Studii şi Cercetări Ştiinţifice
Chimie şi Inginerie Chimică, Biotehnologii, Industrie Alimentară

2018, **19** (1), pp. 033 – 044

Scientific Study & Research
Chemistry & Chemical Engineering, Biotechnology, Food Industry

ISSN 1582-540X

ORIGINAL RESEARCH PAPER

CONSIDERATIONS ABOUT THE USE OF LOVAGE LEAVES TO IMPROVE THE QUALITY OF EDIBLE VEGETABLE OILS AND OIL BLENDS

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Received: January, 24, 2018
Accepted: March, 05, 2018

Abstract: We studied four edible vegetable oils and nine oil blends based on refined sunflower oil, in order to improve the quality characteristics of sunflower oil. The oils used for blends were linseed oil, grapeseed oil, and coconut oil. The physico-chemical properties demonstrated the superior features for oil blends, like lower acidity (measured by acid value) and higher stability to autoxidation (measured by peroxide value and refractive index). The best combination for sunflower oil was with coconut oil (lower acidity, higher stability to autoxidation). For a supplementary improvement of properties, especially for the preservation of oils and oil blends, we tested the lovage (*Levisticum officinale*) extract as additive. The obtained additivated mixtures demonstrated better quality characteristics, which recommend them for the human consumption.

Keywords: *acid value, coconut oil, grapeseed oil, Levisticum officinale, linseed oil, peroxide value, sunflower oil*

INTRODUCTION

Significant amount of oils and fats (about 117 million tons) is produced worldwide every year. They are recognized 17 commodity oils, of which only 4 are of animal origin, 13 of them being from vegetable sources. Between the vegetable oils only castor oil is used solely for industrial and medicinal purposes, approximately 80 % of the total production of oils and fats being used for food purposes (edible oils) [1]. The vegetable oils are mainly used for cooking and frying, as well as in food formulations, representing one of the main ingredients in many food products, like ice cream, sausages, margarine, etc. [2]. Edible oils are vital constituents of human daily diet, providing energy and essential fatty acids, which function as a carrier of fat soluble vitamins. Their important role in the human diet is due to the nutritional and sensory properties [3].

The sunflower oil is the fourth largest oil source in the world, after soybean, palm, and canola (edible rapeseed). It is one of the most popular vegetable oils, in some countries being preferred to soybean, cottonseed and palm oils. Despite of its qualities, the sunflower oil lacks oxidative stability in frying applications [1]. The oxidation of oils leads to the development of off-flavor compounds and undesirable chemical changes of foods. These changes also mean the decrease of nutritional value of foods and potential food safety problems [2].

The coconut oil is a lauric oil, characterized by its high level of shorter and medium fatty acid chain lengths (C6–C14), which reach 80 % in coconut oil, while in the non-lauric vegetable oils they are below 2 %. The major fatty acids are lauric (12:0) and myristic (14:0), at about 48 % and 18 % respectively, while no other fatty acid is present at more than about 8 %. The heavy preponderance of lauric acid gives coconut oil the sharp melting properties and a low melting point. Because of its low unsaturation, the coconut oil is also very stable to oxidation and resistant to rancidification. The outstanding properties of coconuts oil determine its use in the edible field [1]. Although the coconut oil is very extensively used for food products, last studies have advised about the risks of cardiovascular disease. So, only a limited consumption is recommended [4].

The linseed oil production has hardly changed over the past 30 years. The linseed oil is mainly used as industrial oil based on its high unsaturation, but it is increasingly consumed as food oil. It is seed oil rich in the linoleic acid [1].

More studies demonstrated that, because of their specific chemical and physical properties, most vegetable oils have limited technological application in their original forms. Thus, for enhancing their commercial application, vegetable oils are often modified using four different methods, *i.e.* hydrogenation, interesterification, fractionation and blending [5]. The blending of vegetable oils with different properties is one of the simplest methods to create new specific products with desired textural and oxidative properties. The vegetable oil blending has been a common acceptable practice in many countries. For example, the blending of sunflower oil with canola oil or palm oil, soybean oil with hydrogenated soybean oil or corn oil with high-oleic sunflower oil have been widely used. Furthermore, it has recently been permissible to blend common edible oil with unconventional oils such as rice bran oil to achieve different aims to reduce the cost and meet industry demands [5]. The blending of edible oils is not only a simple physical process of mixture. In the blends, the interesterification of edible oils and fats, involving the redistribution of fatty acids among the triacylglycerols within the same molecule or among different molecules, is considered an alternative to produce commercial fats free of *trans* fatty acids and to improve the physical properties of fats and oils [6].

The additivation is another way to improve the quality of edible oils. It is a worldwide trend to avoid, or at least minimize, the use of artificial food additives because of the grown concern over the potential hazards of synthetic antioxidants. Thus, the interest in the use of naturally occurring antioxidants is renewed. The advantages of natural antioxidants include their relatively unquestioned safety, the higher concentrations allowed, their worldwide acceptance, and their lower volatility in heated foods [7].

The lovage (*Levisticum officinale* L.) is a perennial plant which belongs to *Umbelliferae* family, cultivated in temperate climate zone. All the lovage parts (seeds, leaves, stems and roots) are strongly aromatic, with a characteristic earthy, celery-like flavor and smell, containing compounds which can act as natural antioxidants [8]. The monoterpenes (β-phellandrene, α-terpinyl acetate in high concentration, α- and β-pinene, *trans* β-ocimene, terpinene, α-terpineol) were identified in the composition of essential oil of lovage leaves [9]. The addition of the lovage leaves and stems extracts to unrefined rapeseed oil can prevent the oxidation and decrease the acidity [8].

We used the lovage leaves for preserving and improving the properties of vegetable oils and mixtures of oils. We propose the use of fresh leaves and the trituration of oil – lovage mixtures, without a preliminary extraction of essential oils from leaves. In a first stage of the study, we characterized 4 edible vegetable oils. To improve the properties of sunflower oil, 9 blends of these oils were prepared and characterized. The lovage leaves were added in oils and oil blends and the resulted edible flagrant oils were characterized.

MATERIALS AND METHODS

Materials

The vegetable oils (*i.e.* sunflower oil, linseed oil, grapeseed oil and coconut oil) and the lovage were purchased from the Romanian local market.

The high purity reagents (ethanol, chloroform, potassium hydroxide, glacial acetic acid, potassium iodide, sodium thiosulfate, phenolphthalein, starch) were obtained commercially from Sigma-Aldrich and used as received, without further purification.

Methods

The oils and their mixtures were analyzed and characterized on the basis of IUPAC [10], American Oil Chemists' Society (AOCS) [11] and national/international standards.

Organoleptic analysis (appearance, odor, taste) was carried out according to Romanian standard STAS 12/1-72 [12].

Determination of relative density
The relative density was determined according to IUPAC method 2.101, with the appropriate conversion factor [10].

Determination of acidity
The oils acidity was determined on the basis of neutralization with a KOH solution, according to ISO 660:2009 [13], in concordance with AOCS method Ca 5a–40 (Free Fatty Acids) [11].

Determination of peroxide value
Peroxide value (PV) was determined by the iodometric assay according to IUPAC standard method 2.501 [10], in concordance with AOCS Cd 8b-90 (03) [11] or ISO 3960:2001 [14].

Rheological measurement
The viscosity was determined according to SR EN ISO 3104 [15], using a capillary viscometer Ubbelohde with suspended level from Schott AG, Mainz, Germany (type 501 21, K = 0.4989).

Refractive index
The refractive index was determined according to Romanian standard STAS 145-67 [16], in concordance with ISO 6320:2000 [17] and AOCS Cc 7-25 (02) [11], using an Abble-Zeiss refractometer from Atago Co., Japan (model NAR-2T).

The additivation with lovage
The lovage leaves were washed with water and dried. 1 g of lovage leaves, cut in small pieces, was putted in a mortar. 10 mL of vegetable oil/blend of oils are added over the lovage leaves. The mixture was triturated intermittently and left overnight.

Statistical analysis
All results were expressed as mean ± standard deviation (SD) of triplicate determinations. The evaluations were performed by one way ANOVA and statistical significance by Student's t-test was carried to test any significant difference among means. The correlation values were assessed using Pearson correlation. Differences at $p < 0.05$ were considered significant.

RESULTS AND DISCUSSION

Our study involves three stages: (i) the characterization of 4 vegetable oils, commercially available; (ii) the preparation and characterization of vegetable oil blends; (iii) the preparation and characterization of vegetable oils/oil blends additivated with lovage.
The quality standards applied to the vegetable oils in a state for human consumption are described in "Standard for named vegetable oils, CODEX STAN 210-1999" [18]. The standards are applicable to coconut oil (derived from the kernel of the coconut, *Cocos nucifera* L.), grapeseed oil (derived from the seeds of the grape, *Vitis vinifera* L.), and

sunflower seed oil (sunflower oil, derived from sunflower seeds, *i.e.* seeds of *Helianthus annuus* L.), as they are defined by CODEX STAN 210-1999 [18]. "CODEX STAN 19-1981 for edible fats and oils not covered by individual standards" [19] is applicable to linseed oil (derived from the seeds of the flax plant, *Linum usitatissimum* L.).

Characterization of vegetable oils

The organoleptic analysis (appearance, odor, taste) was performed for the vegetable oils (Table 1). The organoleptic characteristics matched the CODEX STAN 210-1999 [18] and CODEX STAN 19-1981 [19]. Thus, the odor and taste of each product are characteristic. The oils are also free from foreign and rancid odor and taste.
We also tested other quality characteristics, like density, viscosity, acid value, peroxide value, and refractive index (Table 1).

Table 1. *The quality characteristics for vegetable oils*

Characteristics	Sunflower oil	Linseed oil	Grapeseed oil	Coconut oil
Appearance	transparent	transparent	transparent	transparent (liquid)
Odor	odorless	odorless	odorless	odorless
Taste	tasteless	tasteless	tasteless	coconut, characteristic
Relative density [x °C/water at 20 °C]	0.9180 (x = 25 °C)	0.9292 (x = 25 °C)	0.9194 (x = 25 °C)	0.9210 (x = 40 °C)
Viscosity [$mm \cdot s^{-2}$]	30.312	20.6818	26.4417	27.4385
Acid value: [mg KOH/g]	0.48	0.80	0.48	0.48
[% free oleic acid]	0.24	0.40	0.24	0.24
Peroxide value [meq O_2/kg]	8	8.5	9	8.8
Refractive index (n^{20})	1.4732	1.4806	1.4741	1.4781

Density
The density of vegetable oils depends on their chemical composition. It was demonstrated that the liquid density of vegetable oils can be estimated by using mixture properties corresponding to the fatty acid composition and a correction for the triglyceride form by the modified Rackett equation [20, 21]. For the edible vegetable oils, the CODEX STAN 210-1999 [18] and CODEX STAN 19-1981 [19] do not indicate anything about the value of density. We found the highest value for linseed oil and the lowest value for sunflower oil.

Viscosity
The rheological properties of vegetable oils are also important for their characterization, because they depend on the chemical composition. In vegetable oils, viscosity is a function of molecules dimension and orientation. The viscosity increases with chain length of triglyceride fatty acids and decreases with unsaturation, so it increases with hydrogenation [22].
The decrease in the oil viscosity was correlated in other studies with an increased portion of 18:2 fatty acids and a decreased portion of 18:1 fatty acids, so oils with more double bonds have lower viscosity. The explanation involves the differences in structure

of fatty acids. Thus, the fatty acids with more double bonds do not have a rigid and fixed structure, being loosely packed and more fluid-like. The flow behavior and its variation with temperature are very important in the utilization of edible vegetable oils in frying [23].

The higher value of viscosity was determined for sunflower oil (30.312 $mm\cdot s^{-2}$), meaning the lower concentration of polyunsaturated fatty acids. On the contrary, to the linseed oil, with lowest viscosity (20.6818 $mm\cdot s^{-2}$), can be assigned the highest quantity of polyunsaturated fatty acids.

A correlation between the density and viscosity of studied oils is obvious from Table 1. Thus, a lower density means a higher viscosity. For the coconut oil, the density and viscosity were determined in liquid state, at 40 °C.

Acid value (AV) measures the free fatty acids content (FFAs) in vegetable oils. AV is considered to be one of main parameters to reflect their quality, degree of refining, and also the quality change during storage. AV is expressed as the amount of KOH (in milligrams) necessary to neutralize free fatty acids contained in 1 g of oil, according to AOCS official method (the titration analysis). The content of FFAs can be also expressed as percent of oleic acid, dividing AV by 1.99 [11, 13, 24, 25]. The FFAs are compounds naturally present in low amounts in vegetable oils. They are a product of hydrolytic degradation of triglycerides, thus their amount is an important quality index for oils. In the deterioration of the oil, supplementary quantities of FFAs are forming, so the AV values increase. The threshold values of AV in vegetable oils are provided by regulations, and vary according to the oil type and the commercial class [24]. According to CODEX STAN 210-1999 [18] and CODEX STAN 19-1981 [19], the AV for refined oils must be up to 0.6 mg KOH/g oil. Except to linseed oil, the AV values for studied oils match with standards. For a proper utilization, a decrease of AV for linseed oil is required. The higher value of AV can be correlated with the lowest viscosity, thus the highest quantity of polyunsaturated fatty acids.

Peroxide value (PV)

The oxidation of lipid consists in a set of autocatalytic reactions. The primary oxidation compounds are the hydroperoxides which are decomposed, giving rise to a variety of secondary oxidation products. The peroxide value is used for the evaluation of the oxidation status in vegetable oils [26]. In CODEX STAN 210-1999 [18] and CODEX STAN 19-1981 [19] were established values up to 10 milliequivalents of active oxygen/kg oil for refined oils. The lowest value of PV, thus lowest value for hydroperoxides was determined for sunflower oil (8 meq O_2/kg), while the highest value of PV, meaning the highest concentration of hydroperoxides, was determined for grapeseed oil (9 meq O_2/kg). Intermediate values were noticed for linseed oil and coconut oil (Table 1).

Refractive index

The refractive index is not established as a quality characteristic in CODEX STAN 210-1999 [18] and CODEX STAN 19-1981 [19]. But, the refractive index of vegetable oils can be correlated with the acid value and peroxide value, therefore with the chemical composition of the oils [24]. The refractive index is considered as an objective method for evaluation of rancidity in edible oils, which can be also correlated with peroxide value. Thus, the refractive indices of oils have been reported to increase on autoxidation. However, the patterns of changes in refractive indices and peroxide values are different. Peroxide values increase at a steady rate over the primary induction period and, after the onset of rancidity, the rate was slightly higher. The changes in refractive indices are not significant during the induction periods, but increase in the time of the detection of perceptible rancid odor. Some correlations between the pattern of peroxide development and changes in refractive indices of the autoxidized oils can be highlighted. It was demonstrated that refractive index changes according to the three known stages of autoxidation of fats and oils, *i.e.* the primary induction period, the secondary stage of relatively more peroxide formation, and the tertiary stage of peroxide decomposition. The increase in refractive index with autoxidation can be due to conjugation, which precedes hydroperoxide formation in the secondary stage, and polymerization of partially oxidized fats, in the tertiary state of autoxidation. Consequently, both conjugation and polymerization are reported to result in increased refractive indices of oils and fats [24, 27].

Blends of vegetable oils

The use of unmixed vegetable oils, which may have low physical, chemical and nutritional properties or poor oxidative stability, can be unfavorable for consumers. The consumption of vegetable oil blends can be a simple way to take advantage of the different characteristic properties of each oil [5]. We mixed the vegetable oils in order to improve the characteristics of sunflower oil, the most common edible oil in Romania. Therefore, we blended sunflower oil (SO) with linseed oil (LO), grapeseed oil (GO), and coconut oil (CO), using the following ratios (v/v): 90:10 (1); 80:20 (2); 70:30 (3).
The physico-chemical characteristics for blends based on sunflower oil are shown in Table 2.

Table 2. *The physico-chemical characteristics for blends of sunflower oil with linseed, grapeseed, and coconut oils*

	Density [g·cm^{-3}]	Viscosity [mm·s^{-2}]	Acid value [mg KOH/g oil]	Peroxide value [meq O_2/kg oil]	Refractive index (n_{20})
Sunflower oil – linseed oil (SO-LO)					
SO-LO 1	0.9197	29.564	0.48	8.1	1.4734
SO-LO 2	0.9219	25.916	0.32	8.2	1.4738
SO-LO 3	0.9226	21.2184	0.24	8.7	1.4744
Sunflower oil – grapeseed oil (SO-GO)					
SO-GO 1	0.9189	29.4351	0.16	8.3	1.4730
SO-GO 2	0.9190	28.628	0.19	8.6	1.4733
SO-GO 3	0.9192	24.32	0.24	8.8	1.4740
Sunflower oil – coconut oil (SO-CO)					
SO-CO 1	0.9182	29.3016	0.16	8.2	1.4741
SO-CO 2	0.9184	28.4373	0.19	8.5	1.4752
SO-CO 3	0.9186	27.6416	0.24	8.6	1.4759

A correlation between the characteristics of individual vegetable oils and their blends is shown in Figure 1 and Figure 2.

a.
b.
c.

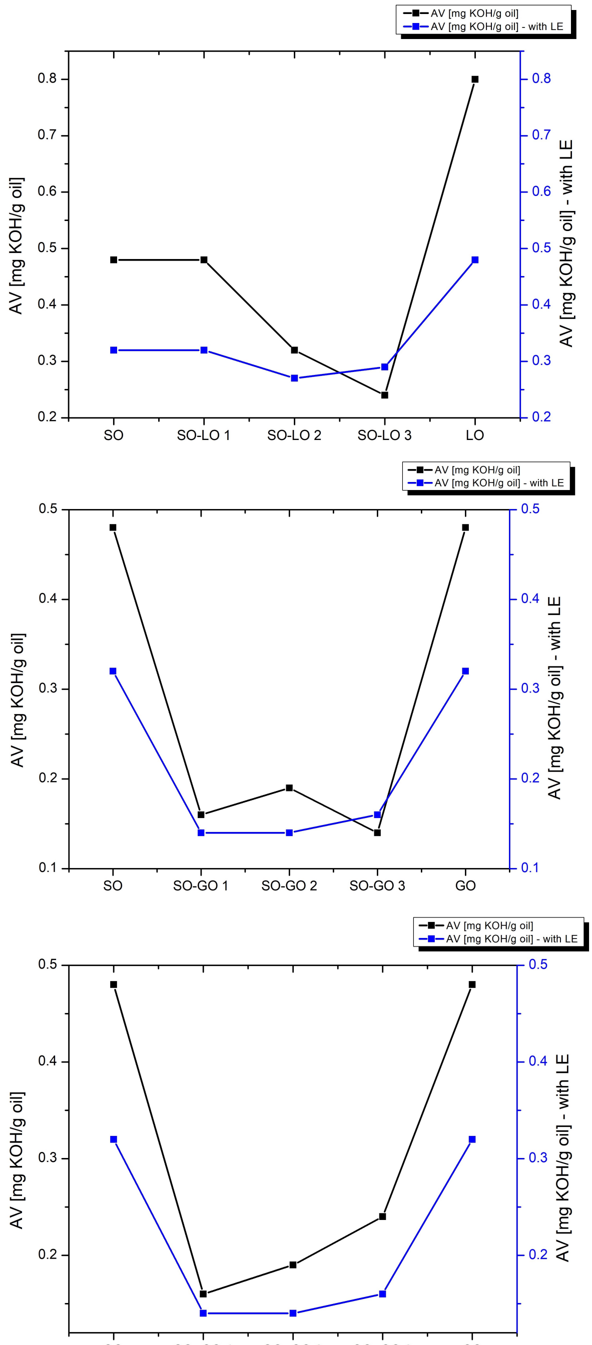

Figure 1. *AV values for sunflower oil based blends in comparison with those of individual oils, with and without lovage addition (LE): SO-LO (a); SO-GO (b); SO-CO (c)*

From Figure 1, the decrease of AV for blends compared to individual oils can be noticed. The variation of AV for blends depends on the oils nature. Thus, by mixing SO with LO, the AV values decreased with the increase of LO quantity, starting with 80:20 ratio, after an unchanged value in comparison to pure SO for 90:10 ratio. This feature indicates an interaction between free fatty acids of LO (LO has the higher quantity of FFAs) and SO, with the decrease of total quantity of FFAs. By mixing two oils with the same AV value (SO+GO, SO+CO), mixtures with lover acidity were obtained. Although the same trend of acidity decrease was observed for both series of mixtures, the dependence of AV on the oils ratio differs for GO compared with CO, as minor component in the blends. The differences can be due to the different chemical composition and different interactions between FFAs and other components of oils.

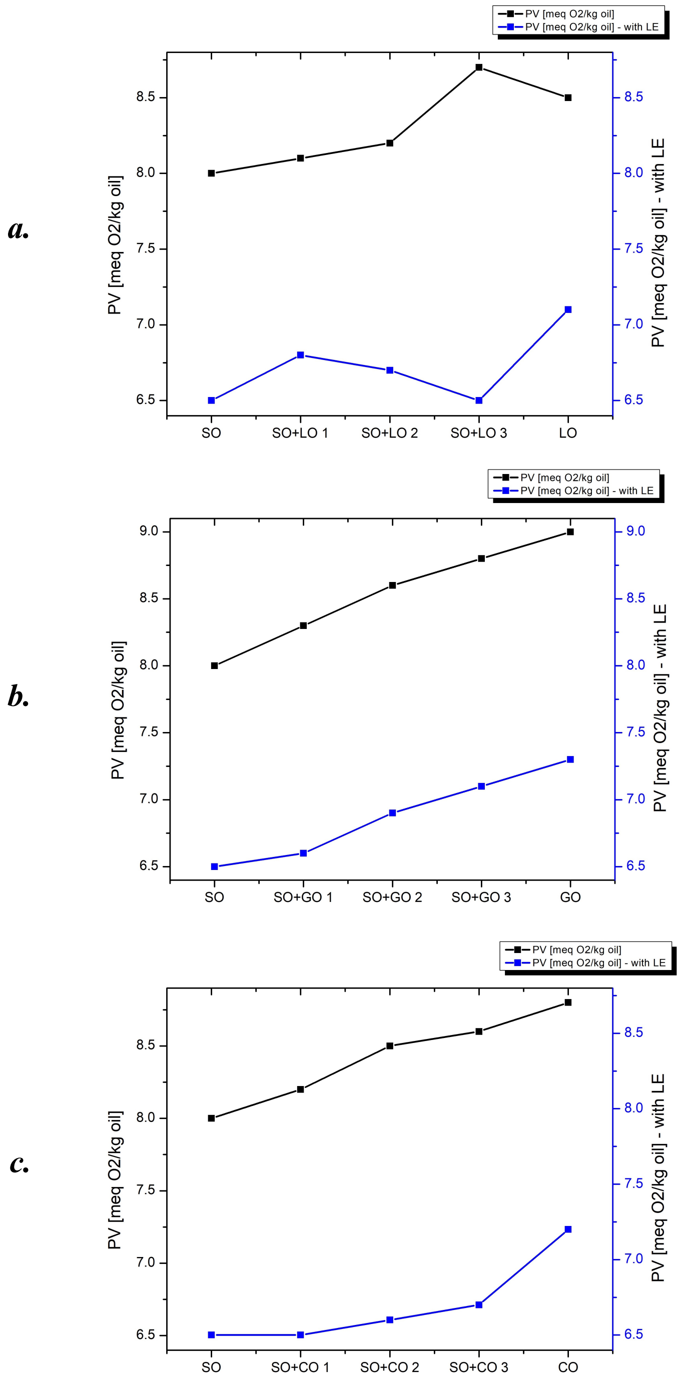

Figure 2. *PV values for sunflower oil based blends in comparison with those of individual oils, with and without lovage addition (LE): SO-LO (a); SO-GO (b); SO-CO (c)*

From Figure 2 and Table 2, a regular variation of PV for oil blends can be observed. The values are between those for individual oils, increasing with the increase of quantity of oil with higher PV. Having in view that PV for SO was the lowest, the blending with oils characterized by higher PVs seems to be a disadvantage in this regard. An unexpected high value of PV for SO-LO 3 was obtained.

Additivation of oils and blends with lovage leaves

The physico-chemical characteristics for additivated oils and blends are shown in Table 3. For all the additivated vegetable oils and blends, the color was yellow-greenish. The green shade demonstrated the extraction of chlorophyll in oils.

Table 3. *Acid values and peroxide values for oils and blends of oils additivated with lovage*

	Acid value (AV) [mg KOH/g oil]	**Peroxide value (PV) [meq O_2/kg oil]**
Vegetable oils:		
Sunflower oil + lovage	0.32	6.5
Linseed oil + lovage	0.48	7.1
Grapeseed oil + lovage	0.32	7.3
Coconut oil + lovage	0.32	7.2
Blends of vegetable oils:		
Sunflower oil + linseed oil		
SO-LO 1 + lovage	0.32	6.8
SO-LO 2 + lovage	0.272	6.7
SO-LO 3 + lovage	0.29	6.5
Sunflower oil + grapeseed oil		
SO-GO 1 + lovage	0.144	6.6
SO-GO 2 + lovage	0.144	6.9
SO-GO 3 + lovage	0.16	7.1
Sunflower oil + coconut oil		
SO-CO 1 + lovage	0.144	6.5
SO-CO 2 + lovage	0.144	6.6
SO-CO 3 + lovage	0.16	6.7

As a general observation, the addition of lovage in oils and oil blends decreased the acidity. Two irregularities, for SO-LO 3 and SO-GO 3 were observed. Obviously, the most important effect of the lovage addition is the decrease of PV values. Without exception, the values of PV for oils and oil blends additivated with lovage are significantly smaller compared with oils/blend of oils without lovage. Interesting, a low value for PV was obtained by adding lovage in SO-LO 3, a blend with unexpected high value for PV. The decrease of PVs is due to the antioxidants from lovage leaves.

The Pearson correlation was applied to evaluate the relationship between AV and PV. Generally, positive correlations were observed between acidity and peroxide value in this study (Table 4). A strong and significant ($p < 0.05$) correlation ($r = 0.860$ and $r = 0.798$) was found between AV and PV for SO-LO 2 and SO-CO 1, and a moderate correlation for other oil blends, meaning that the addition of lovage and the ratio of oil mixture can improve the quality of the vegetable oils.

Table 4. *Correlation coefficient of acidity and peroxide index of oil blends*

Person correlation	**Correlation coefficient (*r*)**
SO-LO 1 + lovage	0.580 ($p < 0.05$)
SO-LO 2 + lovage	0.860 ($p < 0.05$)
SO-LO 3 + lovage	0.432 ($p < 0.05$)
SO-GO 1 + lovage	0.546 ($p < 0.05$)
SO-GO 2 + lovage	0.398 ($p < 0.05$)
SO-GO 3 + lovage	0.432 ($p < 0.05$)
SO-CO 1 + lovage	0.798 ($p < 0.05$)
SO-CO 2 + lovage	0.474 ($p < 0.05$)
SO-CO 3 + lovage	0.511 ($p < 0.05$)

* Correlation is significant at $p < 0.05$

CONCLUSIONS

We obtained and characterized nine oil blends, based on sunflower oil, by adding linseed oil, grapeseed oil, and coconut oil in ratios of 90:10, 80:20, and 70:30 (v/v). The oils used for blends were analyzed and the blends properties were reported to those of individual oils. The obtained blends are characterized by lower values of acidity compared with pure oils, even compared with less acid component. The values of PV for blends are less remarkable, they being enclosed between the values of pure oils.

The lovage leaves were added in oils and oil blends and the extraction of chlorophyll and other phytochemicals was done directly in oils. The extracted phytochemicals slightly decreased the values of acidity, but the decrease of PV values was important for all studied oils and oil blends.

The results highlighted the importance of oils blending, mainly for the decrease of acidity, and also of the fresh herbal addition, for the reduction of the autoxidation processes, both of them improving the quality of edible vegetable oils. The addition of lovage as fresh leaves is an easy and efficient procedure, which avoid the use of chemicals.

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