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ADSORPTION PURIFICATION OF SUNFLOWER OIL USING ACID-ACTIVATED MONTMORILONITE AND QUALITY ASSESSMENT

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This study investigates the process of sunflower oil adsorption purification using acid-activated montmorillonite from the Cherkasy deposit. The paper considers the change in sunflower oil quality indicators resulting from adsorption treatment with natural dispersed minerals.

This work reports a comprehensive solution to the problem of substantiating and devising efficient technology for vegetable oil purification that simultaneously removes impurities, oxidation products, as well as heavy metal cations. Process efficiency was evaluated using physicochemical methods, including determination of acid, color, peroxide values, mass fraction of phosphorus-containing substances, moisture, volatile matter, as well as chromatographic-mass spectrometric analysis of oil composition.

The results of experimental and pilot-scale studies demonstrate that the use of acid-activated montmorillonite at a dosage of 1% by oil mass, a temperature of 90–110°C, and a contact time of 20–30 min, ensures a reduction in the peroxide value to 3.0–3.9 mmol O2/kg, the color value to 6–9 mg I2, and the mass fraction of phosphorus-containing substances to 0.01–0.05%.

It has been shown that adsorption purification does not affect the basic triacylglycerol composition of the oil but promotes the removal of oxidized compounds that deteriorate its consumer properties and oxidative stability. The proposed technology could be implemented in industrial settings without significant modifications to existing production schemes; it represents a cost-effective alternative to imported bleaching clays

Keywords: sunflower oil, adsorption purification, bleaching, peroxide value, color value, gas chromatography–mass spectrometry

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1. Introduction

The oil and fat industry plays a key role in providing the people and related industries with vegetable oils.

Sunflower oil is one of the most common types of vegetable oils, widely used in the food industry and everyday nutrition of people. The quality and safety of the finished product largely depend on the efficiency of its purification and refining processes. One of the important stages in refining is adsorption purification, which is aimed at removing pigments, oxidation products, phospholipids, traces of metals, and other undesirable impurities that impair the color, taste, odor, and stability of the oil during storage [1].

The current state of adsorption refining of oil shows that achievements in the theory of adsorption, as well as processes and devices of chemical technology, are still insufficiently used. It is necessary to radically improve and devise new methods for studying the process of clarifying oils with sorbents, to elucidate the possibilities of choosing or obtaining more effective sorbents by activation, to establish optimal hydrodynamic conditions for the bleaching process.

Modern directions of food technology development involve the search for effective, affordable, and environmentally safe adsorbents for the purification of vegetable oils. Of considerable interest are natural clay minerals, in particular montmorillonite, which after acid activation acquires a high adsorption capacity and could be effectively used in the processes of bleaching and purification of oils. The use of acid-activated montmorillonite, in particular from the Cherkasy deposit, is promising from an economic and technological point of view as it contributes to increasing the efficiency of sunflower oil purification and reducing dependence on imported adsorbents.

The bleaching stage (adsorption treatment) is critical for ensuring stable quality indicators of vegetable oil and reducing oxidative processes [2].

In-depth study on the mechanisms of impurity adsorption could ensure stable quality indicators of sunflower oil and increase resource efficiency of production [3]. Therefore, research into scientific substantiation of the effectiveness of adsorption purification technologies for vegetable oils is relevant both from a theoretical and an applied point of view.

2. Literature review and problem statement

Vegetable oils are multicomponent systems, in which, along with triacyl glycerides, non-glyceride accompanying substances are present: phospholipids, pigments, waxes, free fatty acids, tocopherols, trace metal impurities, and oxidation products [4, 5]. When treating oilseeds by pressing or extraction methods, free phospholipids are removed together with the oil. Bound phospholipids under the influence of heat, moisture, solvent are partially released from the complexes and also pass into the oil. Papers [6, 7] report studies on the properties of sunflower and rapeseed oils obtained by Soxhlet extraction and microwave extraction. The main goal is to evaluate the physicochemical properties of sunflower and rapeseed oils obtained by two different extraction methods. However, those papers did not substantiate the purification of oil by the absorption method.

The authors of work [8] conducted research on the process of hydration of sunflower oil using electromagnetic treatment, which makes it possible to intensify the deposition of impurities, reduce energy consumption, and improve product quality without the use of reagents applied in the standard technological process. However, issues related to oil purification with adsorbents remained unresolved.

In [9], the process of filtering vegetable oils under ultrasonic cavitation conditions was considered. The efficiency of use was analyzed and the prospects for the use of ultrasonic cavitation were outlined. An algorithm for processing data obtained as a result of experimental studies during filtration under ultrasonic cavitation conditions was described. However, no research was conducted on the quality of the oil.

In [10], the influence of the industrial refining process on the quality and antioxidant activity of sunflower oil was investigated. Changes in the composition of useful components (having antioxidant and nutritional value) were determined when the oil passed through several stages of refining. The study has practical significance for the food industry; the work has a clear methodology and quantitative data for each stage of refining. However, the study concerns a specific technological process and does not fully analyze all types of refining. The work does not consider alternative technologies that could preserve more antioxidants.

Oxidation can be accelerated by the presence of metals of variable valence (in particular, iron and copper), which catalyze chain oxidation reactions and lead to a deterioration in organoleptic properties and a shortened shelf life. Work [11] studies the possibilities of using natural dispersed minerals as adsorbents in the process of bleaching rapeseed oil – an important stage of fat purification in food technology. This work substantiates the prospects of using natural dispersed minerals for oil bleaching, which could contribute to the development of safer, more effective, and environmentally friendly oil purification technologies in the food industry. This is important for optimizing the process of phosphorus removal from rapeseed oil during low-temperature bleaching. The presence of metal oxide in bleaching clays was significant. However, the work does not provide quantitative experimental data on the effectiveness of specific minerals in comparison with traditional bleaching agents.

Study [12] assesses the influence of bleaching conditions on the quality parameters of refined sunflower oil, which is an important stage of the refining process. The aim of the study was to establish the process, how changing factors such as temperature, process duration, bleaching clay concentration, and other parameters affects the quality of the final product. However, the work did not investigate long-term changes in oil quality.

Work [13] is an important resource for understanding modern approaches to purifying sunflower oil from impurities. It provides an analytical assessment of purification technologies, considers the possibilities of using natural adsorbents, and offers technological solutions that contribute to improving the quality of oil at minimal costs and loss of useful components. The efficiency of the adsorption process depends on the chemical nature and concentration of adsorbed substances. Non-carbon sorbents of natural and artificial origin are increasingly used for water purification by adsorption. The use of these sorbents is due to their sufficiently high adsorption capacity, selectivity, and condition-exchange properties of some of them, as well as relatively low cost and availability. The most important representatives of mineral natural sorbents are zeolites and clay materials. However, the work does not provide laboratory or industrial experimental data on the effectiveness of various minerals.

Adsorption bleaching with activated clays is the most important stage of processing as it makes it possible to reduce the content of undesirable impurities, including non-hydrated phospholipids, primary and secondary oxidation products and partly catalyst metals. During the thermal decomposition of hydroperoxides, secondary oxidation products are formed – aldehydes, ketones, which contribute to the further oxidation of the oil. Therefore, during further storage of the oil, the oxidation rate increases, which worsens the stability of the organoleptic indicators of the finished oil.

The authors of work [14] conducted a study on the adsorption of impurity particles from a triglyceride solvent, which represents a model vegetable oil, using atomistic molecular dynamic modeling. It was found that particles containing charged groups, phospholipids, hydrogen bonds can saturate each other on the adsorbent. Overall, the work provides an understanding of the interaction of vegetable oils and their components at the liquid-solid interface, as well as recommendations for the development of effective adsorption materials for vegetable oil purification. However, the work does not include experimental confirmation of adsorption processes under real conditions, the model is limited to one type of triglyceride medium (triolein), which may differ from more complex real oils.

Studies have been conducted to optimize the process of bleaching sunflower oil – investigating the molecular mechanisms of adsorption of impurities in a model of vegetable oil (triglyceride medium) on the surfaces of an inorganic adsorbent (silicate) with different densities of hydroxyl groups [15]. The work provides a fine molecular understanding of the adsorption of impurities in apolar media, which helps to develop effective adsorbents for vegetable oil purification technologies. However, the work was carried out without direct experimental confirmation; the model includes only one type of triglyceride solvent, which differs from the full composition of real vegetable oils. The use of an adsorbent in an amount of 2–3% by mass could negatively affect the quality of the oil, giving it an earthy taste.

In [16], the results of adsorption purification of waste industrial oil in the presence of natural aluminosilicates, bentonite, and mordenite are discussed. However, the results were obtained only for a limited set of conditions and adsorbents, which does not allow them to be unambiguously extrapolated to other types of adsorbents or technical lubricants. There is no kinetic and isothermal analysis of adsorption, which limits the understanding of the mechanisms at the molecular level.

Natural aluminosilicates of the montmorillonite group, activated by acid, are used as sorbents for bleaching vegetable oils [17, 18]. These are the most effective sorbents. They are obtained from bentonites by treatment with various mineral acids. Treatment of bentonite with mineral acids gives a modified product with a large surface area and acidity, which has increased adsorption and catalytic capacity [19]. The relative ease of their activation and availability are the advantages of these sorbents. Therefore, it is necessary to continue research in this area.

In [20], the feasibility and effectiveness of using synthetic zeolite for bleaching unrefined sunflower oil compared to imported bleaching clay were examined. The results of the study showed that the bleaching effects of sunflower oil with synthetic zeolite were similar to those of imported commercial bleaching clay, with zeolite showing slightly higher efficiency in reducing peroxide value. However, the color indices indicate that not all aspects of bleaching were optimized, and the study was limited to laboratory conditions, which is not always transferable to production scale without further optimization.

Studies have been conducted on clay from Merauke Regency and its use as an adsorbent for free fatty acids in the purification of used cooking oil [21]. Adsorption experiments have shown that both natural and acid-activated clays reduce the free fatty acid (FFA) content of waste cooking oil, with acid-activated clay consistently showing higher efficiency. However, the studies focused on reducing fatty acid content. No studies were conducted on the removal of oxidation products. The paper lacks kinetic analysis and adsorption isotherms (Langmuir/Freundlich), which limits a deeper understanding of the adsorption mechanisms. The results may be specific to a certain type of waste oil and Merauke clay, which cannot be universally applied. In addition, the treatment with the adsorbent was carried out for a long time, which has a negative effect on the quality of the oil.

All this gives grounds to argue that it is advisable to conduct a study on the process of oil purification by adsorption method.

Activated sorbents based on montmorillonite are much cheaper than imported ones, so they are currently attracting special attention. The problem is to select affordable and inexpensive natural mineral adsorbents from Ukrainian deposits and establish effective technological regimes that ensure high quality purification without complicating industrial schemes.

Despite the presence of numerous scientific works, most of them focus either on individual stages of refining, or on model systems, or on a narrow list of quality indicators. The issues of comprehensive assessment of the impact of adsorption treatment on oxidative stability indicators, the content of phosphorus-containing compounds and trace metal impurities in industrial settings remain insufficiently studied.

All this allows us to state that it is advisable to conduct a study on the justification and effectiveness of the process of adsorption purification of sunflower oil using activated mineral sorbents based on montmorillonite from Ukrainian deposits.

3. The aim and objectives of the study

The aim of our work is to substantiate the technology of adsorption purification of sunflower oil using acid-activated montmorillonite from the Cherkasy deposit and assess the quality of the purified oil. This will provide an opportunity to substantiate the use of acid-activated montmorillonite in the purification of sunflower oil, establish rational process parameters, as well as improve product quality.

To achieve the goal, the following tasks were set:

– to characterize the quality indicators of industrial batches of the initial neutralized sunflower oil;

– to propose a technological regime of contact adsorption purification;

– to assess the change in oil quality indicators after bleaching;

– to establish qualitative differences in the composition of the oil before and after purification according to the data of the chromatographic-mass-spectrometric approach.

4. The study materials and methods

The object of our study is the process of adsorption purification of vegetable oils using acid-activated montmorillonite from the Cherkasy deposit. The subject of the study is to determine the change in the quality indicators of sunflower oil as a result of adsorption treatment with natural dispersed minerals. The study used industrial samples of sunflower oil (after neutralization) and acid-activated montmorillonite from the Cherkasy deposit, used as an adsorbent.

The principal hypothesis assumes using the acid-activated montmorillonite from the Cherkasy deposit as an adsorbent during adsorption treatment of sunflower oil (neutralized), which could contribute to the effective removal of impurities (pigments, oxidation products, trace metals) and would lead to an improvement in its quality indicators, in particular color, acid, and peroxide value, as well as an increase in the oxidative stability of the oil.

In order to ensure the reproducibility of the experiment and the validity of the interpretation of the results, during the study assumptions were adopted that outline the boundary conditions and methodological framework of the study:

– it was assumed that the acid-activated montmorillonite from the Cherkasy deposit has a stable mineralogical composition and the same adsorption properties throughout the experiment. The main mechanism of sunflower oil purification is the adsorption of coloring substances, oxidation products, and trace metals on the surface of activated montmorillonite;

– it was assumed that the conditions of adsorption treatment (temperature, contact duration, mixing intensity) remain constant.

During the study, a number of methodological simplifications were also accepted, necessary for the unification of the experimental conditions:

– the process of adsorption treatment of sunflower oil was considered uniform throughout the volume of the system, that is, sufficient mixing was assumed, which ensures the homogeneity of the mixture of oil and adsorbent;

– it was assumed that the particle size of the adsorbent is relatively uniform and does not change during the oil treatment process;

– the influence of minor fluctuations in the composition of the initial sunflower oil on the results of the study was not taken into account as industrial samples of the same batch were used.

General organoleptic and physicochemical analysis was carried out according to national standards and methodologies from specialized literature. The list of main methods is given in Table 1.

Table 1

Control methods Indicator	The principle of research method
Sampling of raw materials and finished products and preparing them for analysis	DSTU 4349:2004 «Oils. Sampling methods»
Acid value, mg KOH/g	DSTU 4350:2004 «Oils. Methods for determining acid value»
Color number, mg I_2	DSTU 4568:2006 «Oils. Methods for determining the color number»
Mass fraction of phosphorus-containing substances, %, in terms of stearooleolecithin	DSTU 7082:2009 «Oils. Methods for determining the mass fraction of phosphorus-containing substances»
Peroxide value, mmol O_2/kg	DSTU 4570:2006 «Animal and vegetable fats and oils. Method for determining peroxide value»
Mass fraction of moisture and volatile substances, %	DSTU 4603:2006 «Oils. Methods for determining the mass fraction of moisture and volatile substances»
Fatty acid composition	DSTU ISO 5508–2001 "Animal and vegetable fats and oils. Analysis of fatty acid methyl esters by gas chromatography" (ISO 5508:1990, IDT)

The methodology of chromatographic-mass spectroscopic analysis of complex mixtures of organic substances [22] is one of the most informative, objective, and sensitive to minor impurities of various organic substances in complex mixtures. Its use for the analysis of the fatty acid composition of edible oils is the most promising in devising new technological schemes for the processing of oilseeds and the purification of the resulting waste.

The essence of this methodology is the integrated use of the capabilities of the chromatographic method and mass spectroscopy. The first method achieves the separation of complex mixtures into individual substances, and the second – the identification of these compounds.

In this work, a Hewlett-Packard device with a quartz capillary column 25 m long and an internal diameter of 0.2 mm was used for chromatographic separation. The LKB-209/C device (manufacturer – LKB Instruments Sweden) with a direct ballistic injection system was used as a mass spectrometer. The sample under study is placed in a direct injection ampoule and under conditions of deep vacuum, when the temperature is raised to complete evaporation of the volatile mixture (150°C), several mass spectra are taken. The qualitative composition of volatile substances is then determined from them, and when a reference substance, for example, stearic acid of a known concentration in oil is introduced into the sample, the amount of each component in the oil is estimated. For a comparative analysis of the qualitative differences in the composition of the oil before and after purification, the approach of chromatography-mass spectrometry of organic mixtures with identification of peaks by mass spectra was used.

More than a hundred occurrences of bentonite clays have been found in Ukraine [23]. Given the relatively low cost of the studied clays, compared to adsorbents offered by foreign companies, it is planned to continue research into their activation to set up Ukrainian enterprises for the production of adsorbents for the oil and fat industry.

The characteristics of the adsorbent used are given in Table 2.

Table 2

Characteristics of activated montmorillonite – a mineral from the Cherkasy deposit

Indicator	Unit of measurement	Normative value
Humidity	%	≤ 8–12
pH of aqueous suspension	–	2.5–4.5
Bulk density	g/cm^3	0.4–0.6
Specific surface area	m^2/g	180–350
Porosity	–	High, meso- and micropores
Mass fraction SiO_2	%	60–70
Mass fraction Al_2O_3	%	15–20
Mass fraction Fe_2O_3	%	≤ 5
Mass fraction CaO + MgO	%	≤ 5
Bleaching ability	% color reduction	70–95
Soap adsorption	%	≥ 90
Particle content < 75 microns	%	≥ 90

High bleaching capacity (70–95% reduction in color) and significant soap adsorption efficiency (≥ 90%) confirm the feasibility of using the specified adsorbent at the stage of clarification of sunflower oil after neutralization. In addition, the finely dispersed composition (content of particles < 75 μm not less than 90%) provides a developed contact surface and intensification of mass transfer processes.

The set of physicochemical characteristics (Table 2) indicates that the studied montmorillonite is a promising adsorbent for increasing the efficiency of adsorption purification of sunflower oil and improving the quality of the finished product.

5. Results of investigating the process of adsorption purification of sunflower oil

5. 1. Qualitative indicators of industrial batches of the initial neutralized sunflower oil

Industrial samples of sunflower oil were obtained according to typical technological regimes at the line of PrAT "Vinnytsia Oil and Fat Plant" (Ukraine).

Table 3

Qualitative characteristics of the described samples from different batches of original (neutralized) sunflower oil

Sample No.	Mass fraction of phosphorus-containing substances (calculated as stearooleolecithin), %	Acid value, mg KOH/g	Mass fraction of moisture, %	Color number, mg I_2	Peroxide value, mmol O_2/ kg
1	0.2	0.29	0.11	22	16.55
2	0.18	0.25	0.10	25	18.20
3	0.17	0.26	0.08	18	12.42
4	0.12	0.20	0.07	15	11.10
5	0.14	0.22	0.09	15	12.30

Qualitative characteristics of industrial batches of the initial neutralized sunflower oil are given in Table 3.

Each measurement was performed three times, and the results are represented as the mean value along with the corresponding standard error. Data analysis was performed using Microsoft Excel 2016 (Microsoft Corp., USA) and Mathcad 14 (PTC Inc., USA). To determine the presence of significant differences between the samples, one-way analysis of variance (ANOVA) was used. Statistical significance was established at a p-value of less than 0.05.

The analysis results showed that all sunflower oil samples are characterized by a low content of phospholipids and differ from each other in color and peroxide values.

5. 2. Technological mode of contact adsorption purification

Experimental and industrial tests of adsorption purification were performed by the contact method in vacuum bleaching apparatuses with subsequent filtration. The recommended bleaching mode is given in Table 4.

Rational technological parameters of the process have been established, in particular the use of 1% activated montmorillonite with a particle size of 0.07–0.08 mm, a temperature of 90–110°C, and a contact time of 20–30 min.

The use of pretreatment with citric acid and conducting the process in a vacuum of 2–6 kPa ensures the intensification of the removal of impurities and the stabilization of oil quality indicators.

Table 4

Technological regime of adsorption purification of sunflower oil by contact method

Parameter	Value	Time, min
Adsorbent consumption (activated montmorillonite), %, to the weight of oil	1	–
Granulometric composition, mm	0.07–0.08	–
Citric acid solution consumption, kg/t of oil	1	–
Oil temperature when supplying citric acid solution, °C	60	20
Oil temperature when supplying adsorbent, °C	80	10
Bleaching temperature	°C	90–110
Contact duration	min	20–30
Working pressure (vacuum)	kPa	2–6

5. 3. Investigating oil quality indicators after bleaching

Based on our experimental and industrial studies on the process of purification of sunflower oil by the contact method, it was established that the use of activated montmorillonite makes it possible to obtain high-quality oil that meets the necessary requirements (Table 5).

Adsorption purification of sunflower oil with activated montmorillonite from the Cherkasy deposit makes it possible to obtain a higher degree of purification both from peroxide compounds and by other quality indicators.

Our research established a decrease in the mass fraction of phosphorus-containing substances to 0.01–0.05%, peroxide value to 3.0–3.9 *mm*ol O_2/kg, and color value to 6–9 *mg* I_2.

Table 5

Qualitative characteristics of the presented samples of bleached sunflower oil

Sample No.	Mass fraction of phosphorus-containing substances (calculated as stearooleolecithin), %	Acid value, mg KOH/g	Mass fraction of moisture, %	Color number, mg I_2	Peroxide value, mmol O_2/ kg
1	0.05	0.2	0.06	9	3.5
2	0.04	0.2	0.05	8	3.9
3	0.04	0.2	0.05	7	3.1
4	0.01	0.2	0.05	6	3.0
5	0.02	0.2	0.05	8	3.1

The acid value remained stable at 0.2 *mg* KOH/g, and the moisture content did not exceed 0.05–0.06%, which confirms the preservation of the hydrolytic stability of the product.

The obtained data (Table 5) confirm the feasibility of using activated montmorillonite to ensure a high degree of purification and compliance of the oil with regulatory quality requirements.

5. 4. Chromatographic-mass spectrometric study of changes in the composition of sunflower oil after adsorption purification

The analysis of purified oil was performed using the chromium-mass spectroscopy (CMS) method [22].

The efficiency of purification of sunflower oil with activated montmorillonite was studied using the chromium-mass spectroscopy (CMS) method. Fig. 1 shows chromatograms of the initial sunflower oil, which was selected at the Vinnytsia Oil and Fat Plant after the neutralization stage. Fig. 2 shows chromatograms of oil purified with montmorillonite. Fig. 3 depicts mass spectra of individual oil fractions before and after purification with clay.

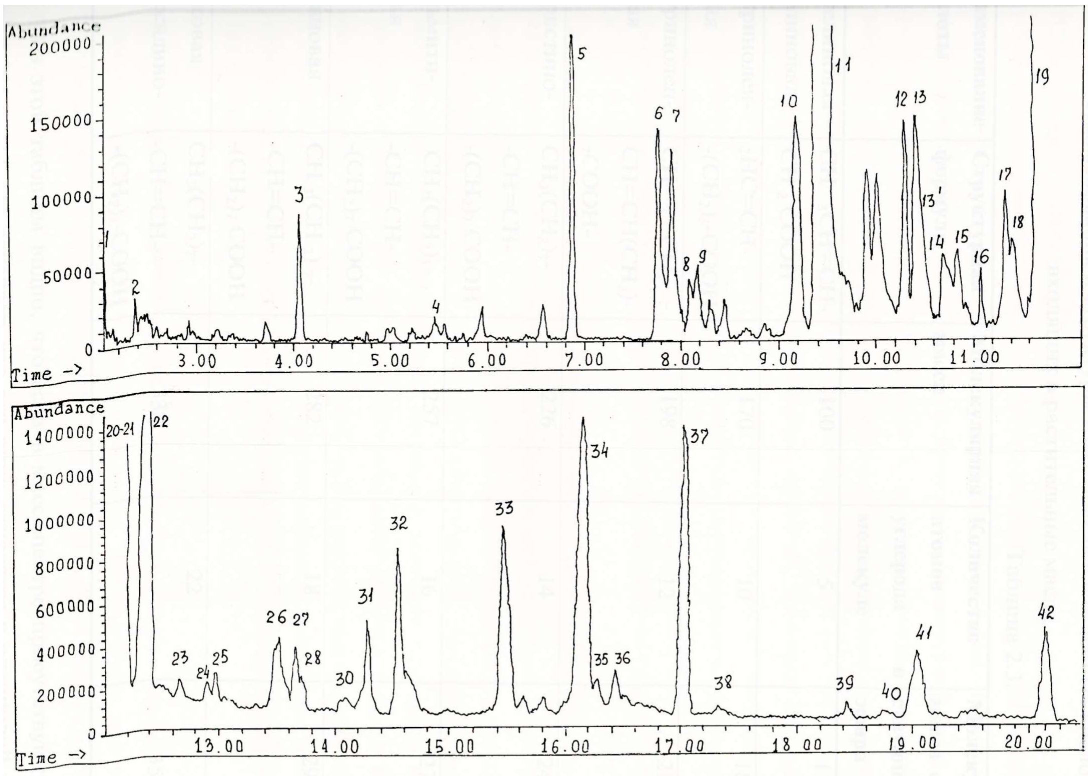

Fig. 1. Chromatogram of the separation of transesterification products of the initial sunflower oil (output of the software of the gas chromatograph with a mass spectrometric detector)

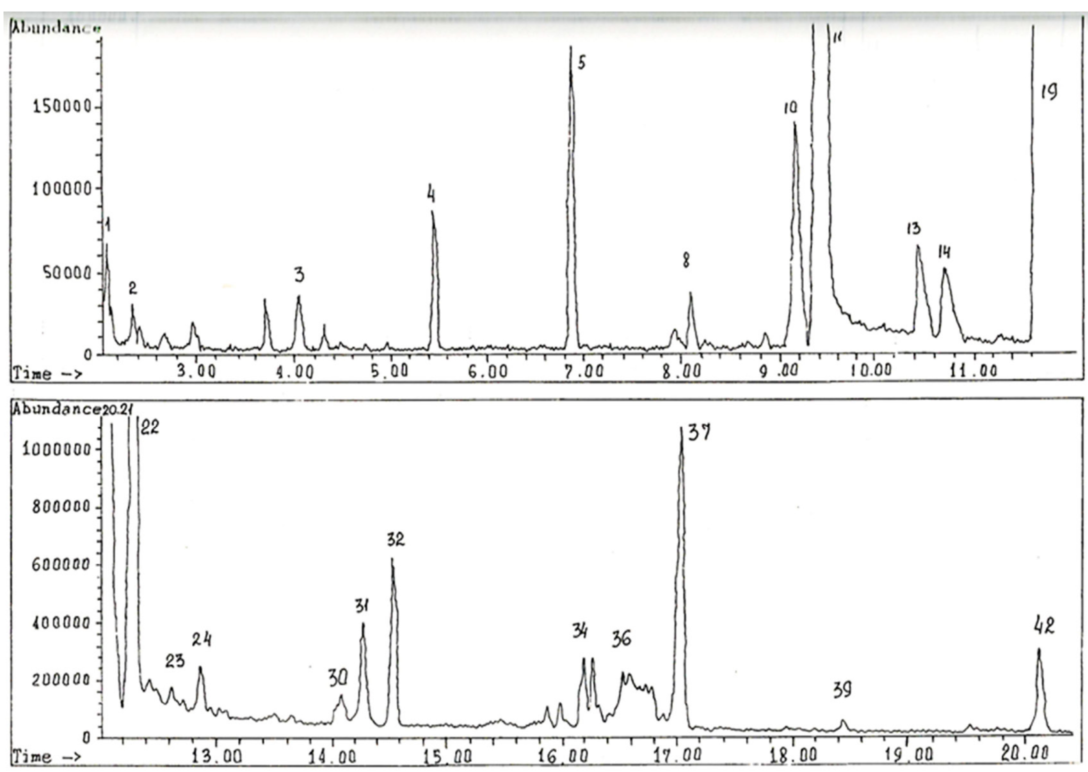

Fig. 2. Chromatogram of the separation of transesterification products of sunflower oil purified with activated montmorillonite (output of the software of a gas chromatograph with a mass spectrometric detector)

Since the establishment of the structural-group composition of oils was not the task of this study, we shall focus only on the qualitative differences in the compositions of the oils before and after their purification with bleaching clays. Therefore, we shall dwell in more detail on the analysis of chromatograms, based on the identification of the observed peaks.

Qualitative analysis of the chromatogram of the original sunflower oil shows that it contains at least 42 compounds. The most intense peaks (No. 11 and 19), according to mass spectroscopic analysis, belong to palmitic (3.5%) and linolenic acid (62%). Intense peaks No. 20 and 22 belong to oleic (28%) and stearic acid (3.3%). Relatively intense peaks No. 34 and 37, which relate to pentadecanoic and docosanoic (0.5%) acids, respectively. Other peaks, correspondences, and concentrations of other acids and related substances are much smaller.

Some peaks (no more than 2) could not be unambiguously identified even by mass spectroscopy.

Comparison of chromatograms of sunflower oil before and after purification with bleaching clays (Fig. 1, 2) shows that most peaks have practically not changed. This indicates that the main part of the structural-group composition of oils does not undergo any changes in the process of oil purification.

However, some peaks undergo significant changes and not only in the direction of their intensity reduction after purification. Some substances completely disappear from the oil during the purification process. These are mainly oxidized compounds. This applies to peaks No. 6, 7, 12, 17, 18, 26, 27, 33, 34, 41 after treatment of sunflower oil with montmorillonite, and peak No. 3 decreased in intensity.

Analysis of changes in the chromatograms of oils treated with activated montmorillonite reveals that after purification, substances with unsaturated bonds disappear. Probably, they are primarily subject to oxidation and the appearance of harmful impurities, which are removed by adsorption purification of bleaching clays.

According to the results of chromatographic-mass spectrometric analysis, it was found that the fatty acid profile after purification does not change significantly, while the intensity of the peaks of oxidized impurities decreases or they disappear completely.

6. Discussion of results based on the study of adsorption purification of sunflower oil with activated montmorillonite

Our results give grounds to argue that the use of acid-activated montmorillonite is an effective solution for adsorption purification of sunflower oil after the neutralization stage. A sequential analysis of the results according to the tasks set revealed that the proposed technology provides a reduction in the content of phosphorus-containing substances, peroxide compounds, and colored impurities without significantly disturbing the basic fatty acid profile of the oil.

As can be seen from the data in Table 3, the initial samples differed in color number, peroxide number, and mass fraction of phosphorus-containing substances, which indicates the heterogeneity of the quality of the raw material after neutralization. Such differences can be explained by the characteristics of the initial seeds, their storage conditions, the degree of oxidative changes during the previous technological stages, as well as the efficiency of hydration and neutralization. This confirms that after traditional refining stages, the oil may still contain residual phospholipids, primary oxidation products, pigments, and other impurities that degrade its quality and complicate further processing. Therefore, the results in Table 3 actually justify the need for an additional stage of adsorption purification.

According to the data in Table 4, the rational process parameters were the addition of 1% acid-activated montmorillonite to the oil mass, a temperature of 90–110°C, a contact time of 20–30 min, and the process in a vacuum of 2–6 kPa. The results can be explained by the combined effect of the physicochemical properties of the adsorbent and the technological conditions of the process. Acid activation of montmorillonite increases the specific surface area, the number and strength of acid centers, as well as the volume of accessible meso- and micropores, which increases its affinity for polar impurities, oxidation products, and residual phospholipids.

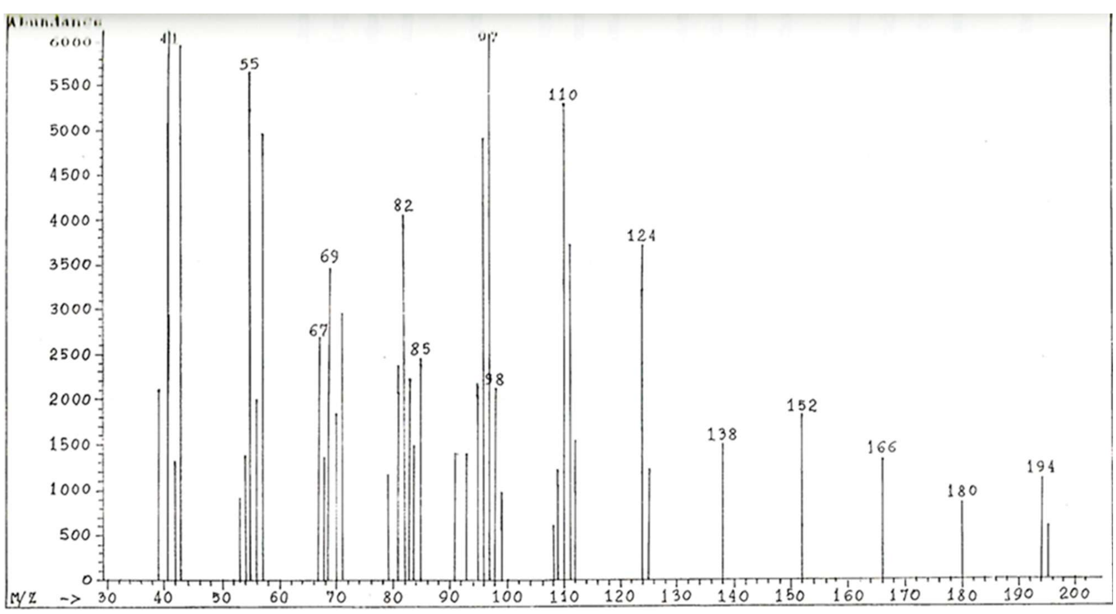

Fig. 3. Mass spectrum of the sunflower oil component corresponding to peak #31 on the separation chromatogram (output from the software of the gas chromatograph with mass spectrometric detector)

The increased temperature reduces the viscosity of the oil and improves the mass transfer between the liquid phase and the surface of the sorbent, and the vacuum limits further oxidation during treatment. The preliminary introduction of citric acid probably enhances the binding of trace amounts of catalyst metals, which are capable of accelerating oxidative processes. Taken together, this creates conditions for intensive extraction of undesirable components without significant deterioration of the main composition of the oil.

According to the data in Table 5, after purification, the mass fraction of phosphorus-containing substances decreased to 0.01–0.05%, the peroxide value to 3.0–3.9 mmol O_2/kg, and the color value to 6–9 mg I_2. At the same time, the acid value remained practically stable, and the mass fraction of moisture did not exceed 0.05–0.06%. Such results can be explained by the selective adsorption of precisely those compounds that most determine the color and oxidative instability of the oil. The decrease in the content of phosphorus-containing substances indicates the extraction of residual phospholipids that were not completely removed at the previous stages. The decrease in peroxide value indicates the effective removal of primary oxidation products, and the decrease in color value indicates the adsorption of pigments and related colored impurities. At the same time, the stability of the acid value confirms that the proposed regime did not cause significant hydrolysis of triacyl glycerides and did not impair the hydrolytic stability of the product.

Comparison of chromatograms in Fig. 1 and Fig. 2, as well as analysis of mass spectra of individual components in Fig. 3, showed that most of the main peaks are preserved, while some of the minor peaks after purification decrease in intensity or completely disappear. This allows us to explain the mechanism of action of the adsorbent: purification is not accompanied by a deep transformation of the basic fatty acid profile but provides preferential removal of minor oxidized compounds and other undesirable components that negatively affect the consumer properties and oxidative stability of the oil. Thus, the results in Figs. 1–3 complement the data in Table 5 and confirm that the improvement of physicochemical parameters is not accidental but is due to the selective removal of undesirable impurities.

The advantages of the proposed technology become more obvious when compared with known solutions. Unlike works that investigated individual stages of refining or only the physicochemical characteristics of oil after extraction [6, 7], this study evaluated the effectiveness of adsorptive purification after neutralization under conditions close to production conditions. Unlike approaches aimed at intensifying hydration or precipitation of impurities without the use of sorbents [8], the proposed solution allows for a targeted reduction in the content of peroxide compounds, pigments, and phospholipids. Unlike studies that considered mainly hydrodynamic or cavitation aspects of filtration [9], our work proved a real improvement in the quality indicators of purified oil. Unlike publications that analyzed changes in the composition of oil at individual stages of industrial refining without testing alternative sorbents [10], this study substantiated the use of a specific acid-activated mineral adsorbent and established rational parameters for its application.

It should be emphasized that, unlike the studies performed mainly on model systems or within the framework of molecular dynamics modeling [14, 15], the results reported here were obtained on industrial samples of sunflower oil. This is what gives the study practical value since it allows us to assess the real technological efficiency of the adsorbent, and not just theoretically predict its ability to bind impurities. In addition, unlike the works where individual types of clay or zeolite were compared mainly by one or two indicators [20, 21], this work carried out a comprehensive assessment of the purification efficiency by several criteria at the same time: peroxide number, color number, content of phosphorus-containing substances, and chromatographic-mass spectrometry data. It is such a multi-criteria assessment that allows us to more fully characterize the efficiency of the proposed technology.

Available studies lack a comprehensive assessment of the impact of adsorption treatment on oil quality in industrial settings, as well as insufficient data on the use of available mineral adsorbents from Ukrainian deposits. In this work: the effectiveness of acid-activated montmorillonite for the purification of neutralized sunflower oil has been confirmed; a rational technological process regime has been established (Table 4); the positive effect of treatment on key quality indicators has been proven (Table 5); it is shown that purification does not destroy the basic composition of the oil but mainly removes oxidized minor components (Fig. 1–3). It is the totality of these results that indicates that the goal of the study has been achieved: bleached sunflower oil meets the requirements of regulatory documentation [24].

The practical significance of the proposed solution is also the possibility of its implementation without significant reconstruction of existing production schemes. The contact purification technique with subsequent filtration is well consistent with typical bleaching stages in the oil and fat industry. Another advantage is that an effective result is achieved with a relatively moderate dosage of the adsorbent – 1% by weight of oil. This is important, since in many known technological solutions [25–27] an increase in the degree of purification is associated with an increase in clay consumption, which, in turn, increases oil losses and the overall cost of the process. In this case, the advantage is provided by a combination of a sufficiently high adsorption activity of acid-activated montmorillonite and an optimal mode of contact with oil.

Thus, the results of our study prove that acid-activated montmorillonite can be an effective adsorbent for the purification of sunflower oil after the neutralization stage. The proposed technological regime provides a significant improvement in physicochemical quality indicators, promotes the removal of oxidized impurities, and does not cause significant changes in the basic composition of the oil. This allows us to consider the proposed technology as justified and practically promising for increasing the efficiency of vegetable oil purification.

However, the study has certain limitations that must be taken into account in the practical application of the results and during further theoretical developments. First, the study was conducted for sunflower oil after neutralization; therefore, the direct transfer of the obtained regularities to other types of vegetable oils requires additional verification. Second, the assessment was conducted for one type of acid-activated montmorillonite, while the properties of natural raw materials may vary depending on the deposit, mineralogical composition, and activation mode. Third, the work did not study the long-term oxidative stability of the oil during storage; therefore, it is impossible to fully assess how much the resulting decrease in peroxide value will affect the shelf life of the product. Fourth, a quantitative assessment of oil losses on the adsorbent, which is of direct importance for the industrial economics of the process, was not performed.

Further studies plan to investigate the influence of the proposed purification regime on the oxidative stability of sunflower oil during long-term storage. It is also promising to compare acid-activated montmorillonite with other types of natural and modified adsorbents under the same technological conditions. Special attention is required to assess the regeneration and reuse of the adsorbent spent, as this is important for resource efficiency and environmental safety of the technology.

7. Conclusions

1. It has been established that among the natural dispersed minerals from the Cherkasy deposit, the most effective adsorbent for the purification of sunflower oil is acid-activated montmorillonite.
2. A contact adsorption purification mode (1% adsorbent; 90–110°C; 20–30 min; 2–6 kPa) has been proposed, which is suitable for industrial implementation without significant reconstruction of production lines.
3. After purification, a decrease in the color number to 6–9 mg I_2 and a peroxide number to 3.0–3.9 mmol O_2/kg was achieved, as well as a reduction in phosphorus-containing substances to 0.01–0.05%.
4. Chromatographic-mass spectrometric comparison data indicate the preferential removal of oxidized impurities while preserving the main triglyceride and fatty acid profile of the oil.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

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Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

Authors' contributions

Larisa Fialkovska: Conceptualization, Methodology, Investigation; **Valentyna Bandura**: Conceptualization, Management, Project administration; **Marina Serdyuk**: Methodology, Resources, Writing – review & editing; **Maksym Hudzenko**: Writing – original draft; Data curation; **Artem Antonenko**: Visualization, Validation; **Vitalii Mihailik**: Formal analysis; **Olga Vasylyshyna**: Formal analysis; Resources.

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