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ILMIY AXBOROTNOMA

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EFFECTIVE METHODS FOR BENTONITE ENRICHMENT WITH MONTMORILLONITE AND THE IMPACT OF THERMAL TREATMENT ON CATION EXCHANGE CAPACITY

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Abstract. This study focuses on the development of effective methods for bentonite enrichment with montmorillonite and the evaluation of the effect of thermal treatment on its cation exchange capacity (CEC). The enrichment process was carried out using mechanical separation, sedimentation, and sodium carbonate (Na_2CO_3) treatment to increase the montmorillonite content and improve adsorption properties. The mineralogical and physicochemical characteristics of the samples were analyzed using X-ray diffraction (XRD), thermogravimetric analysis (TG-DTA), scanning electron microscopy (SEM), infrared spectroscopy (FTIR), and granulometric analysis.

The results demonstrated a significant increase in montmorillonite content from 44.5% to 91.8%, while quartz content decreased to 0.2%. The proportion of fine fractions (≤1 μm) increased, leading to enhanced dispersity and improved adsorption performance. The enriched bentonite exhibited a CEC value of 104 mg-eq/100 g, with sodium ions dominating the exchange complex (88.6 mg-eq/100 g). Thermal treatment above 100–115 °C resulted in a noticeable decrease in CEC, indicating structural transformations within the clay mineral layers. The findings confirm that the proposed enrichment method effectively improves the physicochemical and adsorption properties of bentonite, enabling the production of high-performance organobentonites suitable for environmentally safe applications.

Keywords: Bentonite, montmorillonite, enrichment, sodium carbonate, cation exchange capacity (cec), thermal treatment, x-ray diffraction (xrd), organobentonite, adsorption, dispersity.

Bentonitni montmorillonit bilan boyitishning samarali usullari va termal ishlov berishning kation almashinuvi quvvatiga ta'siri

Аннотация. Ushbu tadqiqot bentonitni montmorillonit bilan boyitishning samarali usullarini ishlab chiqishga va uning kation almashinuv qobiliyatiga (CEC) termik ishlov berishning ta'sirini baholashga qaratilgan. Boyitish jarayoni montmorillonit miqdorini oshirish va adsorbsiya xususiyatlarini yaxshilash uchun mexanik ajratish, cho'ktirish va natriy karbonat (Na_2CO_3) bilan ishlov berish yordamida amalga oshirildi. Namunalarning mineralogik va fizik-kimyoviy xususiyatlari rentgen difraksiyasi (XRD), termogravimetrik tahlil (TG-DTA), skanerlash elektron mikroskopiyasi (SEM), infraqizil spektroskopiya (FTIR) va granulometrik tahlil yordamida tahlil qilindi. Natijalar montmorillonit miqdorining 44,5% dan 91,8% gacha sezilarli darajada oshganini, kvarts miqdori esa 0,2% gacha pasayganini ko'rsatdi. Mayda fraksiyalarning ulushi (≤1 mkm) oshdi, bu esa dispersiyaning kuchayishiga va adsorbsiya ko'rsatkichlarining

yaxshilanishiga olib keldi. Boyitilgan bentonitning CEC qiymati 104 mg-ekv/100 g ni tashkil etdi, almashinuv kompleksida natriy ionlari ustunlik qildi (88,6 mg-ekv/100 g). 100–115 °C dan yuqori haroratda issiqlik bilan ishlov berish CEC ning sezilarli darajada pasayishiga olib keldi, bu esa loy mineral qatlamlari ichidagi strukturaviy o'zgarishlarni ko'rsatadi.

Tadqiqot natijalari taklif qilingan boyitish usuli bentonitning fizik-kimyoviy va adsorbsion xususiyatlarini samarali ravishda yaxshilaydi va ekologik xavfsiz qo'llanilish uchun mos keladigan yuqori samarali organobentonitlarni ishlab chiqarish imkonini beradi.

Kalit so‘zlar: Bentonit, montmorillonite, boyitish, natriy karbonat, kation almashinuv sig'imi (cec), termik ishlov berish, rentgen difraksiyasi (xrd), organobentonit, adsorbsiya, dispersiya

Эффективные методы обогащения бентонита монтмориллонитом и влияние термической обработки на катионообменную емкость

Аннотация. Данное исследование посвящено разработке эффективных методов обогащения бентонита монтмориллонитом и оценке влияния термической обработки на его катионообменную емкость (КОЕ). Процесс обогащения проводился с использованием механической сепарации, седиментации и обработки карбонатом натрия (Na_2CO_3) для увеличения содержания монтмориллонита и улучшения адсорбционных свойств. Минералогические и физико-химические характеристики образцов анализировались с помощью рентгеновской дифракции (РД), термогравиметрического анализа (ТГ-ДТА), сканирующей электронной микроскопии (СЭМ), инфракрасной спектроскопии (ИК-Фурье) и гранулометрического анализа. Результаты показали значительное увеличение содержания монтмориллонита с 44,5% до 91,8%, в то время как содержание кварца снизилось до 0,2%. Доля мелких фракций (≤1 мкм) увеличилась, что привело к повышению дисперсности и улучшению адсорбционных характеристик. Обогащенный бентонит показал значение катионообменной емкости (КОЕ) 104 мг-экв/100 г, при этом в обменном комплексе преобладали ионы натрия (88,6 мг-экв/100 г). Термическая обработка при температуре выше 100–115 °C привела к заметному снижению КОЕ, что указывает на структурные преобразования внутри слоев глинистого минерала.

Полученные результаты подтверждают, что предложенный метод обогащения эффективно улучшает физико-химические и адсорбционные свойства бентонита, позволяя получать высокоэффективные органобентониты, пригодные для экологически безопасного применения.

Ключевые слова: бентонит, монтмориллонит, обогащение, карбонат натрия, катионообменная емкость (кое), термическая обработка, рентгеновская дифракция (рд), органобентонит, адсорбция, дисперсия.

1.Introduction. 1.1 Background of the Study.

The relevance of research on natural clays and their modification methods is driven by the need to develop new high-performance adsorbent materials for solving environmental and industrial problems. Particular attention is paid to studying the mechanisms of interaction between organic modifiers and montmorillonite, which makes it possible to significantly improve the adsorption properties of organobentonites. In recent years, extensive research has been conducted on the adsorption of organic compounds using bentonite [1]. However, unmodified bentonite is not capable of effectively adsorbing organic compounds because it exhibits hydrophilic properties, whereas organic compounds are generally hydrophobic in nature [2]. To alter the surface properties of bentonite from hydrophilic to hydrophobic, surfactants are commonly employed [3]. Consequently, surface modification of bentonite significantly enhances its ability to adsorb organic substances [4].

Organic modifiers, both low-molecular-weight and high-molecular-weight compounds, play a key role in improving clay properties and in the formation of organobentonites. These substances, including cationic ammonium salts and anionic surfactants, are used to alter hydrophilicity and enhance the adsorption characteristics of aluminosilicate materials. Low-molecular-weight modifiers are characterized by good water solubility and effectiveness in producing hydrophobic organobentonites for removing organic compounds from aqueous media. High-molecular-weight organic modifiers are represented by polymers and organic compounds with high molecular weight, complex structures, and numerous functional groups. These modifiers can be incorporated into the structure of aluminosilicate materials in various forms, including polymer layers, coatings, or network structures. They possess unique properties such as high stability, flexibility, and thermal resistance. These characteristics contribute to a significant improvement in the mechanical, thermal, electrical, and adsorption properties of aluminosilicate materials and enable the creation of new functional materials based on them [5].

In general, organobentonites can be obtained using three methods: dry, wet, and pre-gelation methods [6]. The dry method is simple to implement but requires high temperatures to melt surfactants, which increases energy consumption. Compared to the dry method, the wet method requires less equipment and is widely used; however, ion exchange between the surfactant and bentonite may take a long time to reach equilibrium. In addition, obtaining stable organobentonites with desired performance characteristics requires filtration, washing, drying, and grinding steps, which complicates the process [7].

Therefore, it is important to develop new strategies that reduce reaction time and simplify the preparation of organobentonites. A novel method for producing organobentonites, described in [8], represents a hybrid between the dry and wet approaches. In this method, a surfactant and bentonite are mixed with a small amount of water, stirred for 2 hours at room temperature, and then dried at 100–110 °C to obtain organobentonites. Hexadecyltrimethylammonium bromide (HDTMA), a cationic surfactant, is used as the modifier. Compared to the dry method, this approach eliminates the need for melting the surfactant, and compared to the wet method, it avoids filtration steps. As a result, it is a more efficient and cost-effective process that helps prevent secondary contamination by surfactants. This approach is referred to as the semi-dry method [9,10].

Within the framework of these studies, the adsorption activity of HDTMA-modified bentonite (HDTMA-Bent) toward 2,4-D was investigated. The results were compared with those obtained using activated carbon. In addition, the modified bentonite was characterized by zeta potential measurements, particle size determination, and transmission and scanning electron microscopy (TEM and SEM) to elucidate the adsorption mechanism of 2,4-D on the modified materials [11]

2.Materials and methods. Research on bentonite enrichment aimed at increasing the content of the primary mineral montmorillonite is of considerable importance, as it enhances the adsorption properties of clay, promotes environmentally safe applications, and opens new opportunities in nanotechnology. Improved bentonite can replace synthetic materials, reducing costs and environmental impact while providing economic benefits and meeting specific industrial requirements.

The following methods were employed to enrich bentonite for adsorbent production:

2.1 Mechanical separation and elutriation.

This helps separate larger particles and remove non-clay impurities. Mechanical separation and sedimentation.

This method enables the separation of coarse particles and the removal of non-clay impurities, sand, and other undesirable materials, including heavier minerals. Sedimentation is based on differences in surface activity and particle size to separate bentonite from impurities.

2.2 Treatment with a sodium carbonate solution.

This process is aimed at increasing the montmorillonite content by removing calcium and magnesium salts.

The procedure begins with preliminary cleaning of the clay from coarse impurities and visible contaminants, followed by drying in a drying oven at 105 ± 5 °C until complete moisture removal. The dried clay is then ground in a laboratory mill and sieved to isolate fractions smaller than 1 mm. The finely ground clay is mixed with distilled water in a ratio sufficient to completely cover the clay and left to swell for 48 h. Subsequently, an additional volume of water equal to twice the suspension volume is added, and the mixture is stirred thoroughly for 30 min. After standing for 15–20 min, decantation of the upper layer (approximately 20 cm) is carried out to separate sandy particles from montmorillonite. Sodium carbonate is then added to the collected upper layer, with the required mass calculated according to the following equation:

$$M = 0{,}01\left(\frac{m - V}{0.6}\right),$$

where M is the mass of the modifier (Na_2CO_3), g; m is the mass of the suspension, g; V is the suspension volume, mL; and 0.6 is a correction coefficient dependent on the density of dry clay (2.5 g cm^{-3})

3. Results and discussion.

3.1 Bentonite (BK) analysis.

This method yields montmorillonite-enriched bentonite with minimal sandstone impurities. X-ray phase analysis confirmed that enrichment increased the content of montmorillonite, clinoptilolite, and illite due to a reduction in non-clay minerals, primarily low-temperature trigonal quartz. The mineralogical composition of bentonite (BK) and its modified forms was analyzed using X-ray diffraction (Shimadzu XRD-6100 with CuKα radiation), thermogravimetric analysis (Paulik–Paulik–Erdey derivatograph), and scanning electron microscopy (Carl Zeiss EVO MA10), enabling a comprehensive evaluation of structural and physicochemical changes during modification.

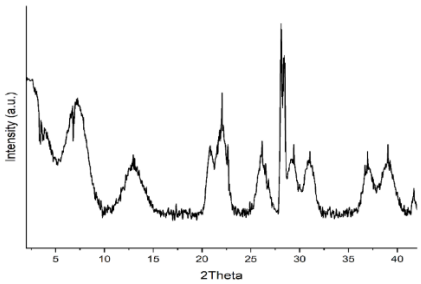

Figure 1. X-ray diffraction pattern of the raw bentonite sample (BK).

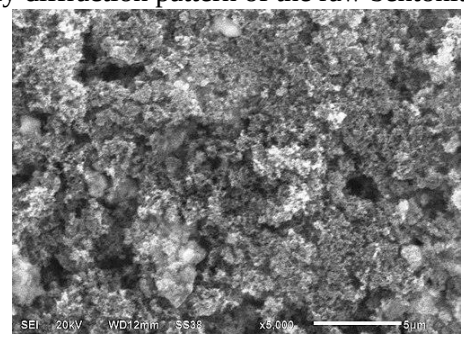

Figure 2. Scanning electron microscopy (SEM) image of the raw bentonite sample (BK).

The initial mass loss observed on the TG and DTG curves, along with the corresponding endothermic peak on the DSC curve at approximately 50–60 °C, is attributed to the removal of physically adsorbed water from bentonite. A subsequent significant mass loss and endothermic peak

on the DSC curve in the temperature range of 400–500 °C indicate the release of structural water from layered silicates such as montmorillonite.

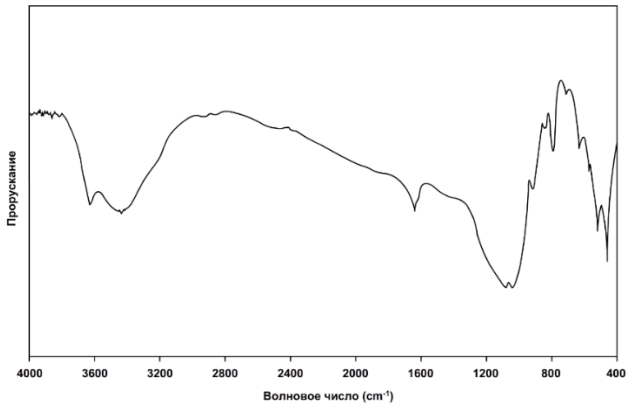

Figure 3. Infrared (FTIR) spectrum of the raw bentonite sample (BK).

The infrared (IR) spectrum of bentonite exhibits several characteristic absorption bands. Bands at 3629 and 3437 cm^{-1} correspond to O–H stretching vibrations of water molecules and hydroxyl groups in the bentonite structure. The band at 3250 cm^{-1} is associated with hydrogen bonding in interlayer water or structural hydroxyl groups, while the band at 2923 cm^{-1} indicates the presence of aliphatic C–H bonds, possibly originating from organic impurities.

X-ray diffraction patterns of enriched samples with particle sizes below 10 μm show a decrease in the intensity of low-temperature quartz reflections (0.428, 0.182, and 0.154 nm) and a 2–4-fold increase in the intensity of montmorillonite reflections (1.349, 1.253, 1.257, and 0.173 nm). The diffraction line of alkaline montmorillonite appears at $2\theta \approx 12.4°$, corresponding to an interlayer spacing of approximately 7.1 Å. Quartz exhibits a characteristic diffraction line at $2\theta \approx 26.6°$, while illite is identified by a diffraction line at $2\theta \approx 8.7°$ (interlayer spacing ≈ 10.3 Å), indicating a higher potassium content and greater water incorporation.

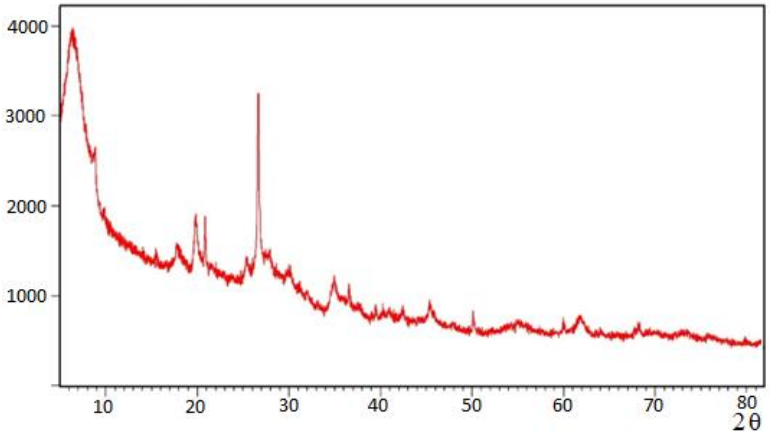

Figure 4. X-ray diffraction pattern of the montmorillonite-enriched bentonite sample.

X-ray diffraction patterns of enriched samples with particle sizes below 10 μm show a decrease in the intensity of low-temperature quartz reflections (0.428, 0.182, and 0.154 nm) and a 2–4-fold increase in the intensity of montmorillonite reflections (1.349, 1.253, 1.257, and 0.173 nm). The diffraction line of alkaline montmorillonite appears at $2\theta \approx 12.4°$, corresponding to an interlayer spacing of approximately 7.1 Å. Quartz exhibits a characteristic diffraction line at $2\theta \approx 26.6°$, while illite is identified by a diffraction line at $2\theta \approx 8.7°$ (interlayer spacing ≈ 10.3 Å), indicating a higher potassium content and greater water incorporation.

3.2 Changes in the mineralogical composition of clays.

Table 1. Mineralogical composition of raw and enriched bentonite samples (%)

Sample	MM	Quartz	Kaolinite	Illite	Chlorides	impurities
Raw	44,5	10,1	9,8	23,5	2,3	9,8
Enriched	91,8	0,2	0,9	6,1	0,1	0,9

Analysis of the mineralogical composition of the clays reveals significant changes after enrichment. In the enriched sample, the montmorillonite content increases markedly from 44.5% to 91.8%, demonstrating the effectiveness of the enrichment process in concentrating this key mineral component. The quartz content decreases sharply from 10.1% in the raw sample to 0.2% in the enriched sample, indicating successful removal of this mineral, which does not contribute to the adsorption properties of bentonite.

The kaolinite content also decreases, though to a lesser extent, from 9.8% to 0.9%. A reduction in illite content is likewise observed (from 23.5% to 6.1%), which further enhances the adsorption performance of bentonite. Changes in mineralogical composition significantly affect the dispersity of the samples; enriched forms exhibit a several-fold increase in dispersity (Table 2).

Table 2. Particle size (granulometric) distribution of the enriched bentonite sample (%).

Particle size (μm)					
≤1	1-5	5-10	10-63	63-100	100-1000
18,3	30,1	15,2	17,3	11,2	7,9

Analysis of the granulometric composition of the enriched bentonite, in comparison with the raw material, shows a noticeable increase in the fine fraction (≤1 μm). The enriched bentonite contains a higher proportion of particles ≤1 μm, indicating an increased content of fine particles, which is known to enhance adsorption properties. In addition, the fraction of particles in the 1–5 μm range also increases in the enriched bentonite sample. The method used to determine the total content of calcium and magnesium cations is based on their replacement by sodium cations through treatment of clay samples with a sodium chloride solution. Following this exchange process, the released calcium and magnesium cations are quantitatively determined by titration using a solution of disodium ethylenediaminetetraacetate (EDTA, also known as Trilon B) in the presence of Eriochrome Black T as an indicator.

Table 3. Composition of the cation exchange complex of the enriched bentonite sample (mg-eq/100 g).

Na^+	K^+	Ca^{2+}	Mg^{2+}	Total cations	pH of aqueous extract	Exchangeable acidity, mmol-eq/100 g
88,6	11,8	3,1	0,5	104,0	8,1	0,09

As can be seen from the data in Table 3, sodium cations constitute the largest fraction of the cation exchange capacity (CEC), amounting to 88.6 mg-eq/100 g, which indicates a high degree of sodium saturation of the bentonite. Calcium and magnesium are present in relatively small amounts (3.1 and 0.5 mg-eq/100 g, respectively), confirming the effectiveness of the enrichment process in reducing their concentrations.

3.3 Determination of organobentonite cation exchange capacity (CEC).

Determination of the cation exchange capacity (CEC) of clays is of critical importance in the development of organobentonites. CEC is a key indicator of the ion-exchange ability of clay materials, which is essential for organobentonite synthesis. This property enables the clay to exchange its inorganic cations for organic cations, resulting in the formation of organobentonites. A higher CEC allows the production of more efficient organobentonite materials. For further studies, a rapid and simple method for determining the total amount of exchangeable cations using cobalt ions was applied. This approach allows the determination of the total exchangeable cation content but does not provide information on individual cation types. In this procedure, 1 g of bentonite sample was mixed with 20 ml of a 0.025 M $[Co(NH_3)_6]Cl_3$ solution. The mixture was shaken at 20 °C for 24 h to allow cation exchange. Subsequently, the concentration of cobalt ions in the solution was measured by spectrometry. The CEC values were calculated from the concentration difference, and the results obtained for the raw bentonite showed good agreement with calculated reference values.

Figure 5 illustrates the variation in cation exchange capacity (CEC) of two bentonite samples as a function of thermal treatment temperature. Literature data were used as a reference for comparison. It can be observed that the CEC of both samples decreases with increasing treatment

temperature, with a particularly pronounced decline occurring at temperatures above approximately 100 °C.

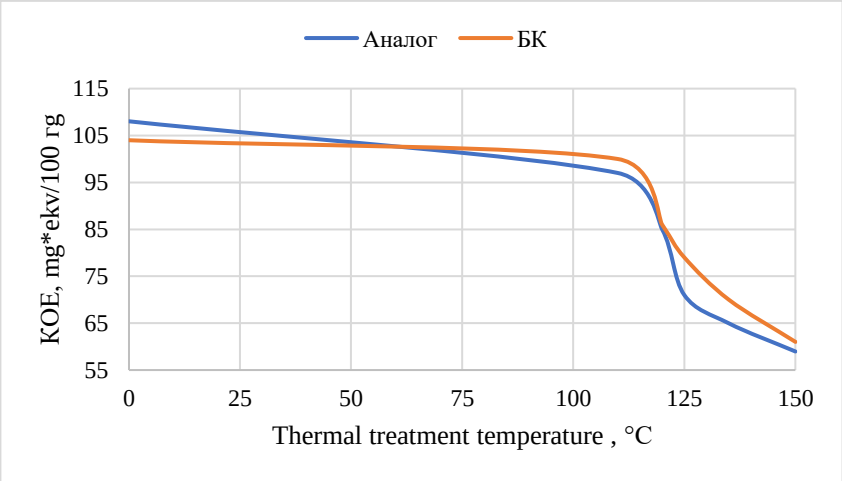

Figure 5. Variation of the cation exchange capacity (CEC) of bentonite samples as a function of thermal treatment temperature (24 h).

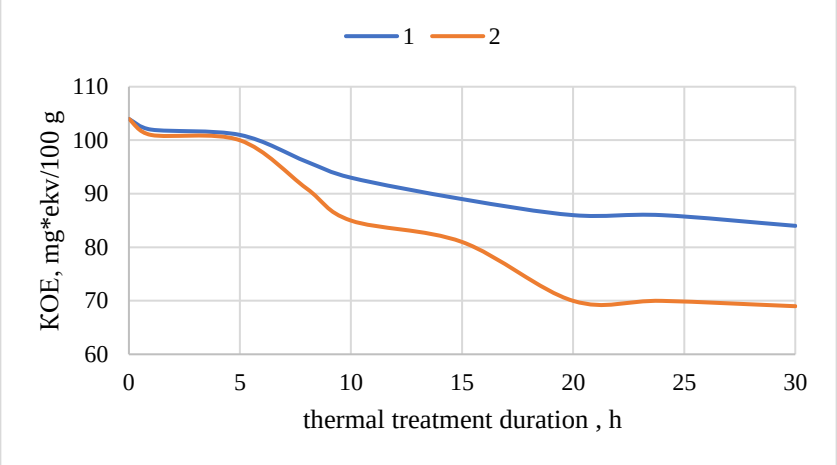

Figure 6. Effect of thermal treatment duration at different temperatures on the cation exchange capacity (CEC) of the bentonite sample (BK): 1 – 120 °C; 2 – 135 °C.

A significant decrease in CEC begins at temperatures above approximately 115 °C. For the 104-BK sample, this decrease is more pronounced, indicating more active structural changes during heating. These results confirm that thermal treatment is an effective method for controlling the cation exchange capacity of bentonite.

4. Conclusion. X-ray phase analysis confirmed that montmorillonite is the dominant mineral component of the studied bentonite. Chemical analysis revealed high contents of silica and aluminum, indicating the silicate and aluminosilicate nature of the material. Bentonite enrichment aimed at increasing montmorillonite content was achieved using mechanical separation, sedimentation, and sodium carbonate treatment, which effectively removed non-clay impurities and concentrated montmorillonite. This was confirmed by X-ray diffraction analysis, which showed an increased montmorillonite content and reduced quartz and accessory mineral phases in the enriched sample.

The granulometric composition of the enriched bentonite demonstrates an increase in fine fractions and overall dispersity, leading to improved adsorption properties and higher specific surface area. Changes in chemical composition, including reduced silica content and increased aluminosilicate components, further enhance bentonite performance. A significant reduction in iron and titanium contents indicates effective removal of these impurities. Sodium cations dominate the cation exchange

complex, reflecting a high degree of sodium saturation and the efficiency of the enrichment process. Thermal treatment was shown to be an effective means of controlling CEC, with decreasing values observed at higher treatment temperatures. The cation exchange capacity of bentonites directly influences their structural characteristics, particularly interlayer spacing. Samples with different CEC values exhibit variations in interlayer distance, affecting pore structure and surface acidity.

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