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ORIGINAL RESEARCH ARTICLE

Study of the processes of sorption and ion exchange of bentonite clays of Georgia

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ABSTRACT

For rational use of natural resources and address environmental issues, it is necessary to create new or modernize existing technological processes One such resource is bentonite clay, but there is practically no information on its use in water treatment processes. It should be noted that bentonites differ from each other depending on their deposit. Therefore, when using them, it is necessary to conduct research according to their field of application. Studied was the process of sorption by bentonite clay (on the example of Ascanite clay of Georgia), which showed that it can be used as an ion exchanger. Established was the total exchange capacity of clay, which was 500 g-eq/m3 (wet cation exchanger) (100 meq/100g of clay). Determined was experimentally the exchange coefficient of Ca-Na ions on clay, which amounted to 1.59 m3/m3 (wet cation exchanger/water). To check the confidence of the obtained value of the ion exchange coefficient, the Pearson criterion was used. It showed that the obtained value describes the ongoing technological process with more than 95% reliability. When calculating the Pearson criterion, a developed new method for determining the number and length of grouping intervals was used.

Keywords: Na-bentonite clay; sorption; ions exchanged capacity; ion exchange coefficient; water treatment

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1. Introduction

Water is one of the main and irreplaceable resources on Earth. Various pollutants disrupt the ecological balance of the environment, as a result of which species decrease or disappear self-cleaning ability of the environment and, in particular, water bodies. The deterioration of the quality of natural water is a problem on a global scale, which causes various diseases and complicates the technology of purifying natural water for drinking and industrial use. Nowadays, there has been increased interest in the creation of environmentally friendly substances, reagents, and materials based on the sorption properties of clays and silicates.

Bentonite clays differ sharply from other pre-existing clay formations and occupy a special position. Due to their specific properties, they are widely used in various industries[1-4].

Bentonites, montmorillonites, kaolins, illite, and other minerals are used in water purification technology due to their developed specific surface areas, chemical and mechanical stability, varying structural characteristics, and low cost.

The specific properties of bentonite clays depend on the structure of the crystal lattice of its constituent minerals[1,3,5]. Bentonite clays should include clays that are composed mainly of aluminum and magnesium members of the montmorillonite group, with a swelling crystal lattice. The content of the montmorillonite component should be at least 60-70%. Montmorillonite is a highly dispersed layered aluminosilicate, in which a negative charge appears due to non-stoichiometric substitutions of cationites of the crystal lattice. This charge is compensated by exchange cations located in the interlayer space. This is due to the high hydrophilicity of bentonite. When water is added to bentonite, it penetrates the interlayer space of montmorillonite, hydrates its surface, and exchanges cations, which causes the mineral to swell. Montmorillonite has high cation exchange and adsorption properties[5-7].

Bentonite has found wide application in the construction, metallurgical, and mining industries due to the above properties. It is used in the production of expanded clay, agglomeration of ores, and in the composition of molding sands in the form of a binder additive. It also contributes to the formation of a viscous gel in the preparation of drilling mixtures and grout cements[5]. It is also used in the chemical industry, as an active mineral additive, in the production of paper, rubber, mastics, and explosives. In agriculture, bentonite is effectively used in the production of animal feed and for the improvement of acidic soils[7]. Moreover, it is known that bentonite is used in more than 200 industries[4,7,8-10].

Currently, bentonite clays are widely used in medicine and pharmacology[11]. Clays are used to successfully treat acute respiratory diseases, bronchitis, pleurisy, peritonitis, cholelithiasis and bladder diseases. Bentonites adsorb toxic substances and can serve as an antidote for poisoning; as an effective anthelmintic agent, they can be part of an antigen introduced into living beings. The ability of bentonite clays to selectively adsorb chemicals plays a significant role in the treatment of diseases of the gastrointestinal tract[11,12]. Georgian scientists report the possibility of using bentonite solution (Askancoll) for diaper rash in children, in cases of cervical erosion, in surgical and urological practice. Based on clinical trials, they came to the conclusion that in the treatment of pyoderma and skin maceration, as complications after wounds, the use of Ascancoll for prophylactic purposes gives good results[11,12].

Attention should be paid to the use of bentonite clay in the purification of natural and waste waters[11]. In ancient times, many peoples of Central Africa and Asia, Australia, China, Burma, and Guinea traditionally used clay in various ways. One famous example was the wearing by the population of small pieces of dried clay, which was added to water and used in meals[12]. But despite this, there is practically no information on the use of clay in water treatment processes for urban or industrial water supply. There are also known technologies for wastewater treatment from dyes, pharmaceuticals, heavy metals in circulating and industrial water supply systems[13,14], and other organic and inorganic compounds[15-17]. The use of montmorillonite as a coagulant during contact water clarification produces disinfection and reduces the concentration of chlorine preparations during further processing.

Bentonite is an environmentally friendly natural product. Due to this, it is actively used for effective clarification and stabilization of fruit juices, wines and similar materials[18,19]. Studies have shown that its use does not adversely affect the physicochemical and organoleptic characteristics of processed drinks. But it should be noted that bentonites differ from each other, depending on their deposit. Therefore, it is recommended that when using them, research should be carried out according to the area in which it is used.

The use of Georgian bentonite clays in municipal and industrial water supply or in wastewater treatment will reduce the cost of technological processes. In addition, the waste generated during water treatment will be safe for the environment, since bentonite is a natural product.

The aim of the study is to investigate the sorption properties of bentonite clays of Georgia, which includes determining the full ion exchange capacity and ion exchange coefficient. The results of the study will allow us to consider the adsorption isotherm and develop recommendations for the use of bentonite clay in the processes of cleaning natural and waste water.

2. Materials and methods

2.1. Material characteristic

Bentonite clays are widespread in Georgia. The total amount of predicted reserves exceeds 130 million tons[1], which was formed as a result of the deposition of ash from underwater volcanoes in seawater twenty million years ago. The products of Georgian deposits are represented by two types of bentonite - alkaline and alkaline earth. In turn, they are divided into two mineralogical zones: alkaline earth and alkaline earth-alkaline (with a subzone of alkaline bentonite), among which we should note the deposit in the village of Askana, which name was given to bentonite clay.

The Askana group of bentonite deposits is located in a mountainous area at the foot of the South Caucasus ridge near the village of Askana, the city of Ozurgeti, and consists of several isolated areas: Tsikhis-Ubani, Vanis-Kedi, Mtis-Piri, Uchkhubi, etc., located at a distance from each other the first up to several kilometers.

All these bentonite deposits are genetically interconnected and form a group of deposits of alkaline earth and alkaline bentonites of volcanic origin. The source rocks for the formation of Ascanian bentonites were thick strata of volcanic rocks that underwent secondary changes.

It should be noted that local clay is considered the best natural adsorbent, which has high sorption activity even in its natural state, which is especially worthy of attention. Its natural chemical composition (**Table 1**) exceeds the properties of analogs and similar materials (**Table 2**) [20].

Table 1. The chemical composition of the bentonites of the Askan field

(data from the report on geological exploration of the Askan field[20]).

Type of Clay		**Quarry Tsikhis-Ubani**			**Quarry Vanis Kedi**
		Askangel	**Askanglina**	**Askangel 1**	**Askanglina**
Chemical components, wt. %	SiO2	54,20	51,40	59,50	53,14
	Al2O3	17,12	16,86	18,60	18,0
	Fe2O3	3,95	3,29	3,50	4,61
	FeO	1,00	1,00	н/д	1,08
	TiO2	0,33	0,25	0,40	0,40
	MnO	0,06	0,04	н/д	0,25
	CaO	1,50	2,0	2,30	4,55
	MgO	3,98	4,71	4,50	4,65
	Na2O	2,80	0,65	2,40	1,15
	K2O	2.0	0,5	1,30	2,8
	SO3	0,17	0,16	0,18	0,96

Of the deposits of the Askan group, the most studied are the Tsikhis-Uban and Vanis-Kedsk areas. Bentonite from the Tsikhis-Ubansky site is represented by both the sodium variety, askangel, and the calcium variety, askanglina. The reserves of the Tsikhis-Uban deposit were approved in 1953 and on its basis the production of bentonite products for a wide range of applications was organized: for drilling, for foundry, for the ceramic industry, acid-activated bentonite, as additives in animal feed, etc.

Despite the widespread use of Georgian bentonites in various fields of industry, there is practically no information on their use for the purification of natural and wastewaters.

Table 2. Composition and properties of bentonite at the Askan deposit (in comparison with bentonite from Milos island (Greece) and Wyoming bentonite MX-80, USA1[20]).

№	**Indicators**	**Bentonites**		
		Askan, Georgia, uch. Tsikhis-Ubani	**Ca-bentonite, about. Milos, Greece**	**Na-bentonite MX – 80, Wyoming, USA**
1	Mineral composition (XRD – X-ray diffraction analysis)			
1.1	Smectite, (%)	85-95	75-85	65-75
1.2	Illit, (%)	2-6	< 4	2-4
1.3	Quartz, (%)	< 4	< 4	5-7
.4	Calcite, (%)	3-5	8 — 12	6 – 9
1.5	Other, (%)	< 4	< 4	5-16
2	Physical properties			
2.1	Cation exchange capacity, mg.equiv./100gr	95 — 105	80 — 90	70 — 80
2.2	Swelling Index, ml. / 2g.	22 — 26	6 — 10	28 — 32
2.3	Water adsorption (Enslin-Neff), %	480 — 520	110 — 150	650 — 800

2.2. Experimental part

Ion exchange, like any chemical reaction, obeys the general thermodynamic laws of equilibrium[21–23]. The penetration of water into the grain of the ion exchanger causes the dissociation of ionogenic groups to a lesser extent than in solution, but it is sufficient for the ion to diffuse freely. Therefore, the matrix can be considered as an analog of an ion in an electrolyte solution. The ion exchange process is reversible:

$$[\text{cat}] \cdot Na_2 + Ca^{2+} \rightleftarrows [\text{cat}] \cdot Ca + 2Na^+ \qquad (1)$$

where [cat]· Na_2 - the designation of the cation exchanger in the Na-form, i.e. with sodium ions in the diffusion layer;

[cat]·Ca - designation of cation exchanger in Ca-form.

Applying the mass action law for equation (1), we obtain an expression for the ion exchange constant or, in other words, the distribution coefficient

$$K = \frac{a_{Ca} \cdot C_{Na}^2}{a_{Na}^2 \cdot C_{Ca}} \qquad (2).$$

where a_{Ca}, a_{Na} - the concentration of ions in the solid phase (ion exchanger), g-eq/m^3 (wet charge of the cation exchanger);

C_{Na}, C_{Ca} – equilibrium concentrations of ions in water, g-eq/m^3

The total sum of equivalents of ions sorbed on the cation exchanger remains constant and represents the value of the total exchange capacity of the cation exchanger E (g-eq/(m^3 of wet cation exchanger);

$$E = a_{Na} + a_{Ca} = const \qquad (3)$$

During the exchange process, the sum of cation equivalents in solution A also remains constant:

$$A = C_{Ca} + C_{Na} = const \tag{4}$$

Therefore, substituting expressions (3) and (4) into equation (2), we obtain

$$K = \frac{a_{Ca} \cdot (A - C_{Ca})^2}{(E - a_{Ca})^2 \cdot C_{Ca}} \tag{5}$$

This resulting expression, called the adsorption isotherm, shows the relationship between the calcium concentration in solution and the equilibrium concentration on the cation exchanger. Thus, solving the isotherm equation with respect to a_{Ca},, we obtain

$$a_{Ca} = E + B\left(1 - \sqrt{\frac{2 \cdot E}{B} + 1}\right) \tag{6}$$

where $B = \frac{0{,}5 \cdot (A - C_{Ca})^2}{C_{Ca} \cdot K}$

Then, by giving C_{Ca} different values within 0 ÷ A, one can calculate the corresponding values of a_{Ca} and plot the adsorption isotherm curve. The adsorption isotherm obtained based on experimental data is shown in **Figure 1**.

Thus, to determine the total exchange capacity of ascanit clay, 5 g of Na-bentonite clay (in powder form), humidity 10-12%, bulk density 0.9-1.1 g/cm^3, was placed in a beaker with a volume of 200 ml, and added a 4% solution of $CaCl_2$. The solution was prepared with distilled water. In the initial solution, the concentration of Ca ions was determined by using the standard method according to Russian State Standard GOST 20255.1-89, which amounted to 727 mg-eq/l. Then, after stirring and settling (the procedure was carried out several times), the concentration of calcium ions in the solution was again determined. When the content of calcium in the original and spent solution differed, the precipitated clay was placed in a new beaker, the original solution was added, and the amount of Ca in the original and spent solutions was again determined. This procedure was repeated until the amount of calcium in the initial and spent solutions reached the same value. After that, the amount of calcium ions retained by the clay was determined. The experiments were carried out under standard conditions at a temperature of 19-21 ^{0}C. Ten parallel cycles conducted showed that the average value of the total exchange capacity of ascanit clay was 500 meq/l (100 meq/100g of clay).

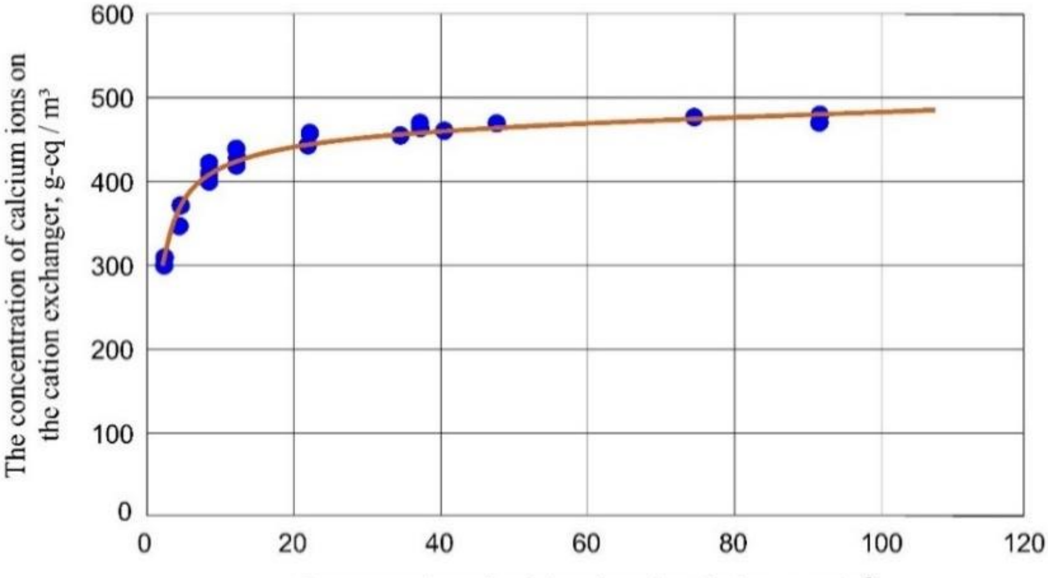

Figure 1. Dependence of the concentration of calcium ions on the cation exchanger on the concentration of calcium ions in the solution.

3. Results and discussion

3.1. Defining grouping intervals

One of the tasks of processing experimental data obtained in laboratory conditions is to determine the appropriate ion exchange constant for constructing the adsorption isotherm, dependence (6). To do this, for each ratio of calcium ions in the solution and on the cation exchanger, experimental values of the ion exchange constant were found. The theoretical values of the ion exchange constant were determined using formula (6). Then the error of discrepancy between the experimental and theoretical values was found.

Considering that the distribution of deviations between the points determined by the equation and those obtained experimentally obeys the normal distribution law, therefore, the assessment of the correspondence between the values calculated by the formula and obtained from the experiment (**Table 3**) is carried out using the Pearson correspondence criterion. In this case, discrepancies (errors) should be presented as a percentage in the form of a variation series.

To determine the number of grouping intervals, the following formula is used[24]

$$m = 1{,}9 \cdot n^{0{,}4} \tag{7}$$

where n – the number of points obtained from the experiment.

The above dependence fully satisfies the practical requirements and does not contradict the experimental and theoretical data given in the scientific and technical literature.

In order to prevent two columns of equal value from forming in the center of the histogram, causing artificial smoothing, it is necessary that the number of grouping intervals be an odd number. Following these requirements, the choice of possible values for the number of grouping intervals will be very limited.

To determine the length of the interval, the dependence[24] is used:

$$d = 2\Delta x_m / m \tag{8}$$

where Δx_m - maximum deviation from the center of the extreme point.

For the extreme points not to be beyond the extreme columns of the histogram, the resulting interval length value must be rounded up. The interval length value should be easily halved to establish the coordinates of the center of the columns. Under this condition, the range of the histogram is defined as follows:

$$l = d \cdot m \tag{9}$$

The difference $l - l_1$ is where l_1 - the initial range between the extreme points, is evenly added to the values of the extreme points. The interval must start with an integer, and zero must fall on the boundary of some interval. This is because in the future, when determining the weighted average value of the interval, there would be no loss of a term of the series due to multiplication by zero or reduction of terms when passing through zero.

3.2. Processing of experimental data

The values of the boundaries of the intervals are determined and the corresponding values of the centers of the columns are calculated. Further calculations of the data given in **Table 1**, are kept in tabular form **Table 4**, according to the standard method for calculating the Pearson criterion.

The theoretical frequency of falling into the interval is determined by the dependence:

$$n_i' = \frac{n \cdot \Delta x}{\sigma \cdot \sqrt{2\pi}} \cdot e^{-\frac{\left(x_i - \overline{x_i}\right)}{2\sigma^2}} \tag{10}$$

where x_i – average value of intervals; $\overline{x_i}$ – weighted average of the number of observations; σ - standard deviation of the number of observations; Δx - interval length; n - number of experimental points; m - the number of intervals; n_i - the number of experimental points that fell into the interval (frequency of hitting);

Table 3. Processing of experimental data on the determination of the ion exchange coefficient on bentonite clay.

No.	α_{Ca} **meq/100g of clay**	α_{Ca} **g-eq/m³**	C_{Ca} **g-eq/m³**	$A - C_{Ca}$ **g-eq/m³**	**Obtained from experimental data** $K_{\text{Э}}$	**Calculated by formula (5) (averaged)** K_{T}	$100 \cdot \frac{\left(K_{\text{Э}} - K_{\text{T}}\right)}{K_{\text{Э}}}$	**Variation series**
1	60	300	2,0	18,5	1,28	1,59	-24,22	-43,24
2	62	310	2,0	18,5	1,47	1,59	-8,16	-40,71
3	70	350	4,0	18,2	1,29	1,59	-23,26	-38,26
4	7,1	376	4,0	18,0	1,98	1,59	19,70	-25,20
5	74	370	4,0	18,0	1,77	1,59	10,17	-24,22
6	84,6	423	8,0	15,0	2,0	1,59	20,50	-24,22
7	80	400	8,0	15,0	1,13	1,59	-40,71	-23,26
8	82	410	8,0	15,2	1,46	1,59	-8,90	-21,37
9	88,2	441	11,8	13,7	2,02	1,59	21,29	-21,37
10	84,6	423	11,8	13,7	1,11	1,59	-43,24	-15,22
11	86,4	432	11,8	13,7	1,48	1,59	-7,43	-8,90
12	90	450	22,0	13,0	1,38	1,59	-15,22	-8,90
13	92	460	22,0	13,0	2,21	1,59	28,05	-8,16
14	90	450	22,0	14,1	1,63	1,59	2,45	-7,43
15	93	465	36,9	12,4	1,58	1,59	-0,63	-3,92
16	94	470	36,9	12,4	2,17	1,59	26,73	-3,25
17	94	470	47,3	11,5	1,46	1,59	-8,90	-0,63
18	93,6	468	47,3	11,5	1,28	1,59	-24,22	2,45
19	94,6	473	47,3	11,5	1,81	1,59	12,16	4,79
20	95	475	74,4	10,6	1,15	1,59	-38,26	10,17
21	96	480	74,4	10,6	1,81	1,59	12,16	12,16
22	95	475	74,4	11,3	1,31	1,59	-21,37	12,16
23	96	480	91,5	12,0	1,90	1,59	16,32	16,32
24	94,8	474	91,5	16,4	2,06	1,59	22,82	19,70
25	94	470	91,5	16,4	1,54	1,59	-3,25	20,50
26	89,6	448	21,5	14,1	1,53	1,59	-3,92	21,29
27	91	455	34,2	13,9	1,27	1,59	-25,20	22,82
28	92	460	40,3	13,5	1,31	1,59	-21,37	26,73
29	92,4	462	40,3	14,5	1,67	1,59	4,79	28,05

Table 4. Pearson criterion calculation table.

No. of interval	**Boarders of Intervals**			**Empirical frequency** n_i	**Mean interval value**	$t = \frac{x_i - \overline{x_i}}{\sigma}$	$\varphi(t)$	**Theoretical frequency** n_i'	$\chi^2 = \frac{(n_i - n_i')^2}{n_i}$
1	-44	;	-33	3	-38,5	-1,69	0,0957	1,5	0,75
2	-33	;	-22	4	-27,5	-1,15	0,2059	3,22	0,15
3	-22	;	-11	3	-16,5	-0,62	0,3291	5,15	1,54
4	-11	;	0	7	-5,5	-0,08	0,3976	6,22	0,09
5	0	;	11	3	5,5	-0,47	0,3571	5,59	2,23
6	11	;	22	6	16,5	1,01	0,2396	3,64	0,92
7	22	;	33	3	27,5	1,55	0,1200	1,88	0,42
								Σ	6,1

Applying the normal density function of the standard normal distribution law

$$\varphi(t) = \frac{1}{\sqrt{2\pi}} \cdot e^{-\frac{t^2}{2}} \tag{11}$$

where; $t = \frac{x_i - \overline{x_i}}{\sigma}$ the values given in **Table 2** are calculated.

According to Pearson's tables of values, for the number of degrees of freedom equal to 4 and the significance level of 0.05, we determine $\chi^2 = 9.5$, which exceeds the found sum of 6.1.

Therefore, it appears that the discrepancies between the empirical and theoretical frequencies are random. This means that the obtained value of the Ca-Na ion exchange coefficient on bentonite (ascanite) clay describes the ongoing technological process with more than 95% accuracy.

4. Conclusion

The above studies of the sorption processes of bentonite (ascanite) clay of Georgia showed that it can be used as an ion exchanger. The total exchange capacity of clay was established, which was 500 g-eq/m^3 (wet cation exchanger) (100 meq/100g of clay). The coefficient of exchange of Ca-Na ions on clay was experimentally determined, which amounted to 1.59 m^3/m^3 (wet cation exchanger/water). To check the confidence of the obtained value of the ion exchange coefficient, the Pearson criterion was used, which showed that the obtained value describes the ongoing technological process with more than 95% reliability. When calculating the Pearson criterion, a previously developed new method for determining the number and length of grouping intervals necessary for constructing a histogram was used[27-28].

Author contributions

Conceptualization, ZM; data curation, ID and ND; formal analysis, ID, TJ and LB; investigation, LB and DCh; project administration, ZM and ND; resources, ZM, ND and ID; supervision, ID; writing — original draft, ID and DCh; writing — review and editing, ID, ZM and TJ. All authors have read and agreed to publish version of the manuscript

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Conflict of interest

The authors declare no conflict of interest.

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