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PURIFICATION FLOW OF COSMETIC CAMELINA OIL

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Abstract

Camelina oil is obtained by pressing the seeds of Camelina sativa. Their pressing is done by a cold process. The oil samples used were obtained from the Camelina sativa - Mădălina variety, which was cultivated at the Belciugatele farm, with a working point Moara Domneasca (Găneasa commune, Ilfov county) between 2017-2018. The present project aims at the use of camelina oil in cosmetic preparations, using 4 types of oil grown in an ecological system, but their storage was different namely: some of the samples were kept at ambient temperature, in plastic dark blue cans, their capacity being 10l, but they were filled with 5l oil, in parallel being kept experiments at the refrigerator in brown glass containers, fully filled, in order to obtain the purest oil, but also the most complete one in terms of preservation of the biochemical qualities. The determinations made consisted of: appearance, odor, color, determination of iodine and saponification indices, as well as composition in fatty acids. All these determinations have led to the most efficient flow of camelina oil purification used in the cosmetic industry.

Key words*: bentonite, Camelina sativa, purification, volcanic tuff, zeolite.*

INTRODUCTION

Camelina is an oilseed plant belonging to the *Brassicaceae* family, which has low agronomic requirements (Putnam et al., 1993). *Camelina sativa* (L.) Crantz (2n = 40) is part of the same families with mustard, rapeseed, shepherd's purse, but also the plant that was used as a model for research in molecular genetics: *Arabidopsis thaliana* (Vollmann and Eynck, 2015).

Camelina sativa has a high adaptability to climate conditions, being able to grow in temperate climate, semi-arid environments, and short season regions (Ehrensing and Guy, 2018). Due to its increased adaptability, this plant can be found and developed in various countries, so the plant is distributed almost throughout the North American continent and in Europe.

From the fatty acid profile point of view, this is unusual with a high alpha-linoleic acid content and relatively low erucic acid concentrations (Zubr and Matthaus, 2002).

The applications for camelina oil include paint industry, biodiesel fuels as well as cosmetics. (Bonjean and Goffic, 1999; Bernardo et al., 2003).

The presence of polyunsaturated fatty acids gives sensitivity to camelina oil to lipid oxidation, but remains sufficiently stable during storage due to antioxidants present in the seed (Ni Eidhin et al., 2003; Abramovič and Abram, 2005). Camelina oil is the main compound obtained from Camelina seeds, and its yield is between 30-40% DM (Budin et al., 1995; Zubr, 2003). The oil obtained from Camelina seeds has a varied content in fatty acids, having a supply of 50-60% unsaturated fatty acids, 35-40% content of omega 3 as well as a content of 15-20% omega 6. It has a content high in omega 3, being one of the richest plant sources related to this fatty acid (Ratusz et al., 2016). Some of the most important attributes of camelina oil refer to its high productivity, which does not require a special and complicated cultivation technology, as well as its multiple uses in various industries (Toncea et al., 2013).

In one research made by Popa et al. (2019) about physico-chemical characterization of oil from *Camelina sativa* seeds grown in Romania, they had analyzed three types of oil (romanian and spanish oil).

The analysis of the oil extracted from the three Camelina varieties lead to the conclusion that the samples are very similar. The both are similar from the point of view of the type of extraction and the fatty acid profile. Another result is about the fertilization and the use of fertilizers, which do not conduct to significant quantitative or qualitative changes in the oils. They all are in accordance with the physico-chemical parameters specified by the European Pharmacopoeia for virgin linseed oil (Popa et al., 2019). Other studies on chemical composition have referred to the amino acids content. Camelina cracks were analyzed and the results indicated a high content of essential amino acids with values between 3.89% and 16.12% (Bătrâna et al., 2019). Another very important aspect regarding the composition of amino acids, refers to the essential amino acids that were found in all three varieties analyzed, namely: 38% for Madalina 37.9% in Calena variety, and for the local variety of BUASVMT 38.58%, along with 9 other non-essential amino acids (Bătrâna et al., 2019). It is well known that only plants can synthesize essential amino acids, so animals must obtain them from food. Their lack can significantly affect the quality of life and the appearance of certain diseases, therefore their importance should not be neglected.

Crăciun et al. (2018) made a research about the benefits and effect of the *Camelina sativa* oil for the skin. They had a revolutionary discover namely: Camelina oil, through its rich composition in fatty acids, accelerates the dermo-epidermal "de novo" synthesis, stimulating the keratinocytes differentiation and turn-over and also fibroblasts cellular division and collagen synthesis (Crăciun et al., 2018).

The linoleic and linolenic acids found in camelina oil have some interesting effects following topical application, as: tissue regeneration, involvement in membrane lipid transport and protective effect against chemical and enzymatic agents (Crăciun et al., 2018).

MATERIALS AND METHODS

For this project, we used the oil that was obtained by pressing the seeds of *Camelina sativa* from the variety Mădălina. This variety was cultivated in an ecological system at Belciugatele farm, having a working point Moara Domnească from Găneasa commune, Ilfov county during 2017-2018. The oil filtration was performed by a cold process, followed by the retention of the 4 samples being performed differently. Two of these (samples 1 and 2) were stored at room temperature in cans with a capacity of 10 l, but partially filled.

Contrary to this, the other two samples (samples 3 and 4) were kept in the refrigerator at temperatures of 4 degrees Celsius in brown glass containers, fully filled. A cold mechanical press was used in order to obtain the oil.

The extraction equipment used is called IEU-00 from the National Institute of Research and Development for Machinery and Installations designed for Agriculture and Food Industry-INMA Bucharest. The seeds were conditioned before pressing to eliminate impurities, using an ICS equipment from INMA-Bucharest. The oil was left at room temperature for 48 hours in order to decant, after that the oil being separated from the sediment.

Camelina oil purification method

The filtration was performed with the help of 3 adjuvants, namely: zeolite, bentonite and volcanic tuff. The samples were filtered differently, namely:

- Sample 1 - stored at ambient temperature - pretreatment with 0.05% bentonite - treatment with 0.05% bentonite - stored at 4°C;
- Sample 2 - stored at ambient temperature - pretreatment with 0.05% zeolite - treatment with 0.05% bentonite - stored at 4°C;
- Sample 3 - stored at 4 °C - 0.05% bentonite treatment - stored at 4°C and
- Sample 4 - stored at 4°C - 0.05% zeolite treatment - stored at 4°C.

Two of the 4 samples were filtered through a single stage and the other 2 samples were pre-treated. For one-step purification, the samples were treated with bentonite respectively zeolite at a concentration of 0.05%.

Purification performed by two steps introduced a pretreatment with 0.05% zeolite, respectively, to subsequently perform a filtration with 0.05% bentonite (Figure 1).

The working protocol used was aimed at removing as much of the impurities as possible to obtain an oil as pure as possible.

Figure 1. Camelina oil filtration

The filtering method (Table 1) was the same for all samples as follows:
Seeds => Seed pressing (V1) => Adding hot water (V1 + 10 mL) => Centrifuging oil (V2) => Adding bentonite or zeolite => 30 minute rest => Oil filtering (V3)
The pH is determined both before and after filtering the oil.
The addition of hot water over the oil sample aims to "wash" the oil of impurities. Once the hot water is added, rotating movements are performed for a few minutes in order to carry out the oil washing process as well as possible. After this process, the oil together with the water is distributed in vacuums and centrifuged at 5000 rpm for 5 minutes. After centrifugation, when the water has separated from the oil, a rapid movement to remove the water from the oil is performed. When all the vacuums have been emptied of oil as well as the removed water, over the oil collected from the centrifugation, the 0.05% bentonite and zeolites will be added, leaving to stand for 30 minutes. When the 30 minutes of rest had elapsed, the oil was filtered resulting in the amount of oil remaining after filtration, oil that will be analyzed and used in cosmetic preparations (Figure 2).

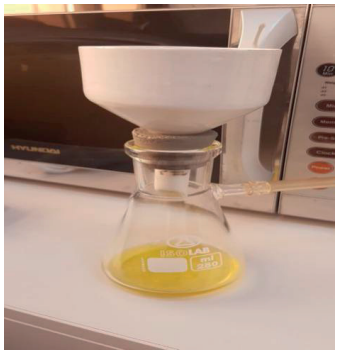

Figure 2. Camelina oil after filtration

The storage of samples before filtration was different, so that samples 1 and 2 were stored in plastic cans at room temperature, and samples 3 and 4 were stored in a dark color container at a temperature of 4°C. After filtration, storage was done in dark colored bottles in the refrigerator, at a constant temperature of 4°C (see Table 1).

Table 1. Filtering protocol (Copaci et al., 2019)

No.	Seeds (g)	V1 (ml)	V1+ 10 ml	V2 (ml)	Bentonite (g)	Zeolite (g)	Rest (min)	pH1	pH2	V3 (ml)	The color of the oil
1	400	90	100	79	0.4	0	30	5	5.5	70	light yellow, no deposits, clear
2	400	90	100	83	0.4	0	30	4.5	5.5	65	light yellow, no deposits, clear
3	200	50	60	48	0.22	0	30	5	5.5	40	dark yellow, no deposits, opaque
4	200	53	63	48	0	0.402	30	5	5	31	dark yellow to green, no deposits, opaque, water bubbles

RESULTS AND DISCUSSIONS

Following these purification processes, the main purpose is to find and optimize the best filtration method as well as the oil purification. The pretreatments applied were with bentonite or zeolite in a concentration of 0.05%, having the same methodology as the subsequent filtration. In the case of oil samples 1 and 2 after the pretreatment with bentonite and zeolite respectively, a treatment with 0.05% concentration bentonite was applied. For sample 3, only 0.05%

bentonite treatment was applied, and for sample 4 zeolite 0.05% treatment.

For the analyzed parameters, the values can be found in Table 2. In the literature, until now, the camelina oil has not been well individualized for use in cosmetic products. Due to the fact that it is not individualized, there is no standard in the European Pharmacopoeia, but also in the American Pharmacopoeia (USP), being analyzed as a newly introduced ingredient.

In order to be able to characterize each parameter, it will be analyzed in comparison with the oils already used and standardized for the cosmetic industry. The macroscopic purpose of these filtrations was to remove the existing physical impurities following the pressure, and at the physio-chemical level not to affect their chemical properties, namely: relative density d2020, refractive index nD20, acidity index (mg KOH/g), the iodine index (g I/100 g), the saponification index (mg KOH/g) and the peroxide index (meq O_2/kg).

Based on the analyzes performed at the physio-chemical analysis center S.C. BIOTEHNOS SA, the obtained results could be comparable with those already existing in the literature, determined by Abramovič and Abram (2005) for *Camelina sativa* oil from Slovenia: density at 20°C being 0.9207 ± 0.0001 g/cm^3, index of refraction at 25°C having a value of 1.4756 ± 0.0001, the peroxide determination being 2.38 ± 0.01 meq O_2/kg, and the acid value -6.2 ± 0.1.

Of the 4 samples, with regard to color, only the oil in sample 4 (without pretreatment + treatment with 0.05% zeolite) had small changes compared to the rest of the samples, namely "weak opalescent liquid", but it complied with the accessibility conditions. imposed. The yellow color and the specific odor of the plant were fully satisfied by all the samples regardless of the filtration method applied. Ullmann's encyclopedia did not report color changes or odor respectively, so that the oil was not degraded by filtration with zeolite or bentonite.

For the sample without pretreatment + treatment with 0.05% zeolite the peroxide index was within the allowed limits, having a value of 9.44 meq O_2/kg. The storage time of vegetable oils depends mainly on the phospholipid profile and the presence of antioxidants. External factors such as temperature, air and light are also decisive factors (Zubr and Matthaus, 2002). In the specialized literature, a peroxide limit of maximum 10 meq O_2/kg was set by SON (Standard Organization of Nigeria) (2000) and NIS (Nigerian Industrial Standard) (1992) (Zahir et al., 2017). Its high α-linoleic acid (ALA) composition is well known, and this high content indicates the susceptibility of camelina oil to oxidation.

Camelina sativa has a much higher content of fatty acids compared to other oilseeds, having a high content (over 50%) in polyunsaturated fatty acids especially OMEGA-3 type where we find α-Linolenic acid (18: 3n - 3) in a percentage of 38% and 15% linoleic acid (18: 2n - 6), followed by other important acids such as: oleic acid and eicosenoic/gadoleic acid.

Table 2. Chemical and physical characteristics of Camelina oil
(Certificate of analysis no. 77 / FC1 / 05.05.2019, S.C. BIOTEHNOS S.A.)

Physical chemical characteristics	Conditions of admissibility	Results			
		Sample 1	Sample 2	Sample 3	Sample 4
Peroxide index, meq O_2/kg	Max 10,0	**188,59**	**188,56**	**37,78**	9,44
Color	yellow	yellow	yellow	yellow	yellow
Acidity index, mg KOH/g	Max 10	1,462	1,409	1,730	1,997
Relative density, d2020	0,9100-0,9300	0,9278	0,9270	0,9233	0,9232
Refraction index, nD20	1,4700-1,4800	1,47751	1,47753	1,47747	1,47748
Saponification index, mg KOH/g	160-200	183,08	183,95	181,63	180,21
Iodine index, g I/100 g	130-170	145,02	141,82	146,70	147,28
Aspect	Clear or slightly opalescent liquid	Clear liquid	Clear liquid	Clear liquid	slightly opalescent liquid
Odor	plant specific	plant specific	plant specific	plant specific	plant specific

Another parameter analyzed was the pH of the four samples before and after filtering (Figure 3). In the case of sample 1 (stored at ambient temperature - pretreatment with 0.05% bentonite - treatment with 0.05% bentonite - stored at 4°C)

the initial pH was 5, and after treatment, the pH increased to 5.5.
For sample 2 (stored at ambient temperature - pretreatment with 0.05% zeolite - treatment with 0.05% bentonite - stored at 4°C) the initial pH was 4.5, identifying a pH of 5.5 after filtering. As with the other 2 samples, at sample 3 (stored at 4°C - 0.05% bentonite treatment - stored at 4°C) the initial pH of 5, increased to 5.5.
Last but not least, sample 4 (stored at 4°C - 0.05% zeolite treatment - stored at 4°C), the sample that did not show pH changes after the filtration. As can be seen from the above data, the filtration with zeolites without an effective pretreatment did not change the pH of the oil, a parameter that has utility for choosing the best filtration method (Table 3).
pH is very important no matter in which industry the oil is used, but for the cosmetics, food and pharmaceutical industry, this parameter is vital, and can have toxic and harmful implications for the human body. Fortunately, no filtration method has significantly altered the quality of the oil and thus the pH.

Figure 3. pH analysis of samples

Table 3. pH determination

Sample	Treatment	pH before filtration	pH after filtration
1	stored at ambient temperature - pretreatment with 0.05% bentonite - treatment with 0.05% bentonite - stored at 4°C	5	5.5
2	stored at ambient temperature - pretreatment with 0.05% zeolite - treatment with 0.05% bentonite - stored at 4°C	4.5	5.5
3	stored at 4°C - 0.05% bentonite treatment - stored at 4°C	5	5.5
4	stored at 4°C - 0.05% zeolite treatment - stored at 4°C	5	5

CONCLUSIONS

From our results we can see that the best method of purification was illustrated by sample 4 (without pretreatment + 0.05% zeolite treatment), samples 1, 2 and 3 do not correspond to a very important parameter, respectively, the "Peroxide index". As it is not possible to speak of a complete description of the camelina oil for its use in cosmetic products, the filtration technology applied to sample 4 (without pretreatment + treatment with 0.05% zeolite) had the best results in terms of maintaining the qualities nutrients, lipid profile, resistance to oxidation.
Oxidative stability is a significant distinguishing feature for camelina oil, which makes the emphasis not only on the filtration method but also on the pre- and post-filtration conditions.

Although the storage conditions after filtering were the same, a defining parameter was temperature prior to this process. For samples 3 and 4 even though they were kept at the same storage conditions, the zeolite filtration was much better able to meet all the required parameters.
Although the other samples were not significantly affected by the pH adjuvant filtering, it is still considered the best method that had no influence. As we said, the best results for maintaining the pH were recorded for sample 4, which further strengthens the selection of this method as the most suitable for preserving the oil properties after filtering.

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