Fatty acids composition and oil characteristics of linseed (Linum Usitatissimum L.) from Romania

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Received: 29 April 2012; Accepted: 10 June 2012

Abstract
Linseed oil characteristics were evaluated to determine whether this oil could be exploited as an edible oil. Petroleum ether extraction of linseeds produced yields of 30% (w/w) oil.

The chemical composition, including moisture, total oil content and ash, was determined. Iodine value, saponification value, acid value and peroxide value of obtained linseed oil was analyzed.

The fatty acids composition was analyzed with GC-MS method according to AOAC standards. Linseed oil was found to contain high levels of linolenic (53.21%) followed by oleic (18.51%), and linoleic (17.25%), while the dominant saturated acids were palmitic (6.58 %) and stearic (4.43%).

Keywords: fatty acids, linseed oil, GC-MS, chemical composition

1. Introduction
Consumption of flax (Linum usitatissimum) seeds is beneficial for human health. Flax seeds, containing about 36-40 % oil are the richest (among crop plants) source of polyunsaturated fatty acids (PUFA) essential in the human diet. PUFA are highly susceptible to oxidation [3]. Flax (Linum usitatissimum L.) is a multi-purpose crop. Its’ seeds containing about 36 to 40 % of oil, have long been used in human and animal diets and in industry as a source of oil. Recently there has been a growing interest in the probiotic properties of flax and in its beneficial effects on coronary heart disease, some kinds of cancer and neurological and hormonal disorders [9].

Flaxseed is abundant in many nutrients, such as polyunsaturated fatty acid, protein, and lignans [11]. In comparison with other vegetable oils, linseed oil is distinguished by the highest content of α-linolenic acid, i.e. 26±60%, which since recently has been found as especially important for human organism. Unfortunately, a high content of α-linolenic acid induces a poor oxidative stability of linseed oil [8].

Flax is an annual plant belonging to the genus Linum and the family Linaceae. Different varieties of linum have been developed for production of fibre oilseed. Varieties of Linum bred for fibre use are called flax, whereas the oilseed varieties are called linseed, linseed flax or just flax.

Linseed contains 26-45% oil. Approximately 22% of the oil is located in the seed coat and 4% in the embryo. The oil is present mainly as triacylglycerols in oil bodies having an average diameter. Approximately 70% of all the linseed oil produced worldwide is destined for technical applications and 30% is for food production [6].
The Codex Alimentarius does not set specific requirements for linseed oil, but it must the general quality requirements for food oils, especially regarding hygiene and safety. Linseed oil is derived from the seeds of flaxseed (*Linum usitatissimum* L.), a plant widely cultivated in Europe for fiber or oil for industrial use [2]. The most important linseed producing countries are Canada, Argentina, USA, China, India and Europe [5]. Generally linseed contains 40% oil, 30% diet fiber, 20% protein, 4% ash and 6% moisture [10,12].

Oomah has published a review concerning flax as a functional food source, presenting a collection of studies concerning the medical health benefits of linseed oil, whole flaxseed and its fractions. According to that review, most of the known biological activities of flaxseed have been assigned to alfa-linolenic acid [6,7].

Flax (*Linum usitatissimum* L.) is grown either as an oil crop or as a fiber crop, with fiber (linen) derived from the stem of fiber varieties and oil from the seed of linseed varieties [4].

2. Materials and Method

*Plant material.* Linseeds from the Western Plain of Romania and were purchased from a local market. To obtain oil samples by solvent extraction, chopped linseed were extracted with petroleum ether (Merck, 40-60°C) in a Soxhlet apparatus and the remaining solvent was removed by distillation. After extraction, the oil samples were filtered and stored. Extracted oil samples had a light yellow color and a very characteristic flavor.

*Chemical analysis of linseeds.* Moisture was determined by oven drying at 105°C for 3 hours. Ash and total fat contents were determined according to AOAC Official Methods [1].

*Physicochemical characteristics of linseed oil.* The ordinary oil constants, e.g., acid value, iodine, saponification, and peroxide number, and refractive index, were estimated according to the AOAC Official Methods [1].

*The fatty acids profiles* were determined by GC-MS. Fatty acid methyl esters were prepared using BF₃ methanolic solution and extracted with hexane.

*Gas Chromatography coupled with Mass Spectrometry Analysis.* A 2 µL volume of each sample was injected in a HP6890 Series Gas Chromatograph coupled with a Hewlett Packard 5973 Mass Selective Detector. The gas chromatograph was equipped with a split-splitless injector and a Factor Four™ Capillary Column VF-35ms fused silica column of 5% phenyl-methylpolysiloxane, 30m*0.25 mm, film thickness 0.25 µm. The GC oven was set to a temperature range of 100 to 300°C with 6°C/min, and a solvent delay of 7 min. The injector temperature was maintained at 230°C. The carrier gas of the sample was helium at 1.0 mL/min and the sample was injected in the splitless mode. The MS conditions were the followings: ionization energy, 70 eV; electronic impact ion source temperature, 200°C; quadrupole temperature, 100°C; scan rate 1.6 scan/s; mass, 40-500 amu. For the identification of the compounds the mass spectra of the samples were compared with those of the NIST/EPA/NIH Mass Spectral Library 2.0.

3. Results and Discussion

In Table 2 and Figures 1 and 2 representing GC-MS chromatogram and mass spectra, are shown the results obtained for samples of fatty acids from linseed oil and also Table 1 have physicochemical characteristics of linseed oil and mass spectrum of methyl palmitate (a), of methyl stearate (b), of methyl linolenate (c), of methyl oleate (d), of methyl linoleate (e). It noted that samples of linseed oil, are major unsaturated fatty acids (88.97%), while saturated fatty acids were at a rate of 11.01%. Predominantly in the composition of unsaturated fatty acids is linolenic acid (53.21%), also the category of unsaturated fatty acids were oleic acid (18.51%) and linoleic acid observed (17.25%).

The experimental values obtained are comparable to those in the literature and it notes that the composition of oleic acid obtained in this work do not differ significantly from literature data, namely (18.51%) to (20.9-24.4%) by El-Beltagi, to (15.81-27.99%) by Nykter and to (16.1) by Hosseinian., also in terms the composition of linoleic acid, values obtained not differ much from the literature, namely (17.25%) to (17.4-19.2%) by El-Beltagi, to (11.18-16.13%) by Nykter and to (14.7%) by Hosseinian.
Table 1. Physicochemical characteristics of linseed oil

<table>
<thead>
<tr>
<th>Properties</th>
<th>Mean value</th>
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<tbody>
<tr>
<td>Refractive index</td>
<td>1.469</td>
</tr>
<tr>
<td>Iodine value (g I₂/100g oil)</td>
<td>177</td>
</tr>
<tr>
<td>Saponification value (mg KOH/g oil)</td>
<td>190</td>
</tr>
<tr>
<td>Acid value (mg KOH/g oil)</td>
<td>0.80</td>
</tr>
<tr>
<td>Peroxide value (meqO₂/kg oil)</td>
<td>0.95</td>
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</tbody>
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Table 2. Composition in fatty acids (saturated and unsaturated) for linseed oil

<table>
<thead>
<tr>
<th>Fatty acids</th>
<th>Retention time (min)</th>
<th>Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Palmitic acid (C16:0)</td>
<td>18.34</td>
<td>6.58</td>
</tr>
<tr>
<td>Stearic acid (C18:0)</td>
<td>21.45</td>
<td>4.43</td>
</tr>
<tr>
<td>Oleic acid (C18:1)</td>
<td>21.35</td>
<td>18.51</td>
</tr>
<tr>
<td>Linoleic acid (C18:2)</td>
<td>21.50</td>
<td>17.25</td>
</tr>
<tr>
<td>Linolenic acid (C18:3)</td>
<td>21.85</td>
<td>53.21</td>
</tr>
</tbody>
</table>

Figure 1. The chromatogram from the GC-MS analysis of the linseed oil
Figure 2. Mass spectrum of methyl palmitate (a), of methyl stearate (b), of methyl linolenate (c), of methyl oleate (d), of methyl linoleate (e).
4. Conclusion

Regarding fatty acid composition, the saturates composed an average of 11.01% of the total fatty acids and of 88.97% unsaturated acid. The major saturated fatty acid was palmitic (16:0) and stearic acid (C18:1).

The major unsaturated fatty acid in the oils samples was linolenic (18:3), followed by oleic (18:1) and linoleic (18:2). The composition of oils confirms that found by the literature [2,6,8].

References

6. Nykter, M.; Kymäläinen, H.R., Quality characteristics of edible linseed oil, Agricultural and Food Science 2006, 15, 402-413