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THE INFLUENCE OF THE ELECTROMOTOR FREQUENCY, THE TEMPERATURE OF THE PRESS HEAD AND THE ATTACHMENT FOR THE CAKE OUTLET DURING THE PRESSING OF LINSEEDS ON THE OIL YIELD

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Summary

Linseeds are rich in oil and protein and have a high proportion of unsaturated fatty acids. Raw linseed oil has a dark yellow color and a strong specific smell and taste. In this study, cold-pressed linseed oil was produced on a continuous screw press and the influence of the process parameters during the pressing of linseed on the utilization of cold-pressed oil was investigated. The following parameters were tested: the size of the attachment for the cake output, temperature of the press head heater and the electromotor frequency. The basic parameters of oil quality were determined on the produced cold-pressed linseed oil: peroxide value, free fatty acids, water content and insoluble impurities. Compliance with the Ordinance on Edible Oils and Fats (OG 11/19) was established. The results show that the investigated pressing parameters influence the utilization of linseed oil. The greater quantity of the produced linseed oil was achieved at a press head temperature of 90 °C, frequency of 22 Hz and a press head extension of 6 mm. The results also show that the studied quality parameters of the oil are in accordance with the values of the regulation, with the exception of insoluble impurities, which have slightly increased, and that it is necessary to extend the settling time of the crude oil. The fatty acid composition of linseed oil is well in accordance with the literature data, with linolenic fatty acid (Ω-3) predominating.

Keywords: linseed oil, screw press, process parameters, yield

Introduction

Linseed, *Linum usitatissimum* L., was already cultivated 3000 years ago. It is the oldest and former most important textile plant in areas with a colder climate. Today, linseed is used in many different industries such as chemical, textile, livestock, paper, pharmaceutical and food industry (Kołodziejczyk et al., 1995). A new direction of processing, which is still under development, is the use of linseed as a raw material for biofuels (Ayhan, 2009). It is an important oilseed grown in more than 50 countries, mainly for fiber and oil. Today, Canada is the leading producer, followed by Argentina, China, USA, Russia and India. Linseeds contain about 45% lipids, 30 % fibers and 20% proteins (Przybylski and Shahidi, 2005). The current interest in linseeds is due to its protective effect against coronary heart disease (Nagaraj, 2009; Boucher et al., 2012). In the diet, linseed is known as a rich source of α-linolenic acid (ALA), which can account for 52-60 % of total fatty acids (Daun et al., 2003). Several studies have shown that ALA may have beneficial effects on dyslipidemia, hypertension, atherosclerosis, platelet aggregation and cardiac arrhythmias (Shahidi, 2005). Linseeds are also known for their content of phytoestrogens, the so-called lignans, which are said to have a health-promoting and anti-cancer effect (Božan and Temelli, 2002). On the food market, linseed is mainly available in the form of whole seeds, ground seeds, partially defatted linseed cake or cold-pressed linseed oil. Depending on the type of raw material used and the growing conditions, linseed oil can be dark yellow, brown or dark amber color (Rubilar et al., 2010). Linseed oil is the natural edible oil mostly consumed without additives. This oil is one of the most unstable vegetable oils in relation to oxidation. The oxidative stability of oils may be influenced by many factors, such as light, metal ions, oxygen, temperature, and enzymes (Nawar, 1985). Because of the polyunsaturated fatty acids, vegetable oil is susceptible to oxidation (Braddock et al., 1995). The effect of fatty acid composition on the oxidative stability of oil has been studied by a number of investigators (OKeefe et al., 1993). The production of edible cold pressed oil is done with a continuous screw press without the use of organic solvents. The processing of oil through the cold pressing procedure ensures the maximum retention of active compounds in oil, like essential fatty acids, phenolic and flavonoid compounds, tocopherols, tocotrienols, phytosterols, and others (Teh and Birch, 2013), as well as the preservation of characteristic sensory properties of the oil. This pressing procedure results in crude oil, which

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must undergo the procedure of removing insoluble solid particles (through sedimentation, filtration and/or centrifuge) in order to obtain final cold pressed oil. One of the by-products in the process of pressing is cake, which retains a certain amount of oil, significant proteins, minerals, fibre, and other ingredients (Zubr, 1997; Quezada and Cherian, 2012). Jokić et al. (2014) investigated the optimisation of the production of cold pressed walnut oil with a screw press and determined that the processing parameters of the pressing process have an effect on the utilisation of oil. Moslavac et al. (2014) indicated that the process parameters of cold pressing have an effect on the utilisation of camelina oil (*Camelina sativa* L.).

The aim of this study was to investigate the influence of the process parameters during the pressing of linseeds on the utilization of cold pressed oil. The cold pressed oil was produced using a continuous screw press. Of the pressing process parameters, the influence of the attachment, i.e. the size of the opening for the cake outlet (6, 8 and 11 mm), the electromotor frequency (22, 28, 34 and 40 Hz) and the temperature of the press head (90, 100, 110 and 120 °C) on the oil utilization was investigated. The composition of the fatty acids and basic quality parameters of the produced cold pressed oil were determined: peroxide value, free fatty acids, water content, insoluble impurities, iodine and saponification value, and compliance with the Regulation on edible oils and fats (NN 11/19) was established.

Materials and methods

Materials

The raw material for the production of cold pressed linseed oil were cleaned, dried and unground linseeds purchased from the OPG Luka Ivoš, Croatia. All other chemicals and reagents were of analytical reagent grade.

Methods

Determination of oil and water content

The initial oil content in linseeds and cake residual oil (CRO) were measured using the automatic extraction system Soxterm by Gerdhart with *n*-hexane (Aladić et al., 2014). The initial water content (moisture) of the linseeds was determined according to the AOAC Official Method 925.40 (AOAC, 2000). The determination of moisture in the defatted cake was done using the modified standard HRN EN ISO 6496 (2001). The measurements were performed in duplicate.

Cold-pressing

The linseed oil was obtained by pressing, using different process conditions. The linseeds (1 kg) were pressed in a screw expeller (power of the electric motor 1.5 kW, press capacity 20-25 kg/hour). The produced crude oil was collected in a graduated cylinder and the volume and temperature were measured. After the precipitation of crude oil (nine days), vacuum filtration was carried out to remove insoluble particles from the oil.

Oil quality parameters

The peroxide value (PV) of produced oil was determined according to HRN EN ISO 3960 (1998) and expressed as mmol O_2/kg of oil. Free fatty acids (FFA) were determined using HRN EN ISO 660 (2020) and expressed as % of oleic acid. Insoluble impurities and moisture content were determined according to HRN EN ISO 663 (1992) and HRN EN ISO 662 (1999), respectively. All measurements were carried out in duplicate.

Parameters for oil identification

Saponification number and iodine number of produced cold pressed linseed oil were determined by ISO methods HRN EN ISO 3657 (2013) and HRN EN ISO 3961 (2018), and expressed as mg KOH/g and g I_2/100 g of oil, respectively.

Determination of fatty acids

The fatty acids methyl esters (FAMEs) were prepared with cold methanolic potassium hydroxide solution according to the procedure described in Annex X B of the Commission Regulation No 796/2002 (EC, 2002). FAMEs were afterwards separated on a Shimadzu GC-2010 Plus gas chromatograph equipped with a flame ionization detector (FID) and fitted with a SH-Rtx-Wax capillary column (30 m, 0.25 mm i.d. and 0.25 μm thick stationary phase). Nitrogen was used as carrier gas, flowing at the constant linear velocity of 1.33 ml/min. The split/splitless injector was set at 250 °C, split ratio was 1:10, and the injection volume 2 μl. Initial column temperature of 110 °C was held for 2 minutes, then gradually increased 10 °C/min until temperature of 175 °C that was hold for 8 minutes, followed by gradual increase 5 °C/min until 210 °C hold for 5 minutes, and temperature increase to final temperature of 230 °C by rate of 5 °C/min. Final temperature was hold for 7 minutes. Flame ionization detector temperature was 300 °C. Identification of separated FAMEs in samples was achieved based on the comparison of retention times with the retention times of certified reference standard (Supelco F.A.M.E. Mix, C4-C24, St. Louis, USA) analyzed under the same conditions. The results were expressed as a percentage of identified fatty acid on total fatty acids (%).

Results and discussion

Table 1 shows the results of determining the basic quality parameters of linseeds. The resulting values were for the oil content 39.72 ± 0.12 % and for the moisture content 6.69 ± 0.09 %.

Table 1. Linseed oil and moisture content

Parameter	Content (%)
Oil	39.72 ± 0.12
Moisture	6.69 ± 0.09

The results of testing the influence of pressing process parameters on the utilization of cold pressed linseed oil are shown in Tables 2-4. Table 2 shows the influence of the temperature of the press head on the utilization of cold pressed linseed oil. The tested temperatures of the press head were 90, 100, 110 and 120 °C. The extension on the head of the press for the cake output used in these tests was 8 mm, and the frequency regulator was set at 22 Hz to ensure a constant speed of the auger. By pressing linseeds at a press head temperature of 90 °C, a volume of crude oil of 400 mL and a temperature of 47 °C were obtained. After settling (sedimentation) of the produced crude oil for nine days and vacuum filtration, in order to remove residual insoluble impurities, the obtained volume of cold pressed oil was 330 mL (final oil). The share of residual oil in the cake was 14.67%. By applying higher temperatures of the press head heater (100, 110 and 120 °C), a gradually decreasing volume of crude oil (390, 385 and 370 mL) was obtained with a gradual increase in the temperature of the crude oil. It is evident that the largest volume of crude oil was obtained at 90 °C, and that at higher temperatures of the press head heater, the volume decreases. After sedimentation and vacuum filtration of the crude oil, a decreasing amount of final oil (cold pressed oil) was obtained. Based on the obtained results, it can be concluded that the tested temperatures of the press head do not have a significant impact on the amount of oil obtained. At higher temperatures of the press head, the volume of produced cold pressed linseed oil is slightly smaller. This phenomenon can be explained by the composition of the linseed, which affects the utilization of oil during pressing at the mentioned process parameters. Moslavac et al. (2016), Vladić et al. (2020) and Martinez et al. (2013) published in their papers that the increase in the temperature of the head increases the process pressure and lowers the viscosity of the oil, which results in greater oil draining as well as greater utilization during pressing. The temperature of the produced crude linseed oil increased as the heating temperature of the press head increased. At press head temperatures of 110 and 120 °C, the temperature of the raw oil slightly exceeded the requirement for the cold-pressed oil category (50 °C) (Regulation on Edible Oils and Fats, NN 11/19). The content of remained oil in the cake was highest (15.72%) at the highest temperature of the press head (120 °C), and the lowest at 90 °C (14.67%).

Table 2. The influence of press head heater temperature on oil yield

	Linseeds (kg)	Crude oil (mL)	Temp. crude oil (°C)	Cold pressed oil (mL)	Cake (g)	Oil content in cake (%)	Water content in cake (%)
N = 8 mm *T* = 90 °C *F* = 22 Hz	1	400	47	330	634.10	14.67	7.71
N = 8 mm *T* = 100 °C *F* = 22 Hz	1	390	49.5	320	641.15	14.78	7.55
N = 8 mm *T* = 110 °C *F* = 22 Hz	1	385	51	315	641.80	14.93	7.18
N = 8 mm *T* = 120 °C *F* = 22 Hz	1	370	52	306	644.44	15.72	7.30

N - extension on the press head that defines the diameter of the cake output (mm),
F - frequency regulator, regulates the screw speed of the press (Hz),
T – the temperature of the press head heater at the outlet of the cake (°C).

Table 3 shows the influence of the electromotor frequency (screw speed) during pressing on the utilization of the cold pressed linseed oil. The electromotor frequencies used for pressing were 22, 28, 34 and 40 Hz. The temperature of the press head heater at the cake outlet was 90 °C and the extension on the press head, which defines the diameter of the cake outlet, was 11 mm. Pressing linseed at 22 Hz produced the largest amount of crude oil (370 mL). On the other hand, at 28 Hz the crude oil volume was 360 mL, at 34 Hz was 340 mL and at 40 Hz the smallest amount of oil was produced (335 mL). The results show a direct influence of the electromotor frequency on the amount of crude oil extracted. The slower the screw was at the tested speeds, the greater the amount of linseed oil was extracted. Jokić et al. (2016) found the same phenomenon in their study, in which increasing the frequency resulted in a lower quantity of cold pressed hazelnut oil. After nine days of settling and vacuum filtration, the influence of the screw speed on the amount of the obtained cold pressed oil was clearly observed, as the most oil was obtained at 22 Hz (310 mL) and the least at 40 Hz (260 mL). The temperature of the crude oil corresponds to the conditions for cold pressed oils (Regulation on Edible Oils and Fats, NN 11/19), as it is below 50 °C in all cases. The proportion of residual oil is lowest in the cake obtained by pressing at 22 Hz (15.26%). When pressing at 28 Hz, the proportion of residual oil in cake was 17.73%, at 34 Hz 18.69% and at 40 Hz 20.79%. The direct influence of the electromotor frequency on the amount of oil remaining in the cake is therefore visible. The lower the frequency, the lower the amount of residual oil in the cake, and thus the greater utilization of oil by pressing the linseeds, thus the greater the amount of linseed oil produced.

Table 3. The influence of electric motor frequency (screw speed) on oil yield

	Linseeds (kg)	Crude oil (mL)	Temp. crude oil (°C)	Cold pressed oil (mL)	Cake (g)	Oil content in cake (%)	Water content in cake (%)
N = 11 mm *T* = 90 °C *F* = 22 Hz	1	370	46	310	650.40	15.26	8.60
N = 11 mm *T* = 90 °C *F* = 28 Hz	1	360	48	295	666.83	17.73	8.13
N = 11 mm *T* = 90 °C *F* = 34 Hz	1	340	44	270	676.46	18.69	8.17
N = 11 mm *T* = 90 °C *F* = 40 Hz	1	335	45	260	687.15	20.79	7.89

N - extension on the press head that defines the diameter of the cake output (mm),
F - frequency regulator, regulates the screw speed of the press (Hz),
T – the temperature of the press head heater at the outlet of the cake (°C).

Table 4 shows the influence of the attachment on the press head, which determines the diameter of the outlet opening of the cake, on the utilization of the linseed oil. The used attachments had a diameter of 6, 8 or 11 mm. The temperature of the press head heater used in this test was 90 °C and the electromotor frequency was 22 Hz. When pressing, the largest volume of crude oil was obtained with a 6 mm diameter attachment and amounted to 420 mL. The highest process pressure was generated here during pressing, which leads to greater extraction of the crude oil. A slightly lower amount of crude oil was obtained with an 8 mm (400 mL) attachment. The smallest volume of crude oil was obtained using 11 mm diameter attachment and amounted to 370 mL. After nine days of sedimentation and vacuum filtration of the crude oil, the largest volume of final cold pressed oil was 340 mL using the 6 mm diameter attachment. The temperature of the crude oil at the outlet is within the limits of the Regulation on edible oils and fats (NN 11/19). The proportion of oil remaining in the cake was lowest with the 6 mm attachment and amounts to 12.55%. Cvetković et al. (2020) and Moslavac et al. (2016) explain that reducing the size of the cake outlet on the press head increases the working pressure during seed preparation.

Table 4. The influence of extension on the press head, which defines the diameter of cake outlet, on oil yield

	Linseeds (kg)	Crude oil (mL)	Temp. crude oil (°C)	Cold pressed oil (mL)	Cake (g)	Oil content in cake (%)	Water content in cake (%)
N = 6 mm *T* = 90 °C *F* = 22 Hz	1	420	47	340	620.37	12.55	8.15
N = 8 mm *T* = 90 °C *F* = 22 Hz	1	400	47	330	634.10	14.67	7.71
N = 11 mm *T* = 90 °C *F* = 22 Hz	1	370	46	310	650.40	15.26	8.60

N - extension on the press head that defines the diameter of the cake output (mm),
F - frequency regulator, regulates the screw speed of the press (Hz),
T – the temperature of the press head heater at the outlet of the cake (°C).

The determined quality parameters of the cold pressed linseed oil were in accordance with the Regulation on edible oils and fats (NN 11/19), and are presented in Table 5. The results show that the peroxide value (PV), free fatty acids (FFA) and moisture content comply with the prescribed values. The share of insoluble impurities in the oil (0.16%) was slightly higher than specified in the regulation (max. 0.05%) but increased sedimentation time of the crude oil could be implemented to separate the solid particles from the oil. The values of the parameters for oil identification (saponification value and iodine value) corresponded to the values given in the literature.

Table 5. The basic quality parameters of produced cold pressed linseed oil

Oil quality parameters	Content
Peroxide value (PV), mmol O_2/kg	0
Free fatty acids (FFA), %	0.39
Moisture, %	0.067
Insoluble impurities, %	0.16
Saponification number, mgKOH/g	191
Iodine number, g I_2/100g	176

Table 6 shows the composition of the fatty acids contained in cold pressed linseed oil. The analysis was carried out using gas chromatography with an FID detector. The analysis of the composition of the fatty acids in a sample of linseed oil showed that polyunsaturated linolenic acid (C18:3) dominated with 54.21%, followed by monounsaturated oleic acid (C18:1) with 18.11% and linoleic acid with 16.65%. The saturated fatty acids are dominated by palmitic acid (C16:0) 6.28% and stearic acid (C18:0) 4.73%. These data were consistent with the literature (Dimić, 2003).

Table 6. Fatty acid composition of cold-pressed linseed oil

Fatty acid	Content (%)
Palmitic acid C16:0	6.28
Stearic acid C18:0	4.73
Oleic acid C18:1	18.11
Linoleic acid C18:2	16.65
Linolenic acid C18:3	54.21

Conclusions

The results show that the pressing parameters investigated influence the utilization of cold pressed linseed oil. The heating temperature of the press head has a minor influence on the oil utilization when pressing linseeds. By pressing linseed at a press head temperature of 90 °C, a larger quantity of raw and cold pressed linseed oil was obtained. On the other hand, the electromotor frequency had larger effect on oil utilization. When using a lower frequency (22 Hz), a larger amount of linseed oil (raw and ready pressed) was obtained and the amount of residual oil in the cake was lower. The use of a different attachment on the head of the press for dispensing the cake affects the use of the oil when pressing linseeds. The smaller the diameter of the nozzle (6 mm), the greater the amount of oil was produced. The results show that the studied quality parameters of the oil comply with the values of the Regulation on edible oils and fats (NN 11/19), with the exception of insoluble impurities, which have slightly increased, and that it is necessary to increase the settling time of solid particles from the crude oil. The temperature of the obtained crude oils complied with the conditions for cold pressed oils (Regulation on Edible Oils and Fats), as they were below 50 °C, with the exception of the press head temperatures of 110 °C and 120 °C. The fatty acid composition of linseed oil corresponds to the information in the literature. It was found that linolenic fatty acid (omega-3) dominates.

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