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GRADE OF THE FLOCCULANTS' EFFICIENCY IN WASTEWATER TREATMENT

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ABSTRACT

The study aims to develop an efficient and sustainable method for treating industrial wastewater using locally sourced bentonite and palygorskite clays modified with chemical activators (e.g., sulfuric acid) and combined with coagulants ($Al_2(SO_4)_3$, $FeCl_3$) and flocculants (PAA, CMC). The research seeks to optimize sorption properties, reduce chemical oxygen demand (COD), and enhance the economic viability of wastewater treatment in manufacturing enterprises. Bentonite and palygorskite clays were activated via acid treatment (H_2SO_4, 10–20%) at 95°C for 3–6 hours, followed by washing, drying, and milling. Physicochemical Analysis: X-ray diffraction (XRD), IR spectroscopy, and derivatography were used to study structural and thermal properties. Sorption Testing: Clay powders were evaluated for wastewater treatment efficiency under varying conditions (temperature, acid concentration, activation time). Coagulation-flocculation-sorption experiments were conducted using $Al_2(SO_4)_3$, $FeCl_3$, PAA, and CMC to assess COD reduction and oil/particle removal. Activated palygorskite-bentonite composites exhibited superior sorption activity (52.5–67.5%) due to increased porosity and polarity. IR spectra confirmed functional group interactions (e.g., SiO_4^{2-}, OH^-), enhancing sorption. The combined method (sorbent + coagulants + flocculants) achieved high treatment efficiency with minimal sludge production. Local sorbents matched or outperformed imported alternatives, reducing treatment time to 15–20 minutes—first application of acid-activated palygorskite-bentonite blends in wastewater treatment, leveraging local Uzbek raw materials. Novel combined chemical approach integrating sorption, coagulation, and flocculation, optimizing resource use. Demonstrated economic feasibility through reagent recycling (e.g., acidic filtrate reuse). Environmental Benefits: Reduces reliance on imported sorbents and minimizes hazardous sludge generation. Industrial Adoption: Scalable for gas/oil, textile, and food industries, aligning with circular economy principles. Policy Support: Provides a model for sustainable wastewater management in resource-limited regions.

Keywords: Wastewater Treatment, Bentonite, Palygorskite, Sorption-Coagulation, COD Reduction, Local Sorbents.
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INTRODUCTION

The global population growth has led to a substantial rise in the demand for environmentally sustainable and high-quality food products.[1,2] In particular, the production of high-purity vegetable oils necessitates advancements in sorbent technology to enhance sorption efficiency.[3,4] The development of novel bleaching agents, particularly those derived from locally sourced raw materials, is crucial for the processing of these products. Their application in the refining process within the oil industry holds significant importance in ensuring quality and sustainability.[4,5] Wastewater treatment of industrial enterprises is one of the necessary processes in industrial production. More precisely, such a process is called adsorption treatment; its main task is to clean various colloidal micro-dispersed particles and sorbable substances in wastewater.[6-8] The very name of adsorption treatment indicates that this process is carried out with the help of sorbents - substances that can absorb impurities in wastewater.[9,10] The sorbents used in wastewater treatment meet the following requirements. The sorbent should, as much as possible, sorb various compounds. However, given that sorbents are selective, the task is to choose them depending on the type of compounds. In our case, the use of soils such as phloredins, as well as activated carbon, as sorbents for wastewater treatment is widely used. Sorbents should have high activity; that is, the lower the amount of sorbent used per unit volume of wastewater, the higher its activity. This indicator

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is important because the sorbent absorbs part of the wastewater. The sorbent should not undergo various chemical changes - processes such as polymerization, decomposition, oxidation, that is, it should have chemical indifference. Bentonite clay obtained from the Tamditou mine in the Navoi region is crushed, and a suspension is prepared in water. Then, mineral acid (for example, sulfuric acid) is introduced in the amount of 35-50% of the dry clay mass obtained for activation. The acid concentration in the activator should be between 10-20%. The activation process is vigorous and takes place at a temperature of 95°C for 3-6 hours. At the end of activation, the reaction mass is diluted with cold water, filtered, then washed 2-3 times with water and filtered again. Acidic water from the first two filters is collected and reused to activate the clay.[1-3] The weakly acidic water passed through the third filter is collected in the neutralizer and is neutralized with soda (Na_2CO_3) and used in the preparation of a suspension that is sent to the sewer or activated. A suspension with a dry matter content of 9-10% is prepared from the activated clay that has passed through the third filter. The resulting suspension is sent to spray drying and mixed with air until a homogeneous mass is obtained. Then the dried sorbent is crushed in a hammer or ball mill with a moisture content of 7-10%. As adsorbents, substances such as sand, activated carbon, and adsorbent-silica gel are mainly used. The structure of clay powders with sorption properties is shown in Fig.-1 below.

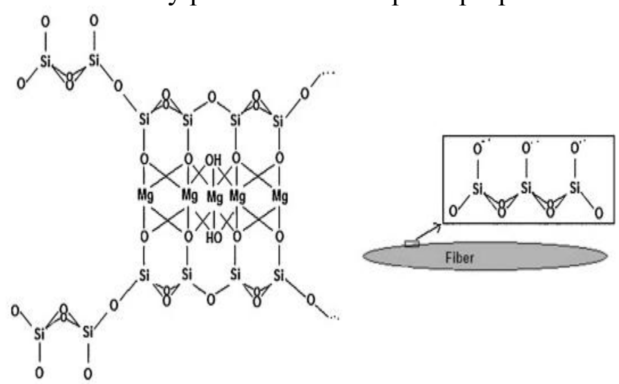

Fig.-1: Structure of Clay Powders with Sorption Properties

Elemental atoms in clay powders exhibiting sorption properties have different compositions, and their structure is mainly octahedral.

EXPERIMENTAL

Material and Methods

The research objective was formulated based on a thorough review of scientific literature relevant to the field, encompassing studies published both domestically and internationally. The primary scientific hypotheses and the accuracy and reliability of the conclusions were corroborated through the application of modern physicochemical techniques, including infrared (IR) spectroscopy, TG and DSC analysis, and X-ray diffraction (XRD).

IR Spectroscopy

The infrared (IR) spectra of the bentonite samples were recorded using an "IRAffinity-1S" Fourier transform infrared (FTIR) spectrometer (Shimadzu, Japan) at the "Chemistry and Technology of Composite Materials" research laboratory named after Professor O.M. Yoriyev, Faculty of Natural Sciences and Agrobiotechnology, Bukhara State University. The spectrometer's parameters were as follows: spectral range of 4000-400 cm^{-1}, signal-to-noise sensitivity ratio of 30,000:1, and a scanning speed of 20 spectra per second.

TG and DSC Analyses

Thermogravimetric (TG) and differential scanning calorimetric (DSC) measurements of bentonite samples were performed on a Setaram Labsys Evo thermal analyzer (Lyon, France) with a flow rate of 99.999% nitrogen at 90 ml/min. The samples used in the form in which they were obtained were weighed in Al_2O_3 crucibles with a volume of 100 µl and heated from 25 to 600 °C at rates of 5, 10, 15, and 20 °C/min. The initial data was corrected and processed using Calisto Processing (version 2.02). The analyzer was calibrated using a multipoint method using seven certified reference samples (CRMS). The samples contained approximately 8 mg with a particle size of 180 microns.

EDXRF Analyses

Elemental analysis of all samples was performed at the Tashkent Chemical-Technological Institute using a high-performance energy-dispersive X-ray fluorescence spectrometer – a Rigaku NEX CG EDXRF Analyzer with Polarization in set - 9022 19 000 0 (Japan).

XRD Analyses

The phase composition of both the initial raw materials and the resulting experimental masses was determined using X-ray diffraction (XRD). Phase identification and subsequent calculations were conducted with reference to established tables, handbooks, and a digital database of mineralogical species. A Bruker AXS D8 Advance diffractometer (Bruker, Germany) was employed for the identification of crystalline phases. The parameters for XRD analysis were set as follows: Cu-Kα radiation, a step size of 0.05 degrees, and a scan rate of 2 degrees per minute. Interpretation of the obtained diffraction patterns was performed utilizing the Match! Software package (Crystal Impact GbR, Bonn, Germany).

RESULTS AND DISCUSSION

To develop an effective technology for treating wastewater from gas processing plants, a combined chemical treatment approach was employed. X-ray structural analysis of the dry residue obtained following treatment with a composite of bentonite, kaolin, and aluminum sulfate revealed the presence of crystalline spheres in the residue (Fig.-2).

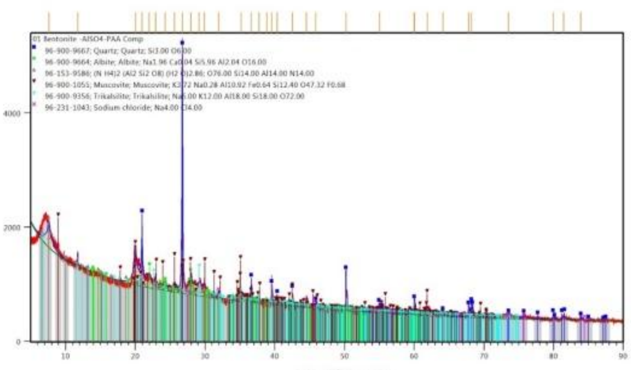

Fig.-2: X-ray Structural Analysis of Navbahor Bentonite

The results obtained indicate that the diffractograms of the dry residue and bentonite samples (Fig.-3) are nearly identical.

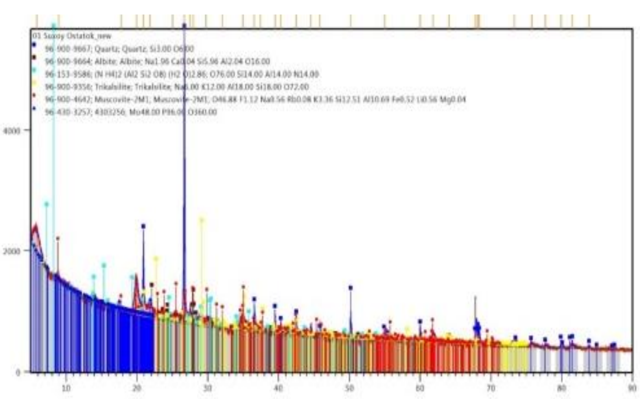

Fig.-3: X-ray Structural Analysis of Thermal-Activated Palygorskite - Navbahor Bentonite Sorbent

This observation suggests that the crystal spheres identified in the study are components of the mineral composition of bentonite.[12,13]

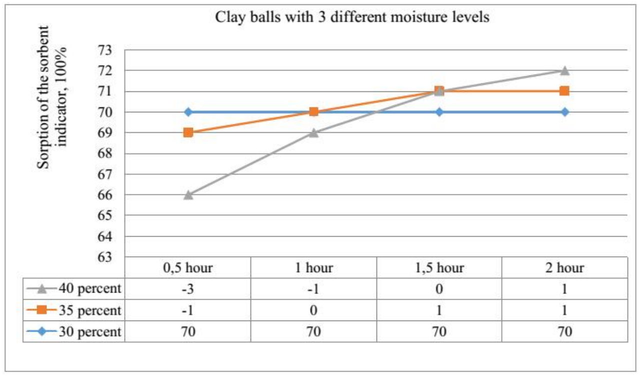

Fig.-4: Effect of Drying Temperature of Palygorskite Clay Powder at 110°C on Sorption Properties

It can be seen from the obtained data that a relative increase in porosity and polarity is caused by humidity.

Moisture content is determined before activation of palygorskite clay powders obtained from the Navbahor mine of the Navoi region. It is also determined whether it is in the state of hydrates or crystalline hydrates. Figure-5 below shows the effect of PK on the sorption property of water retention. It should be noted that the temperature of clay powders changes with increasing hardness and density. If the composition of clay soils consists mainly of organic substances, the temperature of chemical processing is high.

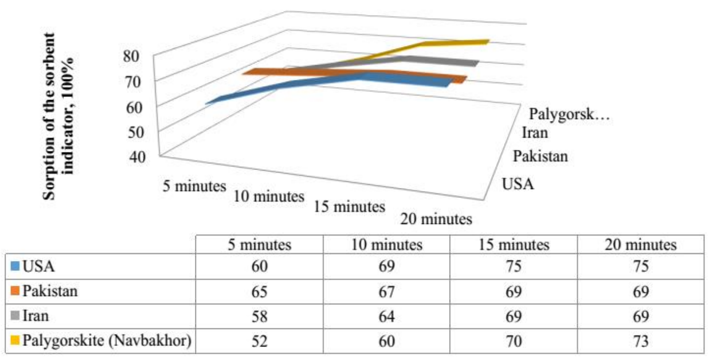

Fig.-5: Comparative Indicators of Domestic and Imported Sorbents for Wastewater Treatment

Currently, effective sorbents are imported from abroad. When comparing the sorption properties of domestic and imported sorbents, the following results were obtained, and the indicators are presented in Fig.-5. In the given picture, the wastewater treatment process was studied at temperatures of 20-60°C. Based on the obtained data, it has been proven that the cleaning time with a local sorbent is 15 to 20 minutes. Thermal analysis of Navbahor mine bentonite and activated sorbents was carried out. Thermogram results are shown in Fig.-6 and 7 and in Table-1. Also, their combined state was studied by thermogravimetric thermal analysis methods.[9-12]

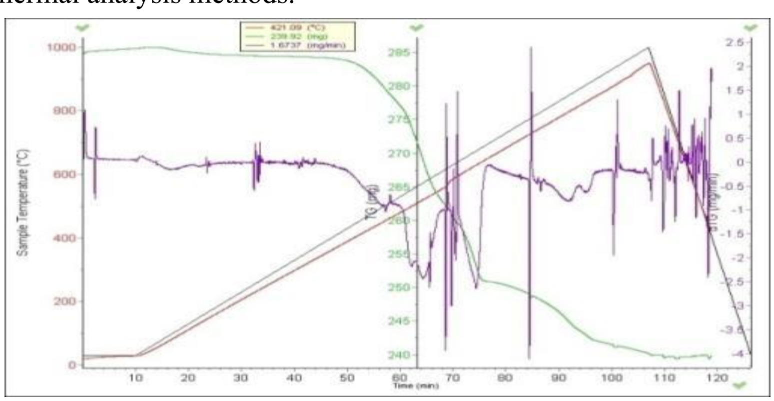

Fig.-6: Navbahor Bentonite Thermogram

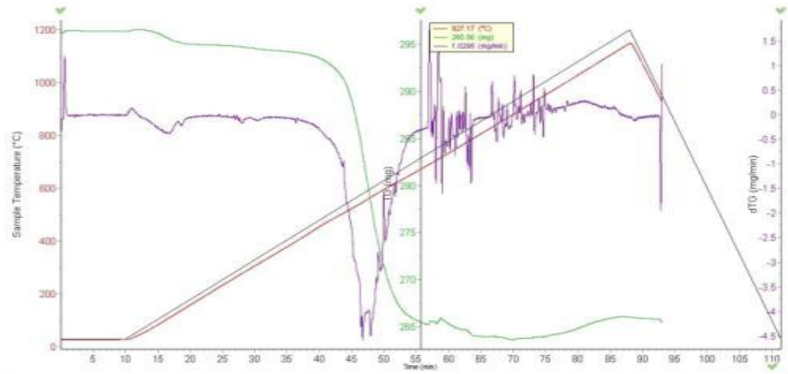

Fig.-7: Navbahor (activated palygorskite-bentonite sorbent) Thermogram

Table-1: In the Derivativeogram (activated palygorskite-bentonite sorbent), Occurrence of the Starting Intervals of Decomposition

Bentonite	Weight of substance %					
Temperature °C	100	200	300	400	500	550
Fragmentation	295	294	293	293	292	286
95°C	99,7	99,3	98,9	98,9	98,5	96,6
Temperature °C	600	700	800	900	1000	
Fragmentation	278	266	266	263	262	
95°C	93,9	89,8	89,8	88,8	88,4	

When studying the thermal stability of natural and activated clays, it was shown that as a result of increasing the temperature to 80-280°C, molecules of adsorbed water contained in clay powders completely disappear by evaporation. In the temperature range of 200-660°C, decomposition of hydrolyzed starch and cellulose occurs with the observation of endo effects. In the derivatograms of citric acid of similar bentonite, changes in its components during thermolysis are observed. The composition and structure of clay powders with sorption properties are not the same. Figure-8 below shows the IR spectroscopic analytical structure of natural palygorskite and activated palygorskite bentonite powder (Fig.-9).

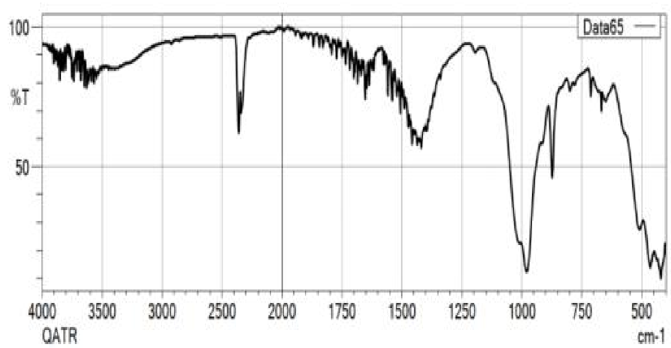

Fig.-8: Navbahor-palygorskite Bentonite IR Spectra

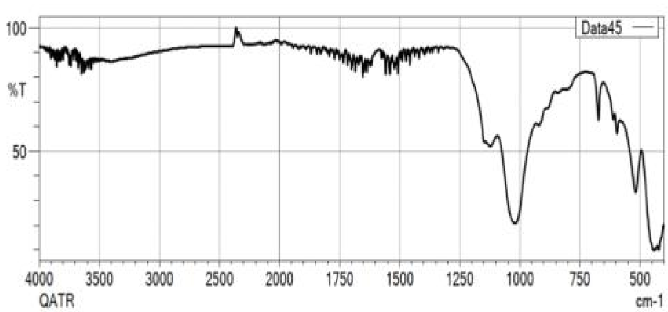

Fig.-9: Navbahor-Activated palygorskite-Bentonite Sorbent IR Spectra

Metal and metal oxides in the composition of the sorbent obtained from palygorskite powder with hydrogen cations that formed a small amount of acidic medium have the following absorption lines in their IR spectra: 3830-3610 cm^{-1}, 1710 cm^{-1}, 1655 cm^{-1}, 1543 cm^{-1}, 1000 cm^{-1}, 467 cm^{-1}. These absorption lines include partially remaining crystallization water molecules in the activated mass, reduced active metals, and metal hydroxides.[14,15] Deformation vibrations of OH^-, H^+, and SO_4^{2-} group (1655-1543 cm^{-1}), low-frequency shifted. The width of 1100-1000 cm^{-1} can be said to correspond to $(SiO_4)^{4-}$ and SiO_2, and corresponding lines also appear at 800 cm^{-1}.

Table-2: The Dependence of the Chemical Composition and Sorption Properties of Bentonite and Palygorskite (3:7) Clays on Activation Factors

Elements affecting the sorption property				SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	Sorption activity, %
Initial clay powder				61,8	15,1	7,9	10,8	2,1	1,5	1,2	
Sample	H_2SO_4 (%)	Activation (°C)	Duration (s)								
1	20	110	4	68,7	10,8	2,5	1,6	1,3	<0,5	0,4	52,5
2	30	100	5	69,6	9,2	2,4	1,7	0,8	<0,5	0,3	67,5
3	40	100	4	69,8	8,7	2,3	1,7	2,4	<0,1	0,1	60

The effect of acid and temperature on activation can be proved by changing its chemical composition.[16,17] When analyzing the chemical composition of natural and activated clay powders, we can see that the quantitative index of SO_2 increases, and that of CaO decreases.[18,19] The method of chemical cleaning with only coagulants is associated with a large consumption of chemicals and gives a lot of slurry, which dries poorly due to its ointment-like consistency. A comprehensive strategy for the chemical treatment of wastewater is presented, employing coagulation or a combined sorption-coagulation technique. The proposed approach, which centers on coagulation, is distinguished by its operational simplicity and the elimination of multiple purification stages. In this process, iron sulfate serves as the coagulant, undergoing reactions that generate iron hydroxide and iron salts. This results in the formation of a light, viscous slurry that accumulates as a surface layer.[20-24] The separated layer is then collected and subjected to treatment with sulfuric acid. This treatment converts the contained fatty acids into soap, and the resultant acidic iron sulfate solution is subsequently recycled and reused as a coagulant in the process.

CONCLUSION

The optimal dosage of reagents for the removal of oil products, suspended solids, and the reduction of chemical oxygen demand (COD) in wastewater was established. Both the existing treatment method and the proposed combined approach demonstrated a high degree of wastewater purification through the application of a sorbent in conjunction with coagulants—aluminum sulfate ($Al_2(SO_4)_3$•$18H_2O$) and ferric chloride ($FeCl_3$•$6H_2O$) - as well as flocculants polyacrylamide (PAA) and carboxymethyl starch (CMS). The implementation of this combined wastewater treatment technology in industrial practice resulted in significant economic benefits. In conclusion, this study highlights the effectiveness of various flocculants and coagulation agents in enhancing wastewater treatment performance, particularly in reducing contaminants such as oil, suspended particles, and organic pollutants measured by COD. The findings demonstrate that integrative approaches combining sorbents, coagulants, and flocculants can achieve superior treatment outcomes while offering cost-efficiency, thereby providing valuable insights into optimizing flocculant selection and dosages for industrial wastewater purification.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to SDG-6: *Clean Water and Sanitation.* All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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