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PIF (India) = 1.940
IBI (India) = 4.260
OAJI (USA) = 0.350

SOI: [1.1/TAS](#) DOI: [10.15863/TAS](#)

International Scientific Journal Theoretical & Applied Science

p-ISSN: 2308-4944 (print) e-ISSN: 2409-0085 (online)

Year: 2021 Issue: 09 Volume: 101

Published: 30.09.2021 <http://T-Science.org>

QR – Issue



QR – Article



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OBTAINING CLAY ADSORBENTS FOR BLEACHING VEGETABLE OIL BASED ON BENTONITS OF UZBEKISTAN

Abstract: This article considers the possibility of obtaining bentonite adsorbents from local bentonites by acid activation for the purification of vegetable oils. Based on the study, the adsorption of benzene and water vapours on activated bentonite adsorbents established that the value of the activated Krantau and Askamar samples is almost 2.5 times higher than the initial ones. It is necessary to pay special attention to the fact that, even at low relative pressure values ($P / P_s = 0.2$), the adsorption of benzene vapour increases sharply due to the montmorillonite hydrophilicity. Based on the study, the adsorption of benzene and water vapours on activated bentonite adsorbents established that the value of the activated Krantau and Askamar samples is almost 2.5 times higher than the initial ones. It is necessary to pay special attention to the fact that, even at low values of relative pressure ($P/P_s = 0.2$), the adsorption of benzene vapour increases sharply due to the montmorillonite hydrophilicity. Therefore, the activated samples' adsorption values of benzene vapours are much higher than those of the original illustrations. It confirms that the activator and activation conditions are correctly selected. Furthermore, it was established that biological samples of Askamar and Krantau bentonite belong to micro- and mesoporous adsorbents since they have a large amount of the latter.

Key words: vegetable oils, bleaching, acid number, bentonite, peroxide number, activation, adsorption.

Language: English

Citation: Ismoilova, M. A., Salixanova, D. S., Sadullaeva, M. S., Ruzmetova, D. T., & Agzamova, F. N. (2021). Obtaining clay adsorbents for bleaching vegetable oil based on bentonites of Uzbekistan. *ISJ Theoretical & Applied Science*, 09 (101), 780-786.

Soi: <http://s-o-i.org/1.1/TAS-09-101-112> **Doi:**  <https://dx.doi.org/10.15863/TAS.2021.09.101.112>
Scopus ASCC: 1600.

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Introduction

The oil and fat industry is one of the foremost leading sectors of the food industry in Uzbekistan and determines the country's food security. Vegetable oils, both used directly for food and sent for processing, must be subjected to a complete refining cycle to remove substances harmful to the body, improve presentation, increase organoleptic characteristics, and ensure resistance to oxidation[1].

Adsorption refining (bleaching) is the most crucial stage in the purification of vegetable oils from pigments and the residual amount of phospholipids, salts of fatty acids remaining in oils after the previous refining stages and metal ions.

As adsorbents, particular activated bleaching earth are used, which have selectivity concerning the accompanying substances of vegetable oils.[2].

Currently, bentonite bleaching earth is most widely used in the oil and fat industry. The primary producers are the United States, Malaysia, and China, which determines their high cost on the market. In connection with the above, improving the technology of adsorptive refining of vegetable oils using bleaching clay based on local clays. [3].

As is known [4], the nature of aluminosilicates forming various crystalline forms determines their physicochemical properties and the possibility of selective absorption of gases, vapours or liquids from the environment. Thus, aluminosilicates can act as effective sorbents, which, in turn, are subdivided into groups: natural, inorganic, organo-inorganic, synthetic.

Such compounds are widely used in many industries. However, unfortunately, in Uzbekistan, there is no base for the industrial production of high-quality adsorbents. Therefore, studies of sorption properties, primarily natural aluminosilicate materials (NAM), are of great interest.[7].

High rates of sorption of various substances from solutions are distinguished by the NAM of the montmorillonite (MM) group.[8]. If the material contains by weight not less than 70% of the mineral of the MM group, it is called bentonite.

MM is a clay mineral belonging to the class of layered silicates. Its structure is based on a three-layer package (2: 1). Two layers of silicon-oxygen

tetrahedra facing each other with their vertices cover a layer of aluminium-hydroxyl octahedra on both sides.[9]. In the crystal lattice of MM, the basal oxygen surface of one packet interacts with a similar one due to the van der Waals forces (energy 8–12 kJ/mol). Moreover, in comparison with kaolinite, the packets in the lattice are bound several times weaker. Therefore, water and other polar liquids can penetrate between the MM packages and push them apart, which manifests itself in the intense swelling of such NAMs compared to kaolinite ones, in that the MM lattice can split. The interplanar spacing for MM is not as rigid as for kaolinite and can vary from 1.0 nm in a dry state to 14.0 nm, that is, until the complete separation of layers with intense water saturation and the predominance of sodium cations in the absorbed complex.[10].

Methodical part.

Chemical analysis of bentonite clays was carried out following SS 21216-2014 [11], the mineral composition of clays using the Dron-4.0 device, CuK α - and CoK α radiation, Ni - filter [12-13], the adsorption characteristics of vegetable oils before and after their purification were determined according to SS R 52465-2005. Sunflower oil specifications.

Results and discussion.

To understand the reason for the value of bentonite as a promising material for processing into effective sorbents, let us consider the structure of the main mineral in more detail.

Of great interest from the point of view of varying sorption properties is MM-containing clay Askamar (Navoi), Krantau (Karakalpakstan). Therefore, the chemical and mineral composition of MM was studied to obtain a high sorption adsorbent from it.

Chemical analysis of fine clay fractions was carried out following GOST 21216-2014 [11], according to which the weight percentages of SiO₂, TiO₂, Al₂O₃, Fe₂O₃, MgO, MnO, CaO, Na₂O, K₂O and P₂O₅ were determined. From the data of the conducted chemical analysis, the studied clay is rich in alkali metal ions.

Table 1. Chemical composition of mineral clays

Clay	Content, % on dry matter											
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃ +FeO	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O	P ₂ O ₅	SO ₃	CO ₂	b.l.
Askamar	60,5	19,9	5,20	0,67	1,8	1,95	2,9	2,7	0,04	0,01	0,07	5,2
Krantau	60,3	16,8	6,6	0,4	1,1	1,9	3,6	2,4	0,2	0,3	0,1	6,7
Dehkanabad	57,91	14,04	5,10	0,8	0,48	1,84	1,53	1,75	0,43	0,75	0,2	15,97

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According to the data, the studied samples differ in the content of basic oxides; however, one of the main active oxides, sodium oxide of Askamar and Krantau, is greater than that of the NAB sample allows you to get adsorbents with higher adsorption properties. The mineralogical composition investigated on a Dron-4.0 device, radiation from CuK_{α} and CoK_{α} , Ni - filter. Sample preparation for X-ray phase analysis carries out following the guidelines.[12,13]. The objects under study preliminarily dried to constant weight, then they were ground in an agate mortar and sieved through a sieve of size 006.

Based on the studies [14], the Askamar bentonite contains montmorillonite, palygorskite, quartz. As well as Askamar bentonite is of the highly clayey montmorillonite type, i.e. forms bydelite. The chemical composition of bentonite also indicates that it contains 15% alumina.

Comparison of X-ray structures identified phase compositions.[15]. Analyzes show that in the clay sample of the Krantau deposit (KR), which contains mainly sodium montmorillonite, besides the lines characteristic of montmorillonite, there are also lines showing the presence of illite, kaolinite, hydromica, feldspar. [16].

Thus, based on a comprehensive study of the Askamar and Krantau deposits' bentonites, the main component is the mineral montmorillonite. The clay of these deposits differs from other clays of Uzbekistan by the low content of harmful impurity non-clay materials. Montmorillonite is the main constituent of the bentonite mineral. It distinguishes by many fine fractions and a unique structure, which demonstrates qualitative aspects: high adsorption properties and the ability to form a stable suspension. Therefore, the dispersion of previously selected samples studied—the results are in Table 2.

Table 2. Fractional composition of local clays

Clay name	Particle size, mm		
	0,06	0,06-0,0015	0,0015мм и менее
Askamar	0,8	2,7	96,5
KR2	0,6	2,0	97,4
Dehkanabad	0,22	2,3	97,4

Table 2 shows that Askamar is a finer dispersed bentonite mineral since there are more small particles. It was found that on average the main fraction (up to 95%) is 0.0015mm, (up to 2.5%) from 0.06-0.0015mm and (up to 0.10%) from 0.06mm in size particles.

Adsorbents for the purification of vegetable oils should: characterized by high activity, low oil absorption, chemical inertness concerning oil - the adsorbent should not cause oxidation, polymerization, decomposition in oil; separate from the oil by technically simple means, such as filtration; do not affect the smell and taste of the oil.

Based on these factors, the acid activation of the selected bentonite samples was carried out, under the conditions the acid concentration (H_2SO_4 , HCl) was from 10 to 20% in an amount of 1: 4, the rotation of the stirrer was 400 rpm, for 4-6 hours. After activation with acid, bentonite neutralized with distilled water. Then bentonite is dried at a temperature of 100-105 °C for 2 hours, and then it is ground to the required size. After acid activation, the structure and, accordingly, the properties of minerals change significantly. In

addition to the well-known montmorillonite, the organosilicon substance also exhibits high sorption capacity about the accompanying implications of vegetable oils. [17].

Next, the adsorption properties of the obtained adsorbents were studied, measured in a high-vacuum installation on a McBenas device with mercury gates and quartz balances at a residual pressure of $1.33 \cdot 10^{-3}$ Pa and a temperature of 298 K. The extension of the springs of the installation monitored using a KM-8 cathetometer.[18,19]. Figure 1-2 shows a diagram illustrating the changes in water and benzene adsorption values on the initial and activated samples of Krantau and Askamar clays.

Based on studying the adsorption isotherms of water vapour, the value of the activated Krantau and Askamar samples is almost 2.5 times higher than the initial ones. It is necessary to pay special attention to the fact that, even at low values of relative pressure ($P/P_s = 0.2$), the adsorption of benzene vapour increases sharply due to the hydrophilicity montmorillonite.

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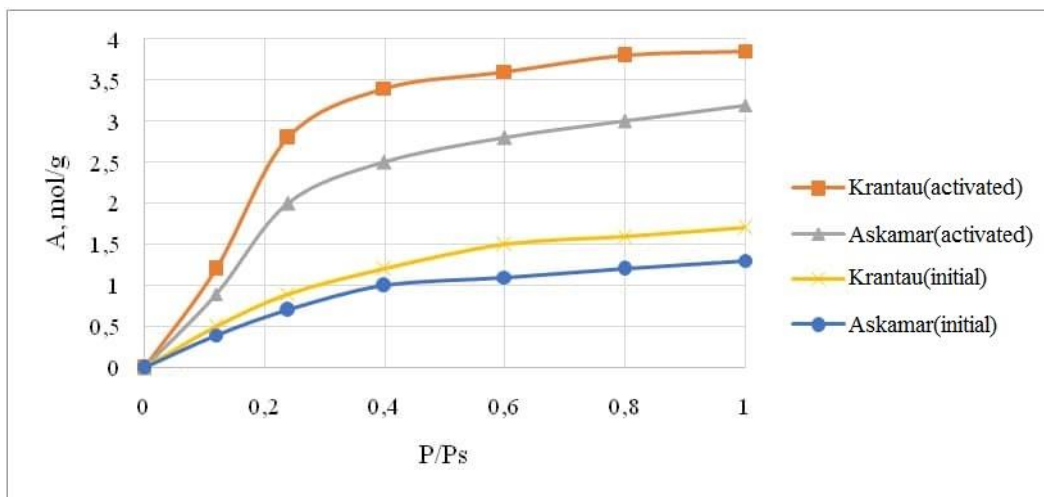


Fig 1. Isotherms of water vapour adsorption on the initial and activated samples of Krantau and Askamar

Figure 2 shows the results of the adsorption of benzene vapour. As the results show, the adsorption

of benzene vapour is much lower than the adsorption of water vapour.

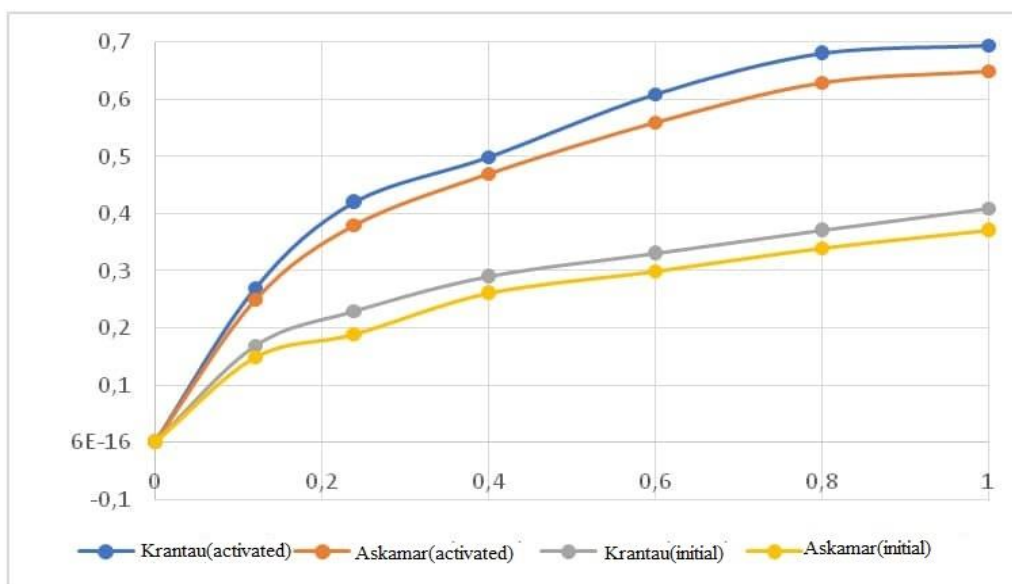


Fig 2. Isotherms of benzene adsorption on the initial and activated samples of Krantau and Askamar.

The activated samples' adsorption values of benzene vapours are much higher than those of the original illustrations. This confirms that the activator and activation conditions are correctly selected. Based on adsorption isotherms, the values of the texture

parameters of the samples under study were calculated using the corresponding equations.

Based on the table results, it was established that biological samples of the Askamar and Krantau bentonite belong to micro- and mesoporous adsorbents since they have a large amount of the latter.

Table 3. Textural characteristics of Askamar and Krantau samples calculated from water vapour adsorption isotherms

№	Name of the adsorbent	Monolayer capacity, A_m	S_{sp} , m^2/g	Saturation adsorption α_s , mol/kg	Micropore volume, W_0 , sm^3/g	Mesopore volume, W_0 , sm^3/g	Saturation volume, $V_s \cdot 10^3$, m^3/kg
1	Krantau	1,929	125,42	3,85	0,47	0,3	0,745

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	(activated)						
2	Askamar (activated)	1,409	91,58	3,2	0,45	0,2	0,66
3	Krantau (initial)	0,653	47,35	1,7	0,17	0,11	0,164
4	Askamar (initial)	0,55	35,77	1,3	0,151	0,09	0,118

Table 4. Textural characteristics of Askamar and Krantau samples calculated from adsorption isotherms of benzene vapors

№	Name of the adsorbent	Monolayer capacity, A _m	S _{sp} , m ² /g	Saturation adsorption	Mesopore volume, W ₀ , sm ³ /g	Monolayer capacity, A _m	S _{sp} , m ² /g
1	Krantau (activated)	0,192	46,35	0,84	0,264	0,107	0,0745
2	Askamar (activated)	0,278	66,83	0,65	0,146	0,09	0,057
3	Krantau (initial)	0,171	41,14	0,41	0,104	0,06	0,041
4	Askamar (initial)	0,15	36,04	0,37	0,087	0,043	0,037

Next, the obtained adsorbents carried out the bleaching of sunflower oil, which shows Table 5. For comparison, the bentonite adsorbents of Pakistan, which are used today in enterprises, are taken. The bleaching conditions are standard, i.e. temperature 60-70 °C, amount of adsorbent 1%.

As shown in Table 5, the adsorbents obtained from local bentonites by acid activation practically do not differ from each other in their adsorption resilience. However, it is noticeable that the adsorbent obtained from the Krantau bentonite oxidizes the oil somewhat less after acid hydration, as indicated by the peroxide number.

Table 5. Oil indicators, before and after their adsorption refining by the obtained activated adsorbents

Indicator name	Indicator value			
	Before adsorption cleaning	After cleaning		
		Adsorbent obtained from Askamar bentonite	Adsorbent obtained from Krantau bentonite	Pakistani adsorbent (control)
Colour number, mg of iodine	10	4	3	4
Acid number, mg KOH/g	0,55	0,17	0,12	0,11
Peroxide number, mmol of active oxygen/kg	8,2	6,8	5,6	6,1

Further, technological characteristics such as filtration rate and oil absorption are studied. The results show in the table. 6.

As can be seen from the above data, oil absorption in the two proposed samples is high because the oil content of the waste bleaching earth should be in the range of 25% to 40%.

The higher the oil absorption value, the higher the oil loss. In the process of filtration, as a result of the compaction of the layer, in the presence of small fractions in it, the resistance of the sediment layer can increase several times.[13]. Then the finely dispersed adsorbent adsorption properties increase; however, the technical characteristics reduce.

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Table 6. Study of the influence of the amount of adsorbent on their properties

Indicator name	Indicator value		
	Adsorbent obtained from Askamar bentonite	Adsorbent obtained from Krantau bentonite	Pakistani adsorbent (control)
Oil capacity, %	35,9	34,3	35,2
Filterability (filtration time 100ml), s	214	248	242

Thus, for acid activation, the chemical, mineralogical, and granulometric compositions of bentonite clays have been investigated, and it has been established that the studied samples differ in terms of the content of basic oxides; however, one of the principal active oxides, sodium oxide of Askamar and Krantau, is greater than that of the NAB sample.

Based on the study of the adsorption of benzene and water vapours on activated bentonite adsorbents, it was established that the values of the activated

Krantau and Askamar samples are almost 2.5 times higher than the initial ones. It is necessary to pay special attention to the fact that, even at low values of relative pressure ($P/P_s = 0.2$), the adsorption of benzene vapour increases sharply due to the hydrophilicity montmorillonite. The adsorption values of benzene vapours of activated adsorbents are much higher than those of the initial samples. This confirms that the activator and activation conditions are correctly selected.

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