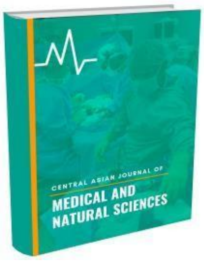

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Article

Alkaline Activation of Bentonite

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Abstract: The results of alkaline activation of natural bentonite from the Logonsky deposit using a 2 wt.% sodium carbonate solution with variation of treatment temperature and duration are presented. The effect of activation conditions on the cation exchange capacity, as well as on the technological properties of the material (moisture content, plasticity number, and pH of a 5 wt.% suspension), was evaluated. It is shown that increasing the temperature to 60 °C leads to an increase in cation exchange capacity, reaching a maximum at a treatment time of 3–5 h, which is associated with intensification of Ca^{2+}/Na^{+} exchange in the interlayer positions of montmorillonite. A further increase in temperature to 70–80 °C is accompanied by a decrease in cation exchange capacity, probably due to partial structural changes and a reduction in the fraction of active exchange sites. An optimal activation regime providing high cation exchange capacity while maintaining satisfactory technological properties of the material was established.

Keywords: Bentonite, Montmorillonite, Alkaline Activation, Sodium Carbonate, Sodium Form, Cation Exchange Capacity, Treatment Temperature, Treatment Time, Plasticity, Suspension pH.

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1. Introduction

Bentonite clays, whose main mineral component is montmorillonite, belong to the most widely available natural aluminosilicates with pronounced sorption and ion-exchange properties [1], [2], [3]. However, natural bentonites are often present in the calcium form and contain associated mineral impurities, which reduces their dispersibility, swelling capacity, and cation exchange activity, and also limits their application in water treatment technologies and in the preparation of inorganic composite materials [4], [5], [6], [7].

One of the most common approaches to improving the functional properties of bentonite is alkaline activation with sodium carbonate, which ensures the conversion of the Ca-form into the Na-form and an increase in cation exchange capacity due to enhanced mobility of interlayer cations and an increase in the number of accessible exchange sites. The efficiency of this approach strongly depends on the temperature–time parameters of treatment, since they determine the rate and completeness of ion exchange and may affect the preservation of the layered structure of montmorillonite [8], [9], [10], [11], [12].

The aim of this study is to evaluate the effect of temperature and treatment duration of natural bentonite from the Logonsky deposit with a 2 wt.% Na_2CO_3 solution on the cation exchange capacity and related technological characteristics (moisture content, plasticity number, and pH of a 5 wt.% suspension), and to substantiate the optimal alkaline activation regime for subsequent sorption and modification applications.

2. Materials and Methods

2.1 Materials

Bentonite clays from the Logonsky deposit were selected as the object of study. Sample preparation and quantitative and qualitative analyses were carried out in accordance with GOST 28177-89.

2.2. Methods

For alkaline activation, the initial natural sample (LB) was used. A 2 wt.% aqueous solution of sodium carbonate was applied as the activating reagent to convert the calcium form of bentonite into the sodium form. The bentonite sample was mixed with the Na_2CO_3 solution at a solid-to-liquid ratio of 1:5. The treatment was performed at 25–80 °C for 1–5 h, depending on the selected regime.

After activation, the suspension was aged for 24 h at room temperature to complete the ion-exchange processes, followed by filtration and washing with distilled water to neutral pH. The obtained material was dried at 80 °C to constant weight, ground, and sieved through a 0.063 mm mesh.

The cation exchange capacity was determined as the sum of the major exchangeable cations. The contents of Na^+ and K^+ were determined according to GOST 3594.3-93, while Ca^{2+} and Mg^{2+} were determined according to GOST 3594.2-93. The total cation exchange capacity of the samples was calculated based on these results.

Moisture content was determined gravimetrically. A 5.00–10.00 g portion of the sample (m_0) was placed in a pre-dried and weighed weighing bottle, dried at 105 ± 2 °C to constant mass, cooled in a desiccator, and weighed (m_1). The moisture content was calculated using the following equation:

$W, \% = (m0 - m1)/m0 \times 100.$

The plasticity index was determined by the Atterberg method as the difference between the liquid limit (WL) and the plastic limit (WP): WP: $IP = WL - WP$.

The liquid limit was determined from the moisture content of the paste at closure of the groove in the Casagrande apparatus (or by the cone penetration method), while the plastic limit was determined from the moisture content of the sample when rolling into threads with a diameter of approximately 3 mm until cracking occurred.

The pH of a 5 wt.% suspension was determined potentiometrically. For this purpose, 5.00 g of the sample (on a dry matter basis) was diluted with distilled water to 100 mL, stirred for 10–15 min, and aged for 30 min. Measurements were performed using a pH meter at 20–25 °C in the supernatant liquid or in a thoroughly mixed suspension. Prior to measurement, the instrument was calibrated using buffer solutions with pH 4.01 and 6.86 (and, if necessary, 9.18).

3. Results and Discussion

Analysis of the data presented in Fig. 1 demonstrates the effect of treatment temperature and duration on the cation exchange capacity of natural Logonsky bentonite treated with a sodium carbonate solution.

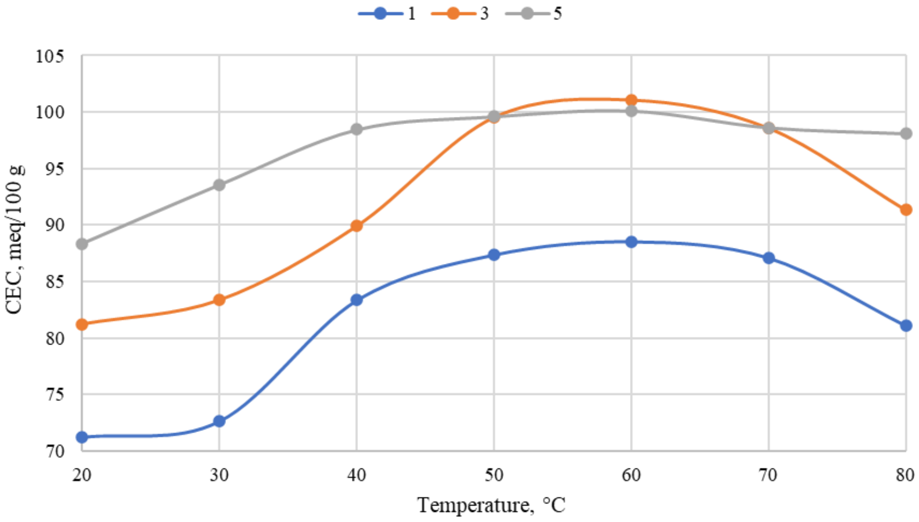

Figure 1. Change in cation exchange capacity during treatment with a Na_2CO_3 solution.

The graph shows that with an increase in temperature within the range of 20–60 °C, the CEC rises in all series, which is associated with the intensification of ion exchange between Ca^{2+} and Na^{+} in the montmorillonite structure. The maximum CEC values are observed at 60 °C: 88.56 mg·eq/100 g for 1 hour, 101.1 mg·eq/100 g for 3 hours, and 100.12 mg·eq/100 g for 5 hours.

Further increasing the temperature to 70–80 °C results in a decrease in CEC, particularly pronounced at shorter activation durations. This may be due to partial destruction of the interlayer structure of montmorillonite and the conversion of some ions into an inactive form, or the formation of poorly soluble compounds. In the case of short-term treatment (1 hour), the activation efficiency is significantly lower compared to longer treatments, confirming the necessity of sufficient time for the completion of ion-exchange processes.

Table 1 presents the changes in the main technological properties and cation-exchange capacity of bentonite samples depending on the temperature of alkaline treatment, with the process duration fixed at 3 hours.

Table 1. Changes in the main characteristics of samples obtained at different temperatures of alkaline treatment.

Properties	**Indicators**					
	OLB-20	**OLB-30**	**OLB-40**	**OLB-50**	**OLB-60**	**OLB-70**
CEC, meq/100 g	81,22	83,36	89,92	99,56	101,1	98,6
Moisture content, %	3,2	5,4	4,6	3,4	3,5	5,3
Plasticity index	31,2	41,3	39,8	36,5	32,6	39,8
pH of 5 % aqueous suspensions	7,1	7,3	7,6	7,4	7,3	7,4

The analysis of the data in Table 1 allows for the evaluation of the effect of alkaline treatment temperature on the main technological properties of activated LB. With an increase in temperature from 20 °C to 60 °C, a steady rise in cation-exchange capacity (CEC) is observed, from 81.22 to a maximum value of 101.1 mg·eq/100 g, which is associated with an increased intensity of ion exchange between Ca^{2+} and Na^{+} in the montmorillonite structure. Further increasing the temperature to 70 °C leads to a decrease in CEC to 98.6 mg·eq/100 g, which may be attributed to partial structural destruction or a reduction in the efficiency of ion exchange due to the formation of secondary phases.

The moisture content varies within the range of 3.2–5.4 %, showing no clear dependence on temperature; however, its increase at 30 °C and 70 °C may be related to differences in the water-retention capacity of the material after drying. The plasticity index reaches a maximum at 30 °C (41.3), then decreases and rises again at 70 °C. This may indicate the complex behavior of the montmorillonite system, where plasticity is determined not only by interlayer moisture but also by the structural mobility of the clay.

The pH of a 5 % aqueous suspension remains within 7.1–7.6, indicating a neutral to slightly alkaline reaction of the medium after treatment and reflecting adequate washing of the samples to remove excess Na_2CO_3. This is important to prevent undesirable effects during further use of the material in sorption or composite systems.

Thus, the optimal temperature for alkaline activation using a 2 % Na_2CO_3 solution is approximately 60 °C, with a treatment duration of 3–5 hours. Under these conditions, the maximum cation-exchange capacity is achieved while maintaining satisfactory technological properties, including plasticity and a stable pH level. The combination of these parameters makes this treatment regime the most effective from the standpoint of inorganic materials technology for preparing natural bentonite for subsequent sorption and modification applications.

4. Conclusion

Alkaline activation of Logon bentonite with a 2 % Na_2CO_3 solution leads to a pronounced increase in cation-exchange capacity (CEC), which is associated with the intensification of Ca^{2+}–Na^{+} ion exchange within the montmorillonite structure. The highest CEC is achieved at a temperature of approximately 60 °C and a treatment duration of 3–5 hours, whereas further temperature increases to 70–80 °C result in a decrease in CEC, likely due to partial structural changes and a reduction in the proportion of active exchange sites.

Technological properties (moisture content, plasticity index) change moderately and do not show a clear dependence on temperature, while the pH of a 5 % suspension remains in the neutral to slightly alkaline range, confirming adequate washing to remove excess reagent. The results obtained justify selecting the regime of 60 °C and 3–5 hours as the most effective for preparing natural bentonite for subsequent sorption and modification applications.

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