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# FLAXSEED *(LINUM USITATISSIMUM)* LOW TEMPERATURE PROCESSING: OIL QUALITY AND FATTY ACIDS PROFILE

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*KEY WORDS:*
*acid value, flax, cake outlet size, optimization, peroxide value, screw press*

ABSTRACT
Flaxseed consumption has been prioritized by health-conscious people all over the world. Its consumption raises interest to it both from a personal and an industrial prospect due to its remarkable fatty acids profile. Besides its high content of unsaturated fatty acids, flaxseed oil is known for its low omega 6/omega 3 ratio, which may possess health improving properties because this oil is precursor of anti-inflammatory molecules. However, the high amount of polyunsaturated fatty acids in flaxseed oil causes its high sensitivity to high temperatures, to light and to oxygen, which may lead to its oxidation and quality degradation. Screw pressing can produce high quality oil but in lesser volume, which could be disadvantage. Therefore, the extraction process must be optimized for maximizing oil recovery, while maintaining oxidation indicators within limits specified by regulations, as well as a potentially health beneficial fatty acid profile. Optimization of screw press parameters for pressing out the flaxseed oil were investigated by Response Surface Methodology. Both size of the cake outlet and the speed of the screw pressing were optimized, and the values that provided the highest experimental oil recovery, 41.4%, were of 1 mm and 155.89 rpm respectively. Although the overall trend in oil extraction showed a rising oil recovery when screw speeds increased from 98.73 up to 213.05 rpm, certain fluctuations were observed in oil extraction with varying outlet cake sizes. However, there was an inversely-proportional function between the oil recovery and the size of the cake outlet, therefore, screw speed provided a minor, non-significant effect while the size of the cake outlet proved to have a strong effect on oil recovery. The highest acid value and peroxide value accounted to 0.71 mg KOH/g and to 7.71 meq/kg respectively. Sediment content (SC) of screw pressed flax oil ranged between 9.12~14%. During the oil extraction at the maximum yield, temperature increased, however the ratio of omega 6 to omega 3 in the obtained oil still remained low.

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# НИЗКОТЕМПЕРАТУРНАЯ ПЕРЕРАБОТКА ЛЬНЯНОГО СЕМЕНИ (*LINUM USITATISSIMUM*): КАЧЕСТВО МАСЛА И ПРОФИЛЬ ЖИРНЫХ КИСЛОТ

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*КЛЮЧЕВЫЕ СЛОВА:*
*кислотное число, лён, размер жмыха на выходе, оптимизация, перекисное число, винтовой пресс*

АННОТАЦИЯ
Потребление льняного семени является приоритетным для людей, заботящихся о своем здоровье, как с личной, так и с производственной точки зрения, особенно из-за замечательного жирно-кислотного состава. Помимо большого количества ненасыщенных жирных кислот, льняное масло известно низким соотношением омега 6/омега 3, которое имеет свойства, улучшающее здоровье человека, поскольку обладает противовоспалительным воздействием. Однако большое количество полиненасыщенных жирных кислот в льняном масле приводит к его высокой чувствительности к высоким температурам, свету и кислороду, что может привести к окислению и снижению качества масла. Шнековым прессом можно производить масло высокого качества, хотя и в меньшем количестве, что является его недостатком. Следовательно, процесс экстракции должен быть оптимизирован для максимального извлечения масла при сохранении показателей окисления в пределах, установленных нормативными актами, а также потенциально полезного для здоровья. Оптимизацию процесса прессования для получения льняного масла исследовали методом поверхности отклика. Размер жмыха на выходе и скорость шнекового пресса были оптимизированы, а значения параметров прессования, которые дали максимальное экспериментальное извлечение масла 41,4%, составили 1 мм и 155,89 об/мин соответственно. Хотя общая тенденция извлечения масла увеличивалась при увеличении скорости вращения шнека с 98,73 до 213,05 об/мин, при извлечении масла наблюдались колебания при различных размерах жмыха на выходе. Однако обнаружена обратно пропорциональная зависимость между извлечением масла и размером жмыха на выходе, поэтому скорость шнека оказывает незначительное влияние на выход масла. Максимальное кислотное число и пероксидное число составили 0,71 мг КОН/г и 7,71 мэкв/кг соответственно. Содержание осадка (SC) в льняном масле шнекового отжима колеблется от 9,12 до 14%. При экстрагировании с максимальным выходом температура повышалась, однако соотношение Омега 6 к Омега 3 полученного масла оставалось низким.

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## 1. Introduction

Flax *(Linum usitatissimum L.)* is one of the most important oilseed crops in the world. It contains lipids, lignans, proteins, fiber, other carbohydrates and micronutrients. Due to its health beneficial components and subsequent nutraceutical potential, the flaxseed oil became a point of interest for healthy food production and pharmaceutical industries throughout the world [1, 2].

Flaxseed oil has been considered as a health supportive food which is a rich source of polyunsaturated fatty acids and other bioactive components [1,2,3]. The most important feature of this crop is its uniquely and comparatively high content of omega-3 fatty acid (α-linoleic acid), which comprises over the half of the amount of its fatty acids [2]. Flaxseed is composed of about 40% of oil, where the specific profile is as follows: α-linoleic acid (ALA) (~53%), oleic acid (~19%), linoleic acid (~17%), palmitic acid (~5%), and stearic acid (~3%). It is necessary to note its favourable omega 6/omega 3 fatty acids ratio (0.3:1) in the flaxseed oil [3,4]. Besides, it also contains antioxidants and phytosterols, which may also boost up a human health [3,5]. It is worth noting, that flaxseed has been reported as a dietary source of lignans [5,6].

However, the high amount of polyunsaturated fatty acids in flaxseed oil leads to its high sensitivity to high temperatures, to light and to oxygen, which may lead to oxidation and oil quality degradation [3,5,7]. Quality degradation may take its origin during the period of growing, harvesting, processing, and/or storage of flax crops, flaxseed, and oil production. Oil degradation may occur both through oxidation and through thermal degradation [3]. It has been reported that oxidative and thermal degradation may be accelerated by the presence of metals such as iron or copper [8,9]. Thermal degradation may be impeded by antioxidants, either the additives like BHA, BHT or TBHQ; or the natural compounds such as polyphenols, especially lignans, which also act as metal chelators, thus providing steric hindrance in the interaction between metals and food components [8].

Pressing is a preferable technique for oil production due to its several advantages: low energy consumption, low cost, and absence of potentially harmful organic solvents; besides, it is also known for the higher quality in the extracted oil [4,10,11]. However, its main disadvantage is a low oil recovery, thus leaving the high share of oil in the remaining cake. Nevertheless, the volume of the obtained oil also depends on process variables, therefore, by adjusting the variable parameters a higher oil recovery may be obtained [10,11].

Quality and quantity of the flaxseed oil depend on pretreatment, processing, and conditions of seeds storage. There have been defined several influencing factors such as dehulling, seed conditioning, steam-treating, enzyme-treating, roasting, hot pressing, dry extrusion, speed of screw pressing and the size of the cake outlet, among the other factors [3,4,11,12]. Although those above-mentioned factors may favorably impact on the quantity of the extracted oil, in some cases quality of the oil may degrade, especially due to an increase in oxidation and thermal degradation of the oil. In any case, pressing technology is preferable for either producing the functional oil (FO), or somewhere in rural places where several species of oleaginous crops are grown, thus the lower yields from one crop may be acceptable [11,12,13,14].

There may be several influencing factors regarding process variables and oil extraction parameters during the various stages of the seed processing (on the laboratory and pilot plant) [14,15]. Therefore, in this study our goal is the optimization of process variables regarding the impact of screw pressing parameters on flaxseed oil production in a pilot plant. The goal was to optimize studies of screw speed and the size of the cake outlet based on Response Surface Methodology (RSM), which outperforms the classical methods. RSM is an effective mathematical and statistical technique for optimization. RSM applying can determine the effect of process parameters on technological response and interaction (function) between them [16].

## 2. Objects and methods

Flax *(Linum usitatissimum)* was brought from Surkhondaryo region, Uzbekistan. The initial and final oil content of the flax and cake, and the moisture content of flax were determined by the formula according to GOST 10857–64[1], GOST 10856–96[2]. Moisture content of the oil was calculated by the formula below:

$$X = \frac{M_i(W_f - W_i)}{100 - W_i} \quad (1)$$

where:

$x$ is the mass of water to be added;
$M_i$ is the initial mass of the sample;
$W_i$ is the initial moisture content of the sample in% w. b.;
$W_f$ is the final moisture content in% w. b.

Moisture content of flax samples was equal to 8.91%.

### *2.1. Parameters for screw pressing flax seeds*

The experiments were run on a Pilot plant screw press by the same methods as were used in our previous studies [17,18,19] (screw press is described below in the Figure 1), normally used for extracting oil from sunflower, soybean, and flax. It is powered by a 16 kW electric motor, with a capacity of 15–75 kg/h. The residual oil in the cake accounts for 9–12%. Internal diameter of the screw shaft is 140±4 mm, and the distance between oil outlet holes is 0.12±0.02 mm. The size of press cake discharging hole can be adjusted from 10.3 to 26.3 mm.

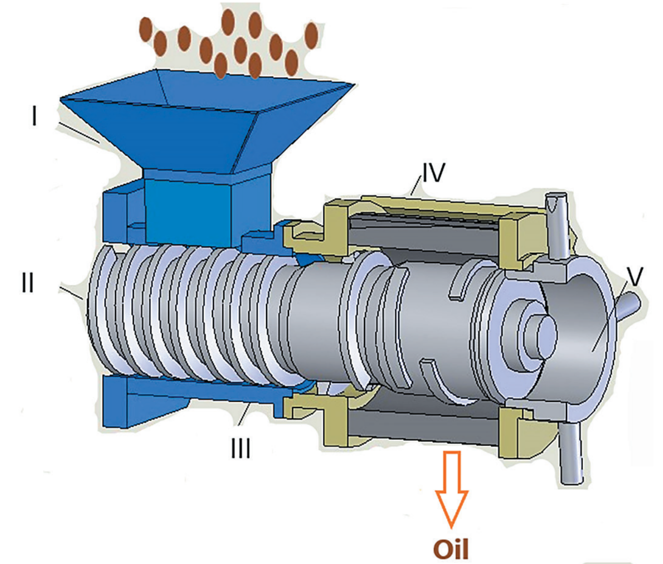



Figure 1. **Pilot plant screw press. (I)-feed hopper, (II)-screw, (III)- press cylinder, (IV)-outlet holes, (V)-cake outlet**
Рисунок 1. **Опытная шнековая пресс-установка (I)-загрузочная воронка, (II)-шнек, (III)- пресс-цилиндр, (IV)- отверстия для выхода масла, (V)-выходное отверстие для жмыха**

Experiments were run at a room temperature of 40±2 °C. Before processing, one kilogram of seed was used to condition the temperature of the equipment cylinder. Experiments were run sequentially by several experimental trials. Several variable parameters were set for each screw pressing. The temperature of the oil was measured by from a close-range distance digital thermometer WT-1 (Elitech, USA). However, the distance from oil dripping point to the collecting surface and an effect of the wind on the measurements were not taken into calculations. Process time was considered as the period from the beginning of the process until the cease of oil flow from the press outlet, which period varied from 15 to 25 min. Both oil and its residues (small oil particles) were collected into a glass container, every experimental trial was run in duplicate. The speed of the screw spinning in the screw-mill was programmed with the help of the inverter CHNT: Nyf2g-7.5/Ts4 Inverter (Czech Republic) [20], while the speed of the screw spinning and the size of the cake outlet were adjusted in accordance with the terms of the experiment, refer to the Table 1). In this study the seeds feeding rate was kept steady and constant.

### *2.2. Sediments content in the oil*

An aliquot of oil was weighed. Sediment particles were isolated from the oil via centrifugation at 1,200 rpm for 30 min (Centrifuge: Tslmn-P10–01-Elekon, Russia), and the supernatant was removed. The pellet was then washed in chloroform, followed by vacuum filtration. Sediment was then dried and weighed at an analytical balance. The sediment content of the oil was determined by calculating the ratio between the weight of the sediment, and the weight of the whole oil.

[1] GOST 10857–64. “Oil seeds. Methods for determination of oil content.”. Moscow: Standartinform, 2010. — 6 p. (In Russian)
[2] GOST 10856–96. “Oil seeds. Methods for determination of moisture content”. Moscow: Standartinform, 2019. — 4 p. (In Russian)

*2.3. Oil recovery calculation*

Oil Recovery (OR) is defined as the ratio of oil weight in the meal to the oil weight in the original seed that was pressed, calculated as follows:

$$OR(\%) = \left(1 - \frac{\text{weight of the meal} \times \text{oil content in the meal}}{\text{weight of the raw material} \times \text{oil content in the raw material}}\right) \times 100\% \quad (2)$$

*2.4. Characteristics of oil quality*

The peroxide value and acid value were determined as quality characteristics of the oil. Although there are no specific regulations for evaluating the quality parameters of flaxseed oil in the food industry, the standards for the virgin oils and cold-pressed fats and oils, set by the Codex Alimentarius Commission (1999) [21], were used to evaluate the oil quality parameters. The peroxide and acid values of the oil samples were determined according to GOST ISO 27107–2016[3] and GOST 31933–2012[4]. All scheduled experiments were duplicated.

*2.5. Response surface methodology*

Response Surface Methodology (RSM) is a collection of statistical and mathematical techniques useful for the modeling and analysis of the problems, where performance response is influenced by several variable parameters. It is a powerful optimization tool that has been widely used in research and industrial sectors. The main goal of RSM is to optimize the response that is affected by various process parameters; the concept of Response Surface Methodology was introduced by George E. P. Box and K. B. Wilson in 1951 [22]. The methodology is based on the matching of a polynomial equation to the experimental data, which must be relevantly selected to represent adequately the behavior of the system. The equation is used to estimate the obtained response and optimize the process. The RSM process involves three primary steps: running the experiment, estimation of the coefficients in a mathematical model, predicting the response and checking the adequacy of the calculated model. RSM is particularly useful when the experimenter is not completely sure of what form the underlying function takes. It provides a means of navigating through the design space using a sequence of designed experiments. These experiments are used to fit a model, predict the response, and determine the settings of the factors that optimize the response. RSM is used to optimize the process parameters to achieve maximum efficiency. In the food industry, RSM is used to optimize the recipe for a new product to achieve the best taste and texture. There are several advantages of RSM application. It helps in the development, improvement and optimization of systems; and it reduces the number of experimental trials needed, thus saving time and resources. Furthermore, it helps visualizing the relations between the response and the independent variables [23]. In this study, RSM, used by *Minitab 19* software, was used for optimizing the flaxseed screw pressing variables. *Minitab* is the software used for data processing, statistical analyses and improvement of workflow processes. It is extensively applied in industries, manufacturing, education and research purposes. Minitab is equipped with tools for performing statistical analysis involving hypothesis testing, regression analysis, ANOVA, and optimization studies [24]. An experimental plan was developed using CCD. Five levels and two factors were used for the following process parameters: screw auger speed (A), and the size of the cake outlet (B). Multiple regression analyses were used to obtain a second-order polynomial equation to predict the rate of screw-pressed flaxseed oil recovery. The general second-order polynomial equation is described below:

$$Y = \beta_0 + \sum_{i=1}^{K}\beta_i X_i + \sum_{i=1}^{K}\beta_{ii} X_i^2 + \sum_{i=1}^{K+1}\sum_{j=1}^{K}\beta_{ij} X_i X_j + \varepsilon \quad (3)$$

where:

$Y$ is response;
$\beta_0$ — intercept coefficient;
$\beta_i$ — linear constant coefficient;
$\beta_{ii}$ — quadratic constant coefficient;
$\beta_{ij}$ — interaction constant coefficient;
$X_i$ and $X_j$ — uncoded process variables;
$K$ — number of the variables;
$\varepsilon$ — random error.

The two independent parameters were: A — screw auger speed; B — the size of the cake (meal) outlet. The coded five levels of factors were −1.41, —1, 0, +1 and +1.414 (Table 1). Response (Y) was evaluated as OR%.

[3] GOST ISO 27107–2016. "Animal and vegetable fats and oils. Determination of peroxide value by potentiometric end-point method". Moscow: Standartinform, 2016–10 p. (In Russian)

[4] GOST 31933–2012. "Vegetable oils. Methods for determination of acid value". Moscow: Standartinform, 2019. — 10 p. (In Russian)

Table 1. **Levels and factors of flaxseed oil screw pressing experiments**

Таблица 1. **Уровни и факторы в экспериментах по шнековому прессованию льняного масла**

| Factors | | Coding | | | | |
|---|---|---|---|---|---|---|
| −1.414 | | −1 | 0 | +1 | +1.414 | |
| Screw speed (rpm) | A | 98.73 | 135.11 | 155.89 | 176.68 | 213.05 |
| Sizes of outlet cake (mm) | B | 1 | 1.25 | 1.5 | 1.75 | 2 |

An experimental design was implemented including 13 treatments (4 cube points, 5 central points, and 4 axial points) for pilot-plant screw pressing. The 13 numbers of results of the experiments were used to create a regression mode (Table 2). Described factors, levels, and responses for oil recovery percentage (OR %), sediment content (SC %), acid value (mg KOH/g), peroxide value (meq/kg) and temperature (°C) of the flaxseed oil are listed below in the Table 2, as well as correspondences to the independent variables (both coded and uncoded).

Table 2. **Central composite design of flaxseed oil pressing experiments**

Таблица 2. **Центральный композитный метод для получения льняного масла с помощью прессования**

| | Coded | | Uncoded | | Quality and quantity properties of the oil | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| № | A | B | A | B | OR (%) | Predicted OR (%) | SC (%) | Acid value (mg KOH/g) | Peroxide value (meq/kg) | Oil Temp. (°C) |
| 1 | 0 | 0 | 155.89 | 1.5 | 38.3 | 37.56 | 11.32 | 0.71 | 5.13 | 37.6 |
| 2 | 0 | 0 | 155.89 | 1.5 | 38.6 | 37.56 | 12.45 | 0.35 | 5.64 | 57.6 |
| 3 | −1.414 | 0 | 98.73 | 1.5 | 34.7 | 34.31 | 13.16 | 0.625 | 4.53 | 85.7 |
| 4 | 0 | −1.414 | 155.89 | 1 | 41.4 | 40.53 | 14 | 0.54 | 4.74 | 102 |
| 5 | -1 | 1 | 135.11 | 1.75 | 26.14 | 27.9 | 9.12 | 0.48 | 4.43 | 46.7 |
| 6 | 0 | 1.414 | 155.89 | 2 | 14.88 | 13.9 | 10.1 | 0.43 | 7.71 | 59 |
| 7 | 0 | 0 | 155.89 | 1.5 | 38.7 | 37.56 | 12.14 | 0.455 | 4.56 | 70.8 |
| 8 | 1.414 | 0 | 213.05 | 1.5 | 33.8 | 33.21 | 10.49 | 0.45 | 4.26 | 91 |
| 9 | 0 | 0 | 155.89 | 1.5 | 38.4 | 37.56 | 11.4 | 0.39 | 4.14 | 69.6 |
| 10 | 1 | 1 | 176.68 | 1.75 | 25.7 | 27.73 | 10.77 | 0.385 | 4.92 | 67 |
| 11 | 1 | −1 | 176.68 | 1.25 | 38.9 | 40.82 | 12.36 | 0.395 | 5.5 | 89.9 |
| 12 | −1 | −1 | 135.11 | 1.25 | 39.8 | 41.45 | 12.74 | 0.395 | 4.05 | 99 |
| 13 | 0 | 0 | 155.89 | 1.5 | 38.39 | 37.56 | 9.7 | 0.395 | 4.56 | 87 |

*2.6. Evaluation of the model congruence*

Using ANOVA, the quality of the model's fitness can be evaluated. The coefficient of determination $R^2$ defines how well the model fits the experimental data.

The regression equation of the oil recovery is described below (4):

$$Y = -37.2 + 0{,}320A + 94.1B - 0.001165A \times A - 41.39B \times B + 0.022A \times B \quad (4)$$

where $Y$ represents oil recovery and $A$ and $B$ represent screw auger speed and the size of the cake outlet, respectively.

Table 3 shows the ANOVA analysis of the predicted model for the screw pressing of flaxseed oil. The effect of the process parameters was evaluated by their significance ($p < 0.05$) with a $R^2$ of 97.16 and adjusted $R^2$ of 95.12.

Table 3. **Analysis of variance of results of flaxseed oil obtained by pilot plant screw press**

Таблица 3. **Дисперсионный анализ результатов получения льняного масла с помощью опытной установки шнекового пресса**

| Source | DF | Adj SS | Adj MS | F-Value | p-Value |
|---|---|---|---|---|---|
| Model | 5 | 686.935 | 137.387 | 47.81 | 0.000 |
| Linear | 2 | 41.032 | 20.516 | 7.14 | 0.020 |
| A | 1 | 3.765 | 3.765 | 1.31 | 0.290 |
| B | 1 | 27.067 | 27.067 | 9.42 | 0.018 |
| Square | 2 | 154.119 | 77.060 | 26.82 | 0.001 |
| A*A | 1 | 20.950 | 20.950 | 7.29 | 0.031 |
| B*B | 1 | 152.994 | 152.994 | 53.25 | 0.000 |
| 2-way interaction | 1 | 0.053 | 0.053 | 0.02 | 0.896 |
| A*B | 1 | 0.053 | 0.053 | 0.02 | 0.896 |
| Error | 7 | 20.113 | 2.873 | | |
| Inconsistency | 3 | 20.004 | 6.668 | 243.18 | 0.000 |
| Pure Error | 4 | 0.110 | 0.027 | | |
| Total | 12 | 707.048 | | | |

*2.7. Volatile acids profile / analysis of the fatty acid composition of the oil*

Further analysis was executed by Gas Chromatography — Mass Spectrometry (GC–MS) on the selected samples of flaxseed oil. The specific column in the Table 2 proves that this amount of the oil pressed in the mode No. 4 was obtained at the highest extraction temperature and with the highest MO. Besides, flaxseed oil extracted by supercritical fluid extraction method, as described in [25], was also analyzed for its comparison.

Previous to their analysis by GC–MS, oil samples were derivatized, and fatty acid methyl esters (FAMEs) were prepared: 0.1 g of the oil sample was used under nitrogen shield; 1 ml of toluol, and 1 ml of BF3-MeOH reagent were added into the flask. The solution was heated in water bath and maintained at 60 °C for 10 min; then it was cooled down to the room temperature. Double distilled (1 ml) water was added to the resulting solution and mixed for 5 minutes. The injection volume of the sample was 0.1μl.

Conditions for GC–MS analyses were as follows: The fatty acid composition / volatile acids profile of oil samples was determined by the device Agilent Technology (USA) GC 7890B/ MS7000D, equipped with a DB-FAT-WAX UI capillary column (30 m x 0.25 mm i. d., 0.25 μm film thickness). Helium was used as the carrier gas. The temperature program of the column oven started at 170 °C was hold during 2 min, and then it was raised at a rate of 4 °C /min to 280 °C and was kept at this level for 5 min. The temperatures of the detector and the injector were both of 280 °C. The detected compounds were identified by their comparison with WILEY275 Mass Spectral Database library.

## 3. Results and discussion

*3.1. Effects of process variables on oil recovery*

Significant effects were observed on the oil recovery (as shown in the Table 3) in the following sources: the size of the cake outlet provided the linear effect (also displayed below in the Figure 1) with ($p < 0.05$).

Experiments with high rotational speeds were run by the author [26]. Researcher [26] studied the effect of the temperature inside the cylinder for the seeds of *Jatropha curcas* L.

Generally, pressure increases along the cylinder toward the outlet inside of the screw press. The highest temperature is registered in the compression zone and close to the cake outlet. Outlet size of the press impacts on oil recovery as well as in productivity. If the outlet size decreases, it affects the flow of the seeds inside the cylinder; as a result, pressure and temperature rise up, oil recovery improves, and oil content in the cake decreases. On the contrary — if the size of the cake outlet is expanded, it may decrease the pressure in the cylinder, and reduce the rate of the oil recovery. Therefore, the outlet size is one of the variables that we have studied.

Screw speed is another process parameter that may affect oil quality and quantity. An increase of screw speed may reduce the oil amount; this is explained by the shorter time of pressing and shorter friction, as well as by viscosity of the phase.

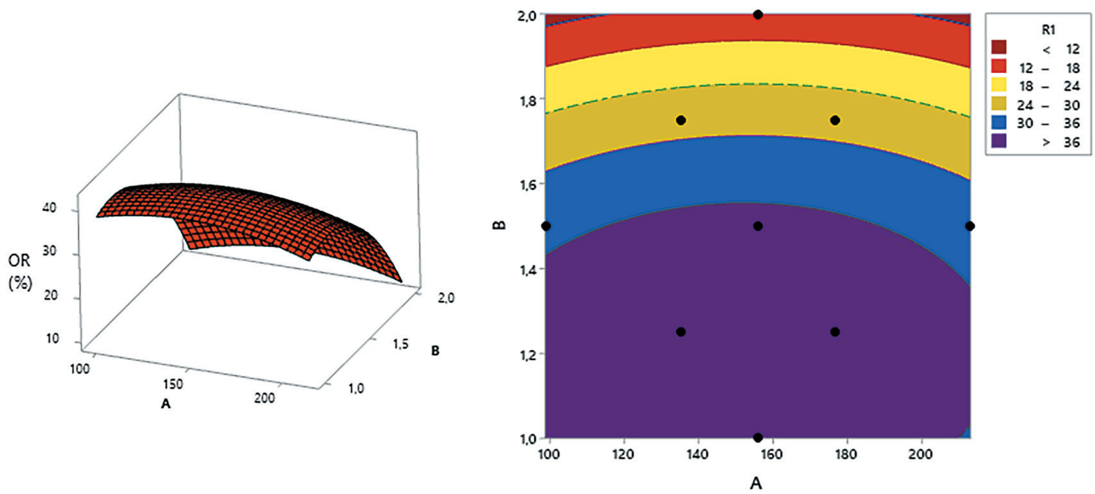
At slow screw speeds oil recovery increases but temperature of the oil and cake rises too due to the longer pressure effect between the screw and the inner surface of the cylinder plus friction among seeds. Therefore, we have chosen the aforementioned variables of the mechanical screw press machine for their optimization [27].

We selected a higher screw speed based on the assumption that the shorter pressing and shorter friction time would result in lower temperatures in the cylinder, thus leading to less oxidation and thermal effects on the oil, especially considering that flaxseed oil contains high levels of polyunsaturated fatty acids. Besides, it would prevent clogging within the pressing chamber. Our aim was the optimization of higher screw speeds along with the various sizes of the cake outlet, considering that the [21] did not impose any limitations on the quality characteristics of oil at the lower screw speeds or the higher process temperatures.

The Table 2 shows the experimental and predicted responses of the screw pressing of flaxseed oil under the considered conditions. According to experimental results, the highest oil recovery was 41.4% at a screw speed of 155.89 rpm (A), and at the size of the cake outlet of 1 mm (B) (Table 2 No. 4). Prediction model suggested different values on both A and B values (135.11 rpm and 1.25 mm respectively) (Table 2 No.12), what would have yielded a 41.45% oil recovery in the trial run No. 12 (where, under experimental conditions, OR% accounted to 39.8%).

Although the overall trend in oil recovery showed a rising yield behavior when screw speeds increased from 98.73 up to 213.05 rpm, fluctuations were observed in the oil recovery with varying the size of the cake outlet. However, it's interesting that no significant effects of the screw speeds on the oil recovery were observed (Table 3). However, there was an inversely-proportional correlation between the oil recovery and the size of the cake outlet. This may be due to the linear significant effect of the size of the cake outlet on the oil recovery ($p < 0.05$) (Table 3), caused by changes in friction forces; probably due to the pressure increase in the third zone of the cylinder, which is the closest to the cake outlet. In summary, screw speed provided a minor effect while outlet cake size provided a strong effect on the oil recovery (Figure 2 (a) and (b)). This could be due to the moisture content of the seed which may affect the interaction between friction force and rate of sliding of the seed material inside of the cylinder at the tested screw rotation speeds. These results are consistent with the previous reports of a similar study to optimize the sesame seed oil extraction by screw-pressing [28]. Therefore, the practical meaning of RSM findings (Figure 1) is that despite preserving the other conditions of the process, the changes in the screw pressing parameters during extraction may give the optimal percentages of the oil recovery. As shown in the Table 2, the oil recovery rate was the highest in the trial run No. 4, and the lowest one was observed in the trial run No. 6 (14.88%). It is worth mentioning that during the experiment, two jammings occurred (in the run No. 4 and No. 12), which could have been caused by the higher temperatures during the process.

The aim of this study was to maximize the OR% by adjusting conditions at the flax screw press operation. Other studies have reported the higher recoveries of the oil after the optimization of conditions for screw pressing of the vegetable materials. At their study, Marcela et al obtained 71.1 ± 2.8% OR in sesame seed, after adjusting screw press speed, the size of the cake outlet and moisture content [29]. For obtaining green juice from cassava at a recovery percentage of 81%, Latif et al found the optimal conditions at 18 rpm screw speed, and outlet diameter of 4 mm [15]. However, those studies were performed on different vegetable materials,




a) b)

Figure 2. **Effects of the screw speed and the size of the cake outlet on flaxseed oil recovery at the pilot screw pressing plant (surface (a) and contour plot (b))**

Рисунок 2. **Влияние скорости вращения шнека и размера отверстия для выхода жмыха на объем извлекаемого льняного масла на опытной промышленной шнековой пресс-установке для отжима масла (поверхностная (а) и контурная диаграмма (b))** as well as at pilot-scale equipment with high screw speeds, thus, results may not be easily transferred to the flaxseed oil obtained from flaxseed meal. According to the model, for our study, the optimal predicted conditions are as follows: the screw speed is 148.38 rpm, and the size of the cake outlet is 1.17 mm (with a predicted oil recovery of 41.91%).

*3.2. Oil quality and characteristics*

PV (peroxide value) and AV (acid value) are commonly used as the quality indicators of the vegetable oils. Hydroperoxides are primary oil oxidation products formed during the initial stages of oxidation and they are toxic to human health, whereas AV measures the amount of secondary oxidation products which contribute to unpleasant flavors and odors in oil. PV and AV should be within specific limits to ensure that oil quality is good enough for its safety consumption [21]. After changing the different variables in the experiment, the average temperature of the oil changed as well (Table 2); however, although indicators of oil quality such as AV and PV did show significant differences among the various runs, specified in the Table 2, they still remained within the limits specified at the [21].

Sediments content (SC) of oil, one more important factor which may be considered in oil consumption, varied from 9.12 to 14%. The highest value, in the run No. 4, may have been related to the higher internal friction force caused by a smaller size of the cake outlet; thus, the number of fine particles within the oil may have increased.

Omega-6 and omega-3 fatty acids are both essential for human health; however, their intake should be balanced. According to several studies, the proper omega 6/omega 3 ratio should vary between 1:1 and 4:1, as it was in the diet of our ancestors in the pre-industrial period (paleodiet). However, the current modern diet contains an extremely high amount of omega-6, where omega 6/omega 3 ratio may reach 20:1 or more. This is explained by the high consumption of red meat, vegetable oil, and highly processed food which contains omega-6 in very high quantities, plus a lack in the consumption of omega-3 rich foods such as fish. An imbalance in the intake ratio of omega 6/omega 3 can cause negative consequences for health, as omega-6 fatty acids are the precursors of inflammatory molecules, while omega-3 are the precursors of anti-inflammatory molecules. As a consequence of this unbalanced ratio, the risk of cardiovascular disease, diabetes, arthritis, Alzheimer's, and some types of cancer, has increased. Therefore, it is favorable to have a low ratio in our diet, as it may help reduce inflammation and cure some chronic diseases [30]. Flaxseed oil is known for its low omega 6/omega 3 ratio, thus a potential functional food with health improving properties [3,4,31]. However, the high amount of unsaturated fatty acids may make it sensitive to oxidation and inclined to degradation [3]; therefore, the effect of temperature during the extraction on the omega-6 and omega-3 fatty acids on flaxseed oil obtained by screw pressing was evaluated by analyzing the fatty acid profile of oil obtained from the run No. 4, where temperature reaches its maximum.

While typically the temperature of oil extracted by cold pressing and screw pressing should not exceed 50 °C and 70 °C, respectively [10]; the oil, obtained from the run No. 4 reached a high temperature of 102 °C. However, its fatty acid profile, as determined by GC–MS and compared to the oil extracted by supercritical fluid extraction (SFE) at 50 °C, 30 MPa, showed that in both cases, polyunsaturated fatty acids (PUFA) are the most abundant acids, followed by saturated fatty acids (SFA), and finally the list of acids is closed with the monounsaturated fatty acids (MUFA); plus, ALA as the most abundant individual fatty acid, with a percentage of over 60% in both cases. When compared to the fatty acid profile obtained by cold pressing of flaxseed [4], the results are similar as well, regarding ALA as the most abundant individual oil, and PUFA as the most abundant type of oil in general (Table 4). However, in this last case, it is not possible to compare these profiles, because the source of flaxseed used for oil extraction was different.

Table 4. **Fatty acid profile (in area percentage of total chromatogram). * refers to [4]**

Таблица 4. **Профиль жирных кислот (в процентах площади от общей хроматограммы) *[4]**

| Fatty acid | Cold extraction* | Run No. 4 | SFE |
|---|---|---|---|
| Palmitic | 6.95 | 8.17 | 8.92 |
| Linoleic | 15.2 | 9.56 | 8.45 |
| Linolenic | 52.58 | 63.64 | 68.83 |
| Oleic | 19.98 | 10.89 | 8.57 |
| Stearic | 5.2 | 7.44 | 4.56 |
| Behenic acid | 0.07 | 0.3 | 0.05 |
| Arachidic | 0.02 | 0 | 0.62 |
| SFA | 12.24 | 15.91 | 14.15 |
| MUFA | 19.98 | 10.89 | 8.57 |
| PUFA | 67.78 | 73.2 | 77.28 |
| omega 6/omega 3 | 0.28 | 0.15021999 | 0.12276624 |

Although the effect of temperature on the fatty acid profile in all cases should be studied, we can still conclude that the high temperature of oil at the outlet did not affect the major quality parameters of oil in the run No. 4 regarding both parameters of food quality as it was defined in the Codex Alimentartius (AV and PV) [21], or the fatty acid profile, specifically related to the ratio of omega 6 to omega 3.

## 4. Conclusions

RSM was applied to optimize the parameters of flaxseed oil screw pressing. The optimal experimental and mathematically predicted values of screw speed and the size of the cake outlet were defined as 155.89 rpm, 1 mm and 148.38 rpm, 1.17 mm respectively. Sediments content in the obtained oil was relatively high. The oil quality parameters were changed by the modifying of the process variables, although they remained within the specifications defined by the Codex Alimentarius; while the fatty acid profile related to potential health benefits on its omega-6 to omega-3 still remained favorable even in the worst-case scenario in terms of temperature.

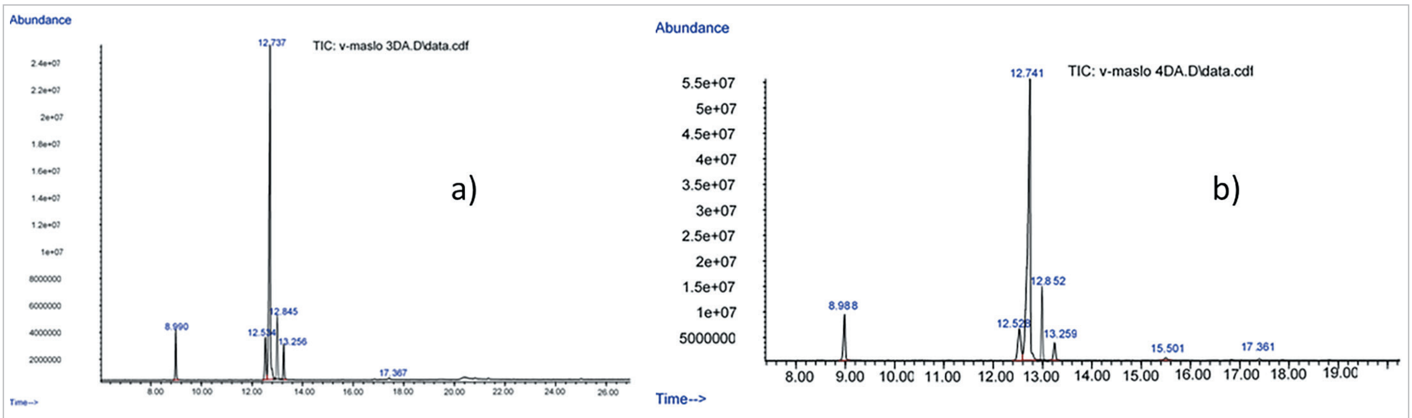



Figure 3. **Chromatograms of the flax seed oils: a) run No 4; and b) SFE**

Рисунок 3. **Хроматограммы масел из льняного семени: а) опыт № 4; и b) СФЭ**

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