POSSIBILITY OF BLEACHING SUNFLOWER OIL WITH SYNTHETIC ZEOLITE

ORIGINAL SCIENTIFIC PAPER

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ABSTRACT:
In this paper, the possibilities and effects of using domestic industrially produced zeolite for bleaching crude sunflower oil, compared to imported commercial bleaching earth, were examined. The parameters of the bleaching process in laboratory conditions were: temperature 95°C, contact time 30 min, and mass fractions of bleaching agent in oil: 0.2, 1, 2 and 3%. The following methods were used to characterize the bleaching agents: XRD, FTIR, BET and SEM/EDS. The bleaching efficiency parameters were determined by laboratory methods prescribed by the relevant ordinance on edible vegetable oils, and included: soap content, peroxide value and fatty acid composition. The results of the research showed that the effects of bleaching sunflower oil with synthetic zeolite are similar to the effects of bleaching with imported commercial bleaching earth, with the former showing a slightly higher effectiveness in reducing the peroxide value.

KEYWORDS: sunflower oil; oil bleaching; synthetic zeolite; bleaching earth

INTRODUCTION

Crude vegetable oil contains undesirable substances such as free fatty acids, gums and dyes, soap residues, phosphatides and traces of metals, and these substances affect the efficiency of its processing process, as well as the quality and market value of the final product [1]. Therefore, in the industrial production of edible vegetable oils, the bleaching procedure of previously neutralized crude oil plays a major role, in which unwanted substances are removed by applying a suitable adsorbent [2], which enables the production of commercial oil of the quality prescribed by relevant national regulations. Natural and active clays are usually used as bleaching agents for edible vegetable oils [3], [4], and clay activation is usually done with inorganic acids such as H_2SO_4 [5], [6] and HCl [7], [8]. The goal of activation is to improve the physical and chemical properties responsible for the removal of unwanted substances from oil [9], and refers, among other things, to the increase of adsorption capacity [10], specific surface area and microporosity of clay [11], [12], [13]. The optimal activation process conditions are specific for each clay [14], which is why different materials are continuously tested in laboratory and semi-industrial conditions.

Producers of edible vegetable oils bleach crude oils under different process conditions (temperature, time, dose of bleaching agent), which mostly depend on the type of oil, type of bleaching agent and equipment used. At the same time, researchers are continuously investigating possibilities of using different natural, synthetic, or waste materials, either unactivated or previously activated, as bleaching agents in order to obtain the required oil quality with greater economic profitability and environmental protection [15], [16], [17], [18].

Among the available adsorbents, zeolites are considered to be inexpensive materials [19]. Namely, although zeolites are available in nature, their industrial synthesis is relatively easy and enables the product to be obtained in a pure form, so it is most commonly practiced [20]. In this paper, the characteristics of the domestic synthetic zeolite and the possibility of its application in the bleaching process of raw sunflower oil were examined, in comparison with imported commercial bleaching earth.

MATERIALS AND METHODS

The following materials were used in the experimental part of the research: synthetic zeolite ZEOflair 100 (Zeochem, Zvornik), imported commercial bleaching earth and raw sunflower oil (Bimal, Brčko), and other reagents and chemicals required for the characterization of bleaching agents. Bleaching of sunflower oil was performed by adding a certain amount of bleaching agent to glasses with 200 ml of raw sunflower oil preheated to 95°C, and then intensively mixing the suspension for 30 minutes. The applied mass fractions of bleaching agents in oil were (w): 0.2, 1, 2 and 3%. At the end of the mixing time, the suspension of oil and bleaching agent was filtered on vacuum filtration equipment.

The following methods were used to characterize the synthetic zeolite and commercial bleaching earth: X-ray diffractometry (XRD), infrared spectroscopy (FTIR), low-temperature nitrogen adsorption (BET), and scanning electron microscopy with energy dispersive spectrometry (SEM/EDS).

X-ray diffractometry was performed on a Rigaku Smartlab X-ray diffractometer, and crystalline phases were identified using Rigaku PDXL 2.0 software with the ICDD PDF-2 2016 database.

Infrared spectroscopy was performed on a Shimadzu infrared spectrophotometer, IRAffinity 1S, using the ATR method (MIRacle 10). With this method, spectra were recorded in the range of wavelengths 4000 - 500 cm^{-1}.

Low-temperature nitrogen adsorption was performed on a Micrometrics ASAP 2010 instrument, which determined the textural characteristics of synthetic zeolite and commercial bleaching earth.

The morphological characteristics of the bleaching agents were determined with an electron microscope JEOL-JSM-6460LV at a resolution of 3-4 nm and a magnification of 500-3000 times. The samples were sputtered with gold on a BAL-TEC SCD 005 device, with a current of 30 mA, from a distance of 50 mm for 80 s. Elemental microanalysis was performed with an energy dispersive spectrometer with analyzer, Noran System Six 200 (detection of elements $Z \geq 5$, detection limit of mass fraction ~ 0.1%, resolution 126 eV).

To characterize sunflower oil before and after bleaching, as well as to determine the effects of bleaching, standardized methods prescribed by the Rulebook on the Quality of Edible Oils, Fats and Mayonnaise in Bosnia and Herzegovina [21] were used, which determined the peroxide value and soap content.

The fatty acid composition of the oil was determined by gas chromatography (GC-FID) on an Agilent 7890 GC gas chromatograph. The retention time for individual fatty acids was determined from the obtained chromatograms, and then their mass fractions in the oil samples were determined based on the area and height of the fatty acid peaks.

RESULTS AND DISCUSSION

RESULTS OF CHARACTERIZATION OF SYNTHETIC ZEOLITE AND COMMERCIAL BLEACHING EARTH

Figure 1 shows the diffractograms of synthetic zeolite samples and commercial bleaching earth. Based on the obtained values of intensity, I (cps) and interplane distances d (Å), and by comparing with the interplane distances, it was determined that the examined zeolite sample has a crystal structure of ZSM-5 (MFI) zeolite.

In the diffractogram of a sample of commercial bleaching earth, in addition to smectite and quartz peaks, peaks of phyllosilicate minerals were also identified, such as palygorskite.

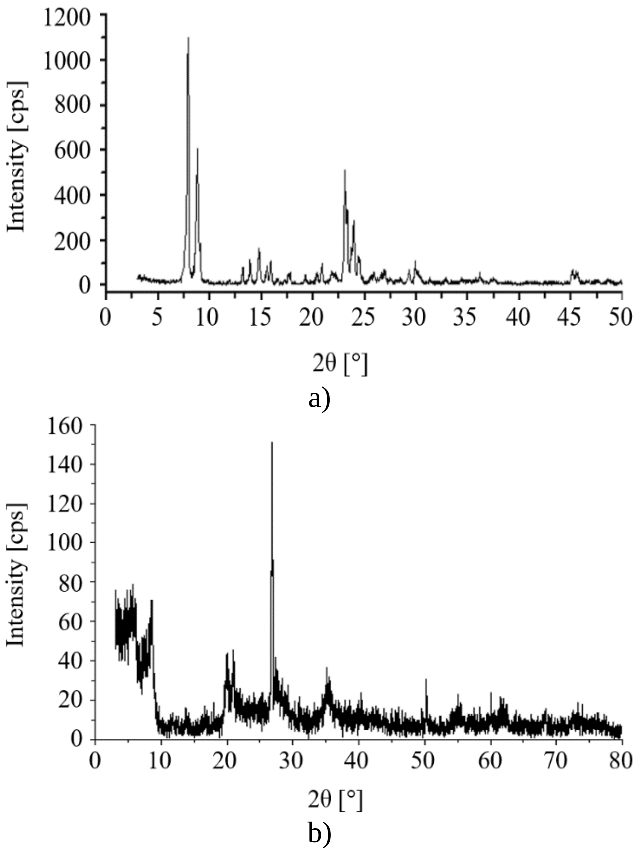


Figure 1. Diffractograms of: a) synthetic zeolite; b) commercial bleaching earth

Figure 2 shows the FTIR spectra of synthetic zeolite and commercial bleaching earth. The FTIR spectrum of the synthetic zeolite shows bands characteristic of ZSM-5 type zeolite. The bands at

~1225 cm^{-1} and ~1070 cm^{-1} originate from external and internal asymmetric stretching vibrations of Si-O-Si bonds, while the band at ~790 cm^{-1} originates from symmetrical stretching vibrations of Si-O-Si bonds. The bands at ~590 cm^{-1} and ~545 cm^{-1} originate from double ring vibrations. The FTIR spectrum of commercial bleaching earth shows bands characteristic of aluminosilicate minerals. The band at ~3600 cm^{-1} originates from stretching vibrations of OH groups coordinated by octahedral Al^{3+}, while the bands at 3400-1630 cm^{-1} originate from stretching and bending OH vibrations of water. The bands in the region of 1020-990 cm^{-1} originate from Si-O and Si-O-Si stretching vibrations, while the band at ~915 cm^{-1} originates from Al-Al-OH bending vibrations. The bands at ~680 cm^{-1} and ~540 cm^{-1} originate from Si-O and Al-O-Si vibrations. A broad band in the range ~790 cm^{-1} to ~750 cm^{-1} indicates the presence of quartz.

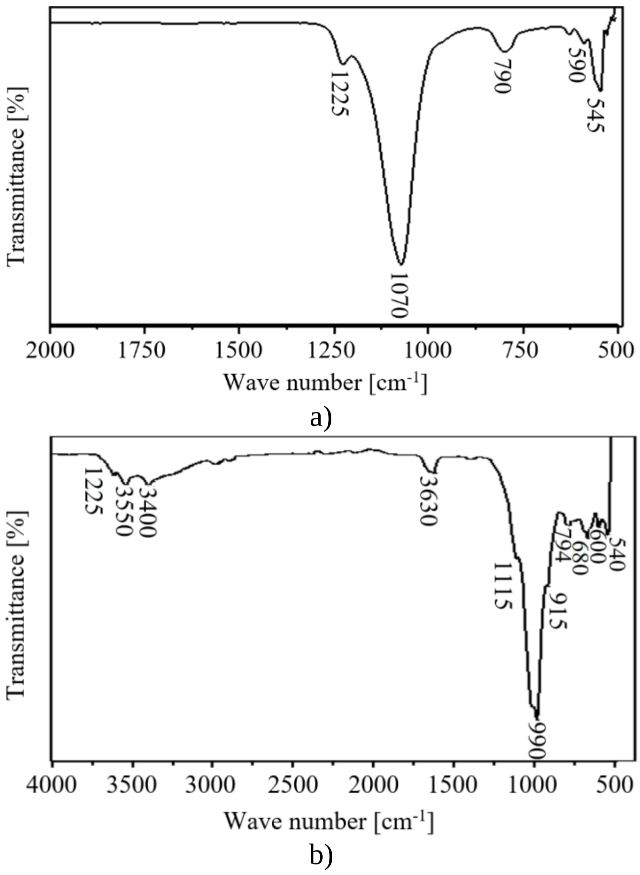


Figure 2. FTIR spectra of: a) synthetic zeolite; b) commercial bleaching earth

Figure 3 shows the adsorption isotherms of synetic zeolite and commercial bleaching earth, and Table 1 shows some of their textural characteristics. The zeolite sample is characterized by a Type I adsorption isotherm for microporous materials [22], where based on the appearance of the isotherm itself and the high value of the specific surface area (325.90 m^2/g), it can be concluded that the sample is dominated by micropores with a certain proportion of small mesopores. Commercial bleaching earth is characterized by a type II adsorption isotherm, which is typical for nonporous or macroporous materials [23].

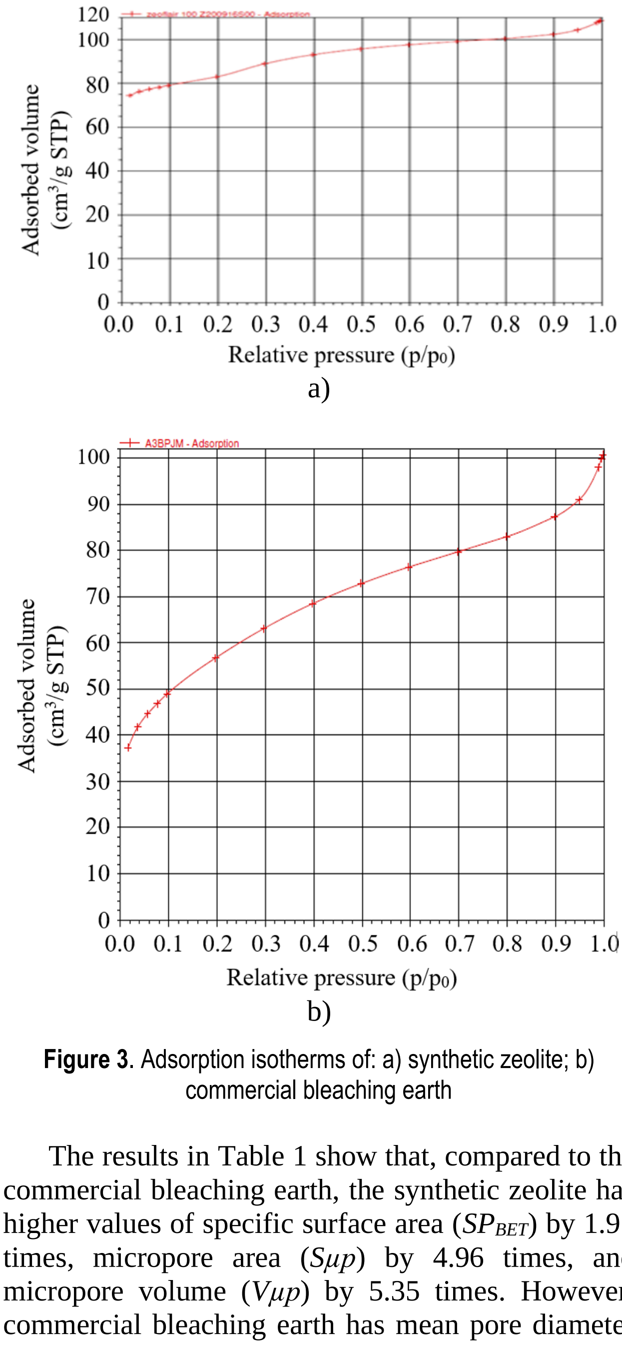


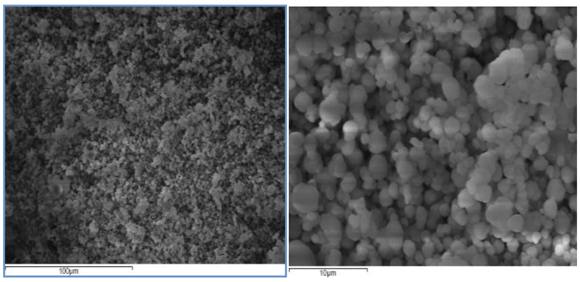
Figure 3. Adsorption isotherms of: a) synthetic zeolite; b) commercial bleaching earth

The results in Table 1 show that, compared to the commercial bleaching earth, the synthetic zeolite has higher values of specific surface area (SP_{BET}) by 1.91 times, micropore area ($S\mu p$) by 4.96 times, and micropore volume ($V\mu p$) by 5.35 times. However, commercial bleaching earth has mean pore diameter (*dp*) and specific external surface area (*SPext*) values 3.11 and 1.09 times higher.

Figures 4 and 5 show SEM micrographs of synthetic zeolite and commercial bleaching earth, at magnifications of 500 and 3000 times. Synthetic zeolite particles are smaller than those of commercial bleaching earth and have significantly higher microporosity, which is confirmed by the fact that the mean pore diameter of synthetic zeolite is smaller than the mean pore diameter of commercial bleaching earth by 3.11 times.

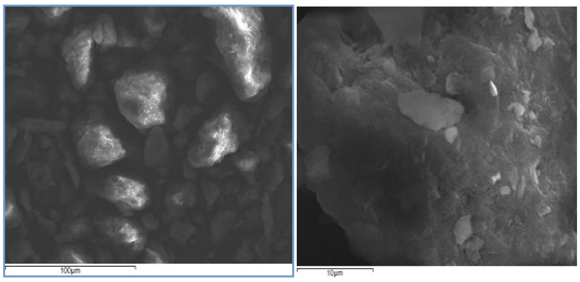
Table 1. Textural characteristics of synthetic zeolite (SZ) and commercial bleaching earth (ZB)

Characteristic	Bleaching agent	
	SZ	ZB
Specific surface area (SP_{BET}), m^2/g	325.90	170.6072
Specific external surface area (*SPext*), m^2/g	113.19	122.9679
Constant (C_{BET})	-66.3216	357.5192
Micropore surface ($S\mu p$), m^2/g	212.702	47.639
Micropore volume ($V\mu p$), cm^3/g	0.111246	0.020798
Mean pore diameter (*dp*), nm	3.8359	11.9390



a) b)

Figure 4. SEM micrographs of synthetic zeolite at magnifications of: a) 500 times; b) 3000 times



a) b)

Figure 5. SEM micrographs of commercial bleaching earth at magnifications: a) 500 times: b) 3000 times

Table 2 shows the mass fractions (*w*) and atomic percentages (*at.*) of individual elements in synthetic zeolite and commercial bleaching earth, obtained by elemental microanalysis (EDS). The zeolite sample has a regular spherical shape and represents silicalite, in which the presence of aluminum and sodium was not detected, and it has a modulus SiO_2/Al_2O_3 >500 (∞). The oxide composition of commercial bleaching earth (*w*) is: 88.32% SiO_2, 7.43% CaO, 1.40% K_2O and 2.85% Fe_2O_3.

Table 2. Elemental analysis of synthetic zeolite (SZ) and commercial bleaching earth (ZB)

Element	SZ, (*w*) %	SZ, (*at*) %	ZB, (*w*) %	ZB, (*at*) %
O	60.03	72.50	68.89	80.93
Al	0.00	0.00	-	-
Si	39.97	27.50	24.46	16.37
Fe	0.00	0.00	3.16	1.06
Ti	0.00	0.00	-	-
K	-	-	0.54	0.26
Ca	-	-	2.94	1.38

THE RESULTS OF SUNFLOWER OIL CHARACTERIZATION BEFORE AND AFTER BLEACHING

The quality of edible vegetable oils in Bosnia and Herzegovina is defined by the relevant Rulebook [21] which prescribes that refined vegetable oils must meet the following requirements: that at a temperature of 25°C they are clear, have a mild and pleasant taste and smell, the color characteristic of the raw material, the mass fraction of free fatty acids (expressed as oleic acid) maximum 0.3%, mass fraction of water and other volatile substances maximum 0.2%, soap content maximum 50 mg/kg (as Na-oleinate) and peroxide value maximum 7 mmol O_2 per kg of oil. The effects of bleaching crude sunflower oil with synthetic zeolite and commercial bleaching earth were determined by methods for determining soap content, peroxide value and fatty acid composition.

The results of soap determination in sunflower oil samples after bleaching with synthetic zeolite and commercial bleaching earth showed that there are no residual soaps in them, i.e. their content was 0.00 mg/kg.

Peroxide value (PV) is a measure of oxidation and change in oil quality (mmol/kg). The determined peroxide value of raw sunflower oil was 6.53 mmol/kg. Table 3 shows the peroxide values of sunflower oil after its bleaching with different mass fractions (*w*) of synthetic zeolite and commercial bleaching earth in oil. The peroxide values of all sunflower oil samples after bleaching with synthetic zeolite and commercial bleaching earth are in accordance with the national regulation on the quality of edible vegetable oils [21]. The results show that increasing the dose of bleaching agent led to a decrease in the peroxide value of the oil, with the synthetic zeolite showing higher efficacy than the commercial bleaching earth. However, a visual comparison of the color of sunflower oil samples after bleaching, with the color of crude and commercial refined oil, showed slightly better effects of changing the color and clarity of the oil using commercial bleaching earth. The reasons for the insufficient change in color and clarity of the oil can be the age of the sunflower oil sample, possibly inadequate filtering equipment, as well as the failure to add activated carbon to the specified bleaching agents.

Table 3. Peroxide values of sunflower oil after bleaching with synthetic zeolite (SZ) and commercial bleaching earth (ZB)

SZ		**ZB**	
(*w*) %	**(*PV*) mmol/kg**	**(*w*) %**	**(*PV*) mmol/kg**
0.2	4.365	0.2	5.75
1.0	3.775	1.0	4.377
2.0	2.475	2.0	3.316
3.0	1.495	3.0	1.229

The types and mass fractions of fatty acids that make up the vegetable oil depend on its type. In tables 4-6. the results of the analysis of the fatty acid composition of sunflower oil samples before and after bleaching with synthetic zeolite and commercial bleaching earth are given. Crude sunflower oil contains the following fatty acids: linoleic, oleic, pamitic, stearic and behenic. In this sample, the mass fractions of saturated fatty acids (SFA), monounsaturated fatty acids (MUFA) and polyunsaturated fatty acids (PUFA) are (*w*): 10.61%, 29.62% and 59.12%. Due to the high content of essential fatty acid (linoleic acid), sunflower oil belongs to the linoleic type of oil [24] and is considered a high-quality edible oil [25],]26].

Table 4. Fatty acid composition of the crude sunflower oil sample

Fatty acid (symbol)	**(*w*) %**
Palmitic (C16:0)	6.43577
Stearic (C18:0)	3.48085
Oleic (C18:1)	29.62076
Linoleic (C18:2)	59.1215
Behenic (C22:0)	0.69442

Table 5. Fatty acid composition of sunflower oil samples after bleaching by synthetic zeolite

Dose of synthetic zeolite ,(*w*) %	**Fatty acid mass fraction, (*w*) %**				
	Palmitic	**Stearic**	**Oleic**	**Linoleic**	**Behenic**
0.2	6.72264	3.57067	28.78736	60.20781	0.71153
1.0	6.68726	3.57808	28.91326	60.1052	0.7162
2.0	6.46943	3.49522	29.57257	59.06908	0.73746
3.0	6.6144	3.56085	29.0606	60.02001	0.74414

Table 6. Fatty acid composition of sunflower oil samples after bleaching with commercial bleaching earth

Dose of commercial bleaching earth, (*w*) %	**Fatty acid mass fraction, (*w*) %**				
	Palmitic	**Stearic**	**Oleic**	**Linoleic**	**Behenic**
0.2	6.60466	3.5589	29.03599	60.10005	0.7004
1.0	6.70676	3.57828	29.04598	59.97751	0.69147
2.0	6.67418	3.57208	29.05314	60.00552	0.69508
3.0	6.61437	3.56525	29.07031	60.06984	0.68023

The results of the analysis of sunflower oil after bleaching with synthetic zeolite and commercial bleaching earth show that there was a slight change in the fatty acid composition, namely: an increase in the content of palmitic, stearic, linoleic and behenic acids, and a decrease in the content of oleic acid. Consequently, there was a slight change in the proportion of fatty acids, i.e. the proportion of SFA and PUFA increased, and the proportion of MUFA decreased.

CONCLUSIONS

In this paper, the possibility of using domestic synthetic zeolite ZEOflair 100 for bleaching sunflower oil, as a possible substitute for imported commercial bleaching earth, was examined. The effects of bleaching sunflower oil with synthetic zeolite were compared with the effects of bleaching with imported commercial bleaching earth. The characterization of synthetic zeolite and commercial bleaching earth was performed using modern test methods, such as: X-ray diffractometry, infrared spectroscopy, low-temperature nitrogen adsorption and scanning electron microscopy with energy dispersive spectrometry. Using the methods of X-ray diffractometry and infrared spectroscopy, it was determined that the examined zeolite sample has a crystal structure of ZSM-5 zeolite, while the commercial bleaching earth consists of smectite and quartz, and phyllosilicate minerals. Using the BET method, it was determined that the sample of synthetic zeolite, as a microporous material, is characterized by adsorption isotherm type I, and commercial bleaching earth by adsorption isotherm type II, which is typical for non-porous or macroporous materials. Based on the results of the textural characteristics, it can be concluded that the synthetic zeolite has values of specific surface, micropore surface and micropore volume that are 1.91 times, 4.96 times and 5.35 times higher than those of commercial bleaching earth. Commercial bleaching earth has higher values of mean pore diameter and external specific surface, by 3.11 times and 1.09 times.

Using the SEM/EDS method, it was determined that the synthetic zeolite is of a regular spherical shape and represents silicalite, in which the presence of aluminum and sodium was not detected, and it has a modulus of SiO_2/Al_2O_3 >500 (∞). The oxide composition of commercial bleaching earth (*w*) is: 88.32% SiO_2, 7.43% CaO, 1.40% K_2O and 2.85% Fe_2O_3.

The quality of edible vegetable oils in Bosnia and Herzegovina is defined by the relevant regulation, and the effects of bleaching raw sunflower oil with synthetic zeolite and commercial bleaching earth are determined by methods for determining its soap content, peroxide value and fatty acid composition. The analysis of sunflower oil samples before and after bleaching found that there are no residual soaps in them, while the peroxide values are significantly lower than those prescribed by the national regulations. By increasing the proportion of bleaching agents, there is a greater decrease in the peroxide value. The fatty acid composition of sunflower oil before and after bleaching with different mass fractions (0.2%, 1.0%, 2.0% and 3.0 %) of individual bleaching agents was determined by the gas chromatography method. The results showed that sunflower oil before bleaching contains: saturated fatty acids (palmitic, stearic and behenic), monounsaturated fatty acids (oleic) and polyunsaturated fatty acids (palmitic). After bleaching the oil with synthetic zeolite and commercial bleaching earth, there was a slight change in the proportion of saturated, monounsaturated and polyunsaturated fatty acids. The bleaching process increased the proportion of saturated and polyunsaturated fatty acids, while the proportion of monounsaturated fatty acids decreased.

The effects of using the researched bleaching agents are similar, with a slightly higher efficiency of synthetic zeolite in reducing the peroxide value of oil. In order to obtain better results and possibly carry out semi-industrial research, laboratory-scale research should be continued using more adequate laboratory equipment and packaging for oil samples, faster analyzes of oil samples after bleaching, as well as using other types of zeolites, combinations of zeolites with different porosity, or combinations of zeolites and activated carbon.

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