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Original Scientific Paper

Characterization of imported olive oil: contribution to efficient verification of declared quality and oil type

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ABSTRACT

The feasibility of applying basic chemical parameters and fatty acid analysis for 26 samples of imported olive oil, coupled with statistical multivariate methods, to confirm the declared quality and type of oil is described. The research included the determination of peroxide value, iodine value, saponification value, free fatty acids expressed as oleic acid, moisture and volatile matter, as well as fatty acid content according to the requirements of the olive oil regulation. Of the 26 olive oil samples, declarations stated 19 were extra virgin, 5 were virgin, while one sample each of pure olive oil and olive pomace oil were tested as control samples. The results showed that, on such a limited number of samples and quality parameters, principal component analysis (PCA) cannot be successfully applied, while linear discriminant analysis (LDA) gave satisfactory results when all tested parameters were included.

1. Introduction

Olive oil (OO) makes up only a small portion of the total consumption of vegetable oils today, but it has tremendous economic importance for producing countries while its nutritional value and peculiar sensory attributes are important for consumers worldwide (*Caporaso and Boscou*, 2021; *Rosati et al*., 2014). Studies have shown that OO has beneficial influence in reducing the risk of coronary heart disease, cancer, inflammation, diabetes, Alzheimer's disease, and metabolic disorders (*Malheiro et al*., 2014; *Beltrán et al.*, 2016, *Rousos et al*, 2025). These properties have their origins in the unique composition of triacylglycerols and fatty acids (FAs) in OO, its richness in monounsaturated FAs, especially oleic acid (*Malheiro et al*, 2014; *Lanca and Ninfali*, 2020), its low levels of polyunsaturated FAs, and its high levels of natural antioxidants. The characteristic composition of OO is responsible for its resistance to rancidity and its antioxidant properties (*Beltran et al*., 2016; *Tsimidou*, 2016; *Mastralexi and Tsimidou*, 2021).

National authorities have legislated olive oil standards and grades (*Official Gazette SCG*, 2004), following the groundwork laid by major OO regulating authorities, such as Codex Alimentarius (*Codex Stand*, 2024), International Olive Council (*IOC*, 2021), European Union (*EU*, 2022), United States Department of Agriculture (*USDA*, 2010) and Australian authorities (*Primefact* 231, 2006). The highest quality OO, so-called virgin olive oil (VOO), is obtained mechanically at monitored temperature to avoid changes in the composition of the oil and loss of quality. VOO for consumption is divided, according to the method of production

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and quality, into extra virgin (EVOO) and common VOO. The price of OO is proportional to the quality. The cheaper grades of OO are pure OO (PuOO) and olive pomace oil (PoOO). There are known cases of cheaper oils being over-declared as higher quality or of their blending in order to achieve economic benefit.

OO is expected to meet some basic physical and chemical quality standards which differentiate it from other oils. Therefore, some of the basic quality parameters should be met, such as peroxide and iodine values, free FA content and FA composition of OO, as are regulated in the legislation concerning OO (*Official Gazette SCG*, 2004; *Codex Stand*, 2024; *IOC*, 2021; *EU*, 2022; *USDA*, 2010; *Primefact 231*, 2006). Data on sterol composition, wax content, trans FA content, UV absorption and triacylglycerol composition are generally used only to determine the adulteration of OO.

One purpose of this research was to determine whether it is possible to confirm the declared quality grade using multivariate analysis on the basic quality parameters of OO. Also, another aim was to determine the significance of the parameters that are essential for OO's grade categorization.

2. Materials and methods

2.1. Chemicals

All standard chemicals and reagents were purchased from Merck KGaA, Darmstadt, Germany. Ultrapure water, ≥18 MΩ, was ELGA DV-25 and ELGA Ultrapure (LabWater, Lane End, High Wycombe, UK).

2.2. Samples

Samples were part of the regular controls of food quality parameters of OO according to regulatory requests (*Official Gazette SCG*, 2004), obtained from retail and from importers. The research included total of 26 samples, mostly virgin olive oils (19 EVOO and 5 VOO). Two samples, one each of PuOO and PoOO, were analysed for the purpose of verifying statistical results. The samples were labelled in the laboratory according to the corresponding declarations on the OOs.

2.3. Chemical parameter determinations

Peroxide value (PV), iodine value (IV), saponification value (SV), free FA (FFA) content, and moisture and volatile content (CW) were determined according to their respective ISO reference methods (SRPS EN ISO 3960:2017, SRPS EN ISO 660:2021, SRPS EN ISO 3961:2019, SRPS EN ISO 662:2017, SRPS EN ISO 3657:2023). Analyses were carried out following the requirements of national regulation for OO.

2.4. GC chromatography

FA determination was performed according to the reference method (ISO 5509:2000). Oils were converted to FA methyl esters (FAMEs) as described by Spirić et al. (2010). FAMEs were determined by capillary gas chromatography on a Shimadzu 2010 gas chromatograph (Shimadzu, Kyoto, Japan) equipped with the flame ionization detector and a capillary HP-88 column 100 m × 0.25 mm × 0.20 μm (J&W Scientific, Folsom, California, USA). The chromatographic peaks in the samples were identified by comparing relative retention times of FAME peaks with peaks in Supelco 37 Component FAME mix standard (Supelco, Bellefonte, Pennsylvania, USA).

2.5. Statistical analysis

Principal component analysis (PCA) and linear discriminant analysis (LDA) of FAs and chemical analysis of OOs were performed using the JMP Statistical Discovery 10 software (SAS Institute, Cary, North Carolina, USA). Descriptive statistics and preparation of data for multivariate statistical analysis were performed in MS Office 2016 Excel.

3. Results and discussion

Results of chemical parameters (IV, PV, FFA, CW and SV) and FA determination of OO samples are presented in Table 1.

Data shown in Table 1 were further subjected to multivariate statistical analysis in an attempt to determine whether it was possible to group the samples into the declared OO quality types, based on the results obtained for chemical parameters and FA composition.

Graphical representation of the PCA results of the data from Table 1 is presented in Figure 1. The first two principal components (PC1 and PC2) accounted for less than 50% of the total data variance (33% and 15%, respectively). The confidence ellipses (at a 95% confidence interval) showed complete overlap of the

Table 1. Fatty acid content and chemical parameters of the olive oils analysed

Label*	C14:0	C16:0	C16:1	C17:0	C17:1	C18:0	C18:1	C18:2	C18:3	C20:0	C20:1	C22:0	C24:0	IV	PV	FFA	CW	SV
E	0.05	12.74	0.70	0.04	0.06	2.73	74.29	6.54	0.79	0.37	0.24	0.12	0.06	89.55	2.56	0.40	0.17	191.03
E	0.04	17.84	1.65	0.10	0.20	2.74	60.92	13.55	0.68	0.31	0.23	0.09	0.10	89.88	1.62	0.33	0.16	192.54
E	0.05	13.19	0.77	0.01	0.01	2.75	74.55	7.21	0.66	0.34	0.21	0.10	0.05	89.79	6.18	0.59	0.09	190.92
E	0.01	11.83	0.72	0.04	0.06	2.73	76.83	6.22	0.44	0.37	0.35	0.13	0.05	78.48	5.13	0.30	0.10	188.08
E	0.01	10.82	0.71	0.01	0.01	2.45	76.81	5.57	0.42	0.41	0.50	0.10	0.01	84.84	5.71	0.70	0.11	187.97
E	0.01	10.71	0.50	0.01	0.01	2.30	78.91	5.35	0.72	0.36	0.01	0.09	0.01	90.26	7.84	0.78	0.17	191.55
E	0.02	10.77	0.57	0.02	0.02	1.91	79.54	4.47	0.77	0.36	0.02	0.08	0.02	80.45	9.82	0.69	0.11	195.54
E	0.02	11.78	0.62	0.02	0.02	2.36	78.09	5.25	0.82	0.39	0.02	0.10	0.02	89.35	4.80	0.99	0.17	185.94
E	0.03	11.49	0.51	0.03	0.05	1.85	77.95	6.10	0.49	0.28	0.19	0.09	0.03	80.06	3.13	0.54	0.07	189.24
E	0.01	13.44	0.88	0.08	0.13	2.18	71.70	9.22	0.60	0.32	0.22	0.10	0.04	75.19	9.83	0.96	0.13	186.96
E	0.02	11.22	0.64	0.07	0.11	2.37	75.21	7.98	0.63	0.07	0.02	0.09	0.04	81.09	6.04	0.72	0.17	193.70
E	0.05	14.28	1.02	0.06	0.10	2.37	69.19	10.92	0.61	0.30	0.02	0.08	0.04	81.96	9.22	0.78	0.16	195.41
E	0.04	11.29	0.50	0.04	0.04	2.27	78.24	5.51	0.45	0.37	0.02	0.11	0.04	83.00	9.72	0.96	0.15	186.18
E	0.02	11.80	0.67	0.05	0.09	3.11	75.71	6.89	0.65	0.47	0.02	0.17	0.02	82.09	9.97	0.85	0.16	189.27
E	0.02	11.66	0.56	0.03	0.02	2.37	78.30	6.09	0.21	0.36	0.01	0.11	0.04	77.24	7.42	0.96	0.13	190.07
E	0.02	10.42	0.44	0.03	0.02	2.09	78.03	7.32	0.43	0.29	0.02	0.08	0.04	93.94	9.60	0.80	0.14	192.48
E	0.02	9.96	0.52	0.06	0.07	3.29	80.00	4.79	0.43	0.37	0.04	0.10	0.03	80.36	7.31	0.54	0.11	186.34
E	0.02	9.69	0.46	0.05	0.09	2.07	81.06	5.18	0.46	0.32	0.02	0.10	0.03	84.31	1.45	0.17	0.10	188.28
E	0.02	10.55	0.49	0.04	0.06	2.28	79.74	5.78	0.47	0.35	0.02	0.10	0.03	82.02	7.64	0.60	0.18	186.99
V	0.01	12.98	1.18	0.10	0.19	2.37	70.96	10.62	0.83	0.33	0.01	0.11	0.01	80.06	8.96	0.30	0.10	187.04
V	0.02	11.58	0.60	0.03	0.05	2.21	78.27	5.93	0.56	0.33	0.31	0.10	0.04	84.94	9.12	0.93	0.18	185.39
V	0.05	11.93	0.57	0.05	0.06	2.57	77.97	4.84	0.53	0.36	0.21	0.11	0.04	81.09	7.54	0.43	0.09	185.52
V	0.02	11.33	0.52	0.03	0.04	2.20	79.95	4.56	0.45	0.32	0.02	0.10	0.04	81.32	9.80	0.98	0.17	188.98
V	0.03	13.67	1.01	0.08	0.16	1.97	73.41	8.48	0.41	0.31	0.02	0.09	0.03	80.52	5.49	0.37	0.07	187.94
O	0.02	11.85	0.61	0.05	0.08	2.60	75.49	7.66	0.41	0.30	0.02	0.08	0.03	93.26	3.71	0.52	0.12	194.43
P	0.05	12.37	0.80	0.09	0.04	2.94	70.59	10.76	0.71	0.46	0.02	0.18	0.08	83.51	5.52	0.40	0.09	182.11

* Olive oil label abbreviations correspond as follows: E – extra virgin olive oil, O – pure olive oil, P – olive pomace oil and V – virgin olive oil. IV, iodine value; PV, peroxide value; FFA, free fatty acid value; CW, moisture and volatile content; SV, saponification value.

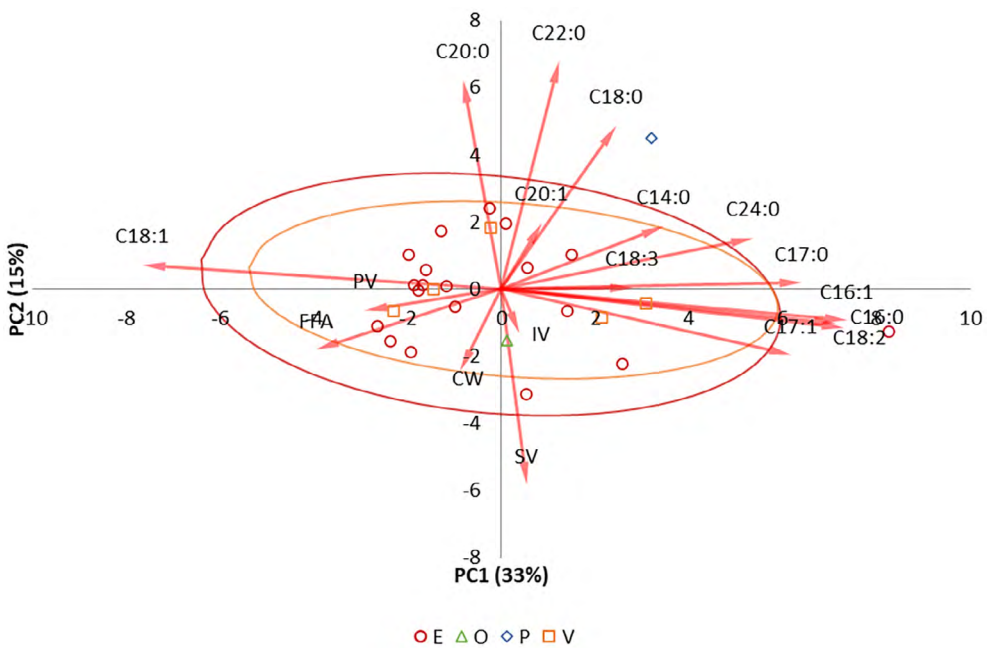

Figure 1. PCA of the examined olive oils' fatty acid content and chemical parameters

data for EVOO and VOO. The control PuOO sample was also included in the confidence ellipses, while the PoOO sample was completely separated from the other groups. The general conclusion is that PCA cannot be applied to determine OO types with the given dataset. The exclusion of PoOO was, in the first place, due to its greater content of saturated FA. The lack of separation is most likely a consequence of comparing the analytical results for samples originating from different geographical climates, produced from different olive cultivars, and with variations in the method and quality of production. All these factors likely contribute to variability in the data that cannot be directly attributed to the analysed parameters.

The results of LDA of chemical parameters and FA content are shown graphically in Figure 2. The first and second canonical functions (canonical 1 and canonical 2) were used for grouping data and obtaining scoring coefficients. Both canonical functions explained 94.6% of the total data variance. Canonical function correlations were 0.984 for canonical 1 and 0.923 for canonical 2. There were no misclassified samples in the analysis (19 EVOO, 5 VOO, 1 PuOO and 1 PoOO). From Figure 2, it can be seen that the groups are completely separated from each other without overlapping. From the LDA results, it can be concluded that chemical parameters have less effect than FAs on the separation of OO groups by type, but they cannot be omitted. The separation of EVOO from VOO was most influenced by differences in the content of the FAs oleic (C18:1n-9), palmitic (C16:0) and palmitoleic (C16:1) acids. LDA of OO by type based only on FA content produced a satisfactory separation, as can be seen in Figure 3. In addition, though, LDA results derived only from FA content had misclassified samples.

In other studies, PCA and LDA were effective in determining the origin and/or type of OOs from individual olive cultivars (*Fuentes et al*., 2018, *Zhang et al*., 2024, *Blasi et al*., 2019, *Roussos et al*., 2025, *Revelou et al*., 2021). Different olive cultivars were effectively grouped according to origin based on FA composition and physicochemical parameters by these statistical methods (*Fuentes et al*., 2018, *Zhang et al*., 2024, *Blasi et al*., 2019, *Revelou et al.,* 2021). The methods were applied to monovarietal OOs (*Fuentes et al*., 2018, *Zhang et al*., 2024, *Blasi et al*., 2019, *Roussos et al*., 2025, *Revelou et al*., 2021).

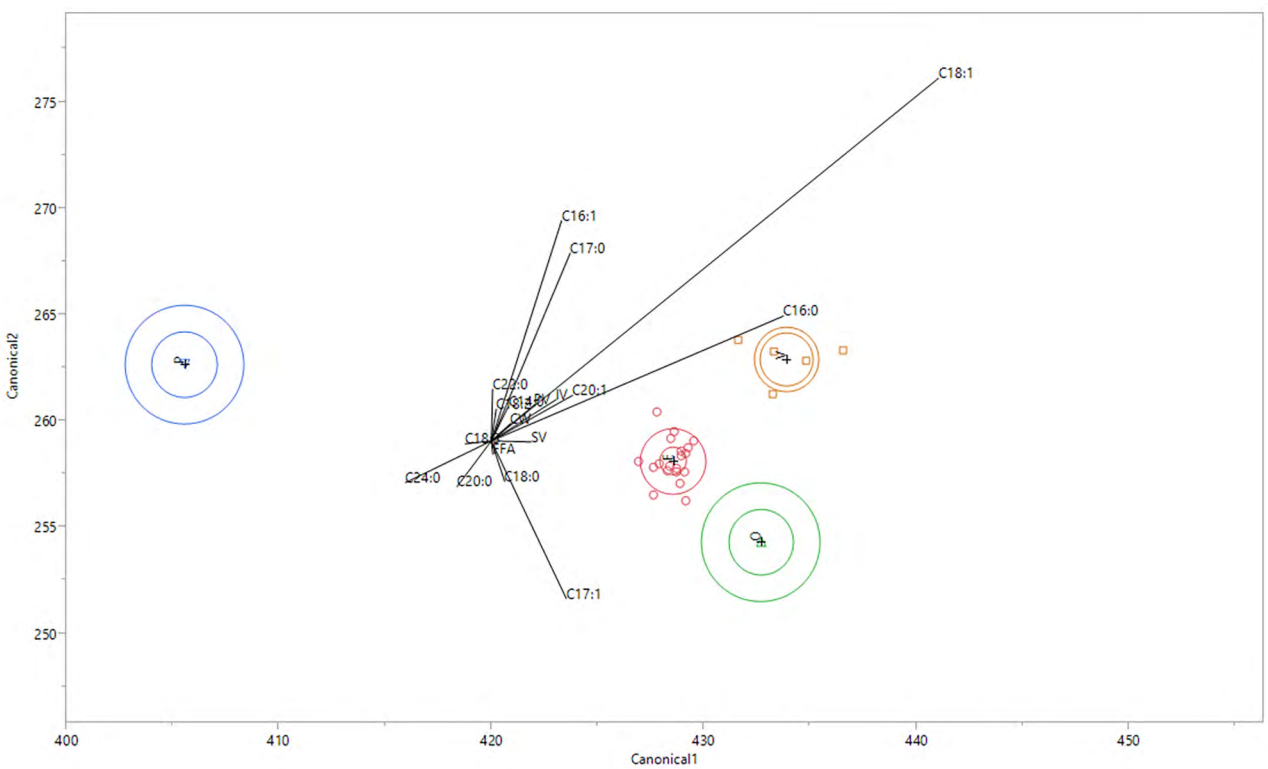

Figure 2. LDA of the examined olive oils' fatty acid content and chemical parameters. Inner circles denoted the 50% contour plot for each group, and outer circles represent the 95% confidence level for each mean

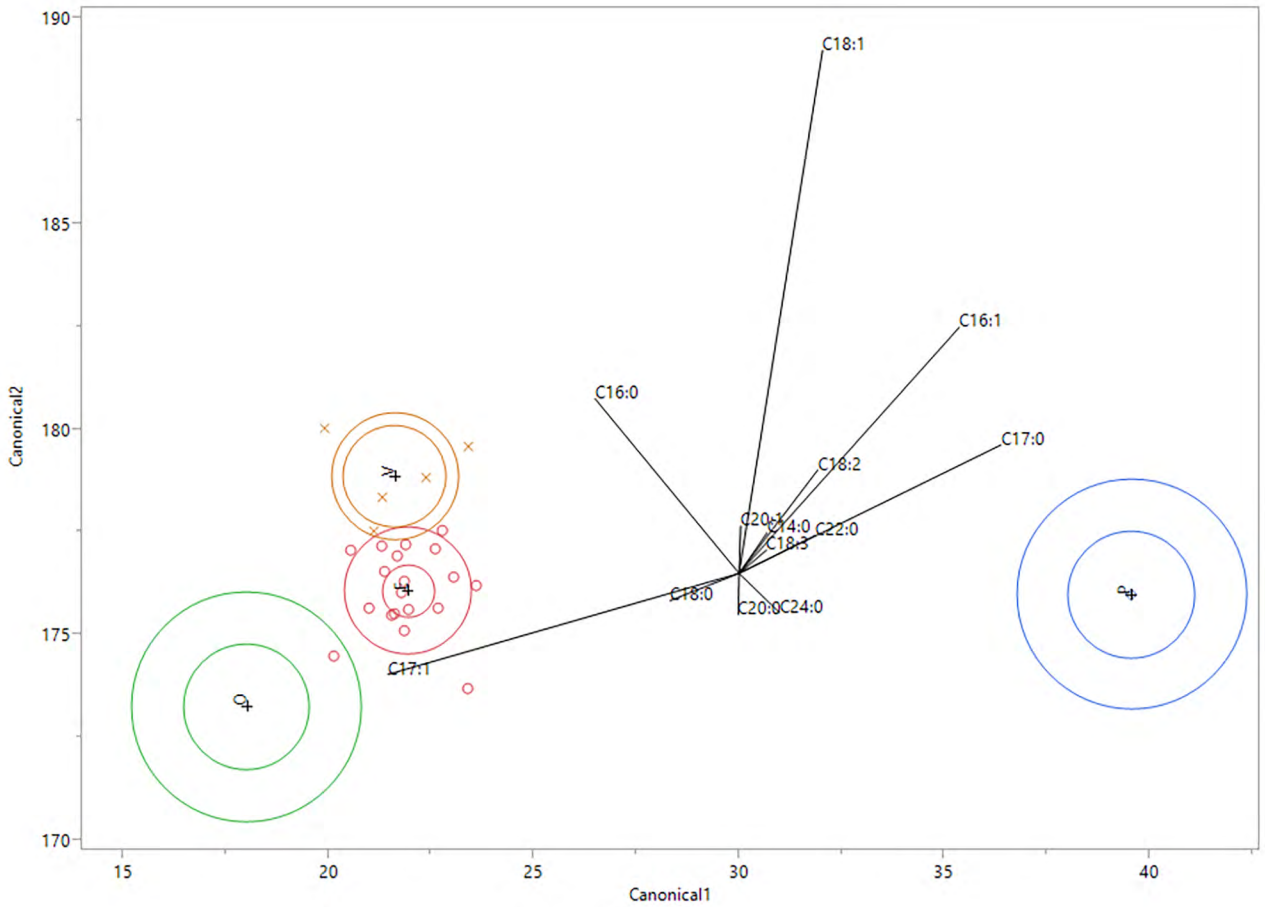

Figure 3. LDA of olive oils based only on fatty acid content. Inner circles denoted the 50% contour plot for each group, and outer circles represent the 95% confidence level for each mean

4. Conclusion

The results of the OO import control based on the requirements of the current regulations are not sufficient to confirm the declared type of OO. This is especially true for EVOO and VOO. However, the results of regular controls can be improved and applied to verify the declared quality and type of OO by processing them with multivariate statistical analysis methods. Although the presented results showed that PCA cannot be successfully applied, LDA proved to be completely satisfactory, even on the small number of OO samples included in the research. Further improvement of the LDA method must be carried out by more extensive validation using a larger number of samples and by including other parameters for testing the quality and adulteration of OO, such as potassium levels, waxes, sterols, etc. This augmentation of analytical techniques would ensure more reliable verification of imported OO, more efficient implementation of legal regulations and better protection of the interests of the domestic market and consumers, from both health and economic aspects.

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