivicultural territory and is frequently linked to "Chemlal" for pollination. It is distinguished by weakly intense early flowering, a high pulp/stone ratio (8.70%), challenging pulp-stone separation, and medium and alternate productivity (ITAFV, 2006).

In numerous fields, fluorescence spectroscopy is a widely recognized and utilized research and analytical instrument. Fluorescence has become more widely used in food analysis in recent years, which is really noticeable (Christensen et al., 2006; Sadecka and Tothova, 2007; Karoui and Blecker, 2011 **;** Sikorska et al.,2014).). The phenomenon of fluorescence is a variant of photoluminescence, wherein a molecule, boosted to a state of electronic excitement by absorbing VIS, NIR or UV radiation, returns to its original state after decaying by emitting a photon.When an excited state is released, its electronic spin is equal to that of the ground state, which is usually equal to zero. This is known as fluorescence. Generally occurring at a rate of $10^8 s^{-1}$, the spin allowed transitions happen slowly (Lakowicz, 2006 ; Sikorska et al., 2014). Furthermore, fluorescence spectroscopy is a highly sensitive, selective and noninvasive method.Other notable advantages include the need for minimal amounts of sample and the lack of reagents and solvents (Lleó *et al.* ,2016).

Fluorescence has been used more often in food analysis over the past ten years, most likely as a result of the widespread adoption of chemometrics (Sadecka J., Tothova J. ,2007). Olive oil quality has been successfully analyzed using fluorescence spectroscopy (Karoui and Blecker, 2011 ; Meenu et al., 2019 ; Baltazar et al.,2020).Olive oil's health benefits are closely linked to its fluorescent compounds, which also contribute to its oxidation resistance and freshness level. These substances include chlorophylls and pheophytins, polyphenols and tocopherols, and products of primary and secondary oxidation (Gorzynik-Debicka et al., 2018 ; Baltazar et al.,2020).

3D-fluorescence spectroscopy, along with chemometrics, is employed with the aim to discriminate between olive oils from three Algerian Varieties extracted with three different systems. The three most predominant varieties cultivated in Algeria were selected for this study : Chemlal, Sigoise and Azeradj. Three different systems were used to extract olive oils : two-phases centrifugation, pressure system and traditional process.

2. Materials and Methods

2.1. Olive fruit samples

Olive samples of Chemlal, Azeradj and Sigoise varieties were harvested In Novembe during the 2015–2016 growing season from the Technical Institute of Fruits Arboriculture and Vine (ITAFV),

located in Emjez Edchiche, Skikda, Algeria.

2.2. Olive oils extraction

Overall, 350 kg of each variety were collected.Following cleaning and homogenization , each sample was split into three parts.The first one (200 kg) was extracted using a pressure system,the second one (100 kg) by a two-phase centrifugation and the third one (50 kg) by a traditional process.Olives were processed within 15 days. The following experimental procedures were followed.

Pressure system : Olives were crushed with a ston-mill. After mixing for 30 minutes, Olive paste was squeezed with a hydraulic press at 100 kg/cm^2. Decanters made of stainless steel were used to separate the obtained liquid (oil and vegetation water).

Two-phase centrifugation : Olives were washed before crushing with a hammer crusher. Olive paste was kneaded without addition of warm water. Then, the paste was separated by a vertical centrifuge, obtaining two final products : The oil and a wet pomace.

Traditional process : Two stones were used to crush the olives .Following its crushing, olive paste was walked on and subsequently combined with water. Decantation is the process used to separate the liquid phases into vegetable water and oil.Before being extracted, three identical portions of olives were exposed to a hot water vapor to evaluate the effects of heat treatment on the quality of olive oil.

We collected twelve samples of olive oil in total :
Samples CP, CC, CTF and CTC were from Chemlal variety;
Samples AP, AC, ATF and ATC were from Azeradj variety;
Samples SP, SC, STF and STC were from Sigoise variety ;
Samples AC, CC and SC were obtained from two-phase centrifugation;
Samples AP, CP and SP were obtained from a pressure system;
Samples ATF, CTF and STF were obtained from a traditional process using fresh olives
Samples ATC, CTC and STC were obtained from a traditional process in which water vapor was used to treat olives olives .
Before being analysed, olive oil samples were immediately put into brown glass bottles and stored at 4 °C.

2.3. Fluorescence spectroscopy analysis

SAFAS Xenius spectrofluorometer (Monaco) outfitted with a light source (xenon lamp), excitation and emission monochromators and a photomultiplier set to 270 V was utilized to measure 3D-fluorescence spectra. The wavelengths of excitation and emission were 284 to 460 nm (step 4 nm) and 300 to 700 nm (step 4 nm) respectively. Slit widths for the monochromator's emission and excitation were both set to 10 nm.The samples were placed in a quartz cuvettes without prior preparation. Every sample was subjected to three replications. Each sample's corresponding data were arranged in a [12 x 101x 45] 3-way cubic array (12 olive oil samples, 101 excitation wavelengths and 45 emission wavelengths). It was subsequently unfolded to creat a (12 × 45 x 45) matrix for the following ICA calculations.

2.4. Chemometrics methods

2.4.1. Independent Component Analysis

The objective of independent component analysis is to separate pure signals from a collection of mixed signals (Bach and Jordan, 2003).

Wang and al.(2008) have described the general model of ICA as follows :

X = AS

Where **X** is the matrix of observed spectra, **A** is the matrix of proportions of the pure signals and **S** is the matrix of pure source signals.

The combinations of multiple independent sources that make up the measured signals should have a higher Gaussian distribution than the sources. Finding the sources that are as far from Gaussian as possible is the aim of ICA. By making the source signals more non-Gaussian. ICA seeks to extract the pure signals (Rutledge and Jouan-Rimbaud Bouveresse, 2013).

A blind identification technique called the JADE (Joint Approximate Diagonalisation of Eigenmatrices) algorithm can be used to reduce Guassian noises and strengthen the non-Gaussian source signals.It is predicated on using the data to create an array of fourth-order cumulants (Rutledge and Jouan-Rimbaud Bouveresse, 2013 ; Maalouly and al., 2013). The JADE algorithm was used to perform ICA calculations for this study.

2.4.2. Determining the optimal number of ICs

ICA-by-Blocks was applied to calculate the optimal number of independent components (ICs). It divides the data into two blocks, compares the ICs found in each block by computing the correlations between each pair of signals taken from the two blocks for a given model, and computes multiple ICA models on each block with ICs ranging from 1 to A ICs. The optimal number of ICs to extract from the data under study is determined by the highest-dimensional model for which ICs obtained in a block are similar to those obtained in another block (Rutledge and Jouan-Rimbaud Bouveresse, 2013).

2.5. Software

The JADE algorithm was employed to perform the independent component analysis. The number of independent components (ICs) was computed using the ICA_by_Blocks method. The data was processed using Matlab (The MathWorks, Natick, USA).

3. Results and discussion

3.1. Optimal number of ICs

The data set was analyzed using the ICA-by-blocks method. For each block, ICA models ranging from 1 to 14 ICs were computed. For a given model,the correlation coefficients between every pair of signals from both blocks were computed in order to compare the ICs in each block.

Fig.1 illustrates that for models extracting 1, 2, 3 ,4 and 5 ICs, correlations are greater than 0.96.The lowest correlation between signals decreases significantly after 8 ICs, which was considered to be the optimal number of ICs.

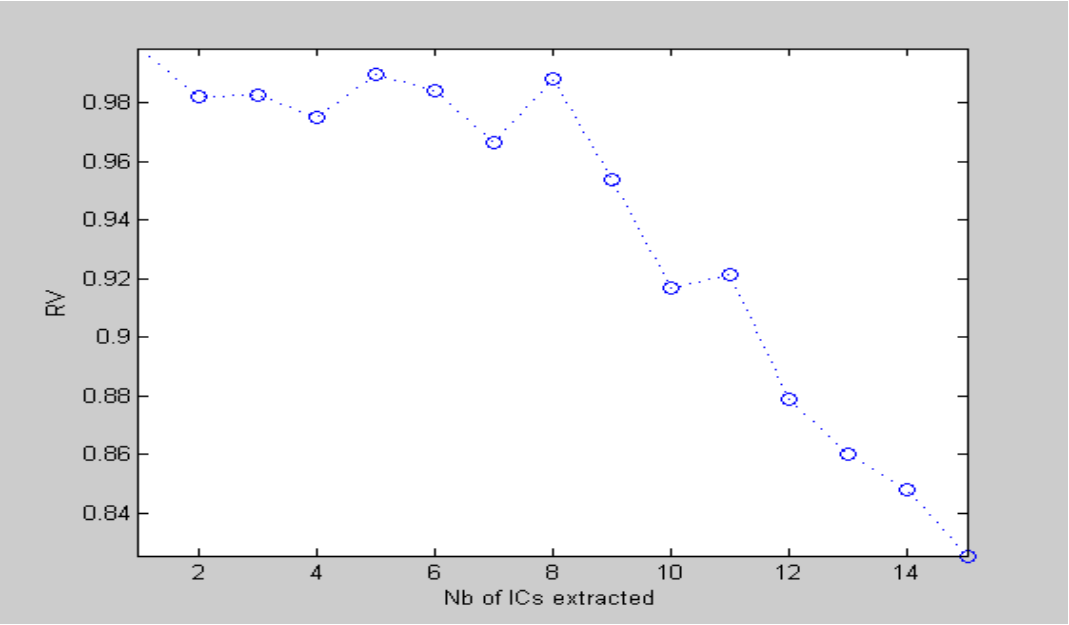

Fig. 1. ICA_by_blocks (2 Blocks, 1 to 14 ICs).

3.2. Extracted independent components

Seven pure signals were extracted from each of the two data arrays (36 x 4545) after applying ICA with eight ICs, as determined by ICA-by-blocks. Seven fluorophore groups are represented by the seven extracted signals.

Comparing the spectra of chemically pure fluorescent components is the primary method used to determine the origin of the specific emission bands (Sikorska *et al.,* 2014). When distinct signals from the corresponding different fluorophores in the sample are released, it results in spectral emission. Previous studies give the main fluorophores in olive oils their excitation and emission wavelength ranges (Lleó *et al.,* 2016).These emission and excitation bands, along with the related references, are compiled in Table 1.

Table 1.Bands of excitation and emission that represent the primary fluorophores in olive oil.

Fluorophores	Excitation (nm)	Emission (nm)	References
Chlorophyll a	430	669	Galeano Diäaz *et al.* (2003)
Chlorophyll b	458	653	Galeano Diäaz *et al.* (2003)
Chlorophyll a and b	355-400	676	Lia *et al.* (2020)
	350-420	660-700	Benabid (2009)
Pheophytin a	406	671	Galeano Diäaz *et al.* (2003)
Pheophytin b	435	658	
Chlorophyll and Pheophytin	380-420	665-680	Lleó *et al.* (2016)
Oxydation products	300-400	400-500	Guimet *et al.* (2004)
	274-400	375-550	Hernández-Sánchez *et al.* (2018)
	320–420	400–500	Ammari *et al.* (2012)
	325-340	434-550	Lia *et al.* (2020)
Vitamin E	325 -375	500- 530	Lleó *et al.* (2016)
Tocopherols	270-310	300-350	Benabid (2009)
	295	386-468	Lia *et al.* (2020)
Polyphenols	260-310	310-370	Zandomeneghi *et al.* (2005)
	280	330	Lia *et al.* (2020)
Tocopherols and Polyphenols	290–315	320–360	Ammari *et al. (*2012)
	270–330	295–360	Sikorska *et al.* (2014)

IC1 (Fig.2 b), shows a band located at 670 nm in excitation and 415nm in emission corresponding to pheophytin a.IC2 (Fig.2 d), present a fluorescence band with excitation wavelengths in the range of 680 to 700 nm and emission spectra from 350 to 420 nm.This band was attributed to chlorophylls a and b. The signal extracted by IC5 (Fig.2 f) was observed at about 430 nm in excitation and 680 nm in emission. This signal was attributed to pheophytin b, according to Galeano Diäaz *et al.* (2003).

The proportions on IC1, IC2 and IC5 (Fig.2 a, c and e), corresponding to all oilve oil samples change in a similar manner. This indicates that chlorophylls (a and b) and pheophytin (a and b) are very similar in chemical structure.The intensity of the fluorescence is typically correlated with the fluorophore concentration. Chlorophylls a and b have lower fluorescence intensities than pheophytin a.Pheophytin a is the main pigment found in olive oil, accounting for approximately 70-80% of all pigments (Galeano Diäaz et al., 2003). The spectral distribution and measured fluorescence intensity are influenced by several factors. These factors are related to the nature, concentration and molecular environment th of fluorophores (Sikorska *et al.*, 2014).

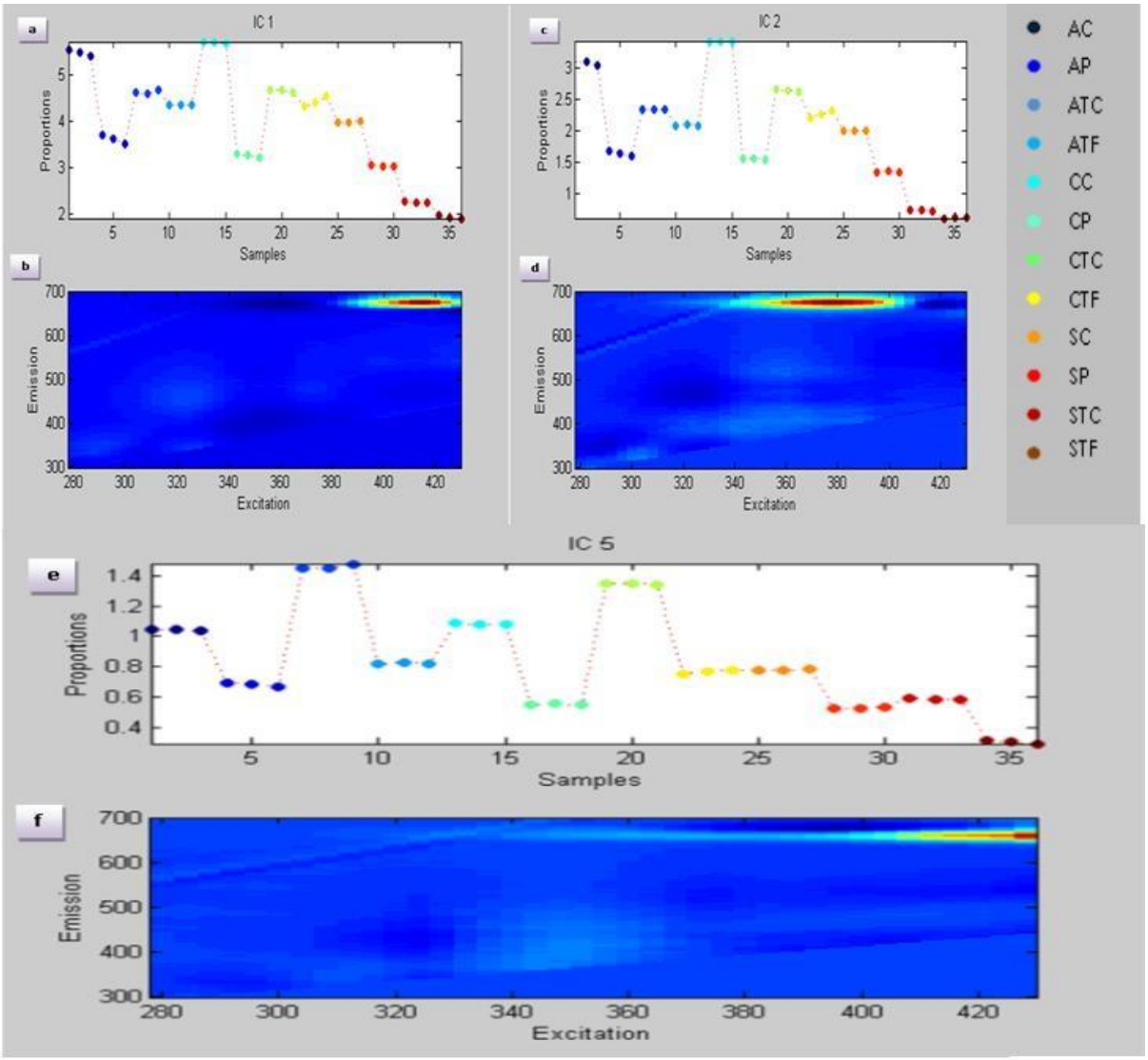

Fig.2. Proportions of olive oil samples (a) and extracted signal (b) on IC1, Proportions of olive oil samples (c) and extracted signal (d) on IC2 and Proportions of olive oil samples (e) and extracted signal (f) on IC5.

The ICA proportions plot (Fig.3) classifies all the olive oil samples in to four groups. The first group included CC and AC samples from Chemlal and Azeradj varieties respectively and extracted by two-phase centrifugation.These two samples have greater proportion values, indicating that the CC and AC samples contain more chlorophylls (a and b) and pheophytin *a* compared with the other samples. The second group contained CTC, ATC, CTF, ATF and SC samples. They have similar proportion value on IC1 and IC2. These six samples were characterised by a high enough concentration of chlorophylls (a and b) and pheophytin *a.* The third group included, AP, CP and SP samples which obtained from the three studied varieties and extracted by a pressure system. They have a lower chlorophylls and pheophytin *a* content than the other samples.The fourth group contained STC and STF samples from Sigoise variety and extracted by a traditional process. They have the lowest chlorophylls and pheophytin *a* content.

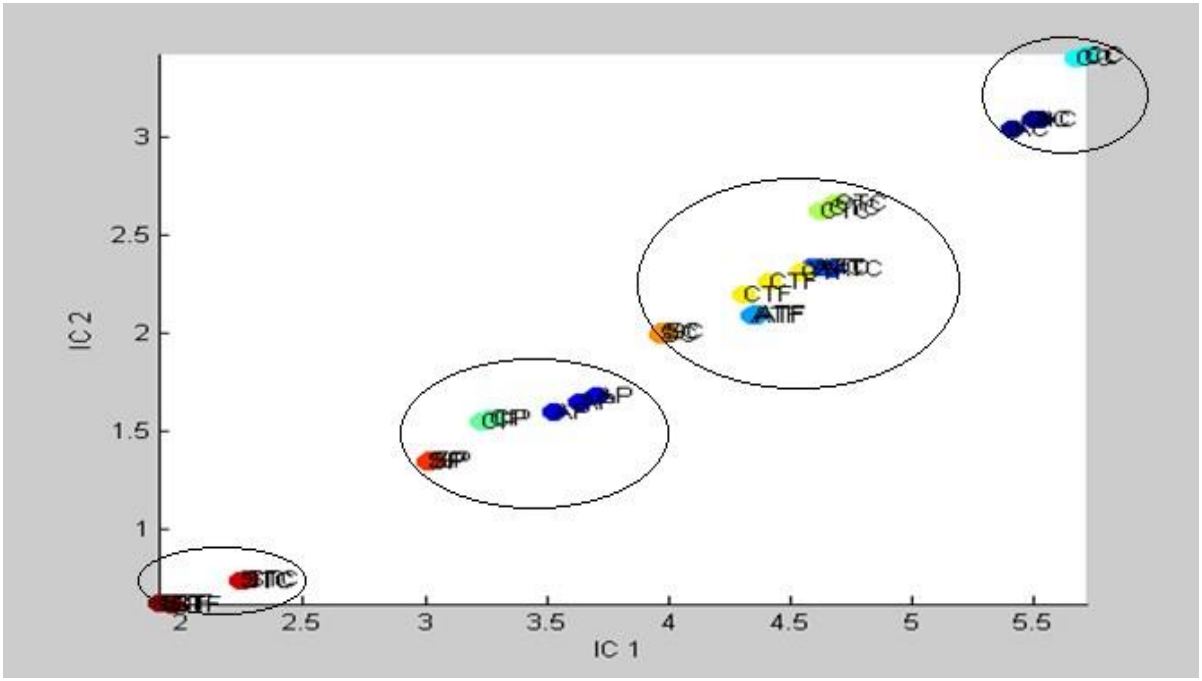

Fig.3. IC1 vs IC2 proportions plot.

Chemlal variety had a higher content of chlorophylls (a and b) and pheophytin (*a and b)* than the other varieties. The chlorophyll content of nine virgin olive oils from a single variety that come from the primary producing regions of Spain was examined by Gandul Rojas and Minguez-Mosquera (1996) .Variations based on fruit variety and level of ripening have been observed in the results.The maturity of olives, the variety, and the olives' geographic origin all affect the amount of pigments in olive oil (Lazzerini et al.2016).

The oive oils from the centrifugation system have higher chlorophylls (a and b) and pheophytin (*a and b)* content than those from the pressure system. Cichelli and Pertesana (2004) and Petrakis (2006) have reported findings that are comparable. We can observe an increases in the concentration of chlorophylls (a and b) and pheophytin (*a and b)* in the olive oils due to heat-treatments of the olive fruits. The results obtained conform to the observations of Jareén-Galaén *et al*. (1999); Luaces *et al.(*2005); Giuliani *et al.(*2011) and Al-Rousan (2017),who discovered that decreased lipoxygenase activity was the cause of the rise in oil pigment extracts from olives heated to a high temperature.

IC 3 (Fig.4 b), shows a characteristic band with excitation at 350-380 nm and emission at 420-550 nm which was collectively associated with the presence of oxydation products, as noted earlier by Guimet *et al.* (2004) and Lia *et al.* (2020). IC 4 (Fig. 4 d), shows three bands,the first band , representing maximum excitation and emission at 310 and 360 nm, respectively, while the second band observed at 670 nm in emission and 405 nm in excitation and broad band emit in the400-550 nm region, with excitation between 380 and 430 nm.These bands belongs to primary and secondary oxidation products. Similar results were found in other studies (Ammari *et al.,* 2012 ; Hernández-Sánchez *et al.*,2018). The proportions on IC3 and IC4 are presented in Fig.4 a and c. STC and STF samples have a higher proportion value than the other samples and AP and CC sampleS have the lowest proportion.

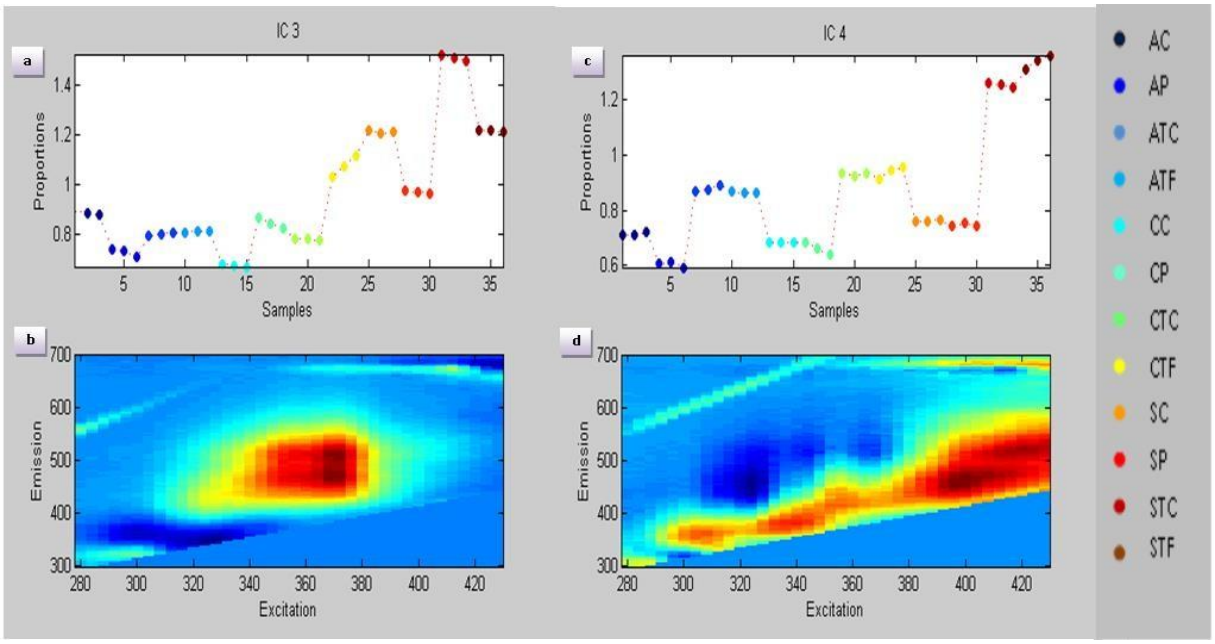

Fig.4. Proportions of olive oil samples (a) and extracted signal (b) on IC3, Proportions of olive oil samples (c) and extracted signal (d) on IC4.

Olive oil samples were split into three groups (Fig.5), the first group formed by six samples (AP, AC, CP, CC, SP and SC), obtained from Azeradj, Chemlal and Sigoise varieties and extracted by pressure system and two-phase centrifugation. These samples were characterized by a lower content of oxydation products. The second group, including ATF, ATC, CTF and CTC samples, obtained from Azeradj and Chemlal varieties and extracted by traditional process.These olive oils have a medium content of oxydation products. The last group, formed by two samples (STC and STF) from Sigoise variety and extracted by traditional process. This group had the highest content of oxydation products.

Chimi (2006), examined the effects of three different processing methods (pressure system, two-phase and three phase centrifugation) on the composition of olive oil. He observed that the oil obtained through two-phase centrifugation had a higher polyphenol content than that obtained through pressure system and three-phase centrifugation. According to Velasco and Dobarganes (2002), Polyphenols and tocopherols are natural antioxidants that inhibit oxidation in virgin olive oils.It should be noted that olive oil from Sigoise variety is more oxidised than the other varieties. Mansouri *et al.* (2014), indicated that oxidative stability is strongly dependent on each olive variety. In the seven Extremadura varieties examined throughout the fruit's maturity, a strong correlation was discovered between antioxidant capacity, total phenolic content, and oxidative stability. This suggests that the amount of phenolic compounds present has a major impact on antioxidant capacity and serves to significantly inhibit lipid oxidation (Franco and al., 2014).

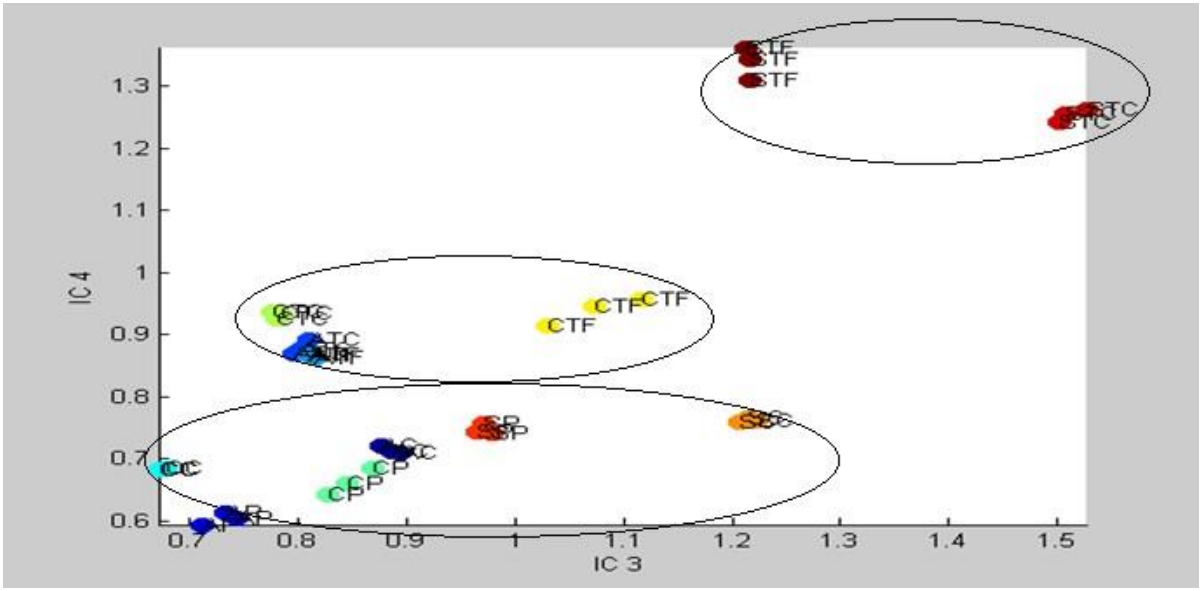

Fig.5. IC3 vs IC4 proportions plot.

IC6 (Fig.6 b) shows a spectral band with excitation at 280-310 nm and emission at 300 340nm. This band could be assigned to tocopherols (Benabid ,2009 ; Lia *et al.* ,2020). IC8 (Fig.6 d) shows a short wavelength band at around 280-300 nm in excitation and at around 300- 330 nm in emission. Zandomeneghi *et al.* (2005) and Lia *et al.* (2020), attributed this band to polyphenols. IC7 (not presented here) shows a Rayleigh scatter line which appears when the emission wavelength is equal to or twice the excitation wavelength.

Fig.6 a illustrate the proportions on IC6. CP and CTF samples have the higher proportions of tocopherols whereas SC and STF have the lower proportions. Sigoise variety had a lower values of tocopherols compared to those obtained from Chemlal and Azeradj varieties. Significant differences were seen in the tocopherol contents between the different varieties.The genetic characteristics and metabolic activity of each variety are likely related to these differences (Sakouhi *et al.,* 2008). Similar results have been reported by Douzane *et al.* (2012) ; Portilla *et al.* (2014) ; Ambra *et al.* (2017).

Regarding Chemlal and Sigoise varieties, olive oils extracted by pressure system have a higher tocopherols content compared to those extracted by two-phase centrifugation. Salvador et al. (2003), reported that the oil extracted using a centrifuge had a higher tocopherol content than the oil extracted using pressure.These results are similar to those found for Azeradj variety. On the other hand, similar tocopherols content was found in this variety extracted with traditionals process (ATF and ATC). For Chemlal variety a higher tocopherols content was found in the traditional cold process (CTF) than in the traditional hot process (CTC). Sigoise olive oils extracted from traditional cold process (STF) obtained values slightly below those extracted with traditional hot process (STC). The effect of extraction process on tocopherols content was not clear.

Fig.6 c, shows the proportions on IC8. Olive olis of Azeradj variety had a higher values for polyphenols than those obtained from Chemlal and Sigoise varieties. These results are consistent with those obtained by Soufi *et al.* (2018) ; Ait Chabane *et al.* (2019). However, Olive olis extracted with traditional process present the lowest values of polyphenols in all the varieties studied. In fact, polyphenols are highly soluble in water. The addition of water during malaxation in the traditional process increases their loss in the vegetative water. For Chemlal and Sigoise varieties, olive olis obtained from two-phase centrifugation have a higher values for polyphenols than those obtained from pressure system.

Chimi (2006), found that oils produced using two-phases centrifugation have higher polyphenols content than oils produced using pressure system. The two-phase centrifugation method preserved the polyphenolic compounds, avoiding the necessity of adding water during malaxation.

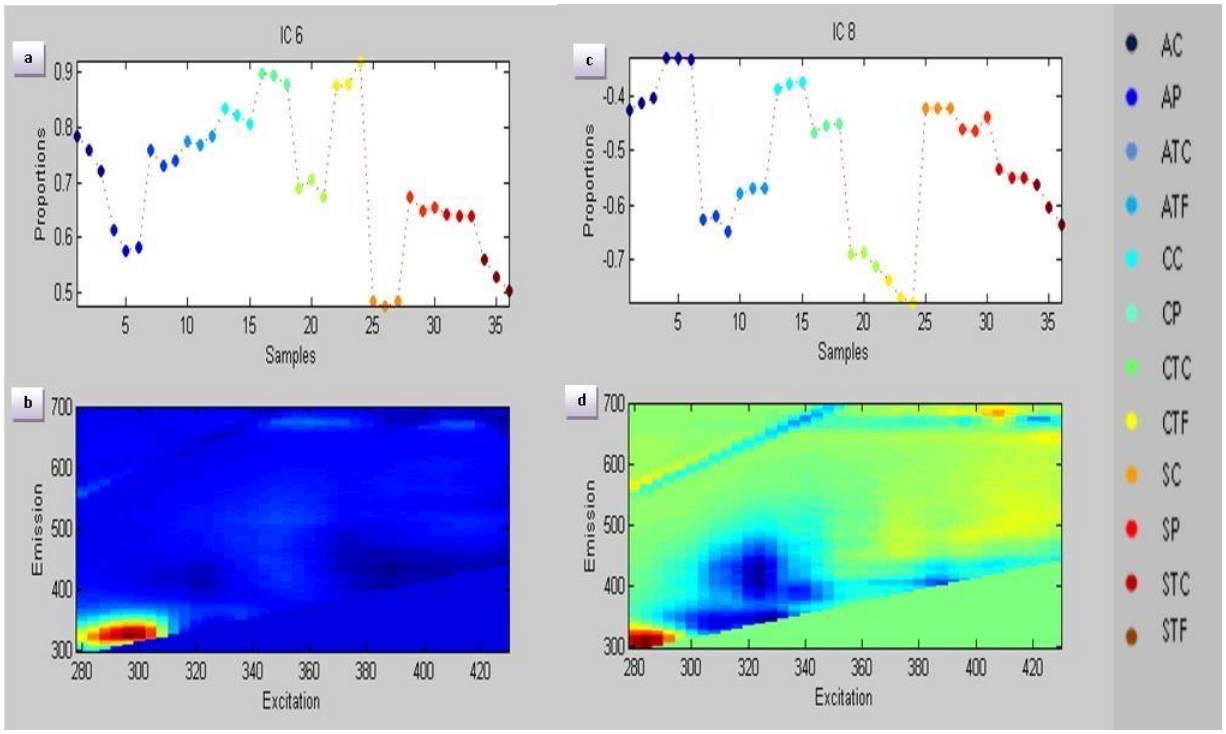

Fig.6. Proportions of olive oil samples (a) and extracted signal (b) on IC6, Proportions of olive oil samples (c) and extracted signal (d) on IC8.

Fig.7, shows very clear differences between all olive oil samples. For the three varieties studied, olive oil samples (AP, AC, CP, CC, SP and SC) extracted with pressure system and two-phase centrifugation presnt a higher values for polyphenols than those extracted with traditional process (ATF, ATC, CTF, CTC, STF and STC). Olive oil samples obtained from Chemlal and Azeradj varieties have higher values of tocopherols than those obtained from Sigoise variety.

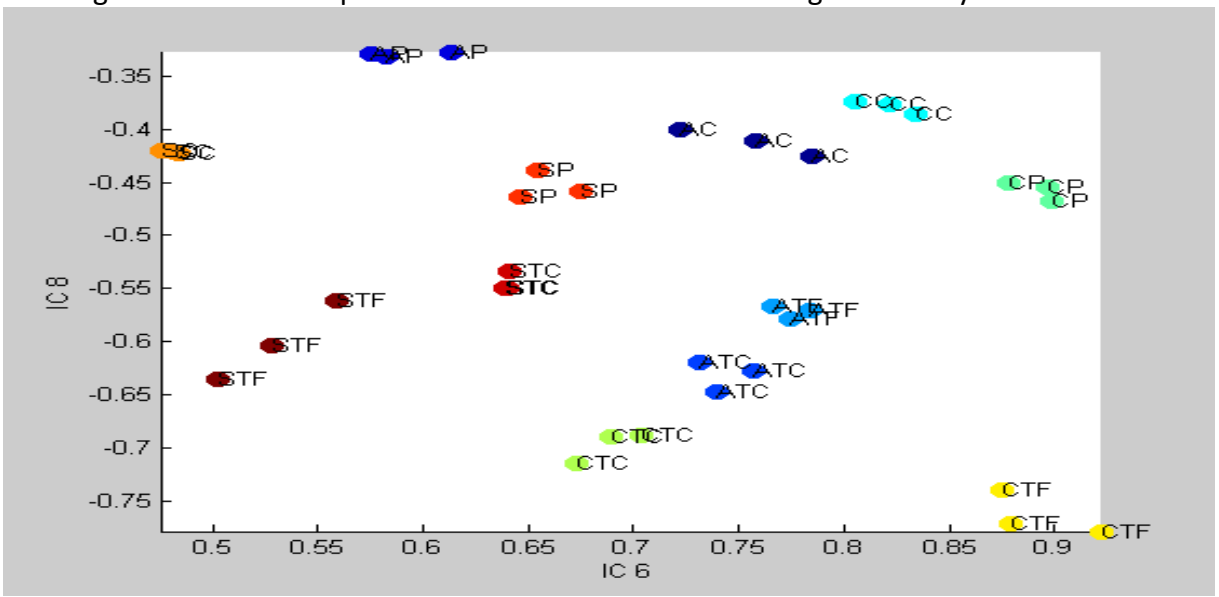

Fig.7. IC6 vs IC8 proportions plot.

4. Conclusions

Independent Components Analysis (ICA) was used to analyse 3D- fluorescence in order to extract pure signals .Seven distinct fluorophores were obtained (pheophytin a, pheophytin b, chlorophylls a and b, polyphenols, tocopherols, primary and secondary oxidation products). Olive oils extracted with two-phases centrifugation were characterized by greater amounts of chlorophylls, pheophytins and polyphenols, lower content of oxydation products compared to other extraction

systems. Olive oils produced with traditional process presnt lower values of polyphenols and higher content of oxydation products than those produced with pressure system.
A large portion of the polyphenols in the olive oil are removed when water is added to the pastes during malaxation in the traditional process . No water needs to be added for the two-phase centrifugation process.which preserve the polyphenol compounds. Higher contents of chlorophylls, pheophytins and tocopherols were observed in olive oils obtained from Azeradj and Chemlal varieties. 3D-fluorescence spectroscopy has shown to be a powerful tool for discerning composition differences between olive oils from different varieties and extracted with different systems.

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