Research of the process of pressing black cotton oil from oil fooses

Yuldosh Savriev, Karim Gafurov and Ulugbek Sevinov
Bukhara Engineering Technological Institute, Bukhara, Uzbekistan

E-mail: kgafurov@yahoo.com

Abstract. In the process of production of vegetable oils, insoluble impurities are removed from the black oil after pressing using sludge traps and filters. Oil sludge separated from the forepress oil has an oil content of up to 50%. For the complete extraction of this oil from the sludge from the sludge separator and filter presses, it is recycled into the upper vats of the roaster of the press units and further for pressing. At the same time, the returnable sludge affects one of the main characteristic of the obtained cottonseed oil – the acid number. Our research has shown that with such a scheme of sludge processing, the sludge acid number obtained after prepressing is 5 mg KOH. This leads to a significant loss of oil to soap stock during oil refining. The solution to this problem is to prevent the sludge separated on the separator from falling back into the frypot, instead the sludge is pressed to produce black cottonseed oil. The degree of influence on the pressing process of influencing factors – the percentage of sludge in the mixture and the duration of mixing the sludge and husk of the mixture - is experimentally studied in this research work. The rational values of these factors are determined. The planning of a two-factor experiment was carried out in order to reveal the effect of the interaction of these factors. The obtained regression equation allows determining with sufficient accuracy the residual oil content of the cake in the investigated range of changes in the influencing factors, which is necessary to optimize the process.

1. Introduction
Currently, the production of vegetable oils is growing all over the world. Therefore, according to [1], the production of vegetable oils (in million tons) in 2016/2017 amounted to 183; in 2017/2018 to 193.3; in 2018/2019 to 198.1; and in 2019/2020 (forecast) to 200.9.

The main volume of vegetable oils production falls on palm and soybean oils. In total, they form 63% of the total world production. The share of cottonseed oil is 2%, which is 4.018 million tons in 2019/2020.

Cottonseed oil is a liquid, which has color from yellow to red-brown. It is a mixture of acid glycerides with the following composition: unsaturated acids - up to 44% linoleic, 34-44% linolenic, 23-35% oleic; saturated acids - 20-22% palmitic, 1-2% stearic, 0.3-0.5% myristic, and 0.1-0.6% arachidic. It also contains up to 3% of saponifiable and unsaponifiable substances, i.e. phospholipids, waxes and waxy substances, sterols, tocopherols (81 mg per 100 g of oil), proteins, carbohydrates, pigments, in particular the toxic pigment gossypol (0.14-2.5%). Useful properties are due to the presence of vitamins B, E, PP in its composition. In addition, monounsaturated and unsaturated acids will enrich the body with beneficial Omega-3 and Omega-6 [2].
According to [3], the world leader in the production of cottonseed oil is China with a production volume of 1396 thousand tons per year. Uzbekistan ranks 6th in the world with a production volume of 30 thousand tons per year.

The technological scheme in the oil extraction plants of Uzbekistan includes preliminary oil degreasing by means of pre-pressing followed by its extraction by extraction with gasoline [4].

The presence of insoluble impurities in vegetable oils obtained after pressing impairs their quality, since oxidative and hydrolytic processes on the surface of the particles proceed faster than in bulk. Therefore, in the process of production of vegetable oils, they strive for the quick and possible complete removal of insoluble mechanical impurities from the oil (filtration) using oil sludge traps and filters.

Oil sludge separated from the forepress oil has an oil content of up to 50%. For the complete extraction of this oil from the sludge separator, it is fed into the re-processing in the upper vats of the roaster of the pressing units and further for pressing. Oil sludge from filter presses is also fed to the upper vats of the roaster of the pressing units for recycling and pressing. At the same time, the returnable sludge affects the acid number – one of the main characteristics of the obtained cottonseed oil.

Our research has shown that with such a processing scheme, the sludge acid number obtained after prepressing is 5 mg KOH. This leads to a significant loss of oil to soap stock during oil refining.

The solution to this problem is to prevent the sludge separated on the separator from falling back into the roaster instead the sludge is pressed to produce black cottonseed oil.

Below are given the definitions of raw materials and half-finished oilseed products [5]:

Oilseeds are seeds and fruits of oil plants, oil-containing waste of essential oil and canning industries, used for the industrial extraction of edible and technical vegetable oils.

Oilseed oil is an intermediate product obtained by crushing an oil kernel or oil raw materials.

Oilseed pulp is an intermediate product obtained in the process of frying oilseed meal or prepress cake.

Oilcake is a product obtained after pressing vegetable oil on presses of various designs from prepared oilseed material.

Oilseed sludge is mainly sediments formed in vegetable oils filtered after their extraction in forpresses from oilseeds or tank sludge released from vegetable oil during its storage and consisting of phosphatides, oil, and moisture and protein impurities.

The settled sludge contains 66-72% oil and 9-21 - phosphatides, filtered - 44-50 and 9-21%, respectively, 3-6% protein; hydration - 35-50% oil, 20-35 - moisture and 15-45% phosphatides. The moisture content of the filtered sludge is 24-36% [6].

During pressing oil-containing crops, the structure of the pulp has a great influence. The structure of the pulp for pressing should ensure plasticity, elasticity, ensure good briquetting of the hush, and develop a sufficiently high pressure during pressing without the pulp coming out of the mills and at the same time obtaining the maximum specified oil content, convergence of the outer and inner surfaces of the pulp particles.

The efficiency of the pressing process depends on the physical and mechanical properties of the material being pressed and the conditions for organizing the pressing. When pressing under the action of external pressure on the material to be pressed, as it moves along the screw shaft in the grain chamber, the external and internal particles of the material will converge, which contributes to the squeezing of the liquid phase from the gaps between the particles, then from the particles themselves. As a result, there is a change in the density, porosity, permeability of the pulp, a change in the physical characteristics of the resulting oil.

Oil sludge itself cannot be pressed due to its unsuitable physical and mechanical characteristics. For pressing, it is necessary to change its characteristics by mixing with the husk of cottonseeds.

The purpose of this research is to determine the rational values of the percentage composition of the mixture, consisting of the husk of cotton seeds and oilseed sludge, to determine the duration of mixing the mixture, providing the maximum yield of black oil after pressing.
The following task of experimental studies follows from the above: determination of the most rational ratio in the percentage ratio of the components of the mixture “oil sludge- cotton seeds husk” and determination of the duration of mixing the mixture to create a uniform distribution of components in the volume, providing the maximum oil yield (minimum oil content in the cake) after pressing.

2. Material for experiments

The following materials were used for the experiments: oil sludge separated at the sludge separator of the press shop of the plant “Evrosnar” LLC of Jondor district of Bukhara region with an oil content of 53% and a moisture content of 25%. Cottonseed husks with a moisture content of 10% (produced on April 15 - 25, 2019).

3. Research methods

Experimental studies were carried out using modern measuring instruments in laboratory and industrial conditions, the planning of experiments and the adequacy of the expression of the regression equation were analyzed using the methods of mathematical statistics.

The oil content of the oil sludge and cake, as well as the moisture content of the sludge and husks were determined by known methods [7, 8].

The main influencing factors on the process are the percentage ratio of the components “oil sludge-cottonseed husk” in the mixture (in%) and the duration of mixing (in minutes), since the structure of the pulp depends on these factors, which affects the efficiency of the press, which determines the residual oil content of the cake (black oil yield).

The first single-factor experiment to determine the rational ratio of the mixture “oil sludge-cottonseed husk” was carried out on a laboratory mixing plant of the horizontal type for mixing the components of the mixture “sludge-husk”. In this case, the amount of sludge in the mixture was kept equal to 10 kg, and the amount of husk in the mixture varied from 2.5 to 23.1 kg, which corresponds to a change in the percentage of sludge in the mixture from 80% to 25% (see table 1). The mixing time for each experiment was taken equal to 30 minutes. Each experiment ended by pressing the resulting mixture in an experimental pressing unit to obtain oil under the same pressing conditions and determining the residual oil content of the cake.

The experimental pressing unit consists of a working chamber, a pressing auger and a drive part, the design and operating principle of which are given in [9].

The results of experiments on the identification of a rational ratio “oil sludge-cottonseed husk”, taking into account the residual oil content of the cake coming out of the press as an output parameter, are shown in table 1.

From the data in table 1 it can be seen that a change in the mixture of the ratio of the husk of cotton seeds and oil sludge significantly affects the residual oil content of the cake obtained after pressing. As the amount of husk increased to 67.2% in relation to the oilseed meal, the residual oil content of the cake obtained after pressing decreased to 8.05%, and a further increase in the added amount of husk more than 70% showed an increase in the residual oil content, for example: when 75% husk was added, the residual oil content of the cake was 8.9%.

Table 1. Results of experiments on identification the rational ratio “high-oil sludge - cottonseed husks”.

| No of the experiment | Amount of husks in the mixture, (kg) | Amount of sludge in the mixture, (kg) | Total quantity of the mixture (kg) | Percentage ratio of husks in the mixture, % | Percentage ratio of sludge in the mixture, % | Residual oil content cake, % |
|----------------------|-------------------------------------|--------------------------------------|-----------------------------------|-------------------------------------------|------------------------------------------|-----------------------------|
| 1                    | 2.5                                 | 10                                   | 12.5                              | 20                                        | 80                                       | 28                          |
| 2                    | 4.3                                 | 10                                   | 14.3                              | 30                                        | 70                                       | 27                          |
| 3                    | 6.7                                 | 10                                   | 16.7                              | 40                                        | 60                                       | 22                          |
| 4                    | 10                                  | 10                                   | 20                                | 50                                        | 50                                       | 18                          |
The second single-factor experiment was carried out on mixing the components of the “sludge-husk” mixture to determine the rational duration of the process, contributing to the uniform distribution of the constituent components throughout the mixture. In this case, the percentage ratio of the components of the “sludge-husk” mixture is taken 67.2% - 32.8%.

The results of experimental studies to determine the duration of mixing the mixture are shown in table 2; the main output parameter is the minimum residual oil content of the cake after pressing.

| No of the experiment | Time of mixing (minutes) | Residual oil content of the cake after pressing, (%) |
|----------------------|--------------------------|------------------------------------------------------|
| 1                    | 10                       | 18.00                                                |
| 2                    | 20                       | 12.00                                                |
| 3                    | 30                       | 8.050                                                |
| 4                    | 40                       | 8.036                                                |
| 5                    | 50                       | 8.036                                                |

From preliminary experiments, the boundary values of the influencing factors were determined:

- The first factor is the percentage of sludge in the mixture. Lower level is \( x_{1\text{min}} = 25\% \); upper level is \( x_{1\text{max}} = 35\% \).
- The second factor is the duration of mixing. Lower level is \( x_{2\text{min}} = 30\text{ min} \); upper level is \( x_{2\text{max}} = 40\text{ min} \).

In the general case, an experiment in which all possible combinations of factor levels are realized is called a complete factorial experiment.

For the first factor, the center of the experiment is:
\[
C_{10} = \frac{C_{1}^{+} + C_{1}^{-}}{2} = \frac{35 + 25}{2} = 30\%
\]

(1)

For the second factor, the center of the experiment is:
\[
C_{20} = \frac{C_{2}^{+} + C_{2}^{-}}{2} = \frac{40 + 30}{2} = 35\text{ min}
\]

(2)

For the first factor, the variation interval is:
\[
\lambda_{1} = C_{1}^{+} - C_{10} = 35 - 30 = 5\%
\]

For the second factor, the variation interval is:
\[
\lambda_{2} = C_{2}^{+} - C_{20} = 40 - 35 = 5\text{ min}
\]

During drawing up even plans for multifactorial and multilevel research, the dimensionless value of the factors is obtained by the formula:
\[
x_{iu} = \frac{C_{iu} - C_{i0}}{\lambda_{i}}
\]

(3)

where \( C_{iu} \) is the value of \( i \)-factor in \( u \)-experiment [10].
According to (1), (2) and (3) we will find the numerical values of the upper $x_i^+$ and lower $x_i^-$ levels of factors in a dimensionless expression [10]:

$$x_i^+ = \frac{c_i^+-c_{i0}}{\lambda_i} = \frac{c_i^+-c_{i0}}{c_{i0}-c_i} = +1;$$  

(4)

$$x_i^- = \frac{c_i^-+c_{i0}}{\lambda_i} = \frac{c_i^-+c_{i0}}{c_{i0}-c_i} = -1.$$  

(5)

First, the plans of the FFE$2^n$ are written in dimensionless expression of the magnitude of the factors, and then a working plan is made using them in the natural dimension of the factors.

The number of experiments of FFE$2^n$ plans corresponds to the number of combinations of $n$ elements when they are changed at two levels:

$$N=2^n.$$  

(6)

For our two-factor experiment, the number of experiments is $N=2^2 = 4$.

The conversion of the factor value into natural dimension is carried out taking into account (3) according to the formula

$$C_{iu} = C_{i0} + \lambda_i x_{iu}.$$  

(7)

### 4. Processing of experimental results

The results of the experiments are shown in table 3. As the output of the process, we consider the residual oil content of the cake after pressing $y$, %.

Based on the results of the two-factor experiment, we will compose an equation in which, in addition to the linear terms, there will be a term that takes into account the effect of pair interfactorial interaction.

| No of the experiment | Factors | Interaction effects of factors | Experimental results | average of the results |
|----------------------|---------|--------------------------------|----------------------|-----------------------|
|                      | $x_1$   | $x_2$ | $x_1x_2$ | $y_1$ | $y_2$ | $y_3$ | $\bar{y}_j$, % |
| 1                    | -       | -    | +        | 12.7  | 13.2  | 14.1  | 13.333          |
| 2                    | -       | +    | -        | 10.5  | 12.2  | 11.7  | 11.466          |
| 3                    | +       | -    | -        | 8.4   | 7.54  | 8.2   | 8.0466          |
| 4                    | +       | +    | +        | 18.3  | 18.7  | 17.5  | 18.166          |
|                      |         |      |          | $\Sigma \bar{y}_j$ | 51.01 |

The regression equation in this case is as follows:

$$y = b_0 + b_1 x_1 + b_2 x_2 + b_{12} x_1 x_2$$  

(8)

The coefficients of the regression equation were determined by the least squares method. The calculation results are shown in table 4.

| Coefficients | $b_0$ | $b_1$ | $b_2$ | $b_{12}$ |
|--------------|-------|-------|-------|----------|
| Values       | 12.75 | 0.35  | 2.06  | 2.99     |

The significance of these coefficients was determined by the Student's test [6]. In this case, we obtain that all the coefficients are greater in absolute value $|b_j|$. Therefore, all coefficients are significant.

Then the regression equation (8) is as follows:
The adequacy of the obtained regression equation was checked using Fisher’s criterion [10]. Converting the values of the factors into natural dimensions and, according to formula (9), using the MatCAD program, we built a graph of the dependence of the process output on the percentage of sludge in the mixture and the duration of mixing (figure 1).

\[
y = 12.75 + 0.35x_1 + 2.06x_2 + 3.0x_1x_2 \tag{9}
\]

5. Conclusion
The obtained regression equation allows determining with sufficient accuracy the residual oil content of the cake after pressing in the investigated range of factors. Using this equation allows to identify the degree of influence of each investigated factor on the result, which is necessary to optimize the process.

As can be seen from the graph (figure 1), with the values of the influencing factors (the percentage of sludge in the mixture \(x_1=33\%\) and the mixing time \(x_2=40\) min) the residual oil content of the cake will be the smallest (8.0%).

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