ORIGINAL SCIENTIFIC PAPER

Adulteration of Oblica Virgin Olive Oil with Edible Sunflower and Refined Olive Pomace Oil

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Summary

Adulteration of virgin olive oils is a common problem on the market which is battled against with different authenticity indicators. This work investigated the efficiency of some legally prescribed indicators (fatty acid composition, trans fatty acids, total wax content and K values) in the determination of adulteration of extra virgin olive oil from variety Oblica with addition up to 20 % of edible sunflower and refined pomace oil, respectively. Fatty acid composition enabled the identification of addition of 20 % of sunflower oil, while trans fatty acids, the content addition of 20 % of pomace oil. Based on results for K_{270} it was possible to detect addition of 10 % of sunflower or the same percentage of pomace oil. The biggest potential for the determination of adulteration with smaller amounts of refined oils (between 1 and 10 %) in extra virgin olive oil from variety Oblica was determined for ΔK indicator.

Key words: Virgin olive oil, adulteration, fatty acid composition, waxes, K values

Sažetak

Patvorenje djevičanskih maslinovih ulja prilikom stavljanja na tržište česti je problem koji se nastoji suzbiti provjerom niza pokazatelja autentičnosti. U radu je provjerena efikasnost nekoliko zakonski propisanih pokazatelja (sastav masnih kiselina, udio trans masnih kiselina, udio voskova i K-brojevi) u utvrđivanju patvorenja ekstra djevičanskog maslinovog ulja sorte Oblica dodatkom do 20 % jestivog suncokretovog odnosno rafiniranog ulja komine maslina. Sastav masnih kiselina omogućio je utvrđivanje dodatka od 20 % suncokretovog, a trans masne kiseline dodatka od 20 % ulja komine maslina. Na temelju K_{270} bilo je moguće registrirati dodatak od 10 % suncokretovog odnosno ulja komine maslina. Najveći potencijal u otkrivanju dodatka malih količina rafiniranih ulja (između 1 i 10 %) u ekstra djevičanskom maslinovom ulju sorte Oblica utvrđen je za pokazatelj ΔK.

Ključne riječi: djevičansko maslinovo ulje, patvorenje, sastav masnih kiselina, voskovi, K-brojevi

Introduction

Fraud in food sector has represented a problem for ages. Wherever there is a food with a higher value, high volume sales or food which represents a premium at the market, someone may be tempted to profit from illegal activity. Episodes of fraud in food products, e.g. in wine, olive oil and dairy products, are highly damaging to the economy and reputation of the country concerned (Jee, 2002). It takes time to regain the trust of consumers.

Virgin olive oil has a special place at the vegetable oil market because of its pleasant taste and growing awareness of its health benefits. It reaches high prices also because of production costs and limited production volume. There are many reports about virgin olive oil adulteration (Watkins, 2011; Poulli et al., 2006; Firestone et al., 1985) mostly with cheap oils like edible vegetable and pomace oils. IOOC (International Olive Oil Council) and Codex Alimentarius Commission have produced standards for each type of olive oil and certain blends of these products (Regulation, 2009; Codex, 2003; European Commission, 1991). Standards includes determination of composition of olive oil glyceride and nonglyceride fraction in order to determine either the addition of lower grade olive oil (refined or pomace olive oil) or other vegetable oil. Despite of present standards, many analysts still make effort to find more reliable and cheaper method to detect described fraud (Maggio et.al, 2010; Ozturk et al., 2010; Angerosa et al. 1997).

In this work several methods were applied in order to become familiar with adulteration of virgin olive oil from Croatian cultivar Oblica. Oblica is a dominant olive variety in the area of Dalmatia and makes about 75 % of all olive trees in Croatia (Zadro & Perica, 2007; Škarica et al., 1996). Fatty acid composition is unique for each oil crop and represents a good base for the detection of adulteration. However, today there are some oil crops with a modified fatty acid composition which are very similar to olive oils. That is why it is necessary to combine this method with others, for instance, with a method for *trans* fatty acids (TFA) determination. TFAs are formed at temperature of approximately 190 °C, which is common for a deodorization process. Therefore, this method is convenient for the detection of fraud with refined vegetable oils. Determination of specific absorbances (K values) can also be used to detect refined oils in virgin olive oil. It verifies the formation of conjugated double bonds of linoleic and linolenic acids which occurs as a result of oil refining. Determination of the wax content is suitable to check fraud with olive pomace oil which contains a high amount of waxes (Koprivnjak, 2006).

The aim of this study was to check the efficiency of several authenticity criteria (fatty acid composition, amount of *trans* fatty acids, wax content and K values), defined by legislation, to detect adulteration of extra virgin olive oil from Oblica variety with up to 20 % edible sunflower oil or refined olive pomace oil. We assume that these findings can improve general knowledge of virgin olive oil adulteration at Croatian market.

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Materials and methods

Oil samples

Extra virgin olive oil was obtained from Oblica olive variety by a three phase centrifugal system. Olives were harvested and oil was produced in the middle Dalmatia region. Quality criteria of this oil sample were: mass ratio of free fatty acids 0.15 %; peroxide number 0.22 mmol O_2/kg; K_{232}=2.13; K_{270}=0.12; ΔK=0.00. Total C40 to C46 waxes mass ratio was 157 mg/kg.

Refined olive pomace oil was purchased from Borges, Spain. Its analytical characteristics were K_{232}=3.45; K_{270}=1.42; ΔK=0.32; total C40 to C46 waxes mass ratio 309 mg/kg. Edible sunflower oil, produced in Croatian oil factory Zvijezda Inc., Zagreb, Croatia, had the following analytical values: K_{232}=2.12; K_{270}=2.01; ΔK=0.76; total C40 to C46 waxes mass ratio 263 mg/kg. Model systems were obtained by addition of 1 %, 10 % and 20 % (w/w) of refined sunflower and refined olive pomace oil in extra virgin olive oil (Table 1.). After the addition, whole samples were vigorously mixed and filled in dark glass bottles.

Table 1. *Samples description*

Sample	Description
EVOO	Extra virgin olive oil obtained from Oblica olive variety
S	Edible sunflower oil
S1	99 % Oblica extra virgin olive oil + 1 % sunflower oil
S10	90 % Oblica extra virgin olive oil + 10 % sunflower oil
S20	80 % Oblica extra virgin olive oil + 20 % sunflower oil
P	Refined olive pomace oil
P1	99 % Oblica extra virgin olive oil + 1 % refined olive pomace oil
P10	90 % Oblica extra virgin olive oil + 10 % refined olive pomace oil
P20	80 % Oblica extra virgin olive oil + 20 % refined olive pomace oil

Analytical methods

All methods used in this experiment were validated. Fatty acid composition was determined using gas chromatography following IOOC methods (2001a; 2001c). Prepared methyl esters were injected in gas chromatograph (Varian CP-3800) equipped with a FID detector and a „Restek“, Rtx-2330 capillary column (30 m × 0.25 mm × 0.2 μm). Helium was used as carrier gas at a flow rate of 1.5 mL/min. The temperature of the injector was set at 220 °C and the temperature of the detector also at 220 °C. The temperature of the oven was programmed to increase at 5 °C/min from an initial value of 140 °C to 210 °C,

Table 2. *Fatty acid composition (%) of Oblica extra virgin olive oil (EVOO), sunflower oil (S), refined olive pomace oil (P) and model systems (S1, S10, S20, P1, P10, P20)**

Fatty acid	EVOO	+/- u_e**	S	S1	S10	S20	P	P1	P10	P20	Limits for EVOO***
C14:0	0.01	0.00	0.04	0.01	0.01	0.02	0.01	0.01	0.01	0.01	≤ 0.05
C16:0	11.3	0.09	5.8	11.0	11.0	10.4	9.7	12.1	11.7	10.9	7.5-20.0
C16:1	0.7	0.01	0.1	0.7	0.6	0.6	0.8	0.7	0.7	0.7	0.3-3.5
C17:0	0.0	0.00	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.1	≤ 0.3
C17:1	0.1	0.00	0.0	0.1	0.1	0.1	0.1	0.1	0.1	0.1	≤ 0.3
C18:0	2.2	0.02	3.0	2.2	2.3	2.4	2.8	2.2	2.2	2.4	0.5-5.0
C18:1	71.6	0.50	26.1	71.0	66.7	61.9	73.3	71.1	71.1	72.0	55.0-83.0
C18:2	12.7	0.05	63.2	13.5	17.8	23.0	11.1	12.6	12.5	12.4	3.5 - 21.0
C18:3	0.6	0.01	0.2	0.6	0.5	0.5	0.6	0.6	0.6	0.6	≤ 1.0
C20:0	0.4	0.00	0.2	0.4	0.4	0.4	0.5	0.4	0.4	0.2	≤ 0.6
C20:1	0.3	0.01	0.2	0.3	0.3	0.3	0.4	0.2	0.4	0.0	≤ 0.4
C22:0	0.1	0.00	0.7	0.2	0.2	0.3	0.0	0.1	0.2	0.2	≤ 0.2
C22:1	0.00	0.00	0.00	0.00	0.00	0.00	0.05	0.06	0.00	0.01	-
C24:0	0.1	0.00	0.3	0.1	0.1	0.1	0.1	0.0	0.2	0.1	≤ 0.2
C18:1t	0.00	0.004	0.02	0.01	0.00	0.01	0.20	0.00	0.02	0.03	≤ 0.05
C18:2t + C18:3t	0.02	0.006	0.04	0.04	0.02	0.04	0.19	0.01	0.03	0.07	≤ 0.05

Results are mean values of two analytical determinations; *samples are described in Table 1; ** expanded uncertainty for the level of confidence of 95 % determined with reference sample of extra virgin olive oil; *** according to Regulation (2009)

and kept at the upper temperature for 30 min Parameters of validation are presented in table 2. Samples were analyzed in duplicate.

The wax content was determined following IOOC method (2003). Analysis was performed by gas chromatography using Varian CP-3800, equipped with a Varian 1079 PTV (Programmable Temperature Vaporizing) injector, FID detector and „Varian" VF-1ms (15 m × 0.25 mm × 0.25 μm) column. The initial temperature of the injector was set at 60 °C and then increased up to 320 °C at 40 °C/min rate. The temperature of the detector was 350 °C. The temperature of the oven was programmed to increase at 30 °C/min from an initial value of 80 °C to 120 °C, and then increase to 340 °C at 5 °C/min rate. Hydrogen was used as carrier gas at flow rate 20-35 cm/s. Limit of detection for the used method was 30 mg/kg and limit of quantification was 50 mg/kg. Samples were analyzed in duplicate.

Ultra-violet absorbency (K values) was determined following standard IOOC method (2001b) using VARIAN Cary 50 Scan spectrophotometer. Established parameters of validation, limit of detection for K_{232} and K_{270} were 0.148 and 0.018, respectively, and limit of quantification for K_{232} and K_{270} were 0.8 and 0.021, respectively. Samples were analyzed in triplicate.

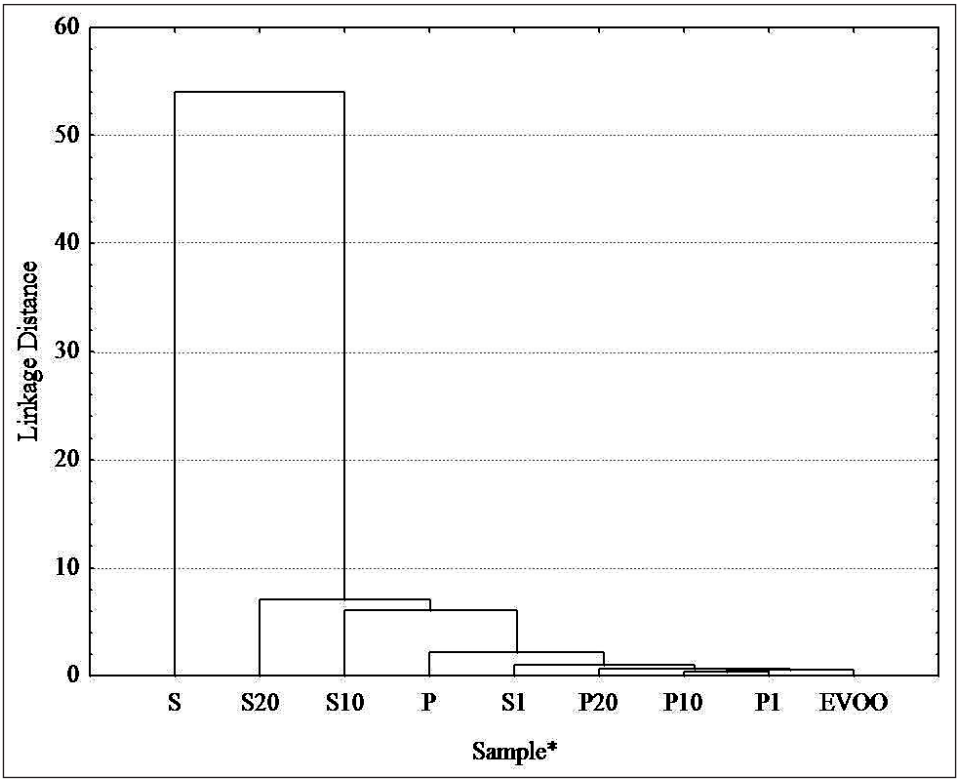

Figure 1. *Differences between the samples based on fatty acid composition obtained by cluster analysis*
**samples are described in Table 1*

Statistical analyses

Cluster analysis was performed to determine differences between the samples based on the fatty acid composition and wax content. Statistical analyses were performed using the software package STATISTICA v. 9 (STATISTICA, 2009).

Results and discussion

Results of previous research on virgin olive oils from variety Oblica (Žanetić et al., 2010; Škevin, 2003) showed that analyzed parameters of quality and authenticity were within the limits defined by national (Regulation, 2009) and international legislation (European Commission, 1991; Codex, 2003). Based on quality criteria, sample of olive oil from Oblica variety used in this research corresponded with extra virgin olive oil category. Also, selected parameters of authenticity, mass yield of total wax, fatty acid composition and amount of *trans* fatty acids (Table 2.) were within the defined limits for this category.

For preparation of adulterated olive oil model systems, two types of oil were chosen: edible sunflower oil and refined olive pomace oil. Price of these oils in world market is at least 50 % lower than of virgin olive oil (Index mundi, 2011), and by adding 20 % of those oils in virgin olive oils illegal profit increases around 10 % above the real value of the product. For that reason, we examined minimum addition of 1, 10 and 20 % of edible sunflower or refined olive pomace oil which could represent an economical interest for adulteration of Oblica variety virgin olive oil.

Sample of sunflower oil had standard fatty acid composition (Table 2.) which was in accordance with national legislation (Regulation, 2009). It means that it had a significantly lower content of oleic acid and significantly higher content of linoleic acid compared with EVOO limits. Addition of 1 % of sunflower oil in EVOO of Oblica changed to a greater extent only the content of linoleic acid but the results were inside the legal frames. Only the addition of 20 % of edible sunflower oil changed the values of linoleic and behenic acid to the levels that could be detected as adulteration.

Refined pomace oil resembled in fatty acid composition to EVOO of Oblica variety. It was confirmed with small linkage distance calculated by cluster analysis (Figure 1). Based on this indicator of authenticity, sample of refined pomace oil was inside the limits of national and international legislation for this type of product (Regulation, 2009; Codex, 2003). For this reason, addition of refined pomace oil to EVOO from variety Oblica did not cause significant changes in fatty acid composition.

Ricci (2011) states that based on the share of *trans* fatty acids, up to 5 % of sunflower oil in virgin olive oil can be detected. Sample of sunflower oil used in this research had relatively low value of *trans* fatty acids considering that it was refined oil. We can assume that it was refined under mild conditions (lower temperature, less concentration of bleaching clay) which have not caused significant *cis-trans* isomerisation of double bonds in the chains of unsaturated fatty acids. For that reason in samples adulterated with sunflower oil no values of *trans* fatty acids above legal limits were determined.

In the case of refined pomace oil, the content of *trans* fatty acids was 10 to 20 times higher than in EVOO from Oblica variety but nevertheless two times under the legal maximum (Regulation, 2009). Theoretical values for *trans* fatty acids in models P1, P10 and P20, which were calculated from analytical data for EVOO and refined olive pomace oil were in the range

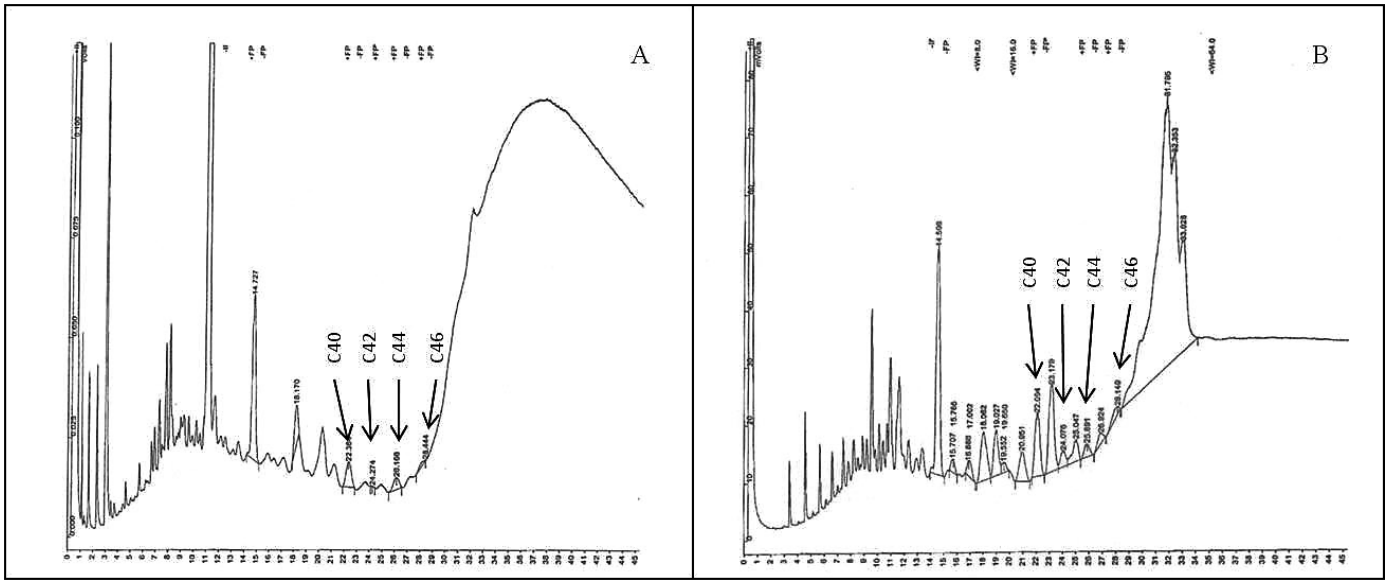

***Figure 2**. Chromatograms of wax composition of Oblica extra virgin olive oil (A) and edible sunflower oil (B)*

between 0.00 and 0.05 (data not shown). That implies a limited ability of adulteration determination of EVOO variety Oblica with olive pomace oil based on the content of *trans* fatty acids. Only analytically determined sum of *trans* isomers of linoleic and linolenic acid of model system P20 (addition of 20 % of pomace oil) reached an upper limit of 0.05 %.

Mass content of waxes in refined sunflower oil is in the range 366-624 mg/kg (Carelli et al., 2002). Since the upper limit for VOO is 250 mg/kg, this indicator can be used for the determination of adulteration with sunflower oil. However, it should be noted that wax content of sunflower differs from the virgin olive oil and olive pomace oil. Dominant waxes in pomace oil have mostly 40, 42, 44 and 46 carbon atoms (Bianchi et al., 1994) while sunflower oil is rich in C36, C37, C40, C41, C46 and C48 waxes (Carelli et al., 2002). The IOOC method for the determination of waxes in olive oils (IOOC, 2003) takes into consideration only waxes between C40 and C46. Therefore, other peeks on GC chromatogram, which represented waxes characteristic for sunflower, were not included in the calculation of total waxes for the sample of sunflower oil (Figure 2).

Consequently, total wax content determined using the above described method in our sample of sunflower oil (263 mg/kg) was lower than what is usual for this type of oil.

Data presented in Table 3 show that sunflower oil had two times higher values of C40 compared to olive oil, while mass content of C42 were much higher for pomace oil than in EVOO from Oblica variety. However, unlike for the total mass content of waxes, national and international market legislations do not prescribe limits for particular waxes. Figure 3 shows parallel analytic and arithmetic (expected) values for total wax content compared with upper limits for VOO. In models with 10 and 20 % of edible sunflower oil, analytical values were higher than arithmetical values. Determined values were in any case lower than the upper limit for VOO prescribed by national and international legislation. It can be concluded that by applying the official method for the total wax content in VOO, it was not possible to establish the presence of 20 % of sunflower oil in EVOO from Oblica variety.

The total wax content for the sample of refined pomace oil used in this study was well above minimum limit for this product category, which is 350 mg/kg. Figure 3 shows that analytically and arithmetically determined values enable the detection of adding of 10 % of refined pomace oil to EVOO of variety Oblica by applying the official method and maximal share of total waxes. In favour of this, there were also large differences determined with cluster analysis for samples EVOO, P10 and P20 (Figure 4.). These results are in agreement with literature which describes a possibility of detection of 6-15 % of pomace oil in virgin olive oil (Ricci, 2011).

Values determined on the base of absorption of solution of oil in UV spectra indicate the presence of conjugated diens (K_{232}) and conjugated triens (K_{270}) of fatty acids. Figure 5 shows that analytically determined values for K_{232} are above those expected (arithmetically determined). Analytically determined value for K_{232} at refined pomace oil was by about 60 % higher than the values determined at sunflower oil and EVOO. Despite this, it was not possible to determine the addition of 20 % of refined pomace oil based on K_{232} values.

Analytical values determined for K_{270} for sunflower oil and pomace oil were 17 and 12 times

***Table 3.** Waxes mass ratio (mg/kg) of Oblica extra virgin olive oil (EVOO), sunflower oil (S), refined olive pomace oil (P) and model systems (S1, S10, S20, P1, P10, P20)**

Carbon number	EVOO	S	S1	S10	S20	P	P1	P10	P20
C40	87.1	154.0	98.8	107.7	121.9	713.6	90.5	147.8	163.5
C42	3.3	36.3	14.9	21.0	25.6	1411.15	31.4	180.5	294.4
C44	48.9	39.1	29.1	38.7	46.2	773.70	29.0	88.6	119.9
C46	17.4	33.5	13.8	9.7	21.8	195.16	13.5	49.2	52.9

Results are mean values of two analytical determinations; *samples are described in Table 1

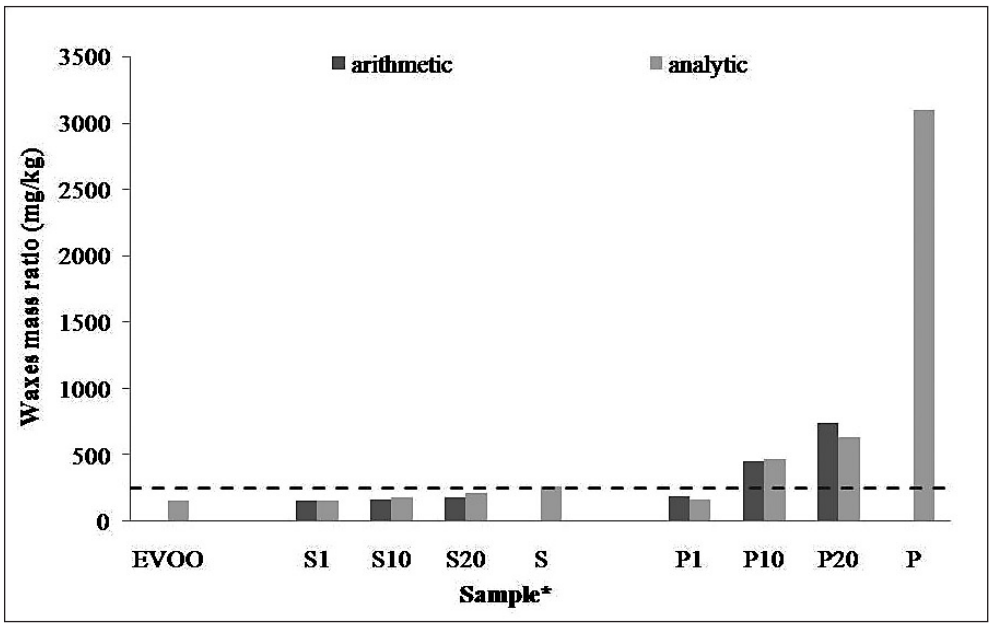

Figure 3. *Comparison of total C40 to C46 waxes mass ratio (mg/kg) determined analytically in model systems S1, S10, S20, P1, P10, P20 and arithmetically from values determined in Oblica extra virgin olive oil (EVOO), sunflower oil (S) or refined olive pomace oil (P). Results are mean values of two analytical determinations. *Samples are described in Table 1. Dashed line represents upper limit for EVOO (Regulation, 2009).*

higher than in EVOO, respectively. Consequently, it was possible to detect adulteration of EVOO with addition of 10 % of both used refined oils.

Nevertheless, the biggest potential for the detection of adulteration of samples of EVOO from variety Oblica with sunflower and refined pomace oil was observed for ΔK values. In this case, analytically determined value reached legally prescribed upper limit even at the sample S1, while values at samples S10, S20, P10 and P20 were well above that limit.

Conclusions

From all stated above, it can be concluded that on the basis of investigated indicators of authenticity (fatty acid composition, *trans* fatty acids, total wax content and K values), it was not possible to detect adulteration of EVOO from variety Oblica with addition of 1 % of sunflower or pomace oil. Fatty acid and *trans* fatty acids gave us a possibility to detect only adulteration with 20 % of sunflower or pomace oil. In order to determine if there was an addition in amount less than 20 %, other indicators of authenticity stated in legislation (Regulation, 2009) have to be examined. In case of adulteration with sunflower oil, based on literature, ΔECN 42 and sterol content are especially useful (possibility of detection up to 1.5 % and 0.7 %, respectively) (Ricci, 2011). Adulteration with refined pomace oil in amounts between 0.5 and 2 % can be detected with data of stigmastadienes content (Ricci, 2011). However, adulteration detection can be done with determination of ΔK which showed a significant potential in revealing the addition of small amounts of refined oils (between 1 and 10 %) in EVOO from variety Oblica. Advantages of this method are its rapidity, simplicity and relatively low price. Therefore, it can be applied in Croatian control laboratories which may contribute to clear picture of olive oil adulteration in Croatia.

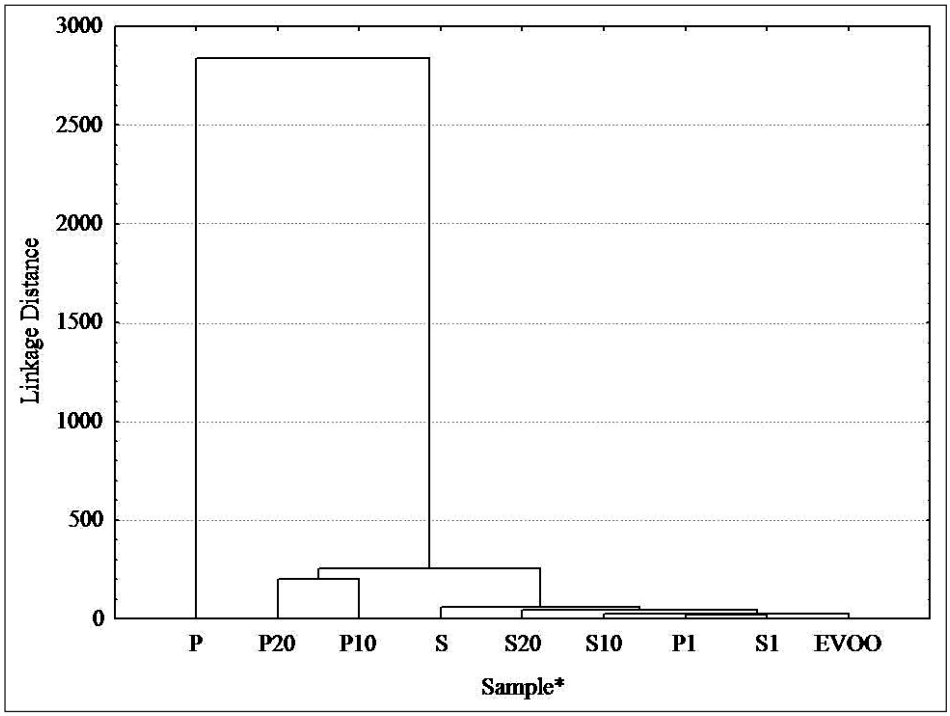

Figure 4. *Differences between the samples based on wax content obtained by cluster analysis *samples are described in Table 1*

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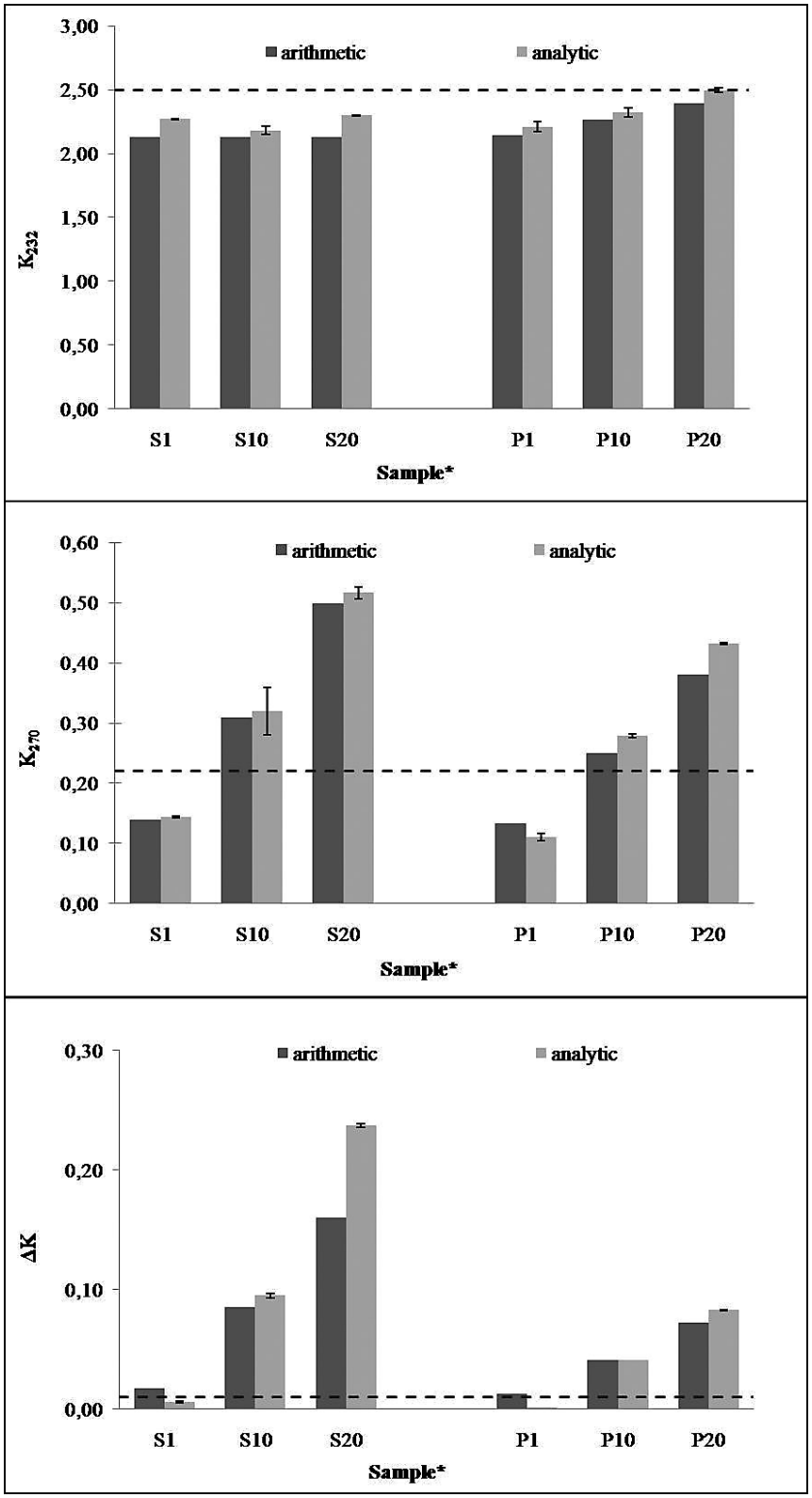

Figure 5. *Comparison of K values determined analytically in model systems (S1, S10, S20, P1, P10, P20) and arithmetically from values determined in Oblica extra virgin olive oil (EVOO), sunflower oil (S) or refined olive pomace oil (P). Results are mean values of three analytical determinations ± SD. *Samples are described in Table 1. Dashed lines represent upper limit for EVOO (Regulation, 2009).*