

ISSN 2181-8622

**Manufacturing technology problems**



# **Scientific and Technical Journal Namangan Institute of Engineering and Technology**

INDEX  COPERNICUS  
INTERNATIONAL

**Volume 8  
Issue 4  
2023**



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## INVESTIGATION OF THE CHEMICAL-MINERALOGICAL COMPOSITION OF BENTONITE OF THE KHAUDAG DEPOSIT AND SYNTHESIS OF WINE FINING AGENTS BASED ON ITS

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**Abstract.** Fining is about removing unwanted material from wine while still in the cellar. It is part of the clarification and stabilisation process and involves adding a substance to the wine that will flush out



certain elements that may cause a wine to look hazy or affect its aroma, colour or bitterness. Fining removes 'colloids', which are molecules that include tannins, phenolics and polysaccharides. The fining agent binds to the unwanted particles in the wine, which means they become sizeable enough to be filtered out. The chemical and mineralogical composition of the Khovdak bentonite clay for the production of sorbents was studied. Microscopic studies of samples of bentonite clays were carried out using an MBS-10 optical microscope in transmitted and polarized light with an installed digital camera with a magnification factor of up to 600 s. The study of the phase composition of samples of bentonite clays of the Khaudag deposit was carried out by X-ray analysis.

**Keywords:** bentonite clay, bentonite, sorption, phase composition, wastewater treatment, sorbent.

**Introduction.** Fining is understood as clarifying, making wine resistant, correcting its acidity, and accelerating the aging process. For this, wetting agents (gelatine, fish glue, egg protein, bentonite, etc.) are added to the wine, which react with the colloidal substances contained in the wine. Proteins and additives in wine react with astringents to form insoluble compounds. These compounds settle down in the sediments, and during the sedimentation, the small particles in the wine become briquettes and make the wine clear [Abdullaev et al., 2012].

Fining is not only to clarify the wine, but also improves its taste and aroma, and increases its resistance to diseases. During wine fining, small particles of wine are adsorbed on the coagulated particles. As a result, under the influence of gravity, it forms bubbles, sinks down and separates from the wine [Abdullayev et al., 2019].

Bentonite, which is used as an adhesive, is considered an inert substance and does not react chemically with substances contained in wine. It adsorbs small particles contained in the wine, forms coagulated patches, sinks down and separates from the wine [Ivanova et al., 2008].

In the world, bentonites and their varieties play a significant role in the form of a mineral supplement, in the breeding of farm animals and poultry, as well as deodorization of premises. The use of bentonite clays is associated with their chemical composition, which includes many vital micro and macro elements.

Various artificial and natural materials are used as sorbents: ash, coke breeze, peat, silica gels, aluminum gels, active

clays, varieties of bentonite, etc. It should be noted that, depending on the nature and content of minerals, their sorption and selective properties of the oils removed from cottonseed related substances. Thus, for example, it is known from the literature that Angren kaolin effectively absorbs gossypol and its derivatives [Nadirov, 1973], and activated carbon absorbs carcinogenic substances (residues of a hydrocarbon solvent, 3,4-benzopyrene, etc.) [Ksenofontov, 2011].

In this study, compounds based on bentonite and organic monomers were synthesized [Eshkurbonov et al. 2022a-c]. Recommendations are given on the use of the synthesized compounds in the food industry.

It should be noted that in order to achieve maximum levels of water purification using sorbents based on bentonite clay, their activation and modification is required. In turn, the organization of the production of adsorbents on an industrial scale requires targeted research and the development of new and highly efficient technologies for the complex processing of bentonite clays based on their chemical and mineralogical composition, structure and sorption properties their further place of application.

In the Jarkurgan district of the Surkhandarya region, there is the Khaudag deposit, where there are reserves of bentonite raw materials in the amount of 1091 thousand tons, which are developed by the MS-MARJON Chamber of Commerce and Industry to meet the needs of farms with their products as agro-ore raw materials and other areas of consumption.



Researchers [Eshkurbonov et al., 2022c] for the first time modified Khaudag bentonite with polymer compounds and obtained a new composition. Most natural clay adsorbents partially or completely do not adsorb the carcinogenic substance 3,4-benzo[a]pyrene from the extraction oil.

**Methods and materials.** This problem has been proven to be partially overcome with the help of the above synthesized compound. In addition, as a result of the research, the polymer product obtained on the basis of dimethylolurea and orthophosphoric acid (DMU+OPA) was reprocessed with bentonite and a composition was obtained. In order to determine the possible areas of application of this mechanical composite, its studied physical and chemical properties were presented in further studies [Eshkurbonov et al., 2022b].

A composition was created on the basis of Khaudag bentonite and a polymer additive, and practical work was carried out on its effectiveness in cleaning vegetable oils. [Eshkurbonov et al., 2022a] Optimum conditions for obtaining an import-substitute composition for oil refining with the help of synthesized compositions were determined. According to it, it was found that the UFPA-1-B sorbent (based on urea-formaldehyde (UF) and phosphoric acid) has higher sorption properties than the UFAP-1-B (based on UF and ammonium phosphate) composition. In addition, it was determined that the optimal conditions for activation are 4 hours of activation using 15% H<sub>2</sub>SO<sub>4</sub> acid. It was found that the use of 2% in oil whitening with the help of the obtained compositions leads to maximum oil purification. The amount of bleaching sorbents used varies from 0.5 to 5%, depending on the amount of dyes in the oil to be treated and the degree of bleaching required.

When an activated adsorbent is used in the bleaching process, a small amount of isomerization and the formation of glycerides containing sequentially linked

fatty acids are observed. This, of course, leads to a decrease in the quality of refined oils and fats and a shortening of their shelf life. The conditions mentioned above and the size of the oil capacity require that the amount of activated earth used for bleaching be reduced as much as possible. At present, activated adsorbents are imported from foreign countries and used for adsorption purification of vegetable oils in various sectors of our oil industry [Mamajonova et al., 2020]. Localization of adsorbents coming from abroad and their use in the food industry remains one of the urgent issues of today.

In this research work, for the first time, studies were conducted on the use of Khaudag bentonite and its composite compound with polymer compounds for wine clarification in the wine industry. Before starting research work, bentonite of Khaudag is acid activated. After that, it is separately modified with polyacrylonitrile (PAN) and DMU.

A comprehensive study of the mineralogical composition of bentonite and its modified forms was carried out by the methods of X-ray, thermogravimetric and electron microscopic analyzes, the use of which makes it possible to explain the influence of changes occurring in the process of modification on their properties.

X-ray diffraction patterns were taken with an XRD-6100 X-ray powder diffractometer (Shimadzu, Japan). CuK $\alpha$  radiation was used ( $\beta$ -filter, Ni, 1.54178 current mode and tube voltage 30 mA, kV) and a constant detector rotation speed of 4 deg/min with a step of 0.02 deg, and the scanning angle varied from 4 to 80°.

The cell for DTA has a recorder of the temperature difference between the test sample and the standard - aluminum oxide Al<sub>2</sub>O<sub>3</sub> and a recorder of the sample temperature. The method of work includes preparing the device for operation, calibration processes, conducting the main test, processing curves, calculations and interpretation of the results obtained.

Electron microscopic studies were carried out on a scanning electron microscope (SEM) EVO MA10 SEM Cari Zeiss, the use of which makes it possible to view a sample on SEM, determine its quantitative elemental composition, and also obtain color microphotographs.

Thermogravimetric studies were performed using a Paulik-Paulik-Erdey derivatograph system. The heating rate of the samples was 10°/min, the mass of the test sample was 0.1 g.

The preparation of samples for X-ray phase analysis was carried out in accordance with the guidelines [Higerovich and Merkin, 1968 ; Gorshkov, 1981 ]. The objects under study were preliminarily dried to a constant mass, then ground in an agate mortar until they completely passed through a 006 sieve. unit cell parameters of clay samples according to the formulas below [Frank-Kamenetsky, 1984]:

$$a = \frac{b}{\sqrt{3}}$$

$$b = 6 * d_{060}$$

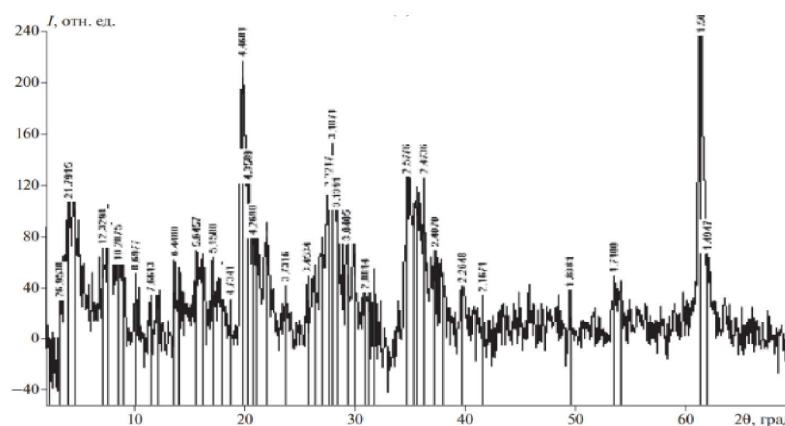
$$\sin\beta = \frac{2d_{020}}{a}$$

$$c = \frac{l * d_{00l}}{\sin\beta}$$

**Results and discussion.**

Experiments have shown that a clay sample from the Khaudag deposit contains mainly sodium montmorillonite (1.4250; 1.3012; 0.4263; 0.2729; 0.2298; 0.1801; 1.4090 nm). The presence of intense lines characteristic of montmorillonite (Fig. 1) proves that in this case it is the main rock-forming mineral. In addition to the lines

characteristic of montmorillonite, there are also lines showing the presence of illite (0.9891; 0.3870; 0.3853; 0.3611; 0.3001; 0.1597; 0.1699 nm), kaolinite (0.6993; 0.26001; 0.1499 nm), hydromicas (0.4606; 0.2971; 0.2487; 0.1677; 0.1561 nm), feldspar (0.3853; 0.2298; 0.1884). The sodium form of montmorillonite is proved by the reflex d001 = 1.301 nm.



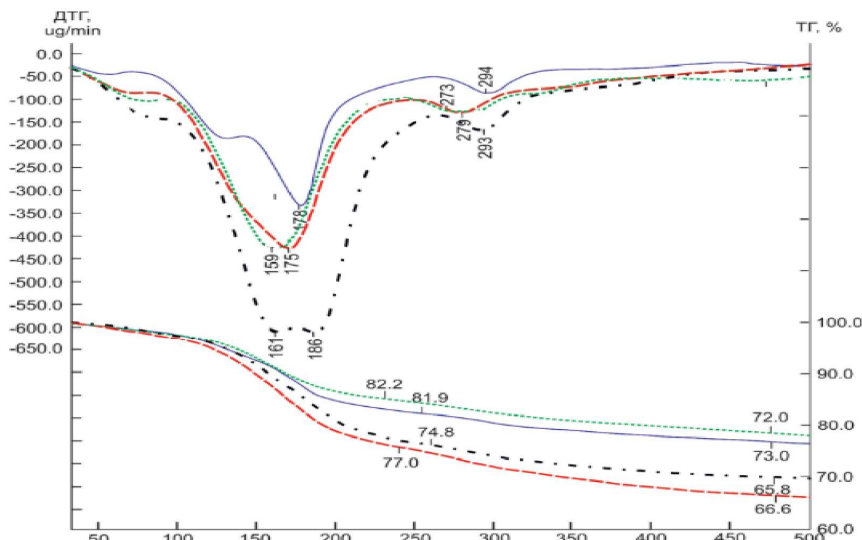
**Fig.1. X-ray diffraction pattern of a Khaudag bentonite sample**

Thermal analysis data are consistent with the results of the previous analysis. On the thermogram of Khaudag bentonite at temperatures of 80-125°C, an intense

endothermic effect is observed, due to the release of adsorption and interlayer molecular water (Fig 2). The presence of an additional effect at temperatures with a

maximum of about 535°C is caused by the removal of structural water. The third weakest endoeffect at temperatures above 790°C corresponds to the destruction of the

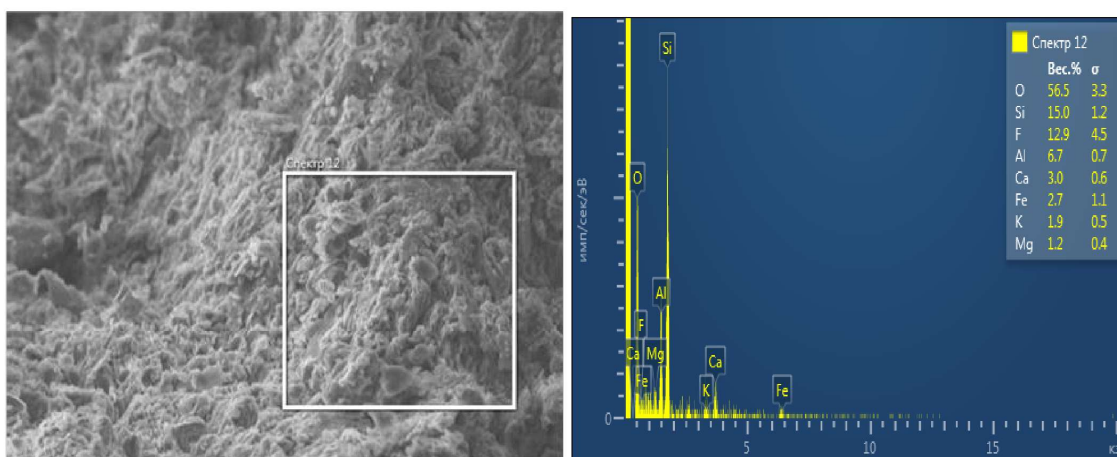
anhydrous modification of montmorillonite and the transformation of the layered aluminosilicate matrix.



**Fig.2. Curve of thermal analysis of Khaudag bentonite sample**

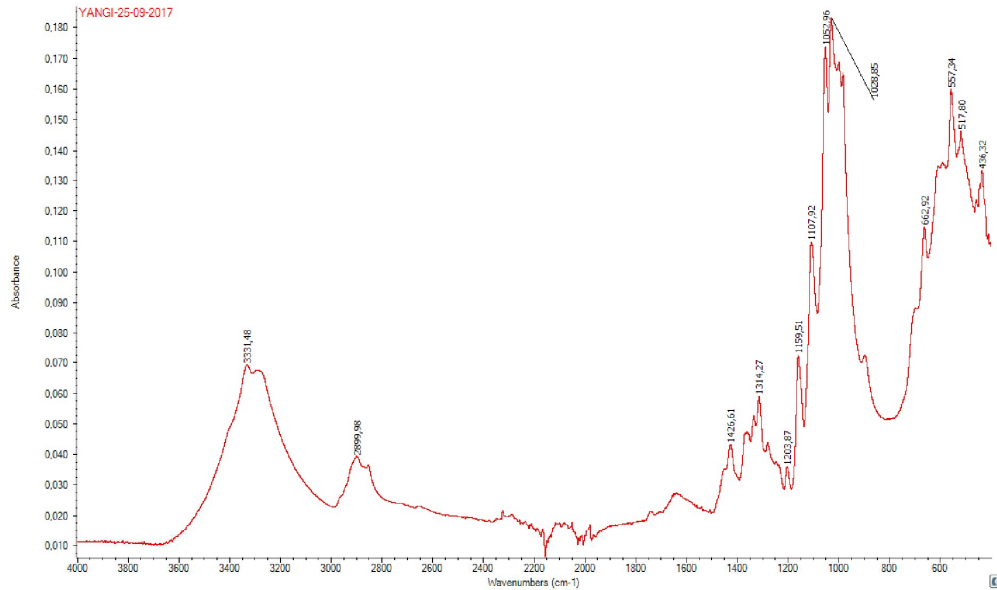
Clay samples were studied by Fourier IR spectroscopy on a Nicolet iS50 Fisher Scientific spectrometer, as well as on a UV-NIR spectrophotometer (spectral region 185-3300 nm) UV 3600 Shimadzu. For spectral tests, samples were prepared in powder form and in the form of tablets. The samples were pre-dried at 105°C in an

oven for more than 7 hours. The following chemical elements are fixed on the energy-dispersive spectrum of clay (in descending order): oxygen, silicon, aluminum, iron, potassium, sodium, etc.(Fig 3) Microdiffraction analysis confirms the results obtained by the method of chemical analysis of the composition.



**Fig.3. Electron microscopic image of a sample of Khaudag bentonite**





**Fig.4. IR spectrum of Khaudag bentonite sample**

The interpretation of the above spectrum shows that the main bands shown on them belong to the valence bonds of silicon with oxygen and hydrogen with oxygen (Fig4). A well-defined broad band at 1028.85 cm<sup>-1</sup> corresponds to the stretching vibrations of the Si-O-Si tetrahedra of the silicon-oxygen framework, and the bands at 517.80 and 436.32 cm<sup>-1</sup> correspond to the bending vibrations of the Me-O bonds. The band in the interval 662.92 cm<sup>-1</sup> corresponds to the Si-O-Si vibrations of rings of SiO<sub>4</sub> tetrahedra. Intense bands in the interval 2899.98-3331.48 and 1426.61 cm<sup>-1</sup> refer to OH-valence and deformation vibrations of free and bound water.

Chemical analysis of fine clay fractions was performed according to GOST 21216-2014 [GOST, 1995], according to which the weight percentages of SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MgO, MnO, CaO, Na<sub>2</sub>O, K<sub>2</sub>O, and P<sub>2</sub>O<sub>5</sub> were determined. It follows from the data of the chemical analysis that the studied clay is rich in alkali metal ions. In tab. 1 also provides data on the chemical composition of Sherobod bentonite, as a control sample and it shows changes in the chemical composition of bentonites from the Khaudag and Sherobod deposits before and after their enrichment.

Table 1

**Chemical composition of clay samples**

Name of chemical components	Khaudag bentonite, %		Sherobod bentonite, %	
	before enrichment	after enrichment	before enrichment	after enrichment
SiO <sub>2</sub>	61,2	55,3	54,91	38,02
Al <sub>2</sub> O <sub>3</sub>	16,3	21,2	14,6	30,5
CaO	3,6	3,8	1,6	1,8
Fe <sub>2</sub> O <sub>3</sub>	2,8	2,3	6,6	6,2
Na <sub>2</sub> O	2,4	2,2	1,9	1,7
MgO	3,6	3,8	1,6	1,8
MnO	0,7	0,8	0,4	0,8
K <sub>2</sub> O	2,6	2,3	1,65	1,4

P <sub>2</sub> O <sub>5</sub>	0,3	0,5	0,56	0,68
CO <sub>2</sub>	0,12	0,10	0,18	0,25
loss after ignition	6,38	7,7	16	16,85

From tab. Table 1 shows that the content of SiO<sub>2</sub> after enrichment of Khaudag bentonite by elutriation decreased from 61.2 to 55.3%, and of Sherobod bentonite - from 54.91 to 38.02%. And vice versa, the content of Al<sub>2</sub>O<sub>3</sub> after enrichment of Khaudag bentonite increased from 16.3 to 21.2%. At the same time, the content of coloring oxides (Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, etc.) in both samples decreased by about 2 times. In terms of Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> content, the enriched bentonite of the Sherobod deposit exceeds the enriched Khaudag bentonite. This favorably affects the quality of the resulting adsorbents and once again confirms the special need for the bentonite enrichment process before their activation.

Above, wine clarification processes were carried out with the help of acid-activated and modified adsorbents. During different periods of time, activated

adsorbents were used in the amount of 2% and clarification processes were carried out. The obtained results are presented in Table 2.

As can be seen from Table 2, the wine indicators clarified with the help of adsorbents obtained as a result of activation for 4 hours showed the best indicator. Therefore, 4 hours was taken as an optimal condition for further activation processes. For our next work, it is planned to carry on Khaudag bentonite, as this bentonite has been found to clean wine better than Pakistani alkaline bentonite.

The maximum amount of adsorbent used for acid concentration and clarification was used above. At the next stage, research was conducted to find the optimal conditions for acid concentration and adsorbent amount. The obtained results are presented in the following tables 3-4.

Table 2.

**Effect of activation time on wine clarification process**

Name of adsorbent	Activation time, hours	Wine color 35 yellow, in a cuvette 12.5 cm thick		Wine output, %
		Red Unit	Blue Unit	
	Initially	14,2	0,3	-
Khaudag bentonite	2	10,1	0,2	97,5
	4	7,6	0,1	97,2
	6	7,2	0,1	98,1
PAN + Khaudag bentonite	2	7,2	0,1	97,1
	4	6,8	0,09	97,6
	6	6,3	0,07	98,2
DMU + Khaudag bentonite	2	7,0	0,9	96,8
	4	6,4	0,07	97,5
	6	6,1	0,05	98,4
Pakistani Bentonite	2	8,2	0,3	96,8
	4	7,8	0,2	96,5
	6	7,6	0,2	97,2

Table 3

**Effect of acid concentration on wine clarification process**

Acid concentration, %	Wine color 35 yellow, in a cuvette 12.5 cm thick		Wine output, %
	Red Unit	Red Unit	
10	7,2	0,15	97,7
15	7,1	0,13	98,3
20	7,4	0,12	97,5
25	7,6	0,14	96,2

Table 4

**The influence of the amount of adsorbent on the wine clarification process**

Amount of adsorbent, mg	Wine color 35 yellow, in a cuvette 12.5 cm thick		Wine output, %
	Red Unit	Blue Unit	
1	7,6	0,12	96,9
1,5	7,3	0,14	97,5
2	7,8	0,15	96,3
2,5	8,2	0,14	96,7

From the tables 3 and 4 above, the most optimal condition for the activation of the adsorbent was determined to be the activation using 15% H<sub>2</sub>SO<sub>4</sub> acid for 4 hours. In addition, it was determined that the consumption of adsorbent for clarification is 1.5%.

**Conclusion.** In conclusion, the optimal conditions for obtaining an adsorbent that replaces import for wine clarification using local Khaudag bentonite were determined. According to it, it was found that Khaudag bentonite has higher adsorption properties than Pakistani

alkaline bentonite. Thus, a comprehensive study of the bentonite of the Khaudag deposit showed that its main constituent is the mineral montmorillonite. The clay of this deposit differs from other clays of Uzbekistan by a low content of harmful impurity non-clay materials.

In addition, it was determined that the optimal conditions for activation are 4 hours of activation using 15% H<sub>2</sub>SO<sub>4</sub> acid. It was determined that the use of 1.5% in wine clarification with the help of adsorbents leads to the maximum purification of wine.

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