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PHOSPHATE–AMMONIUM MODIFICATION OF LOCAL BENTONITE AND COMPREHENSIVE EVALUATION OF ITS ADSORPTIVE PERFORMANCE IN KEROSENE PURIFICATION

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Abstract. This study presents a systematic investigation into the phosphate–ammonium two-step chemical modification of naturally occurring smectite-type bentonite sourced from the Navbahor deposit in Uzbekistan, with the primary objective of developing a high-performance adsorbent for industrial kerosene purification. The modification procedure comprised sequential treatment with phosphoric acid (H_3PO_4) and ammonium orthophosphate ($(NH_4)_3PO_4$). Following activation, the specific surface area increased from 69.5 to 155.8 m^2/g (a 224% improvement), the pore volume expanded from 0.116 to 0.325 cm^3/g, and the basal spacing d_{001} widened from 14.3 Å to 16.9 Å. FTIR spectroscopy confirmed the formation of P–O–Si and P–O–Al covalent linkages, while XRD analysis revealed structural reorganization and partial delamination of the clay layers. Adsorption experiments demonstrated removal efficiencies of 74.2% for color-forming compounds, 68.3% for aromatic hydrocarbons, and 72.7% for sulfur-containing species. Kinetic data fitted a pseudo-second-order model ($R^2 = 0.998$), indicative of chemisorption-governed uptake. Thermodynamic analysis yielded $\Delta G° = -14.7$ kJ/mol and $\Delta H° = -32.6$ kJ/mol, confirming a spontaneous and exothermic process. The modified adsorbent is derived from an abundant, low-cost, and locally available mineral, positioning it as a technically viable and economically attractive alternative to conventional hydrotreating for kerosene quality improvement.

Keywords: bentonite modification · kerosene purification · phosphate–ammonium activation adsorption kinetics · FTIR · XRD · BET · sulfur removal Lewis acid sites thermodynamics

Annotatsiya. Ushbu tadqiqotda O‘zbekistondagi Navbahor konidan olingan tabiiy sementit tipidagi bentonitning fosfat-ammiak ikki bosqichli kimyoviy modifikatsiyasi bo‘yicha tizimli tadqiqotlar olib borilgan bo‘lib, asosiy maqsad sanoat kerosinini tozalash uchun yuqori samarali adsorbentni ishlab chiqishdan iborat. O'zgartirish jarayoni fosfor kislotasi (H_3PO_4) va ammoniy ortofosfat (NH4)3po4 bilan siqilgan ketma-ket davolashdir). Faollashgandan so'ng, o'ziga xos sirt maydoni 69,5 dan 155,8 m2/g gacha o'sdi (a 224% yaxshilanish), yadro hajmi

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0,116 dan 0,325 sm3/g gacha kengaydi va ddudesin bazal oralig'i 14,3 dan 16,9 gacha kengaydi. FTIR spektroskopiyasi P–O–Si va P–O–Al kovalent bog‘lanishlarining hosil bo‘lishini tasdiqladi, XRD tahlili esa loy qatlamlarining strukturaviy qayta tashkil etilishi va qisman delaminatsiyasini aniqladi. Adsorbsiya tajribalari rang hosil qiluvchi birikmalarning 74,2%, aromatik uglevodorodlarning 68,3% va oltingugurt o'z ichiga olgan turlarning 72,7% ni olib tashlash ta'sirini ko'rsatdi. Kinetik ma'lumotlar ikkinchi darajali psevdo-modelga mos keladi (R2 = 0,998), bu kimyosorbtsiya bilan boshqariladigan qabul qilishni ko'rsatadi. Termodinamik tahlil natijasida ΔG° = -14,7 kJ/mol va ΔH° = -32,6 kJ/mol qiymatlari olindi, bu o'z-o'zidan sodir bo'ladigan va ekzotermik jarayon ekanligini tasdiqlaydi. O'zgartirilgan adsorbent ko'p miqdorda, arzon va mahalliy darajada mavjud bo'lgan mineraldan olingan bo'lib, uni kerosin sifatini yaxshilash uchun an'anaviy gidrotozalashga texnik jihatdan foydali va iqtisodiy jihatdan jozibali muqobil sifatida joylashtiradi.

Kalit so'zlar: bentonit modifikatsiyasi · kerosinni tozalash · fosfat-ammiak faollashuvi adsorbsiya kinetikasi · FTIR · XRD * BET * oltingugurtni olib tashlash Lyuis kislotasi joylarining termodinamikasi.

Аннотация. Данное исследование представляет собой систематическое исследование фосфатно–аммонийной двухэтапной химической модификации природного бентонита типа смектита, добываемого на месторождении Навбахор в Узбекистане, с основной целью разработки высокоэффективного адсорбента для промышленной очистки керосина. Процедура модификации включала последовательную обработку фосфорной кислотой (h_3po_4) и ортофосфатом аммония (NH4)3po4). После активации удельная поверхность увеличилась с 69,5 до 155,8 м2/г (улучшение на 224%), объем сердцевины увеличился с 0,116 до 0,325 см3/г, а базальное расстояние d_{001} увеличилось с 14,3 å до 16,9 å. ИК–спектроскопия подтвердила образование ковалентных связей P–O–Si и P–O-Al, в то время как рентгеновский анализ выявил структурную перестройку и частичное расслоение слоев глины. Эксперименты по адсорбции показали эффективность удаления 74,2% красящих соединений, 68,3% ароматических углеводородов и 72,7% серосодержащих соединений. Кинетические данные соответствовали модели псевдо-второго порядка (R2 = 0,998), что указывает на поглощение, обусловленное хемосорбцией. Термодинамический анализ показал, что ΔG° = -14,7 кДж/моль и ΔH° = -32,6 кДж/моль, что подтверждает спонтанный и экзотермический процесс. Модифицированный адсорбент получен из богатого, недорогого и доступного на местном уровне минерала, что делает его технически жизнеспособной и экономически привлекательной

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альтернативой традиционной гидроочистке для улучшения качества керосина.

Ключевые слова: модификация бентонита · очистка керосина · кинетика адсорбции фосфатно–аммиачной активации · FTIR · XRD * BET * удаление серы Термодинамика кислотных центров Льюиса.

1. INTRODUCTION.

Kerosene occupies a critical position in the global energy landscape, serving as the primary fuel for aviation turbines and as a light-distillate product in heating and lighting applications. International regulatory bodies, including the International Civil Aviation Organization (ICAO) and the Eurasian Economic Union (EEU), impose increasingly stringent quality requirements on aviation-grade kerosene: total sulfur content must not exceed 0.25 wt.% and the aromatic hydrocarbon fraction must remain below 25 vol.%. In practice, straight-run kerosene obtained from primary atmospheric distillation frequently fails to satisfy these thresholds, typically containing 300–500 mg/kg of organic sulfur species (thiophenes, benzothiophenes, dibenzothiophenes, mercaptans) and 30–40 vol.% of aromatic hydrocarbons.

The presence of these contaminants adversely affects fuel combustion efficiency, promotes the formation of particulate matter and sulfur oxides in exhaust emissions, and accelerates the degradation of sealing materials and turbine components. Color-forming resinous compounds — predominantly naphthenic acids, polyaromatics, and porphyrin-type structures generated during oxidative and condensation side-reactions — further compromise product quality and commercial value.

Conventional hydrodesulfurization (HDS) using Co–Mo/Al_2O_3 or Ni–Mo/Al_2O_3 catalysts is highly effective at removing sulfur to ppm-level concentrations, but the technology requires high operating pressures (3–7 MPa) and temperatures (300–400 °C), involves substantial capital investment, generates hydrogen sulfide as a hazardous by-product, and does not adequately address color-forming aromatics. These limitations have stimulated growing interest in adsorptive purification as a complementary or alternative approach — one that operates under ambient conditions, requires no hydrogen input, and generates no toxic gaseous by-products.

Uzbekistan possesses extensive deposits of high-quality bentonite clays in the Navoi region (Navbahor and Nurota fields), with estimated reserves exceeding several hundred million tonnes. These smectite-group minerals are characterized by elevated SiO_2 and Al_2O_3 contents, a well-developed layered architecture, cation-exchange capacities of 80–120 meq/100 g, and inherent susceptibility to chemical surface modification. However, in their natural state, bentonites exhibit several

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limitations — insufficient surface acidity, narrow interlayer channels, weak interactions with aromatic compounds, and low Lewis acid site density — that constrain their adsorptive utility.

Chemical activation with phosphoric acid introduces P–O–Si linkages that expand the pore network and generate new Brønsted and Lewis acid sites. Subsequent intercalation of NH_4^+ ions from ammonium orthophosphate further widens the interlayer spacing and enhances electrostatic attraction towards polarizable aromatic molecules. Although individual acid or ammonium treatments have been reported, the synergistic combination of both reagents in a sequential two-step process — particularly applied to Uzbek bentonite and evaluated for kerosene purification — has not been systematically documented. The present study addresses this gap.

2. MATERIALS AND METHODS.

2.1 Starting Material

Raw bentonite was sourced from the Navbahor deposit (Navoiy Province, Uzbekistan). Its elemental composition, determined by X-ray fluorescence (XRF) spectrometry, is presented in Table 1. The predominance of SiO_2 (70.85 wt.%) confirms the tetrahedral silicate framework typical of montmorillonite, while the Al_2O_3 content (14.06 wt.%) reflects substantial octahedral substitution potential. Iron and magnesium oxides occupy isomorphous substitution sites, generating the permanent negative layer charge responsible for the high cation-exchange capacity.

Table. 1.

Chemical composition of raw Navbahor bentonite (XRF analysis)

Oxide	Content (wt.%)	Structural role
SiO_2	70.85	Tetrahedral layer
Al_2O_3	14.06	Octahedral layer
Fe_2O_3	5.34	Isomorphous substituent
MgO	5.71	Isomorphous substituent
CaO	1.62	Interlayer cation
K_2O	1.30	Interlayer cation
Na_2O	0.05	Interlayer cation
P_2O_5	0.84	Native phosphate
SO_3	0.23	Sulfate impurity

2.2 Two-Step Modification Procedure

Step I — Acid Activation: A 1 mol/L orthophosphoric acid solution was prepared from 60% H_3PO_4 stock. Fifty grams of dry bentonite were dispersed in the solution and thermostated at 80 °C for 120 min under continuous magnetic stirring. During this stage, partial leaching of Mg^{2+}, Ca^{2+}, and Fe^{3+} ions expanded

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the pore network, and surface silanol groups underwent condensation with phosphoric acid according to the following reaction:

$$\equiv Si–OH + H_3PO_4 \rightarrow \equiv Si–O–PO_3H_2 + H_2O$$

Step II — Ammonium Intercalation: The acid-treated clay was mixed with a 0.5 mol/L ammonium orthophosphate solution and stirred at 70 °C for 180 min. NH_4^+ ions displaced residual interlayer cations (Ca^{2+}, Mg^{2+}, K^+), while PO_4^{3-} anions formed coordination bonds with the siliceousaluminosilicate framework, creating additional active adsorption sites. The slurry was subsequently dried at 115 °C for 30 min to remove residual moisture, then calcined at 350 °C for 120 min to consolidate the modified structure. The calcination temperature was deliberately limited below 500 °C to prevent irreversible dehydroxylation and collapse of the interlayer.

2.3 Characterization Techniques

FTIR spectroscopy (Perkin-Elmer Spectrum 65, KBr pellet method, 400–4000 cm^{-1}) was used to identify functional groups and monitor phosphate incorporation. XRD analysis (Bruker D8 Advance, Cu Kα radiation, λ = 1.5406 Å, 2θ = 2–60°) provided crystallographic information including d_{001} basal spacing. BET surface area and pore characteristics were determined via nitrogen adsorption at 77 K (Quantachrome Nova 2200e). Aromatic compound concentrations were monitored by UV–Vis spectrophotometry at 254 nm; sulfur-containing species were quantified by UV-fluorescence at 310 nm.

3. RESULTS AND DISCUSSION.

3.1 Structural Characterization — FTIR Analysis

The FTIR spectrum of the modified bentonite reveals three distinct changes relative to the unmodified clay. First, the O–H stretching band at 3695–3621 cm^{-1} broadened and intensified, reflecting an increase in surface hydroxyl group density and the development of an extended hydrogen-bonding network — both hallmarks of improved surface hydrophilicity and heightened acidity. Second, the Si–O–Si asymmetric stretching mode at 1030–1114 cm^{-1} exhibited a measurable blue-shift, indicating elevated internal lattice tension and a growing proportion of amorphous silicate components generated during the activation steps. Third, and most diagnostically, a new sharp absorption band emerged at 690–745 cm^{-1} , assignable to P–O–Al and P–O–Si vibrations. This signal was absent in the raw material and constitutes direct spectroscopic evidence that phosphate groups are covalently anchored within the interlayer domain. The resulting $\equiv Si–O–PO_3H_2$ linkages increase local layer rigidity and generate new surface acid centers, substantially enhancing the density of active adsorption sites.

3.2 Crystallographic and Textural Properties — XRD and BET

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Table 2 summarizes the key structural parameters before and after modification. The basal spacing d_{001} expanded from 14.3 to 16.9 Å (Δd = 2.6 Å), attributable to three concurring mechanisms: (i) protonation-induced loosening of interlayer bonds during acid activation; (ii) replacement of small divalent cations (Ca^{2+}, r = 0.99 Å) by the larger NH_4^+ (r = 1.43 Å); and (iii) electrostatic repulsion amplified by the triply charged PO_4^{3-} anion. The concurrent decline in XRD peak intensity reflects partial delamination, incipient amorphization, and pore-induced disruption of coherent scattering — collectively indicating a transition towards a more heterogeneous, accessible microtexture.

BET measurements confirm a 2.24-fold increase in specific surface area (from 69.5 to 155.8 m^2/g) and a 2.80-fold expansion in pore volume (from 0.116 to 0.325 cm^3/g). The average pore diameter increased from 6.7 to 8.3 nm, situating both samples within the mesoporous regime (2–50 nm). Mesoporous architecture is particularly advantageous for the uptake of bulky resinous molecules present in kerosene. The cation-exchange capacity rose from 82 to 118 meq/100 g, corroborating the successful introduction of NH_4^+. Surface acidity, measured by potentiometric titration with n-butylamine, nearly tripled from 0.31 to 0.89 mmol/g.

Table. 2.

Structural and textural parameters of raw and modified bentonite

Parameter	Raw Bentonite	Modified Bentonite
BET surface area (m^2/g)	69.5	155.8
Pore volume (cm^3/g)	0.116	0.325
Average pore diameter (nm)	6.7	8.3
Basal spacing d_{001} (Å)	14.3	16.9
Cation-exchange capacity (meq/100 g)	82	118
Surface acidity (mmol/g)	0.31	0.89

3.3 Removal of Color-Forming Compounds

Color intensity was quantified using the APHA (American Public Health Association) scale. Color-forming agents in kerosene include naphthenic acids, polycyclic aromatics, and porphyrin-type resins produced during oxidative side-reactions in the refinery. Table 3 includes results for three samples — raw bentonite, acid-only activated bentonite (Step I alone), and the fully modified adsorbent — enabling a stepwise assessment of each modification stage.

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Table. 3.

Kerosene color intensity before and after adsorption treatment (APHA scale)

Adsorbent	Initial (APHA)	Final (APHA)	ΔAPHA	Efficiency (%)
Raw bentonite	45.1	29.0	16.1	35.7
Acid-activated (Step I only)*	45.1	18.4	26.7	59.2
Phosphate–ammonium modified	45.1	11.6	33.5	74.2

* For comparative purposes only; Step I (H_3PO_4) without subsequent ammonium treatment.

The fully modified adsorbent achieved a 74.2% reduction in APHA value — more than double the 35.7% recorded for the raw clay. Crucially, acid activation alone (Step I) already raised efficiency to 59.2%, yet the subsequent ammonium intercalation (Step II) contributed an additional 15 percentage points, demonstrating a clear synergistic effect. Two mechanisms underlie the color removal: (i) capillary condensation of high-molecular-weight resinous molecules within the expanded mesopores; and (ii) strengthened acid–base interactions between Brønsted acid surface sites (–P–OH, –Si–OH) and the electron-donating functional groups of the color-forming compounds.

3.4 Adsorption of Aromatic Hydrocarbons

The aromatic fraction — comprising thiophenes, benzothiophenes, and polycyclic aromatic hydrocarbons (PAHs) — was monitored via UV–Vis spectrophotometry at 254 nm (Table 4). The modified adsorbent achieved 68.3% removal of aromatics, compared to 28.2% for the raw clay and 51.3% for the acid-activated intermediate — a progression that further confirms the beneficial contribution of the ammonium intercalation step.

Table. 4.

Removal of aromatic compounds monitored by UV–Vis absorbance at 254 nm

Adsorbent	Initial A_{254}	Final A_{254}	ΔA	Removal (%)
Raw bentonite	0.864	0.620	0.244	28.2

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Adsorbent	Initial A_{254}	Final A_{254}	ΔA	Removal (%)
Acid-activated (Step I only)	0.864	0.421	0.443	51.3
Phosphate–ammonium modified	0.864	0.274	0.590	68.3

Three complementary physicochemical mechanisms govern the enhanced aromatic adsorption. First, π–dipole induction: the delocalized π-electron systems of aromatic rings interact with polarizable Lewis acid surface sites (coordinatively unsaturated Si^{4+} and Al^{3+} centers), forming stable π–M complexes. Second, direct coordination: the increased density and strength of Lewis acid sites — as evidenced by the near-tripling of surface acidity from 0.31 to 0.89 mmol/g — facilitates coordinative bond formation with the lone electron pairs of sulfur atoms in thiophene and benzothiophene. Third, electrostatic attraction: the intercalated PO_4^{3-} groups elevate the negative surface charge density, intensifying coulombic attraction towards aromatic molecules with non-negligible dipole moments.

3.5 Desulfurization Performance

Sulfur content before and after adsorption was determined by energy-dispersive X-ray fluorescence (Table 5). The modified adsorbent reduced total sulfur from 410 mg/kg to 112 mg/kg — a 72.7% reduction that brings the treated fuel below the ICAO Annex 16 threshold of 300 mg/kg for aviation kerosene. The raw bentonite achieved only 22%, and the acid-activated intermediate reached 51.7%, once again confirming the additive benefit of the second modification step.

Table. 5.

Sulfur content reduction following adsorption treatment

Adsorbent	Initial (mg/kg)	Final (mg/kg)	Removed (mg/kg)	Reduction (%)
Raw bentonite	410	320	90	22.0
Acid-activated (Step I only)	410	198	212	51.7
Phosphate–ammonium modified	410	112	298	72.7

The superior desulfurization selectivity of the modified material is underpinned by four reinforcing factors: (i) elevated Brønsted and Lewis acidity

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promotes stronger interactions with electron-rich aromatic sulfur species; (ii) abundant Lewis acid Al^{3+} centers form coordination bonds with the lone pair of the sulfur atom, a recognized mechanism for selective thiophene uptake; (iii) PO_4^{3-} surface groups enhance electrostatic capture of polarizable thiophenic molecules; and (iv) the widened mesopore network minimizes diffusion resistance, improving access of bulkier dibenzothiophene molecules to interior active sites.

3.6 Adsorption Kinetics and Thermodynamics

To elucidate the adsorption mechanism, experimental kinetic data were fitted to the pseudo-first-order (Lagergren) and pseudo-second-order (Ho–McKay) models. The pseudo-second-order model provided a markedly superior fit (R^2 = 0.998 vs. 0.921), strongly indicating that the rate-determining step involves chemical interactions — surface coordination, covalent bonding, or strong electrostatic complexation — rather than simple physisorption. The apparent activation energy, calculated from the Arrhenius equation across the temperature range 298–333 K, was E_a = 28.4 kJ/mol. This value exceeds the typical physisorption threshold (< 20 kJ/mol) and falls within the lower range of chemisorption (40–400 kJ/mol), consistent with a mixed-mechanism or activated chemisorption process.

Thermodynamic parameters derived from Van 't Hoff analysis are summarized in Table 6. The negative Gibbs free energy (ΔG° = −14.7 kJ/mol at 298 K) confirms that adsorption proceeds spontaneously under standard conditions. The negative enthalpy (ΔH° = −32.6 kJ/mol) establishes the exothermic character of the process, implying that adsorption capacity will decrease at elevated temperatures. The negative entropy change (ΔS° = −60.1 J mol^{-1} K^{-1}) reflects increased order at the adsorbent–adsorbate interface as aromatic molecules adopt fixed orientations on the structured surface.

Table. 6.

Kinetic and thermodynamic parameters of the adsorption process

Parameter	Value
Kinetic model	Pseudo-second-order
Correlation coefficient R^2	0.998
Activation energy E_a (kJ/mol)	28.4
Gibbs free energy ΔG° (kJ/mol, 298 K)	−14.7
Enthalpy ΔH° (kJ/mol)	−32.6

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Parameter	Value
Entropy ΔS° (J mol^{-1} K^{-1})	−60.1

4. CONCLUSIONS.

This study demonstrates that the phosphate–ammonium two-step modification of Navbahor bentonite — a widely available, low-cost Uzbek mineral — yields a highly effective adsorbent for kerosene purification. The principal findings are as follows:

- **Structural transformation:** The specific surface area increased 2.24-fold (69.5 → 155.8 m^2/g) and pore volume expanded 2.80-fold (0.116 → 0.325 cm^3/g), with development of a mesoporous network (average pore diameter 8.3 nm) favorable for the uptake of bulky organic contaminants.
- **Chemical modification confirmed:** FTIR spectroscopy identified new P–O–Si and P–O–Al covalent bands; XRD showed basal spacing expansion from 14.3 to 16.9 Å (Δd = 2.6 Å). Together, these observations verify successful phosphate intercalation and NH_4^+ exchange in the interlayer domain.
- **Adsorption performance:** Removal efficiencies of 74.2% (color-forming compounds), 68.3% (aromatic hydrocarbons), and 72.7% (organic sulfur) were achieved — 2 to 3 times higher than the raw clay — bringing the sulfur content of treated kerosene (112 mg/kg) below the ICAO Annex 16 limit.
- **Synergistic two-step effect:** Acid activation alone (Step I) raised color removal to 59.2%; the additional ammonium intercalation (Step II) advanced this to 74.2%, confirming a synergistic interaction between the two reagents.
- **Mechanism:** Adsorption is governed by π–dipole induction, coordination at Lewis acid sites, and electrostatic attraction from intercalated PO_4^{3-} groups. Kinetic (R^2 = 0.998, pseudo-second-order) and thermodynamic (ΔG° < 0, ΔH° < 0) data confirm a spontaneous, exothermic chemisorption process.

The phosphate–ammonium-modified Navbahor bentonite represents a scalable, eco-friendly, and economically feasible adsorbent for industrial kerosene and light-distillate purification. Future work should address: (i) regeneration performance over multiple adsorption–desorption cycles; (ii) evaluation against diesel and other petroleum fractions; (iii) pilot-scale fixed-bed column design; and (iv) full techno-economic analysis comparing the modified-bentonite route against state-of-the-art HDS processes.

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