



Investigation of trace metals in different varieties of olive oils from northern Cyprus and their variation in accumulation using ICP-MS and multivariate techniques

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Abstract

Determination of trace metals in olive oil is an essential aspect for the assessment of good quality of dietary oil. It is so, because heavy metals are known to have a negative effect on the oxidative rate of the oils and be toxic to its consumers. This study is the first to be carried out in northern Cyprus aimed at determining the content of copper (Cu), cadmium (Cd), chromium (Cr), arsenic (As), lead (Pb), and nickel (Ni) in different varieties of olives in the region. The analysis was carried out using inductively coupled plasma mass spectrometer following microwave digestion with nitric acid and validated using an oil reference material. Two multivariate statistical analysis (principal component and agglomerative cluster analysis) used for the modeling and classification of different varieties of olive oil produced in the northern region of Cyprus. The metal concentrations are: Cu 1.02–3.81, Cd 0.02–0.09, Cr 0.33–0.95, As 0.03–0.75, Pb 0.15–1.48, and Ni 0.22–0.79 (minimum–maximum in µg/g). The content of Cu was observed with a high concentration of about 87% of all varieties of olive oils, while Pb, Cr, and As have a notable amount in a few of the varieties as compared to the standard limits by FAO/WHO expert committee on food and additives and US environmental protection agency. Other metals like Ni and Cd are found to be tolerable in all the varieties of olive oils.

Keywords Olive oil · Heavy metals · ICP-MS · Multivariate techniques · Varieties

Introduction

Olive oil is a fat obtained from the fruit of the *Olea European sativa* (Olive tree). This vegetable oil is from a traditional tree crop of the Mediterranean region and is also a monounsaturated oleic acid with an authoritative source of vitamin E. Olive oil intake has grown over the years, due to its ability to lower incidence of coronary heart diseases and certain types of cancer, which include breast and colon cancers (Nunes et al. 2011). The phenolic present in olive oil inhibits the oxidation of low-density hypo proteins and possesses other potential biological activities (Benincasa et al. 2007). According to Christodoulou (1959) Cyprus had 2.25 million olive trees of which one million were wild,

since in 1937 with grafting and systematic cultivation from 1946 to 1958, olive trees increased in number by 40% and in 2003–2004 local production of olive oil was 4500 tons (1000 kilos per ton) and in 2004–2005 was 6000 tons (Euphrosyne 1992). In 2011 total capacity of olive oil produced amounted to about 450–500 t/day, which is far above the production needs (Tozlu et al. 2011). Heavy metals such as copper (Cu), cadmium (Cd), chromium (Cr), arsenic (As), lead (Pb), and nickel (Ni) present in olive oil above a required level possess significant health hazards and thereby become extremely toxic to both plant and animals. Some of these heavy metals are considered as a potential carcinogen causing cancer in humans (Nunes et al. 2011). Environmental pollution and contamination due to heavy metals are the severe issues in the world today, the entry of pollutant from natural reservoirs into the environment like soil, water, and air is the most significant adverse impact of human activities on the terrestrial and aquatic ecosystem (Lin et al. 2012). A large proportion of plants in industrialized countries contains higher levels of several elements and compounds, which is considered pollution.

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Inductively coupled plasma mass spectrometry (ICP-MS) is an exceptional technique used for elemental and isotopic analysis, which requires all sample to be digested and diluted to limit the amount of organic matter and dissolved solids. Since samples of the olive oil can influence the overall performance causing problems related to instability or even extinction of the plasma (Chaves et al. 2011). Another adverse effect over injector cones and lens blockage is the carbon deposits from incomplete oxidation of the organic matrix. Furthermore, severe spectral and non-spectral interferences could occur with complex matrices in ICP-MS, but these limitations have been overcome by the addition of oxygen into the plasma to aid oxidation of the organic matter or by decreasing the temperature using a cooled spray chamber (Dos Santos et al. 2007; Edlund et al. 2002).

Content of lead, cadmium, chromium, and nickel in olive oil samples cannot be overemphasized due to its high intake by humans mostly around the Mediterranean. These olives are consumed as food daily, because they play a significant role in human nutrition and also in the process of digestion and metabolism; they mostly react with some chemicals like oxygen (Martinez et al. 2003). Copper (Cu) and iron (Fe) are regulated as the criteria for the qualities of edible olive oils and fruits. Cadmium and lead are considered as significant pollutants, primary sources of Cu, Pb, Cd, Fe, Cr, and Ni contamination might be the soil as natural metal sources, and from packaging, manufacturing, and environmental and air pollution (Lepri et al. 2011). The presence of heavy metal composition in olive fruits occurs as a result of natural pollution from the soil, fertilizers, industrial activities, or closeness to the highways or accumulation of heavy metals in the olive plantations. Olive oil may also be contaminated with the heavy metals during the production process and maybe as a result of its contact with storage materials. Levels of heavy metal in olive oil are one of the quality aspects and also an effect efficient on its oxidation and human health. The level of unsaturation and the presence of heavy metal are the major factors that increase the oxidation rate (Dilek et al. 2013; Meira et al. 2011). The heavy metals increase the rate of oxidation of regular edible oils such as olive oils by helping the generation of toxins from body fat or hydroperoxides. The oxidation processes that occur due to the presence of some trace elements may also generate peroxides, aldehydes, ketones, acids, epoxides, and other compounds, which may develop pathological effects on the digestive system and increase carcinogenic effect by reacting with other food components such as proteins and pigments (Castillo et al. 1999). Several researchers have carried out studies on edible oils by the use of microwave digestion of virgin olive, olive, pomace-olive, sunflower, soybean, and corn (Durali et al. 2009; Llorent-Martínez et al. 2011a; Sabier Luis and Pablo 2018); emulsion breaking, ultrasonic extraction, and wet digestion of sunflower, hazelnut, canola, corn, and olive

oils (Dilek et al. 2013) with quantification methods like atomic absorption spectrometry (AAS) (Durali et al. 2009), ICP-MS (Llorent-Martínez et al. 2011a; Sabier et al. 2018), and inductively coupled plasma optical emission spectrometry (ICP-OES) (Dilek et al. 2013).

This research is aimed at the combination of ICP-MS with multivariate techniques for the quantitative analysis of heavy metals (As, Pb, Cu, Cd, Cr, and Ni) in different varieties of olive oils as suggested by Llorent-Martínez et al. (2011a), so as to ascertain the origin of the traceability of the metals and to better understand how the method of harvest affects the quality of the oil. On that basis, it was also aimed to obtain information on the possible selection of the varieties that are more resistant to the uptake of heavy metals. The microwave digester was also employed for the digestion of the oil samples before being analyzed, as this is the first research that is carried out in the region.

Materials and methods

Sample collection

Samples of olives were obtained from the different markets across northern Cyprus, which include different brands. The olive fruits harvested from the ground and directly from the tree from different locations as shown in Fig. 1 were used to extract the oil sample from it. The fruit seeds were extracted after being crushed using n-hexane by placing it on a heating mantle at 65 °C and soxhalation for 4 h and then cooled. The extracts were filtered using Whatman 40 filter paper, the oil was then separated from the solvent and dried at 60 °C for 10 min and the extracted oil obtained was stored at room temperature. The other oil samples were collected directly from the farmers after locally extracting the oils by the cold press method during 2017. The samples collected were carefully packaged in polyethylene bags to avoid contaminations and then stored at 4 °C in the Environmental Research Laboratory of Cyprus International University (ERL-CIU) until the time of analysis.

Apparatus

A microwave digester speedwave MWS-2 BERGHOF (Germany) equipped with advanced composite PTFE vessels was used for the digestion of all the samples. The Inductively Coupled Plasma Mass Spectrometer (ICP-MS) (Agilent Technologies, Santa Clara, CA) run under a linear multi-point calibration. The ICP-MS equipment was turned on using aqueous multi-element standards of 10 ng/mL. Table 1 shows the operational equipment procedures.

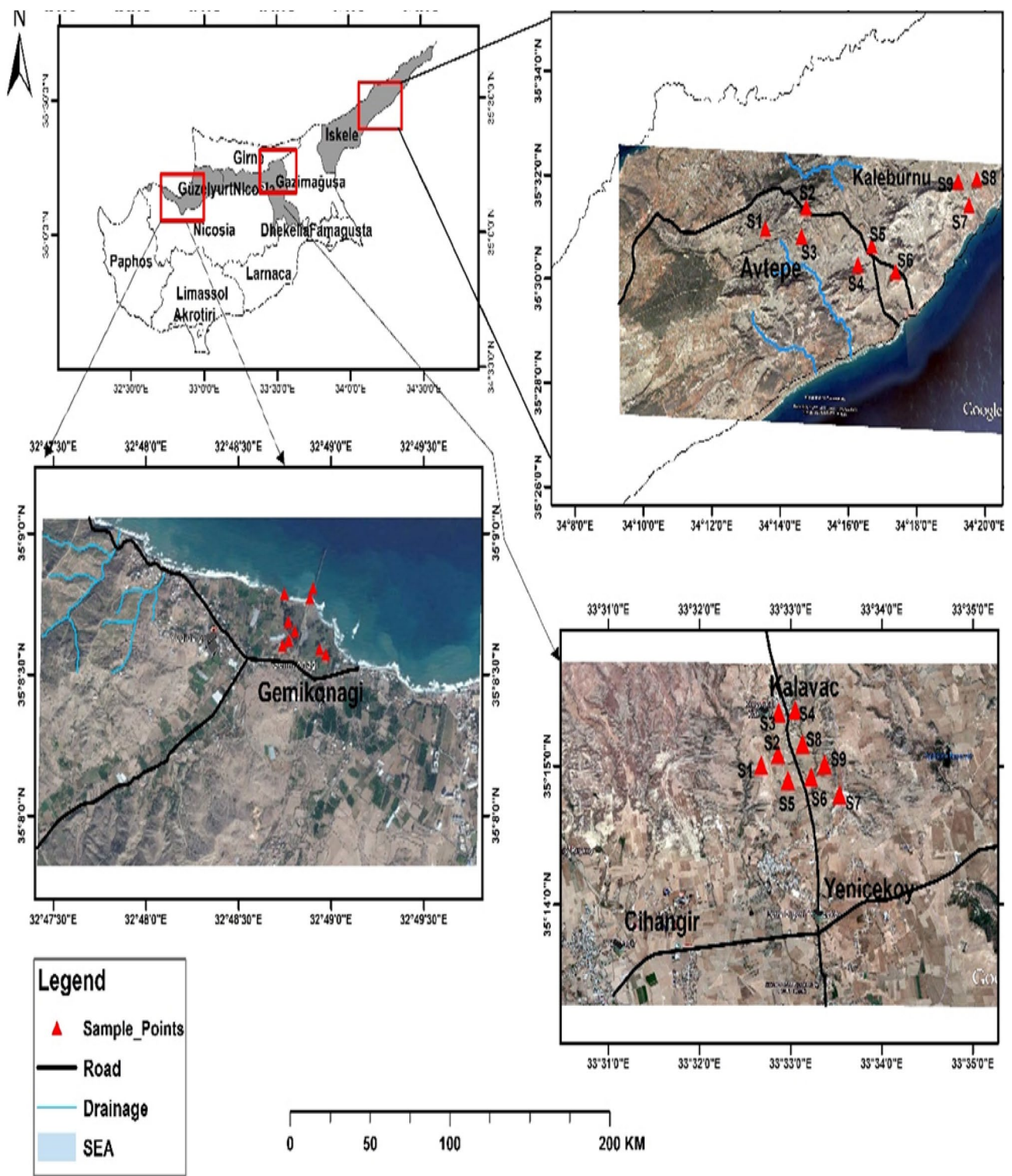


Fig. 1 Map showing the sampling area

Reagents

The reagents used were of the highest available purity and analytical-reagent grade. Double deionized water (18 m cm

resistivity) was used for all the research experimental processes and dilutions. The chemical (HNO₃) used was of super-pure quality (Merck Darmstadt, Germany). The apparatus used like glasswares were washed and cleaned

Table 1 Operating conditions for ICP-MS

Plasma conditions	
RF power	1.2 kW
Plasma Ar flow rate	15 L min ⁻¹
Auxiliary Ar flow rate	0.89 L min ⁻¹
Carrier Ar flow rate	0.95–1.0 L min ⁻¹
Torch horizontal alignment	(0.5–1.0) mm
Torch vertical alignment	0.2–0.5 mm
Sampling depth instrument	6.0–8.0 mm
Sampler cone	Nickel, 1.0 mm orifice diameter
Skimmer cone	Nickel, 0.4 mm orifice diameter
Isotopes measured	⁶³ Cu, ⁷⁵ As, ⁵³ Cr, ⁶⁰ Ni, ¹¹¹ Cd, ²⁰⁶ Pb

Table 2 Operating program for the microwave digestion

Step	Initial T (C)	Final T (C)	Time (min)	Power (W)
1	25	90	5	700
2	90	90	3	600
3	90	170	10	600
4	170	170	7	600

extensively with nitric acid solution and further rinsed with double deionized water, respectively. All the standard solutions used for the calibrations were prepared by diluting a stock of all the elements to be analyzed such as Cd, Cu, Ni, Pb, Cr, and As supplied by Sigma chemical company similar to the work done by Mendil et al. 2009.

Sample preparation and digestion

Microwave-assisted digestion function is being performed to dissolve the oil sample for analysis as mentioned in different researches carried out (Ansari et al. 2009; Llorent-Martínez et al. 2011a, b; Mendil et al. 2009), while targeted microwave-aided digestion function for the same aim has been applied in a few research (Sant'Ana et al. 2007). As shown in Table 2, research has been carried out on the digestion of olive oil using various conditions by microwave digestion.

The samples collected were digested by placing 0.2 g of olive oil in a polytetrafluoroethylene (PTFE) flask, then 5 ml of acid and oxidant mixture was added [HNO₃ (65%)]. The flask was kept at room temperature (18 degrees Centigrade) for 1 h and then transferred to a Teflon container, then placed in a microwave-assisted digestion oven and heated to about 80% with a power of 900 w for 5 min to decompose the organic matter in the olive oil sample. The digestion flask was then removed and allowed to cool for 10 min; the flask was carefully opened inside the fume cupboard and dried. It was then diluted with 0.2 M nitric acid up to 10 mL in

a volumetric flask and the sample was kept as a stock. A blank (without sample) digestion was done using the same method as the sample to obtain a comparable result and also to calibrate the instrumental reading that will be obtained. Some calculations were carried out to get the accuracy and precision of the analytical method by analyzing the certified reference materials.

Physicochemical analysis of olive oils

The most important physicochemical parameters of olive such as UV absorption characteristics at 232 and 270 nm, free acidity, and peroxide indices were analyzed according to the official methods of European Union (Issaoui et al. 2010).

Total phenols analysis

The modified isolation method was used to determine the total phenol content in the olive (Gharsallaoui et al. 2011). 2.5 g of olive oil sample was dissolved in hexane and further extracted with a solution of methanol and water. The concentration of the fraction was estimated using Folin–Ciocalteu reagent at 725 nm and quantitation was achieved using external calibration curve ($r^2=0.996$) made with caffeic acid purchased from Sigma-Aldrich.

Statistical analysis

XLSTAT software was used in the statistical analysis and computation for ANOVA and multivariate analysis involving 15 varieties of olive oil performed through the principal component analysis (PCA) and agglomerative hierarchical clustering analysis (AHCA) to group and analyze clusters of similar characteristics (Ansari et al. 2009; Kazi et al. 2008).

Results and discussion

Free fatty acid

Analysis of variance (ANOVA) applied to the values of free acidity has made a distinction between the oils under investigation. Considering the harvesting mode, oils obtained from olives that remained in contact with the soil for a short period gave a value of free acidity of 2.74%, higher than the expected range of 0.8%–2.0%. Oils derived from olives that harvested directly from the plants gave a value of 0.99%, inside the said range.

Polyphenols

Considering the factor of analyses being the harvesting mode, it is highly evident that the most abundant content of

polyphenols is found in oils produced from olives harvested directly from the plants (Gharsallaoui et al. 2011). This value decreases significantly with increase in the contact of the fruits with the soil. The polyphenols content of oils produced by processing the fruits collected from the ground in the last period of the harvest was very low (17.16 mg/kg).

Fatty acid methyl esters (FAMES)

In general, the fatty acid content is typical of Mediterranean olive oils (Dabbou et al. 2010; Issaoui et al. 2010). However, the oils under analysis were dominated by palmitic acid (C16: 0), stearic acid (C18: 0), and oleic acid (C18: 1), (Table 3). In this study, the observed values do not show a particular pattern that can explain the permanence of the olives with the soil and the incidence of the different water irrigation regimes on the quality of the oils obtained. In fact, the fatty acids of olive oil are independent of the processing and harvesting methods of the olives, but rather depend on genetic factors.

Quality control and quality assurance data of ICP-MS

The detection power for ICP-MS was able to give a very reliable data for the detection of heavy metal (As, Cd, Cr, Cu, Ni, and Pb) in olive fruit seed and oil grown and produced in Gemikonagi, Kalavac and Avtepe/Kelebrunu province of Cyprus after digestion using microwave digester.

A recovery test was conducted using certified reference material ERM.CAO11C containing all the elements under study. The material was characterized using a combination of ID-MS analysis. The sample was spiked with a solution containing enriched ^{206}Pb (NIST SRM 983) to give a predicted ^{208}Pb : ^{206}Pb ratio 1. Duplicate measurements were carried out on each sample. Table 4 shows the results of the recovery test values, which were between 99.1 and 100.3% and they were in good agreement with the certified values. There was also a control of the microwave digester concerning the recovery values.

Statistical analysis of multivariate data

The combination of ICP-MS and multivariate statistics shows a very reliable result in the characterization of data

Table 4 Heavy metal concentrations in certified reference material (ERM.CAO11C)

Element	Certified value ($\mu\text{g/g}$)	Found value ($\mu\text{g/g}$) ^a	Recovery (%)
Cu	1963 \pm 20	1965 \pm 20	100–100.2
Cd	5.31 \pm 0.02	5.25 \pm 0.02	98.8–99.10
Cr	49.3 \pm 0.60	48.79 \pm 0.20	98.9–99.60
Pb	10.02 \pm 0.03	10.04 \pm 0.04	99.7–100.0
As	10.25 \pm 0.10	10.28 \pm 0.20	99.7–100.30
Ni	19.49 \pm 0.04	19.35 \pm 0.03	99.2–100.0

^a(\pm SD, $n=3$)

obtained from the analysis. Principal component analysis (PCA) and agglomerative hierarchical clustering analysis (AHCA) were used to interpret the data collected. The PCA allowed a substantial decrease in some variables and the detection of structure in the correlation between metals and different varieties of olive fruit seeds and oils that give favorable information on the data. As predicted by PCA in Fig. 2, all the varieties contain different amounts of metals, even though the varieties were grown and produced in different areas of the region. The data matrix consisting of different varieties were introduced to PCA for modeling; this was divided into sample codes representing different locations (Avt, Gem, and Kal) and different data set of metals. The samples were selected randomly and the results for As, Cd, Cr, Cu, Ni, and Pb were obtained. Therefore due to the large variability of the result, different categories of the olive seed oil are formed according to the concentration of the heavy metals (Table 5).

From the results shown in Fig. 2, the first three-component shows a clear separation of 15 variables of the olives obtained according to the heavy metal concentrations. Entire PCA accounted for 68.97% of the total variability and were sufficient for the data as shown in Table 6. The first component representing 42.86% of the total variability comprising of Cu, Pb, Ni, and Cr is said to be the dominant variable. This PCA also correlates more strongly with Cu, therefore based on the correlation of 0.534 Cu tends to be mostly accumulated followed by the other metals such as Pb, Ni, and Cr. The second component with 26.11% total representation increases with only two of the variable Cd and As, thus having a low concentration of As. The third

Table 3 Parameters of the quality of olive oil under study

Mode of harvest of samples	Free acidity (% oleic acid)	Polyphenols (% mg/kg)	Stearic acid (%)	Oleic acid (%)	Palmitic acid (%)
From the plant	0.99	61.52	3.63 \pm 0.45	70.60 \pm 0.56	14.77 \pm 0.64
From the ground	2.74	15.16	2.08 \pm 0.36	72.44 \pm 0.80	14.46 \pm 1.19
From the market	0.92	59.22	2.21 \pm 0.62	72.69 \pm 0.02	13.78 \pm 0.52

Values of mean in triplicate ($p \leq 0.05$)

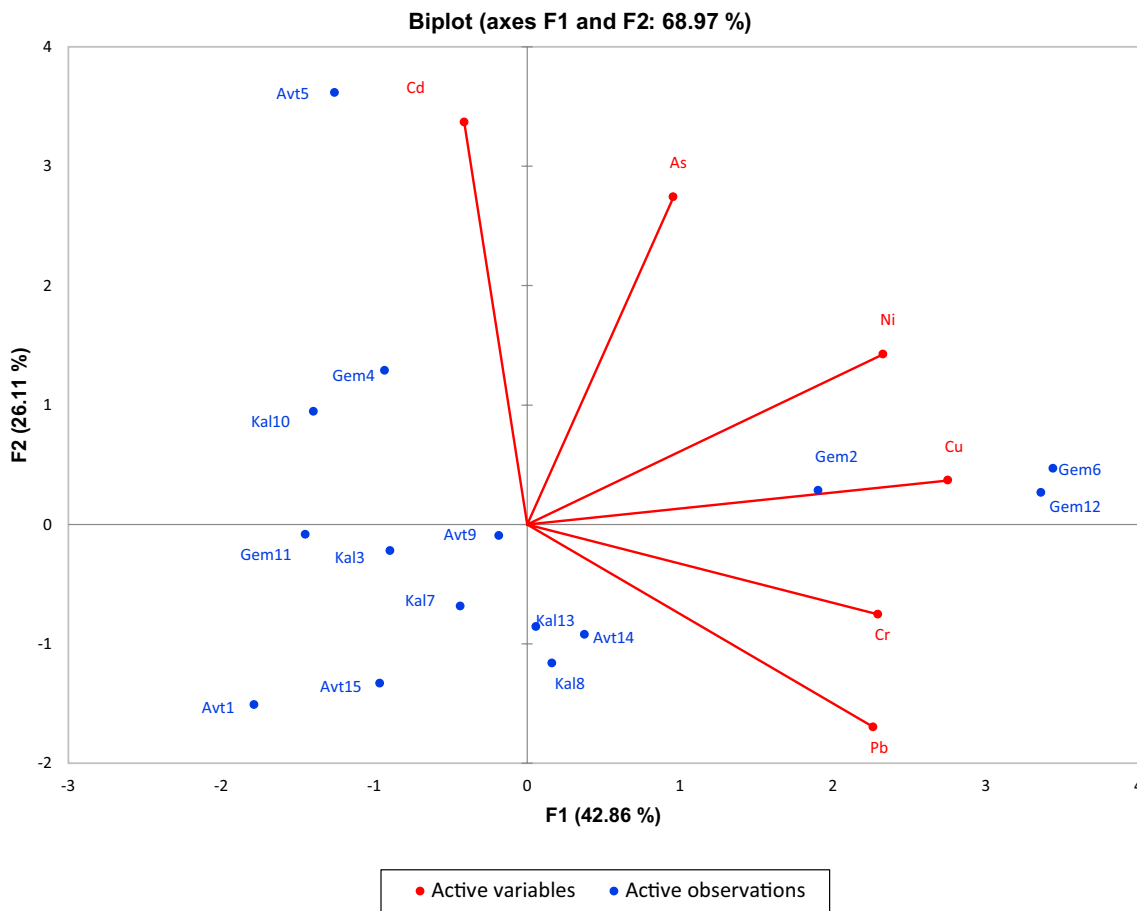


Fig. 2 Biplot of olive fruit seed and oil using Table 4 values

Table 5 Concentration of heavy metals from 15 varieties of olive oil \pm SD ($\mu\text{g/g}$ $n=3$)

Observation	class	Cu	Cd	Pb	Cr	As	Ni
Avt1 ^c	1	1.27 \pm 0.20	0.04 \pm 0.01	0.94 \pm 0.05	0.56 \pm 0.03	0.12 \pm 0.04	0.22 \pm 0.07
Gem2 ^a	2	3.52 \pm 1.35	0.09 \pm 0.04	1.08 \pm 0.40	0.75 \pm 0.01	0.36 \pm 0.04	0.74 \pm 0.04
Kal3 ^a	1	1.95 \pm 0.31	0.05 \pm 0.01	0.79 \pm 0.20	0.52 \pm 0.08	0.45 \pm 0.09	0.44 \pm 0.06
Gem4 ^c	3	1.32 \pm 0.17	0.09 \pm 0.02	0.89 \pm 0.33	0.33 \pm 0.06	0.59 \pm 0.04	0.62 \pm 0.02
Avt5 ^c	3	1.02 \pm 0.30	0.04 \pm 0.03	0.24 \pm 0.12	0.58 \pm 0.08	0.74 \pm 0.02	0.44 \pm 0.02
Gem6 ^a	2	3.81 \pm 1.38	0.09 \pm 0.02	1.20 \pm 0.17	0.82 \pm 0.05	0.75 \pm 0.02	0.79 \pm 0.02
Kal7 ^b	1	1.42 \pm 0.30	0.04 \pm 0.03	1.48 \pm 0.84	0.56 \pm 0.05	0.54 \pm 0.04	0.44 \pm 0.09
Kal8 ^c	1	1.25 \pm 0.40	0.03 \pm 0.02	1.07 \pm 0.63	0.72 \pm 0.02	0.42 \pm 0.08	0.54 \pm 0.02
Avt9 ^b	3	1.51 \pm .039	0.03 \pm 0.01	1.08 \pm 0.28	0.55 \pm 0.04	0.56 \pm 0.05	0.62 \pm 0.02
Kal10 ^a	3	1.71 \pm 0.35	0.04 \pm 0.02	0.15 \pm 0.02	0.41 \pm 0.07	0.71 \pm 0.01	0.58 \pm 0.04
Gem11 ^b	1	1.48 \pm 0.34	0.04 \pm 0.02	0.91 \pm 0.02	0.33 \pm 0.05	0.55 \pm 0.01	0.44 \pm 0.03
Gem12 ^a	2	3.67 \pm 1.12	0.03 \pm 0.02	1.38 \pm 0.04	0.87 \pm 0.05	0.39 \pm 0.03	0.52 \pm 0.05
Kal13 ^c	4	1.24 \pm 0.20	0.03 \pm 0.01	0.79 \pm 0.04	0.79 \pm 0.01	0.09 \pm 0.02	0.59 \pm 0.01
Avt14 ^b	4	1.64 \pm 0.46	0.03 \pm 0.01	1.08 \pm 0.11	0.95 \pm 0.06	0.43 \pm 0.01	0.51 \pm 0.02
Avt15 ^c	1	1.14 \pm 0.19	0.02 \pm 0.01	1.25 \pm 0.41	0.45 \pm 0.05	0.03 \pm 0.01	0.66 \pm 0.04
WHO thresh-hold limits		0.1	0.05	0.1	0.1	0.1	0.2

^aSamples harvested from the ground

^bSamples harvested directly from the olive plantation

^cSample from the supermarkets

Table 6 Eigenvector and Eigenvalues on the correlation matrixes of heavy metals uptake in 15 varieties of olive fruit and oil

	F1	F2	F3	F4	F5	F6
Cu	0.556	0.074	0.166	0.280	-0.293	-0.702
Cd	-0.083	0.681	-0.384	-0.377	0.322	-0.369
Pb	0.458	-0.343	0.277	-0.397	0.661	-0.043
Cr	0.464	-0.152	-0.506	-0.486	-0.465	0.232
As	0.193	0.554	0.636	-0.249	-0.239	0.363
Ni	0.471	0.288	-0.296	0.569	0.317	0.428
Eigenvalue	2.572	1.567	0.857	0.474	0.312	0.218
Variability (%)	42.861	26.110	14.292	7.903	5.201	3.633
Cumulative %	42.861	68.971	83.262	91.165	96.367	100.000

component records a higher concentration of both As and Ni, which also accounts for 13.9% of the total PCA. The other principal component records a lower percentage of the total variability.

AHCA analysis is a multivariate analysis that is used to group and analyze all clusters of similar characteristics and it also builds a hierarchy of clusters. This AHCA was used to quantitatively test the class separation (Ansari et al. 2009;

Fangkum et al. 2011) and also to study the result of the analysis of heavy metals from the different varieties of olive seed and oil.

The dendrogram from Fig. 3 is categorized into four main clusters, the first cluster, which is (C1) comprised of six varieties, Avt1, Kal3, Kal7, Kal8, Gem11, and Avt14. This shows low accumulation of all the six heavy metals as is in the other varieties. The second cluster (C2) with 28%

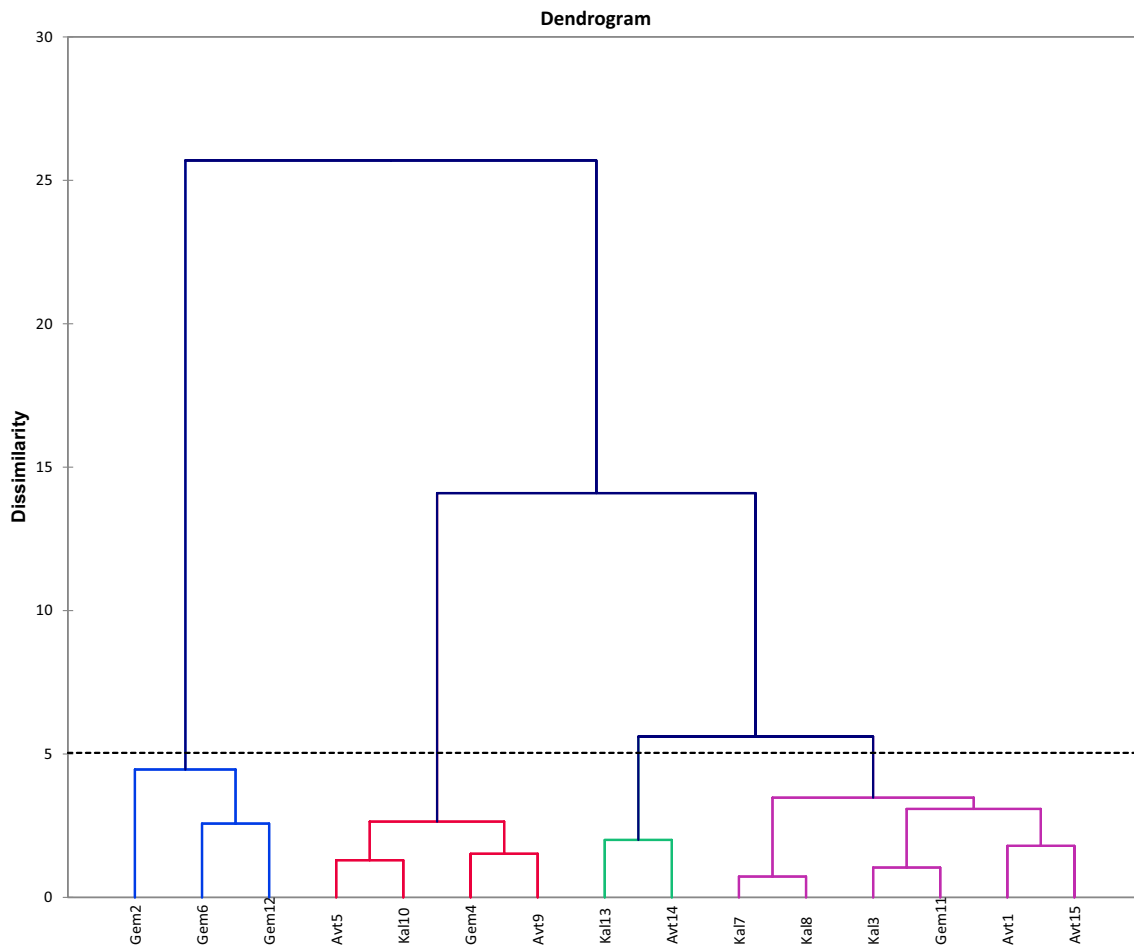


Fig. 3 Dendrogram of agglomerative hierarchical cluster analysis of 15 varieties of olive fruit seed and oil

Table 7 Metal concentration of edible oils from other researchers

Metals	Concentration	Extraction method	Quantification instrument	Oil type	References
(mg/kg)					
Cu	0.570–4.504	Wet digestion, emulsion breaking and ultrasonic extraction	ICP-OES	Sunflower, hazelnut, canola, corn, olive	Dilek et al. (2013)
Cd	0.022–0.058				
Cr	0.126–7.106				
Pb	0.099–0.134				
Ni	0.908–2.182				
(µg/g)					
Cu	0.248–0.276	Microwave digestion	AAS-GF	Olive oil	Fangkum et al. (2011)
Cd	2.39–2.79				
Pb	0.009–0.016				
As	0.008–0.015				
Ni	0.046–0.061				
(µg/g)					
Cu	0.71	Microwave digestion	ASS	Olive oil	Durali et al. (2009)
Cd	0.15				
Pb	0.03				
(ng/g)					
Cr	17–110	Microwave digestion	ICP-MS		Llorent-Martínez et al. (2011a, b)
Cu	15.5				
Pb	5				

ICP-OES inductively coupled plasma optical emission spectrometry, *GF-AAS* graphite furnace atomic absorption spectrometry, *ICP-MS* inductively coupled plasma mass spectrometry

dissimilarity contains Gem2, Gem6, and Gem12 and this shows a high accumulation in all the six heavy metals most, especially recording a high-level uptake of Cu in all the three varieties of the cluster ranging from 3.52 to 3.81 µg/g. The varieties in the third cluster (C3) Gem4, Avt5, Avt9, and Kal10 show a high concentration of As and Ni ranging from 0.6 to 0.9 µg/g with 14% dissimilarity as compared to other varieties in the cluster. Kal13 and Avt 14 have Cr level higher than the other varieties ranging from 0.8 to 1.0 µg/g. These results obtained suggest that the varieties of olive oil belonging to cluster 1 (C1) have lower concentration of all the heavy metals, and thus are said to be suitable for the consumption and production, while the varieties in C2 contain a higher amount of all the heavy metals, because of its harvesting method (having contact with the soil) and are also recommended for further treatment before consumption.

This method of analysis was a better and flexible method of assessment of heavy metal in different varieties of olive oils for suitable consumption, as it is an essential dietary oil in the Mediterranean, which helps in lowering the rates of inflammation, cardiovascular diseases, and also some particular types of cancer in humans. (Karima et al. 2012; Meira et al. 2011).

Evaluation of trace metals in olive oils

The use of multivariate statistical analysis in this study shows how the method of harvesting olives strongly affects the quality of the oils produced. The oils that were produced from the olives harvested directly from the ground were found to be of poor quality compared to that collected from the tree and this is because olives are a source of the fermentation process that can further lead to higher values of free acidity and very low values of phenolic compound. However, considering the contact of olive with the moist soil, the oils which will be produced will surely be marked by the negative attribute of the mold and the ground. The uptake of metals by plants from the soil also depends on plant species; this was proven in this research. The results of trace metals in the varieties of olives grown in the same environment are shown in Table 5. Most of these samples were found to have a high concentration of Cu and Pb, but mostly records a higher concentration of Cu in a sample of olives harvested from the ground. Though much less than the concentration reported by Dilek et al. (2013) with 0.570–4.504 mg/kg for sunflower, hazelnut, corn, and oil, but higher than the concentration of edible oils of olive oil with range of 0.248–0.276 µg/g, the results are attributed to the contact with the soil that could be contaminated.

The high level of Pb in all varieties could be due to wind direction blowing from the traffic around the plantation. Cd records a deficient concentration that is below the Commission Regulation (EU). WHO standard limits mention a range of 0.009–0.016 µg/g, while Durali et al. (2009) forwards this range as 0.15 µg/g (Table 7); while the concentration of other metals such as Cr, As and Ni is due to high affinity for metals existing in agricultural soil.

As range of 0.03–0.75 µg/g of arsenic in all varieties, it is found to be higher than the concentration of As in olive oil reported by Durali et al. (2009), but all below the CODEX STAN 193-1995 of 0.1 mg/kg for As, Pb, and Cd and this permissible limit is also applicable for standard regulation of maximum levels for mercury, lead, and cadmium from Commission Regulations No 1881/2006 and No 629/2008. These conditions depend on flora or the lack of awareness of farmers, about the dangers of the harmful circumstances caused by the use of polluted wastewater, fertilizers, and high application of pesticides. Because of the biological pattern of cultivation, trace quantities of some metals are present in the olives and oil extracted from them. However, such contagion may be kept to the lowest level using appropriate conditions of pre-cultivation; the soil should be investigated and certified, the water used should not be contaminated, and the seeds used for the cultivation should be certified for high tolerance to trace metals.

Conclusions

Six heavy metals (Cu, Cr, Cd, As, Pb, and Ni) in different varieties of olive oil collected from Gemikonagi, Kalavac, and Aytepe/Kelebrunu region of North Cyprus were determined using inductive coupled plasma mass spectrometry following microwave digestion. The results conclude the reliability of microwave digestion of olive oil in the determination of trace metals. The results also conclude the significant differences in some elements' concentrations in different varieties of olive oils. The multivariate analysis using PCA and AHCA shows that all the types of oils contain Cu concentrations. Some contains Pb, Cr, and Ni in which according to the international requirement by WHO, the approved levels of metals in oils are: 0.1 µg/g (Cu, As, Cr, Pb), 0.2 µg/g (Ni), and 0.05 µg/g (Cd) (Zhu et al. 2011). This result poses a threat to the quality of oil and human health as well. Other varieties such as Avt1, Gem2, Kal7, Kal8, Gem11, and Avt14 show a lower concentration of all the heavy metals, thereby declared suitable for consumption.

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