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Investigation of the Structure and Composition of Natural, Enriched, and Na-Modified Clays

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ABSTRACT: This article presents a study of the structure and mineral composition of natural, enriched, and sodium-modified clays in order to evaluate the changes in their physicochemical properties under the influence of various treatment methods. The results showed that sodium modification promotes an increase in specific surface area, improves particle dispersion, and enhances the ordering of the montmorillonite layered structure. Significant changes in cation exchange capacity and sorption characteristics were also observed. The obtained data indicate the high potential of Na-modified clays for use as sorbents and components of barrier materials.

I. INTRODUCTION

Clays are widely used natural materials with diverse applications in environmental protection, construction, and industry due to their unique structural and physicochemical properties. Among various types of clays, montmorillonite-rich bentonites have attracted significant attention for their high surface area, cation exchange capacity, and swelling ability. However, the natural form of clays often requires modification to enhance these properties for specific applications. One of the most effective approaches to improving the performance of clays is sodium modification, which can significantly influence their structural characteristics and surface properties. Enrichment and sodium treatment processes alter the mineralogical composition, increase the dispersion of particles, and improve the sorption capacity, making clays more suitable for use in adsorption, catalysis, and barrier systems. This study focuses on the investigation of the structure and mineral composition of natural, enriched, and sodium-modified clays. The main objective is to evaluate the changes in their physicochemical properties resulting from different treatment methods and to determine the potential applications of Na-modified clays in various technological fields.

II. RESULTS AND DISCUSSION.

Bentonite clays from the Logon (LBG), Kattakurgan (KBG), and Beshtyube (BBG) deposits were selected for the dissertation research. The chemical composition of the fine fraction (<2 μm) was determined according to GOST 21216-2014. The obtained results are presented in Table 1.

Table 1. Chemical composition of the studied samples

Oxides	Samples		
	LBC	KBC	BBC
SiO_2	59,12	62,25	63,45
Al_2O_3	17,12	16,85	18,75
Fe_2O_3+ FeO	5,61	6,12	2,92
CaO	1,08	2,12	3,45
MgO	2,12	1,85	1,18
Na_2O	3,42	1,56	1,65
K_2O	2,26	1,73	0,75

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P_2O_5	0,28	0,45	0,21
SO_3	0,36	0,56	0,56
CO_2	0,33	0,48	0,74
Impurities after calcination	8,3	6,03	6,34

The total SiO_2 + Al_2O_3 content in all three samples ranges from 76% to 82%, indicating the dominance of montmorillonite and minor quartz–feldspar impurities. The highest SiO_2 (63.45%) and Al_2O_3 (18.75%) contents were recorded in BBG, while the lowest SiO_2 content (59.12%) was observed in LBG. As a result, the SiO_2/Al_2O_3 ratio ranges from 3.39 (BBG) to 3.69 (KBG), which is close to the theoretical value. The sum of alkali and alkaline earth oxides is highest in LBG (8.88%), where Na_2O + K_2O account for over 65%, and the Na_2O/CaO ratio is approximately 3.2. This corresponds to a sodium-type bentonite with high swelling capacity and cation exchange capacity (CEC). KBC demonstrates a more balanced Na_2O/CaO ratio of approximately 0.74 and elevated Fe_2O_3 + FeO content (6.12%), while BBG represents a calcium type (Na_2O/CaO ≈ 0.48) with the highest CaO content (3.45%) and low natural swelling ability. MgO varies from 1.18% (BBG) to 2.12% (LBG), reflecting the degree of substitution in the octahedral layer. The high iron oxide content in KBG enhances acid dissociation, while the minimum Fe content in BBG (2.92%) is beneficial for ion-exchange sorbents. Loss on ignition is highest in LBC (8.3%) and lowest in KBG and BBG (6.0–6.3%), which is associated with a higher amount of bound water and organic matter in LBC and its high reactivity and sorption activity. To determine the total cation exchange capacity (CEC) of the <0.063 mm fraction (1.000 g), the sample was shaken twice with 100 mL of 1 M NaCl at 25 °C for 3 hours, then centrifuged (3000 rpm, 10 minutes), and the extract was removed. The salt residues were washed with a 1:1 ethanol–water solution until Cl^- ions disappeared (tested using $AgNO_3$). In the percolate, Ca^{2+} and Mg^{2+} were titrated with 0.01 M EDTA at pH 10 using Eriochrome Black T as the indicator (1 mol EDTA = 1 mol of cation). To determine Na^+ and K^+, the sample was treated three times with 1 M NH_4OAc (pH 7.0); the combined percolate was brought to 100 mL, and the concentrations of Na and K were determined using flame photometry at wavelengths of 589.0/589.6 nm (Na) and 766.5/769.9 nm (K), calibrated with 0.5–5 mg/L standards. The total equivalents of Ca^{2+}, Mg^{2+}, Na^+, and K^+ were recalculated per 100 g of absolutely dry sample and expressed in mmol(+)/100 g with a margin of error of ±2%. The obtained results are shown in Table 2.

Table 2. Cation exchange complex (mg-eq/100 g) of clays

Samples	Na^+	K^+	Ca^{2+}	Mg^{2+}	$\Sigma_{cations}$	pH of aqueous extract
KBC	24,2	9,8	23,2	11,5	68,7	7,4
LBC	38,8	5,6	21,2	8,9	74,5	8,1
BBC	6,5	4,2	31,2	9,9	51,8	7,5

LBC has the highest cation exchange capacity (CEC) at 74.5 mg-eq/100 g, due to its high Na^+ content (38.8) and moderate Ca^{2+} (21.2); Mg^{2+} and K^+ are 8.9 and 5.6, respectively. The dominance of sodium indicates a sodium-type montmorillonite with high swelling ability and water retention capacity. The pH of 8.1 indicates an alkaline environment and suitability for organophilic modification. BBG has the lowest CEC (51.8 mg-eq/100 g), with dominant Ca^{2+} (31.2) and low Na^+ (6.5); Mg^{2+} and K^+ are 9.9 and 4.2, respectively. This is typical for calcium bentonites with limited swelling capacity, which require sodium activation. The pH of 7.5 indicates a slightly alkaline environment with weak buffering capacity. For the enrichment of bentonite clays, gravitational sedimentation, selective flocculation, and centrifugation with low water consumption (<0.04 L/g of raw material) were used. The fine fraction (<63 μm) was dispersed in a tenfold excess of water, allowed to settle for 8–10 minutes, and the colloidal layer was decanted. The dispersion and decantation cycle was repeated 3–4 times, after which the concentrate was flocculated using 0.05% Praestol 2500 and centrifuged at 3000 rpm for 5–10 minutes. The sediment was washed, dried at 25 °C (24–48 hours), and then at 90 °C to constant weight (2–3 hours). For alkaline modification, a stoichiometric amount of Na_2CO_3 was added to the suspension, calculated as two equivalents of Na^+ per equivalent of exchangeable Ca^{2+} + Mg^{2+} (KBC – 34.7, LBG – 30.1, BBG – 41.1 mg-eq/100 g). The calculated masses of Na_2CO_3 per 100 g of clay are presented in Table 3.

Table 3. Calculation of the stoichiometric amount of Na_2CO_3 for complete sodium modification of bentonite samples

Samples	Σ (mg-ekv/100 g)	n (mmol Na_2CO_3)	m (g Na_2CO_3 for 100 g)
KBC	34,7	17,35	17,35·0,106 ≈ 1,84
LBC	30,1	15,05	15,05·0,106 ≈ 1,60
BBC	41,1	20,55	20,55·0,106 ≈ 2,18

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Table 4. Cation Exchange Complex (mg-eq/100 g) of Enriched and Na-Modified Clays

Samples	Na^+	K^+	Ca^{2+}	Mg^{2+}	$\Sigma_{cations}$	pH of aqueous extract
EKBC	29,8	11,6	23,9	9,9	75,1	7,8
ELBC	48,5	6,2	18,9	6,4	79,9	8
EBBC	10,4	4,6	34,3	8,3	57,6	7,6
Na-KBC	56,6	9,9	8,6	5,5	80,6	8,1
Na-LBC	65,8	5,9	6,5	5,9	84,1	8,5
Na-BBC	44,5	4,1	5,9	3,6	58,1	7,9

III. CONCLUSION

After fractional enrichment, the total CEC of all samples increased due to the removal of impurities and enrichment of the smectite phase: for enriched KBG (EKBG) from 68.7 to 75.1 mg-eq/100 g (+6.4), enriched LBG (ELBG) from 74.5 to 79.9 (+5.4), and enriched BBG (EBBG) from 51.8 to 57.6 (+5.8). The main increase was provided by Na^+: in EKBG it rose by 5.6, in ELBG by 9.7, and in EBBG by 3.9 mg-eq/100 g, due to the removal of Ca^{2+} and Mg^{2+}. The pH of the extract increased by 0.4–0.6, indicating an enhancement in surface alkalinity. Additional Na-activation (with Na_2CO_3) further increased the CEC: Na-KBG up to 80.6 (+5.5), Na-LBG up to 84.1 (+4.2), and Na-BBG up to 58.1 (+0.5). The Na^+ content after modification reached 56.6 (+26.8), 65.8 (+17.3), and 44.5 (+37.9) mg-eq/100 g, respectively, while the average reduction in Ca^{2+} + Mg^{2+} was 15–25 mg-eq/100 g. The final pH of 8.1–8.5 indicates complete sodium activation and the formation of a stable alkaline buffer.

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