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Study of the mechanism and efficiency of using mineral-organic bleaching-filtering agents in polishing refining

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<p>Received: January 20, 2026 Peer-reviewed: May 23, 2026 Accepted: June 11, 2026</p>	<p>ABSTRACT In this scientific study, the main objective is to purify vegetable oils stored in the state reserve for an extended period to reach consumable quality based on their physicochemical parameters. In the experiments, polishing refining of cottonseed oils stored for 6 months or longer was carried out. By analyzing the physicochemical parameters of polishing-refined vegetable oils, selective bleaching-filtering agents (BFAs) with various pore structures were obtained for use in the bleaching-filtration stage. The BFAs were obtained through acid activation of bentonitic and opoka-type clays, as well as pyrolysis of sunflower husk. The filtration capacities of each activated BFA toward polishing-refined oils were investigated. The findings show that the highest efficiency was achieved when a composite BFA of the BK grade-prepared at a 1:1 mass ratio of acid-activated Navbahor alkali bentonite (ANAB) and pyrolyzed sunflower husk (P-SH) was added at 1.5-2.0% relative to the oil mass. The research results demonstrate that a composition consisting of polar organic and non-polar inorganic components was successfully obtained, capable of filtering fine soapstock particles while not resisting pressure buildup during the filtration process.</p>
	<p>Keywords: bentonite clay, opoka-type clay, sunflower husk, BET analysis, activation, pyrolysis, composition, polishing refining, bleaching-filtering agent.</p>
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Introduction

Scientific research is being conducted to improve the technologies for producing natural and synthetic sorbents in various fields of the chemical

and food industries. In this regard, special attention is given to improving and testing technologies that utilize modified sorbents possessing micro, meso, and macroporous structures and capable of forming ionic bonds for the removal of coloring

substances from food products and the purification of industrial wastewater in the chemical sector [[1], [2], [3], [4], [5]].

In 2019-2020, global cotton production reached 43 million tons [6], of which Uzbekistan accounted for about 3 million tons. Cottonseed oil belongs to the group of vegetable oils that require full refining.

Refining methods are selected based on the type of oil-bearing raw material and the extraction process used. Oils obtained from low-grade seeds require multi-stage refining procedures [[7], [8], [9]].

Chlorophyll, xanthophylls, carotenoids, gossypol, and soapstock formed during the processing of vegetable oils-along with other soap-like substances, free fatty acids, and peroxides-possess diverse physicochemical properties and vary in their chemical nature. Depending on the nature and concentration of these substances, vegetable oils are refined either through a complete or partial refining cycle [[10], [11], [12], [13]].

Depending on the type of oily raw material and the method of oil extraction, vegetable oils contain various accompanying substances. These properties include the content of phosphatides, moisture and volatile substances, iodine value, color index, refractive index, and saponification value, among others [6].

Cottonseed oil contains biologically active compounds such as antioxidants-including tocopherols, sterols, and flavonoids-as well as a yellow-colored pigment known as gossypol.

The bleaching stage of the vegetable oil refining cycle requires the use of selective sorbents that act through targeted adsorption.

Various mineral-and organic-based adsorbents are used during the bleaching stage [[14], [15]].

Scientific sources have examined the physicochemical mechanisms of the pyrolysis process of sunflower and peanut shells at temperatures of 280 and 350 °C, as well as the effects of the resulting solid carbon-based mass on soil formation and seed germination [16].

Natural mineral sorbents are mineralized clays with high adsorption or ion-exchange capacities, which include natural bentonite, siliceous (opoka) clay, and other similar materials [[4], [14], [17]].

Natural clays are activated through mechanical, physical, and chemical methods. Such treatments modify the physicochemical properties of the clay and enhance its adsorption capacity [[18], [19]].

During the storage of vegetable oils, primary and secondary oxidation products are formed,

which can have adverse effects on the human body [20].

In the first stage of the oxidation process, homolytic cleavage of the covalent bonds within the C-H hydrocarbon chains occurs, resulting in the formation of free hydrogen radicals and free fatty acid radicals. Hydroperoxides, which are primary oxidation products found in vegetable oils, enter the oil as a result of enzymatic processes [[21], [22]].

During the storage of vegetable oils, factors such as temperature and ultraviolet radiation contribute to the degradation of fatty acid molecules. At subsequent stages, oxidation proceeds in a chain mechanism, and the first organoleptic changes in the oil become noticeable. [[23], [24]].

All types of vegetable oils undergo oxidation due to the presence of accompanying substances. Oxidation products, in turn, lead to changes in the standard physicochemical parameters of the oil and a decrease in its nutritional value. Under the influence of internal and external factors, the hydrogen atom adjacent to the double bond of unsaturated fatty acids is removed, resulting in the formation of highly reactive free radicals possessing unpaired electrons. These radicals interact with fatty acid molecules and initiate a new chain oxidation process [25].

The increase in acid value and peroxide value in vegetable oils is attributed to the formation of primary oxidation products. However, secondary oxidation products may also form simultaneously with the primary ones, which ultimately leads to the deterioration and unsuitability of the oil for use [26].

The authors extracted oil from flax and hemp seeds using the cold-pressing method, determined changes in parameters such as acid value, peroxide value, and anisidine value in oils stored at room temperature for 6 months, and analyzed their oxidation kinetics using differential scanning calorimetry (DSC). They emphasized that vegetable oils obtained through cold pressing are highly prone to oxidative degradation [27].

The studies also examined the extraction of oil from non-traditional oilseeds and the physicochemical changes occurring during their storage. In particular, an increase in peroxide and acid values was observed during long-term storage of peanut oil. The rise in these compounds imparts a bitter taste to the oil and leads to the formation

of further oxidation products, which may even contribute to the development of certain diseases [28].

Physicochemical methods are used to remove accompanying substances from all types of vegetable oils. The principles of physical and chemical refining methods, as well as their adverse effects on oils, have been extensively described by numerous authors [[29], [30]].

Vegetable oils stored for long periods typically exhibit elevated acid values, and the micro-sized soapstock particles formed during the polishing-refining stage tend to clog the pores of the filter cloth, thereby hindering oil flow. In this study, to eliminate this problem, the possibilities of obtaining and applying BFAs capable of retaining soap-like substances were investigated.

During the filtration process, the pores of the filter cloth are capable of allowing particles up to a certain size to pass through. Various types of filtration equipment are used for this purpose, including both vacuum and non-vacuum systems, operating in either batch or continuous modes.

The filtration process is carried out using fabric, non-fabric, and metal mesh filters, as well as filtration aids-also known as filtering agents-that facilitate the process. The filtration surface determines the primary geometric characteristics of the filter. The filtration surface consists of a filtering barrier whose porosity directly influences the permeability and overall performance of the filter. The following section presents the results of our studies on the use of bleaching-filtering agents with varying adsorption activities in the filtration of vegetable oils and the removal of soap-like substances after the polishing refining process.

Materials and Methods

Materials

The raw materials used in this study included bentonite clay obtained from the Navbakhor deposit and opoka-type clay collected from the Karmana deposit, both located in Uzbekistan. Sunflower husk was utilized as a pore-forming organic additive. Distilled water was used for sample preparation and processing. A 15 wt.% sulfuric acid (H₂SO₄) solution was employed for the acid activation and modification of the clay materials. All chemicals and reagents were of analytical grade and were used without further purification.

Our experiments were conducted using 10 liters of cottonseed oil obtained from the state reserve, which had been stored for more than six months. The physicochemical characteristics of the oil used in the experiments are presented in Table 1 below.

To bring the oil samples with the above quality characteristics to the level required for polishing refining, the free fatty acids were first neutralized with a 250 g/l caustic soda solution, using an excess alkali dosage of 0.5%. During the neutralization process, the temperature was maintained at 55 °C. The resulting soapstock was precipitated and separated using a saline (water-salt) solution. The physicochemical characteristics of the polished refined oil are presented in Table 2 below.

Table 1 - Physicochemical characteristics of long-term stored cottonseed oil

Sample	Acid value, mg KOH/g	Red units	Transparency	Peroxide value, mmol ½O ₂ /kg	Qualitative test for soap content	Content of moisture and volatile substances, %
Cottonseed oil	0.6	8	Clear	14	Not available	0.21

Table 2 - Physicochemical characteristics of polished refined cottonseed oil

Sample	Acid value, mg KOH/g	Red units	Transparency	Peroxide value, mmol ½O ₂ /kg	Qualitative test for soap content	Content of moisture and volatile substance, %
Cottonseed oil	0.15	8	Turbid	4.2	Available	0.11

As seen from the table data, both the acid value and peroxide value of the oil decrease several times during the polishing refining process. Although these values are close to standard parameters, they are still considered relatively high for oils undergoing secondary refining. In addition, the turbidity of the oil and the results of the qualitative test for soap content indicate difficulties in removing micro-sized impurities from the oil using primary processing methods such as sedimentation or simple filtration. Based on the above findings, it is necessary to explore the potential for developing specialized bleaching-filtering agents to remove micro-sized impurities from polished refined oils.

Obtaining whitening-filtering agents

Our subsequent experiments focused on the activation of bentonite and opoka-type clays, which served as the primary research objects for obtaining bleaching-filtering agents (BFAs). During the sulfuric acid activation of bentonite and opoka-type clays, optimal results were achieved using a 15% acid solution. A hydromodule ratio of 1:2.5 (clay to acid solution) was selected, and activation was carried out with an acid consumption of 40%. The resulting suspension was washed with distilled water until the pH value reached 4. The resulting clay mass was first air-dried under ambient conditions and then further dried in a drying oven at a temperature of 110-120 °C for 2 hours. Considering the effect of particle size on sorption properties, the dried samples were sieved until 90% of the material passed through a 56 µm mesh (10,000 openings per/sm²).

To produce a microporous, carbon-based organic sorbent, sunflower husks were ground to a particle size of up to 1 mm and subjected to thermal treatment (pyrolysis) at 600 °C in an oxygen-free environment. Based on the study of the sorption properties of the obtained solid carbon-based samples, a composite bleaching-filtering agent (BFA) was developed using activated bentonite and pyrolyzed sunflower husk.

During the filtration of polished refined oils using BFAs, the reagent was added at a dosage of 0.5-2.0% relative to the oil mass and stirred for 15-30 minutes at 60 °C. Filtration was carried out in frame filters at a pressure of 0.1-0.5 MPa using a filter cloth compliant with State Standard 332-91. During the process, changes in flow rate with increasing filtration pressure were monitored, and the operation was stopped when the pressure exceeded 0.5 MPa and the flow rate dropped below 0.02 kg s/m². Under these controlled parameters, after 10 hours of filtration, the thickness of the sediment layer on the filter surface reached 10 mm.

The moisture content of cottonseed oil was determined by the gravimetric method according to ISO 662:2016. The color of the oil was measured in red units at a constant 35 yellow units using a Lovibond Tintometer (Model E) with a 13.5 sm cuvette.

The acid value was determined by the titration method in accordance with ISO 660:2020.

The peroxide value was determined by the iodometric titration method in accordance with ISO 3960:2020.

Sorption pores were determined in accordance with IUPAC requirements.

Analysis of specific surface area and porosity was carried out using the method of low-temperature nitrogen adsorption on a Micromerelies Gemini VII 2390t analyzer (USA).

Results and Discussion

The main objective of our study is to remove micro-sized soapstock particles remaining in the oil after polishing refining and to reduce the acid and peroxide values to standard levels, which requires the preparation of effective bleaching-filtering agents (BFAs).

To obtain BFAs for filtering polished refined oils, the sorption properties of Navbahor alkaline bentonite and opoka-type clay from the Karmana deposit were investigated. Their sorption indicators are presented in Table 3 below.

Table 3 - Sorption characteristics of natural bentonite and opoka-type clays

Parameters	NAB	OG
Specific surface area (BET), m ² /g	43.52	153.25
Specific surface area of micropores, m ² /g	2.07	-
Specific surface area of meso- and macropores, m ² /g	41.45	153.25
Pore volume, cm ³ /g	0.014	0.262
Average pore diameter, Å	12.94	98

NAB – Navbahor alkaline bentonite; NOA – natural opoka-type clay.

Analyzing the data presented in the table, it can be observed that the natural bentonite clay exhibits a relatively small specific surface area of 43.52 m²/g, a specific surface area of micropores of 2.07 m²/g, and an average pore diameter of 12.94 Å. Although the opoka-type clay has a relatively high specific surface area of 153.25 m²/g, it lacks significant microporosity, with an average pore diameter of 98 Å. Given that alkali-treated, polished refined oils contain colloidal and micro-sized soapstock particles, the sorption characteristics of these two clays alone are insufficient to yield the desired performance when used as bleaching-filtering agents. Our experiments revealed that acid activation of these clays not only increases their specific surface area but also promotes the formation of micropores within the macro and mesoporous structure under the influence of acid treatment, thereby enhancing the characteristics required for our intended application.

Taking the above considerations into account, the bentonite and opoka-type clays were activated

using a 15% sulfuric acid solution. The obtained results are presented in Table 4.

Table 4 - Sorption characteristics of BFAs obtained through acid activation

Parameters	ANAB	AOC
Specific surface area (BET), m ² /g	176.38	164.36
Specific surface area of micropores, m ² /g	15.25	4.70
Specific surface area of meso- and macropores, m ² /g	161.13	159.66
Pore volume, cm ³ /g	0.108	0.384
Average pore diameter, Å	35.062	74

Acid-activated Navbahor alkaline bentonite, AOC-acid-activated opoka-type clay

The data presented in Table 4 show that although sulfuric acid treatment of bentonite clay resulted in a significant increase in its specific surface area, the majority of the pore structure consists of macro and mesopores (161.13 m²/g), while the surface area of micropores remains comparatively low at 15.25 m²/g. In the acid-activated opoka-type clay, these values were found to be 159.66 m²/g for macro-and mesopores, and 4.70 m²/g for micropores. Although an increase in acid concentration leads to the formation of additional micropores, it simultaneously causes a reduction in the overall specific surface area.

It can be concluded that although acid activation of various clay samples increases their specific surface area, the resulting materials are still insufficient for effectively filtering soapstock and colloidal-sized particles from the studied oils. According to the literature review, plants and plant-derived organic sorbents possess microporous structures.

Based on this information, it was concluded that it is necessary to obtain an organic-based sorbent. Therefore, sunflower husks were subjected to pyrolysis at 200 °C for 120 minutes in an oxygen-free environment. This process consists of several stages, including dehydration, decomposition, carbonization, and the formation of active (oxide) sites. During pyrolysis, the main structural components of the husk-cellulose, hemicellulose, and lignin-gradually decompose, resulting in a carbon-rich solid mass with a ring-shaped carbon structure.

Table 5 below presents the sorption characteristics of sunflower seed husks obtained through the pyrolysis method.

Table 5 - Sorption characteristics of pyrolyzed sunflower husk

Parameters	P-SH
Specific surface area (BET), m ² /g	132.14
Specific surface area of micropores, m ² /g	58.72
Specific surface area of meso and macropores, m ² /g	73.42
Pore volume, cm ³ /g	0.162
Average pore diameter, Å	33.1

P-SH-pyrolyzed sunflower husk

As shown in the table, the obtained organic-based sorbent possesses micropores with a specific surface area of 58.72 m²/g. At this stage, it becomes necessary to study the potential of the obtained organic-based sorbent with microporous structure for filtering long-term stored vegetable oils.

Experiments were conducted to determine the filtration efficiency of polished refined cottonseed oil using the sorbent obtained through the pyrolysis process. Table 6 below presents the results of filtering polished refined cottonseed oil using the sorbent described in Table 5, which possesses the specified sorption characteristics.

Table 6 - Results of filtering polished refined cottonseed oil using P-SH grade BFA

Parameters	P-SH dosage 1,0%	P-SH dosage 1,5%	P-SH dosage 2,0%	P-SH dosage 2,5%
Acid value, mg KOH/g	0.15	0.14	0.14	0.14
Red units	8	8	8	8
Transparency	turbid	turbid	turbid	turbid
Peroxide value, mmol ½O/kg	4.1	4.1	3.8	3.8
Qualitative test for soap content	Available	Available	not available	not available
Content of moisture and volatile substances, %	0.09	0.09	0.09	0.09

As seen from the table, when the P-SH grade BFA was added to the oil at dosages of 1%, 1.5%, 2%, and even 2.5% relative to the oil mass, the filtered oil did not show significant improvement in terms of acid value or peroxide value. At the initial stage of the filtration process, the filtration rate was relatively high; however, over time, a noticeable increase in filtration pressure and a sharp decrease in the filtration rate were observed.

This occurred because the P-SH grade organic-based BFA formed a compressible sediment layer on the surface of the filter cloth. All organic-based sediments belong to the category of compressible deposits.

If the particles in the filtration process possess non-deformable characteristics, they belong to the category of incompressible sediments, and an increase in pressure does not alter their resistance [31].

During the filtration process, an increase in pressure leads to deformation and compaction of particles. Such sediment is compressible, and over time this causes an increase in resistance and a decrease in filtration rate (Figure 1). These factors were taken into account in the present study.

On the one hand, despite the presence of micro-sized particles in the P-SH grade BFA, the soapstock particles tended to clog the pores of the filter cloth, which led to an increase in filtration pressure. On the other hand, the accumulation of soapstock particles on the surface of the filter cloth caused them to swell even under slight moisture, resulting in an increase in their volume. In both cases, the formation of a deformable sediment layer on the filter cloth surface reduced the filtration rate.

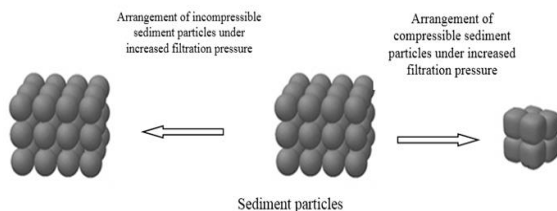


Figure 1 - Change in the void space between particles under the influence of pressure in the filter

It is well known that the permeability of the filter plays a crucial role in the filtration of vegetable oils. The filtration process can be described using the Strouhal criterion as follows (1).

$$S_t = \frac{f \cdot L}{V} \tag{1}$$

Where:

- f - frequency of pressure fluctuations (Hz)
- L - diameter of the filter cloth pores (μm)
- V - flow velocity (m/s)

Based on this, considering that the filtration process consists of the filtrate volume dV_f separated over a time interval dt , the filtration rate S_f can be expressed as follows (2).

$$S_f = \frac{dV_f}{F_f + dt} \tag{2}$$

Where:

- S_f - filtration rate
- F_f - filtration surface area
- dV_f - filtrate volume

The filtration rate is directly proportional to the driving force acting on the suspension and inversely proportional to the resistance of the filtration surface. The resistance on the filtration surface depends on the properties of the filter medium itself and the thickness of the sediment layer formed during the process. Accordingly, the filtration rate can be expressed as follows (3).

$$S_f = \frac{\Delta P}{\mu \cdot R_{fp} + r_o \cdot l} \tag{3}$$

Where:

- ΔP - pressure difference across the filter (driving force), Pa;
- R_{fp} - resistance of the filter medium, m^{-1}
- r_o - specific resistance of the sediment, m^{-2}
- l - thickness of the sediment layer, m

Naturally, both R_{fp} and r_o are variable parameters. The resistance of the filter medium may increase due to partial clogging of the pores or swelling of the fabric fibers under the influence of moisture. The parameter r_o represents a specific resistance value corresponding to a unit thickness of the sediment layer.

P (Pa) represents the driving force in the filter, defined as the difference between the pressure before the filter barrier (P_1) and the pressure at the barrier (P_2) (4).

$$\Delta P = P_1 - P_2 \tag{4}$$

The optimal value of ΔP is achieved by maintaining a sufficiently high filtration pressure (P_1) while ensuring that the resistance of the filter barrier does not lead to an excessive increase in P_2 .

Experimental results show that the filtration process proceeds much more easily when the particles form a non-deformable sediment. Such sediments accumulate on the upper surface of the filter barrier, and the interparticle voids help retain finer particles. In this case, the internal pores of the filter cloth remain unblocked, and the filtration resistance R_{fp} does not change. This extends the effective operating life of the filter. The variable resistance depends solely on r_o , which is determined by the increasing thickness of the

sediment layer. R_{fp} - the resistance of the filter medium - in our case corresponds to the clogging of the internal pores of the filter cloth. Once clogged, these pores are difficult to clean and sometimes require separate treatment, which reduces the overall efficiency of the filtration process.

Considering the above theoretical considerations and the sharp increase in pressure observed during filtration with the P-SH grade BFA, a decision was made to develop a composite material based on ANAB and P-SH, whose sorption characteristics were presented in Table 3. The composite obtained in this manner is composed of polar organic and nonpolar inorganic components.

Experimental research was carried out to develop composite BFA based on ANAB and P-SH for the treatment of long-term stored vegetable oils.

By varying the ratio of the obtained components, it is possible to achieve selective sorption of molecules of different sizes and, when necessary, to form an ion-exchanging, micro and mesoporous structure with adjustable properties across a wide range of pore sizes.

To enable control over the porous structure of the resulting BFA, composites were prepared using various ratios of ANAB and P-SH.

Using the following general mathematical formula (5), it is possible to predict in advance the sorption characteristics of composite BFAs obtained at different component ratios.

$$W_{comp} = \frac{W_1 \cdot m_1 + W_2 \cdot m_2}{m_1 + m_2} \quad (5)$$

Where:

W_1, W_2 - specific surface areas of the components, g/m^2 ;

m_1, m_2 - mass ratios, %.

In our subsequent experiments, the effects of the obtained composites on the bleaching-filtration process of long-term stored cottonseed oil were investigated. It was found that the highest efficiency was achieved when using a composite BFA composed of bentonite clay and pyrolyzed sunflower husk in an equal (1:1) mass ratio.

The composite BFAs obtained are expected to possess polarity that enables the coagulation of fine soapstock particles within the oil, facilitating their aggregation and sedimentation. At the same time, due to their macroporous structure, these agents should prevent clogging of the filter cloth during the filtration process.

The BFA we are developing should not only mechanically retain particles but also be capable of reducing the chemical impurities present in the oil - specifically, the acid and peroxide values as well as the secondary oxidation products - to standard levels. The inclusion of acid-activated bentonite clay with a pH of 4 in the composite provides the ability to form ionic bonds. The sorption characteristics of the obtained composite BFA of the BK grade are presented in Table 7.

Table 7 - Sorption characteristics of the composite BFA

Parameters	BK
Specific surface area (BET), m^2/g	154.24
Specific surface area of micropores, m^2/g	36.36
Specific surface area of meso and macropores, m^2/g	117.27
Pore volume, cm^3/g	0.135
Average pore diameter, \AA	34.08

In the course of our research, the filtration capabilities of acid-activated clays, as well as organic, inorganic, and composite materials, were studied for use in filtering polished refined vegetable oils after long-term storage.

Table 8 presents the changes in the acid value of polished refined cottonseed oil after filtration using various BFAs.

Table 8 - Effect of the amount of BFA obtained by different methods on the acid value

BFA dosage, %	ANAB	AOC	BK
	Acid value, mg KOH/g		
0.5	0.13	0.14	0.11
1	0.13	0.14	0.1
1.5	0.12	0.13	0.09
2	0.11	0.12	0.07
2.5	0.11	0.12	0.07
Standard Deviation	0.01	0.01	0.017888544
Standard Error	0.004472136	0.004472136	0.008
Upper (95%)	Cl 0.13241664	0.14241664	0.110211561
Lower (95%)	Cl -0.10758336	0.11758336	0.065788439

As shown in the table, the addition of all BFA samples at a dosage of 2% relative to the oil mass resulted in the greatest reduction of the acid value. Specifically, this parameter was 0.11 mg KOH/g for ANAB, 0.12 mg KOH/g for AOC, and 0.07 mg KOH/g for the BK composite.

Figure 2 illustrates the variation of the acid value of cottonseed oil with respect to reagent consumption during the filtration process using different BFAs.

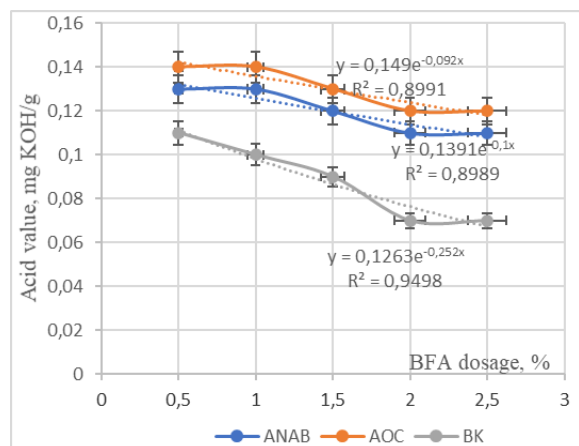


Figure 2 - Change in acid value relative to BFA dosage

As seen from the graph, the acid value consistently decreases for all samples as the reagent dosage increases.

This process is associated with the adsorption of free fatty acids onto the active sites present on the surface of the BFAs. The regression equations for the indicators obtained in the experiments were expressed as follows:

$$\text{ANAB: } y = 0.149e^{-0.092x}, R^2 = 0.8997$$

$$\text{AOC: } y = 0.1391e^{-0.1x}, R^2 = 0.8997$$

$$\text{BK: } y = 0.1263e^{-0.252x}, R^2 = 0.9351$$

For the BK-grade BFA, the value of $R^2 = 0.9351$ indicates that, within this system, the acid value follows an exponential decay pattern with respect to reagent consumption.

Filtration using the BK-grade O-FA resulted in the greatest reduction of acid value and peroxide content in the oil compared with ANAB and AOC. This can be attributed to the fact that both clay samples were activated using an acid-based method, resulting in a final product with an acidic pH environment. Although the acidity of the medium is an important factor for the adsorption of certain substances, temperature and time can also contribute to an increase in the acid value. In the graphs shown in Figure 1, the reduction in the acid value of the oil sample filtered using the BK-grade BFA is attributed to the presence of the P-SH component within its composition.

The capability of the obtained BFAs to remove oxidation products during the filtration of oil

samples was also investigated, and the results are presented in Table 9.

Table 9 - Effect of the amount of BFA obtained by different methods on the peroxide value

BFA dosage, %	ANAB	AOC	BK
	Peroxide value, mmol $\frac{1}{2}$ O/kg		
0.5	3.8	4	3.7
1	3.6	3.8	3.3
1.5	2.9	3.5	2.1
2	2.6	3.2	1.9
2.5	2.5	3	1.9
Standard Deviation	0.59	0.41	0.855569985
Standard Error	0.124096736	0.08	0.397492138
Upper (95%) CI	4.024547777	4.002115608	3.703615102
Lower (95%) CI	3.335452223	3.557884392	1.496384898

According to the table data, filtration using BFA samples at a dosage of 2% relative to the oil mass had the greatest effect in reducing the peroxide value. At this dosage, the peroxide value of the oil filtered with ANAB decreased from 4.2 mmol $\frac{1}{2}$ O/kg to 2.5 mmol $\frac{1}{2}$ O/kg, while filtration with AOC reduced it to 4.2 mmol $\frac{1}{2}$ O/kg. In contrast, filtration using the BK composite resulted in a further decrease of the peroxide value to 1.9 mmol $\frac{1}{2}$ O/kg.

Figure 3 illustrates the variation of the peroxide value of cottonseed oil with respect to reagent consumption during the filtration process using different BFAs.

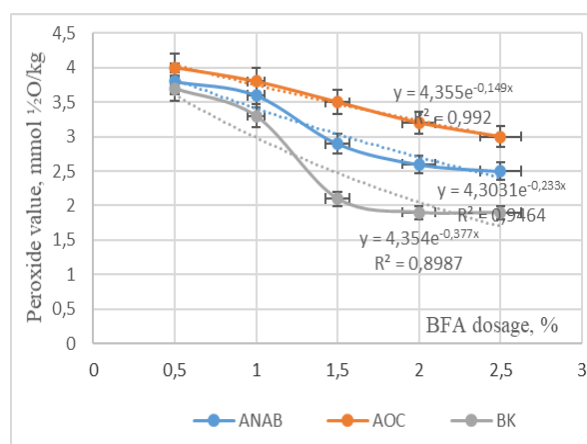


Figure 3 - Change in peroxide value relative to BFA dosage

As observed from the graph, the peroxide value consistently decreases for all samples as the reagent dosage increases. The regression equations describing the relationship between reagent consumption and peroxide value for ANAB, AOC, and BK samples are expressed as follows:

$$\text{ANAB: } y = 4.355e^{-0.149x}, R^2 = 0.9929$$

$$\text{AOC: } y = 4.3031e^{-0.233x}, R^2 = 0.9437$$

$$\text{BK: } y = 4.354e^{-0.377x}, R^2 = 0.8641$$

The results indicate that the R^2 values range from 0.86 to 0.99 which demonstrates that the model shows excellent agreement with the experimental data. In particular, the R^2 value of 0.9929 for the ANAB sample indicates a nearly perfect exponential relationship between the peroxide value and reagent consumption within this system.

Since the pH of the ANAB and AOC samples was higher compared to that of the BK sample, better results in terms of peroxide value reduction were achieved in the oil filtered using the mineral-organic composite BFA. Under the influence of temperature, secondary oxidation products are formed alongside the primary ones. The adsorption of these compounds onto the active sites of the BFAs significantly affects the overall kinetics of the process.

In the BK grade composite, the P-SH component provides sufficient porosity to retain micro-sized impurities from the oil, while the ANAB component prevents clogging of the filter cloth pores under pressure. In other words, it inhibits deformation of the sediment particles and functions as a drainage layer. As a result, both the filtration pressure and the filtration time are reduced. In addition, the presence of ANAB within the BK composition ensures that, during filtration, the acidic environment of the clay promotes both the ionic binding of the oil's coloring substances within the microporous active sites and their adsorption within the meso and macroporous structures.

In this process, the filtration performance of AOC was limited because its adsorption pore structure had a relatively large average pore diameter (74 Å) and a comparatively small specific surface area of micropores (4.70 m²/g). The adsorption pores present in AOC are primarily composed of macro and mesopores. Since the pore size is larger than that of the particles being filtered, this indicates that AOC functions mainly as a transmission channel rather than as an effective adsorbent medium.

Conclusions

The conducted studies revealed that the mineral-organic BFA obtained by combining bentonite and pyrolyzed sunflower husk in a 1:1 mass ratio demonstrated high efficiency in the filtration process of cottonseed oil. A composite BFA of the BK grade was developed based on the synergistic interaction between the mesoporous, mechanically stable structure of ANAB (with a specific surface area of 176.38 m²/g) and the microporous, carbon-based structure of P-SH. This combination resulted in a material with enhanced pressure resistance and high filtration efficiency. The simultaneous processes of filtration and adsorption purification significantly improve the color and clarity of the oil, removing oxidized and suspended impurities. ANAB in the composition reduces the compressibility of the filtered precipitate and increases its permeability, while P-SH effectively sorbs micro-sized molecules and oxidation products. This ensures a stable filtration process speed and prevents pressure increase.

At the same time, the proposed BFA oils with organic and mineral composition act as a drainage layer during the filtration stage, forming an incompressible layer under pressure.

The use of BFAs in the filtration process increases the filtration surface area and filtration rate due to the resistance of the formed precipitate. However, from a structural perspective, preventing this issue is considered quite challenging.

Through the use of BFAs, coagulated soapstock particles in polishing refined vegetable oils are effectively retained. As a result, during a continuous one-shift operation, issues such as the need for additional filter cleaning or filter cloth replacement do not arise.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Минералды-органикалық ағартқыш-сүзгілеуіш агенттерді жылтырату рафинациясында қолдану механизмі мен тиімділігін зерттеу

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Қабылданды: 11 маусым 2026

ТҮЙІНДЕМЕ

Осы ғылыми зерттеудің негізгі мақсаты - мемлекеттік қорда ұзақ уақыт сақталған өсімдік майларын олардың физика-химиялық көрсеткіштері бойынша тұтынушылық сапаға дейін тазарту болып табылады. Тәжірибелерде алты ай немесе одан да ұзақ уақыт сақталған мақта майларын жылтырату рафинациясы жүргізілді. Жылтыратылып рафинацияланған өсімдік майларының физика-химиялық көрсеткіштерін талдау негізінде ағартушы-сүзгілеуші агенттер (АСА) ретінде қолдануға арналған, әртүрлі кеуек құрылымына ие селективті материалдар алынды. АСА-лар бентонитті және опока типті саздарды қышқылмен белсендіру, сондай-ақ күнбағыс қауызын пиролиздеу арқылы алынды. Әрбір белсендірілген АСА-ның жылтыратылып рафинацияланған майларды сүзу қабілеті зерттелді. Зерттеу нәтижелері бойынша ең жоғары тиімділік қышқылмен белсендірілген Навбахор сілтілі бентониті (КНСВ) мен пиролизденген күнбағыс қауызының (П-КК) 1:1 массалық қатынаста дайындалған ВК маркалы композициялық АСА-сы қолданылған кезде байқалды. Бұл қоспа май массасына қатысты 1,5–2,0% мөлшерде енгізілгенде ең жоғары нәтиже көрсетті. Зерттеу нәтижелері полярлы органикалық және полярсыз бейорганикалық компоненттерден тұратын композицияның сәтті жасалғанын дәлелдейді. Бұл композиция сүзу процесі кезінде қысымның артуына кедергі келтірмей, ұсақ сабын тұнбасының бөлшектерін тиімді сүзуге қабілетті.

Түйін сөздер: бентонит сазы, опока типті саз, күнбағыс қауызы, ВЕТ талдауы, белсендіру, пиролиз, помпозиция, жылтырату арқылы рафинациялау, ағартушы-сүзгілеуші агент.

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Исследование механизма и эффективности применения минерально-органических отбеливающе-фильтрующих агентов при полировочной рафинации

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Поступила: 20 января 2026 Рецензирование: 23 мая 2026 Принята в печать: 11 июня 2026	АННОТАЦИЯ В данном научном исследовании основной целью является очистка растительных масел, длительное время хранившихся в государственном резерве, до потребительского качества на основе их физико-химических показателей. В экспериментах была проведена полировочная рафинация хлопковых масел, хранившихся в течение 6 месяцев и более. Путём анализа физико-химических показателей полировочно рафинированных растительных масел были получены селективные отбеливающе-фильтрующие агенты (ОФА) с различной пористой структурой для использования на стадии отбеливания и фильтрации. ОФА были получены путём кислотной активации бентонитовых и опоковидных глин, а также пиролиза подсолнечной шелухи. Были исследованы фильтрационные способности каждого активированного ОФА по отношению к полировочно рафинированному маслу. Полученные результаты показали, что наивысшая эффективность была достигнута при добавлении композиционного ОФА марки ВК, приготовленного в массовом соотношении 1:1 из кислотного-активированного Навбахорского щелочноземельного бентонита (KANB) и пиролизованной подсолнечной шелухи (П-ПШ), в количестве 1,5–2,0 % по отношению к массе масла. Результаты исследования показали, что была успешно получена композиция, состоящая из полярных органических и неполярных неорганических компонентов, способная фильтровать мелкие частицы соапстока и при этом не препятствовать росту давления в процессе фильтрации.
	Ключевые слова: бентонитовая глина, глина опокового типа, шелуха подсолнечника, БЭТ-анализ, активация, пиролиз, композиция, полировальная рафинация, отбеливающе-фильтрующий агент.
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