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Acid Activation of Local Bentonites: Kinetics, Structural Modifications, And Enhanced Coagulation Performance for Water Treatment

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Abstract

The increasing demand for clean water and the environmental concerns associated with conventional coagulants have necessitated the search for sustainable, cost-effective alternatives. This study comprehensively investigates the acid activation of bentonite clays from the Shargun deposit (Uzbekistan) and evaluates their application as coagulants and adsorbents in water treatment. The research combines kinetic analysis of acid decomposition, detailed characterization of structural modifications, and performance evaluation in coagulation and adsorption processes.

Natural bentonite samples were treated with hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) under varying conditions of temperature (30–95°C), acid concentration (5–30%), and treatment duration (15–180 min). The physicochemical transformations were analyzed using X-ray diffraction (XRD), differential thermal analysis (DTA), thermogravimetry (TG), and chemical analysis methods. Kinetic studies focused on the extraction rates of Al_2O_3 and Fe_2O_3, with apparent activation energies calculated using Arrhenius equations.

Keywords: Bentonite, acid activation, coagulation, water treatment, kinetics, structural modification, Shargun deposit, adsorption.

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1. Introduction

1.1. Background and Problem Statement

The rapid growth of industrial activities and urbanization has led to increasing contamination of water resources, creating an urgent need for effective water treatment technologies [1]. Coagulation-flocculation remains one of the most widely implemented methods for removing suspended solids, colloidal particles, and organic impurities from water due to its operational simplicity and cost-effectiveness [2]. Traditionally, aluminum sulfate (alum) and ferric chloride have been the primary coagulants employed in water treatment plants worldwide [3].

However, the extensive use of these metal-based coagulants presents significant challenges. Residual aluminum ions in treated water have been associated with neurological disorders, while excessive iron concentrations can cause aesthetic problems and promote bacterial growth [4]. Furthermore, these coagulants generate substantial volumes of sludge that require costly disposal, and their production relies on energy-intensive processes with considerable environmental footprints [5].

These limitations have stimulated research into alternative coagulants derived from natural, renewable sources.

1.2. Bentonite as a Potential Alternative

Bentonite clays, particularly those rich in montmorillonite, have emerged as promising candidates for water treatment applications [6]. These natural aluminosilicates possess unique properties including high specific surface area, cation exchange capacity, swelling ability, and chemical stability [7]. The layered structure of montmorillonite consists of octahedral alumina sheets sandwiched between two tetrahedral silica sheets, with exchangeable cations in the interlayer spaces [8].

In their natural state, bentonites exhibit moderate adsorption and coagulation capabilities. However, chemical modification through acid activation can significantly enhance their functional properties for specific applications [9]. Acid treatment selectively removes octahedral cations (primarily Al^{3+}, Mg^{2+}, Fe^{3+}), creating structural vacancies and increasing surface acidity while preserving the fundamental layered architecture [10].

1.3. Research Gap and Objectives

Despite extensive research on acid-activated clays globally, studies focusing on Uzbekistan's bentonite resources remain limited. The Shargun deposit contains substantial bentonite reserves with unique mineralogical characteristics that warrant detailed investigation. Furthermore, previous studies have often treated acid activation and application evaluation as separate research domains, lacking integrated approaches that connect activation parameters with specific water treatment performance.

This study addresses these gaps through the following objectives:

1. To investigate the acid activation of Shargun bentonite using HCl and H_2SO_4 under systematically varied conditions.

2. To characterize the structural, mineralogical, and thermal transformations resulting from acid treatment.

3. To analyze the kinetics of acid decomposition and determine activation energies for Al_2O_3 and Fe_2O_3 extraction.

2. Methods

2.1. Sample Collection and Preparation

Bentonite samples were collected from the Shargun deposit (Surkhandarya region, Uzbekistan). The raw material was air-dried, homogenized, and sieved to obtain a fraction with particle size <100 μm. Initial moisture content was determined by drying at 105°C for 24 hours.

2.2. Chemical Reagents

- Hydrochloric acid (HCl, 37%, analytical grade)

- Sulfuric acid (H_2SO_4, 98%, analytical grade)

- Sodium hydroxide (NaOH, pellets, analytical grade)

- Kaolin powder (for synthetic turbid water preparation)

- Distilled water (resistivity >18 MΩ·cm)

3.3. Acid Activation Procedure

Activation experiments were conducted in a 500 mL three-neck round-bottom flask equipped with a mechanical stirrer, condenser, and thermometer. For each trial, 20 g of bentonite was mixed with 200 mL of acid solution at predetermined concentration. The system was heated using a thermostatically controlled oil bath with ±1°C accuracy.

Experimental variables included:

- Acid type: HCl and H_2SO_4
- Acid concentration: 5, 10, 15, 20, 25, 30% (w/w)
- Temperature: 30, 40, 50, 60, 70, 80, 90, 95°C
- Reaction time: 15, 30, 45, 60, 90, 120, 150, 180 minutes
- Stirring speed: 300 rpm (constant)

After treatment, suspensions were vacuum-filtered through Whatman No. 42 filter paper. The solid residues were washed with distilled water until neutral pH (pH 6.5–7.5) and dried at 105°C for 24 hours. Activated samples were stored in desiccators until further analysis.

2.4. Characterization Techniques

2.4.1. Chemical Composition Analysis

- Silicon (SiO_2): Gravimetric method as insoluble residue after HF treatment
- Aluminum (Al_2O_3): Complexometric titration with EDTA
- Iron (Fe_2O_3): Photometric method with 1,10-phenanthroline
- Calcium (CaO) and Magnesium (MgO): EDTA titration
- Sodium (Na_2O) and Potassium (K_2O): Flame photometry
- Loss on ignition: Weight loss after heating at 1000°C for 2 hours

2.4.2. Mineralogical Characterization

- X-ray diffraction (XRD): DRON-2.0 diffractometer with CuKα radiation (λ=1.5418 Å), operating at 40 kV and 30 mA. Scans from 2° to 70° 20 with 0.02° step size and 2 s counting time.
- Qualitative analysis: Identification of mineral phases using ICDD-PDF2 database.
- Semi-quantitative analysis: Relative mineral abundances estimated using reference intensity ratio (RIR) method.

2.4.3. Thermal Analysis

- Differential thermal analysis (DTA) and Thermogravimetry (TG): Paulik-Erdey derivatograph, heating rate 10°C/min from room temperature to 1000°C in static air atmosphere, α-Al_2O_3 as reference material.
- Derivative thermogravimetry (DTG): First derivative of TG curve to identify decomposition temperatures.

2.5. Kinetic Studies

The extraction efficiency (α) of Al_2O_3 and Fe_2O_3 was calculated as:

$$\alpha = \frac{C0 - Ct}{C0} \times 100\%$$

where (C_0) is initial concentration in solid, (C_t) is concentration after time t.

Samples were withdrawn at regular intervals, filtered, and analyzed for Al and Fe concentrations in solution using atomic absorption spectroscopy (PerkinElmer AAnalyst 400). Activation energies were determined from Arrhenius plots:

$$\ln k = \ln A - \frac{E\alpha}{RT}$$

where k is rate constant, A is pre-exponential factor, Eα is activation energy, R is gas constant, T is absolute temperature.

2.6. Coagulation Experiments

2.6.1. Synthetic Turbid Water Preparation

Kaolin suspension (500 NTU) was prepared by dispersing 5 g of kaolin in 1 L of distilled water with 30 minutes stirring followed by 24 hours settling. The supernatant was diluted to desired turbidity levels (50, 100, 200 NTU) for coagulation tests.

2.6.2. Jar Test Procedure

Standard jar tests were conducted using a six-paddle stirrer (VELP Scientifica). Procedure:

1. 500 mL of synthetic turbid water placed in each beaker
2. Coagulant dosage: 10, 25, 50, 75, 100, 150 mg/L
3. Rapid mixing: 150 rpm for 2 minutes
4. Slow mixing: 30 rpm for 15 minutes
5. Settling: 30 minutes quiescent conditions
6. Sampling: 2 cm below surface for turbidity measurement

Turbidity was measured using Hach 2100Q Portable Turbidimeter. Coagulation efficiency was calculated as:

$$\text{Efficiency }\% = \ln A - \frac{To - Tf}{To} \times 100\%$$

where T_0 is initial turbidity, Tf is final turbidity.

2.7. Adsorption Studies for Water Softening

2.7.1. Hard Water Preparation

Synthetic hard water (300 mg/L as $CaCO_3$) was prepared by dissolving appropriate amounts of $CaCl_2 \cdot 2H_2O$ and $MgSO_4 \cdot 7H_2O$ in distilled water.

2.7.2. Batch Adsorption Experiments

- Adsorbent dosage: 1, 2, 5, 10 g/L

- Contact time: 15, 30, 60, 120, 240 minutes

- Temperature: 25°C (room temperature)

- pH: Natural (approximately 7.2)

- Agitation: 150 rpm on orbital shaker

After contact time, samples were filtered (0.45 µm membrane) and analyzed for calcium and magnesium by EDTA titration. Hardness removal efficiency calculated as:

$$\text{Removal }\% = \ln A - \frac{Ho - Hf}{Ho} \times 100\%$$

2.8. Statistical Analysis

All experiments were conducted in triplicate. Data presented as mean ± standard deviation. Regression analysis and model fitting performed using OriginPro 2022 software.

3. Results and Discussion

3.1. Characterization of Natural Bentonite

3.1.1. Chemical Composition

The chemical composition of Shargun bentonite is presented in Table 1. The material is classified as calcium-magnesium bentonite based on exchangeable cation predominance.

Table 1. Chemical composition of natural bentonite from Shargun deposit (wt.%).

Table 1.

Component	Content (%)	Component	Content (%)
SiO_2	60.80 ± 0.35	TiO_2	0.71 ± 0.05
Al_2O_3	13.90 ± 0.25	P_2O_5	0.06 ± 0.01
Fe_2O_3	4.12 ± 0.15	LOI*	5.78 ± 0.20
FeO	0.29 ± 0.03	Na_2O	0.81 ± 0.08
MgO	2.33 ± 0.10	K_2O	2.26 ± 0.12
CaO	0.88 ± 0.07	Total	99.94 ± 0.45

LOI = Loss on ignition at 1000°C

The SiO_2/Al_2O_3 ratio of 4.37 suggests moderate silica content typical of bentonites from volcanic origin. The relatively high iron content (4.41% total as Fe oxides) contributes to the reddish-brown coloration and may influence activation behavior and coagulation performance.

3.1.2. Mineralogical Composition

XRD analysis reveals montmorillonite as the dominant phase, with characteristic basal reflection at 15.4 Å (air-dried) expanding to 18.2 Å after glycol saturation. Semi-quantitative estimation indicates:

- Montmorillonite: 72–78%

- Quartz: 12–15%

- Feldspar (albite): 5–8%

- Hematite: 3–5%

- Calcite: traces (<1%)

The crystallinity index, calculated from the full width at half maximum (FWHM) of the (001) reflection, is 0.85, indicating well-ordered stacking of montmorillonite layers.

3.1.3. Thermal Behavior

DTA/TG curves (Figure 2) show characteristic features:

- Endotherm I (50–120°C): Removal of physically adsorbed water (mass loss: 8.2%)

- Endotherm II (120–220°C): Evolution of interlayer water (mass loss: 4.1%)

- Endotherm III (550–750°C): Dehydroxylation of structural OH groups (mass loss: 3.8%)

- Exotherm (850–950°C): Recrystallization to high-temperature phases (spinel, mullite)

The total mass loss up to 1000°C is 16.1%, consistent with the LOI value from chemical analysis.

3.2. Acid Activation: Effect of Process Parameters

3.2.1. Influence of Acid Type

Comparative activation with HCl and H_2SO_4 under identical conditions (20% concentration, 90°C, 60 min) revealed significant differences:

- HCl treatment: Higher extraction of Al_2O_3 (91.2% vs 87.5%) but lower extraction of Fe_2O_3 (78.4% vs 83.8%)

- H_2SO_4 treatment: More pronounced structural degradation evidenced by greater reduction in XRD peak intensities

- CEC changes: HCl-activated samples retained higher CEC (45 meq/100g) compared to H_2SO_4-activated (32 meq/100g)

The differences are attributed to anion effects: chloride ions show weaker complexation with aluminum compared to sulfate ions, leading to different dissolution kinetics and solid residue characteristics.

3.2.2. Temperature Effect

Illustrates the temperature dependence of Al_2O_3 and Fe_2O_3 extraction. The extraction efficiency increases exponentially with temperature, with particularly sharp increases between 70–90°C. At 95°C with 20% HCl:

- Al_2O_3 extraction: 93.8%

- Fe_2O_3 extraction: 84.8%

The temperature sensitivity (Q_{10} value) was calculated as 2.3 for Al_2O_3 and 2.1 for Fe_2O_3 between 60–90°C, indicating strong temperature dependence typical of chemically controlled processes.

3.2.3. Acid Concentration Effect

Shows extraction efficiency versus acid concentration. A concentration threshold exists at approximately 15%, beyond which extraction increases more gradually. Optimal concentration ranges:

- HCl: 20–25% for maximum extraction with minimal structural collapse

- H_2SO_4: 15–20% due to greater aggressiveness of sulfuric acid

Excessive acid concentration (>25%) leads to rapid structural degradation, reducing the specific surface area due to pore wall collapse.

3.2.4. Time Dependence

Extraction kinetics follow characteristic profiles:

- Rapid initial phase (0–30 min): 60–70% of total extraction occurs

- Slower intermediate phase (30–90 min): Additional 20–25% extraction

- Plateau phase (>90 min): Minimal further extraction

The time to reach 90% of maximum extraction (t_{90}) decreases from 120 min at 70°C to 45 min at 90°C.

3.3. Kinetic Analysis

3.3.1. Kinetic Models

Three kinetic models were evaluated for Al_2O_3 extraction data:

1. First-order model: $\ln(1-\alpha) = -kt$

2. Second-order model: $1/(1-\alpha) = 1 + kt$

3. Diffusion-controlled model: $1 - 3(1-\alpha)2/3 + 2(1-\alpha) = kt$

The first-order model provided the best fit with R^2 values >0.98 for all temperatures, confirming chemical reaction control as rate-determining step.

3.3.2. Activation Energy Determination

Arrhenius plots for HCl activation yield:

- Al_2O_3: $Ea = 50.70 \pm 2.3$ kJ/mol, $A = 3.2 \times 10^6$ min^{-1}

- Fe_2O_3: $Ea = 65.50 \pm 3.1$ kJ/mol, $A = 8.7 \times 10^8$ min^{-1}

For H_2SO_4 activation:

- Al_2O_3: $Ea = 45.20 \pm 2.1$ kJ/mol, $A = 1.4 \times 10^6$ min^{-1}

- Fe_2O_3: $Ea = 58.30 \pm 2.8$ kJ/mol, $A = 2.3 \times 10^8$ min^{-1}

The higher activation energy for Fe_2O_3 extraction reflects stronger bonding of iron in the octahedral sheet compared to aluminum.

3.3.3. Empirical Correlations

Multiple regression analysis yielded predictive equations for extraction efficiency:

For HCl activation (20% concentration):

$\alpha Al_2O_3 = 1.317\ t^{0.95243}$ ($R^2 = 0.986$)

$\alpha\_Fe_2O_3 = 0.2537\ t^{1.3026}$ ($R^2 = 0.974$)

For H_2SO_4 activation (20% concentration):

$\alpha Al_2O_3 = 0.9583\ t + 10.201$ ($R^2 = 0.991$)

$\alpha\_Fe_2O_3 = 0.1403\ t^{1.4272}$ ($R^2 = 0.968$)

These equations are valid for t = 15–180 min, T = 70–95°C.

3.4. Structural Modifications After Acid Activation

3.4.1. XRD Analysis

Progressive acid treatment causes systematic changes in XRD patterns.

1. Basal spacing reduction: d(001) decreases from 15.4 Å to 12.8 Å after moderate activation, indicating partial collapse of interlayer space.

2. Peak broadening: FWHM of montmorillonite reflections increases by 40–60%, suggesting decreased crystallite size and increased structural disorder.

3. Accessory mineral dissolution: Quartz and feldspar peaks diminish significantly after extended treatment.

4. Amorphous halo: Appearance of broad hump at 20–30° 2θ indicates formation of amorphous silica phase.

The extent of structural modification correlates strongly with extraction efficiency. Samples with Al_2O_3 extraction >80% show complete disappearance of the (001) reflection, indicating substantial layer disorder.

3.4.2. Thermal Analysis Changes

DTA curves of activated samples reveal:

- Reduced low-temperature endotherms: Decreased physically adsorbed water content

- Shifted dehydroxylation peak: From 680°C to 620–650°C, indicating weakened structural bonding

- Diminished exotherm intensity: Reduced formation of high-temperature phases due to disrupted structure

- Total mass loss reduction: From 16.1% to 8–12% depending on activation severity

These changes confirm partial destruction of the aluminosilicate framework and formation of less thermally stable phases.

3.4.3. Surface Area Evolution

Specific surface area measurements show complex behavior:

- Initial increase: From 42 m²/g (natural) to 185 m²/g after mild activation (10% HCl, 70°C, 30 min)

- Maximum values: 210–230 m²/g achieved with moderate activation (15–20% acid, 80–90°C, 45–60 min)

- Decrease with severe activation: Dropping to 120–150 m²/g with 30% acid or extended treatment (>120 min)

The surface area evolution reflects competing processes: creation of new surfaces through layer separation versus pore collapse under aggressive conditions.

3.5. Coagulation Performance

3.5.1. Comparison of Natural and Activated Bentonite

Jar test results demonstrate superior performance of acid-activated bentonite across all tested conditions. For initial turbidity of 100 NTU and coagulant dosage of 50 mg/L:

- Natural bentonite: 68.2 ± 2.1% removal

- HCl-activated (20%, 90°C, 60 min): 91.5 ± 1.8% removal

- H_2SO_4-activated (20%, 90°C, 60 min): 94.2 ± 1.5% removal

The enhanced performance is attributed to multiple factors: increased surface charge density, higher specific surface area, and improved dispersion characteristics.

3.5.2. Effect of Activation Parameters on Coagulation

Coagulation efficiency correlates strongly with activation conditions (Table 2):

Table 2. Coagulation efficiency as function of activation parameters (initial turbidity: 100 NTU, dosage: 50 mg/L).

Table 2.

Activation Conditions	Activation Conditions	Turbidity Removal(%)	Optimal Dosage (mg/L)
Natural bentonite		68.2 ± 2.1	75–100
Mild activation	10% HCl, 70°C, 30 min	82.5 ± 1.9	50–75
Moderate activation	20% HCl, 90°C, 60 min	94.2 ± 1.5	25–50
Severe activation	30% HCl, 95°C, 120 min	85.7 ± 2.3	50–75

Moderate activation provides optimal balance between increased surface reactivity and preserved layered structure. Severe activation reduces performance despite higher extraction, likely due to excessive structural degradation and reduced particle size.

3.5.3. Dosage Optimization

Dose-response curves show typical saturation behavior. For acid-activated bentonite:

- Threshold dosage: 10–15 mg/L for significant turbidity reduction
- Optimal range: 25–50 mg/L achieving 90–95% removal
- Saturation: >75 mg/L provides minimal additional benefit

Compared to conventional alum (optimal dosage 20–40 mg/L for similar conditions), acid-activated bentonite requires slightly higher doses but offers environmental advantages and potential cost benefits considering local availability.

3.5.4. pH Dependence

Coagulation efficiency shows strong pH dependence:

- Optimum pH range: 6.5–8.0 (90–95% removal)
- Reduced efficiency at low pH: <6.0 (75–85% removal)
- Significant reduction at high pH: >9.0 (60–70% removal)

The pH optimum corresponds to conditions where both clay particles and colloidal impurities carry moderate negative charges, facilitating charge neutralization mechanisms.

3.6. Adsorption Performance for Water Softening

3.6.1. Hardness Removal Efficiency

Acid-activated bentonite residues demonstrate excellent adsorption capacity for hardness ions (Figure 12). For water with initial hardness of 300 mg/L as $CaCO_3$:

- Natural bentonite: 22.4 ± 1.8% removal (10 g/L, 120 min)
- HCl-activated residue: 88.7 ± 2.1% removal (10 g/L, 120 min)
- H_2SO_4-activated residue: 91.3 ± 1.9% removal (10 g/L, 120 min)

The dramatic improvement is attributed to the amorphous silica-rich composition of activated residues, providing abundant silanol groups (Si-OH) for ion exchange.

3.6.2. Adsorption Kinetics

Hardness removal follows pseudo-second-order kinetics:

$$\frac{\mathrm{t}}{Qt}=\frac{\mathrm{t}}{\mathrm{Kq^2c}}+\frac{\mathrm{t}}{Qe}$$

where Qt is adsorption at time t, Qe is equilibrium adsorption, K is rate constant.

For HCl-activated residue (10 g/L):

- Calcium: Qe = 28.7, mg/g), (K = 0.024, g/mg·min)
- Magnesium: Qe = 24.3, mg/g), (K = 0.018, g/mg·min)

Equilibrium is reached within 90–120 minutes for both ions.

3.6.3. Regeneration and Reuse

Saturated adsorbents were regenerated with 5% NaCl solution followed by rinsing. After five regeneration cycles:

- Capacity retention: 82–85% of initial capacity
- Mass loss: 12–15% due to particle attrition
- Structural stability: XRD confirms maintained

amorphous character

The regenerability enhances practical applicability and economic viability.

4. Conclusion

This comprehensive study demonstrates the significant potential of acid-activated bentonite from Uzbekistan's Shargun deposit for water treatment applications. The research provides detailed insights into activation mechanisms, kinetic behavior, structural transformations, and practical performance.

Key Findings

1. Optimal activation conditions were identified as 20% acid concentration, 90°C temperature, and 60 minutes treatment time, achieving 93.8% Al_2O_3 and 84.8% Fe_2O_3 extraction with HCl.

2. Kinetic analysis revealed first-order behavior with activation energies of 50.70 kJ/mol for Al_2O_3 and 65.50 kJ/mol for Fe_2O_3 extraction, confirming chemically controlled processes.

3. Structural modifications include partial dealumination, increased surface area (up to 230 m^2/g), enhanced surface acidity, and preservation of layered structure under moderate activation.

4. Coagulation performance of acid-activated bentonite significantly exceeds natural material, achieving 94% turbidity removal at optimal dosage of 25–50 mg/L compared to 68% for natural bentonite.

5. Adsorption capacity for water softening reaches 91% hardness removal, with the solid residue after acid treatment showing excellent regeneration potential.

6. Empirical models developed in this study enable prediction of extraction efficiency and performance based on process parameters.

This research establishes a solid foundation for the development of bentonite-based water treatment technologies in Uzbekistan and similar regions with abundant clay resources. The findings contribute to the global effort toward sustainable water management through the utilization of locally available, environmentally friendly materials.

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