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Precipitation of Aluminium Hydroxide by Hydrogen Peroxide from Sodium Aluminate Solutions

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Abstract

In this study, the conditions for the precipitation of aluminium hydroxide from sodium aluminate solutions obtained from the alkaline solution of raw alunite ore by hydrogen peroxide, an eco-friendly reagent, are studied. Factors influencing the precipitation by H_2O_2 and aluminium hydroxide from diluted aluminate solutions (concentrations of hydrogen peroxide and alkali, volume ratios of hydrogen peroxide and aluminate solution, temperature, time, caustic modulus) are investigated and found under optimal conditions (initial concentration of hydrogen peroxide >15 %; temperature 20–40 °C, $V_{H_2O_2}/V_{sol} = (5-20) : 20$ mL, alkali concentration 42.33 g/L (according to Na_2O)) precipitation of ~95 % of aluminium in aluminate solution is achieved. The recovery of alkali (sodium hydroxide) after the precipitation of aluminium hydroxide by H_2O_2 , its return to the leaching of alunite has been studied, and several difficulties have been eliminated in the precipitation of aluminium hydroxide by other methods. The characteristics of the obtained samples are confirmed by means of XRD, SEM/EDS analysis.

Keywords: aluminium hydroxide, alkali, caustic modulus, hydrogen peroxide, green reagent

INTRODUCTION

Bauxite currently accounts for 90 % of the aluminium produced. The rapid development of aluminium industry has pushed the processing of other aluminium-bearing ores, especially alunite and nepheline, to the forefront due to the inability to sustain the development of this field in the future because of the depletion of bauxite reserves in nature. Alunite ore (Zaglik deposit) in Azerbaijan is a potentially suitable raw material for the production of clay and soil. One of the alkaline products of bauxite, nepheline and alunite processing is sodium aluminate solution [1–5].

Depending on the composition of the ore used, its processing sequence, the depth of desilication of aluminate solutions, the degree of purity of the obtained products, there are several methods of separation of aluminium hydroxide from alumi-

nate solutions: decomposition, CO_2 neutralization (thermal neutralization and carbonation), acidification, etc. The long time required to obtain an aluminium-containing sediment by the Bayer process, the low sedimentation effect, and the high mass of seed additives prompted researchers to look for other alternative methods [6].

The decomposition process of aluminate solutions is carried out at low temperatures (50–70 °C), at low values of the caustic modulus ($K_a = 1.6-1.7$) with an alkali concentration of 130–170 g/L. Due to adjusting the equilibrium of hydrolysis to the right, small crystals of active $Al(OH)_3$ (as seed) are added to the solution, and the solution is stored for 2 days (50 h) for the ageing of $Al(OH)_3$ (gibbsite) crystals. One of the main problems in the production of clay-soil (alumina) is the low rate of precipitation and the use of large amounts of seed in the process [7].

To obtain aluminium hydroxide in the form of highly dispersed pseudoboehmite, the researchers [8] neutralized the sodium aluminate solution with nitric and hydrochloric acids. It should be noted that neutralization leads to the loss of aluminium and alkali. Alkali recovery during the deposition of aluminium hydroxide is one of the important parameters from both an environmental and economic points of view.

It is preferable to add active additives from sodium aluminate solutions to grow aluminium hydroxide crystals, increase productivity and gibbsite deposition rate [9]. Zhang *et al.* [10] carried out the precipitation of gibbsite from sodium aluminate solutions using methanol as an additive. They found that methanol, which creates a stable hydrogen bond with water in the aluminium hydrate phase, increases the sediment mass by 70 % in 24 h. Distillation of aluminate solutions and recycling of alcohol complicates the application of this method. The intensification of the process is summarized in studies conducted in 3 areas: activation of particles, the addition of surfactants, ultrasonic and magnetic field activation [11]. The high cost and complexity of the proposed methods limit their industrial application. Alkaline recovery by these methods and techniques is carried out in several stages, with reagent and energy consumption. Neutralization of aluminate solutions by acid streams leads to the loss of alkali and aluminium, doubling the cost of the process.

The synthesis of boehmite from strong alkaline aluminate solutions has been studied at room temperature in the presence of both CO_2 and H_2O_2 [12]. The high alkalinity of the medium leads to the deposition of 50 % of aluminium from the solution, and the presence of caustic modulus 3.3 stops the formation of sediment.

Researchers at the Korea Institute of Rare Metals [13] obtained a 99 % yield of aluminium hydroxide from synthetic aluminate solutions, the NaOH leaching solution of mechanically activated black dross, using H_2O_2 (30 %) as a precipitating reagent.

The authors of [14] studied the precipitation of aluminium hydroxide in the presence of hydrazine and hydrogen peroxide, also compared these precipitators which one is more effective for precipitating aluminium hydroxide.

Researchers from Egypt developed a new method for obtaining crystalline boehmite from a synthetic low concentrated alkaline solution of sodium aluminate by adding an aqueous solution of hydrogen peroxide [15]. But none of these schol-

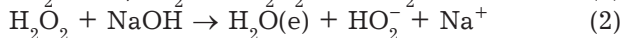
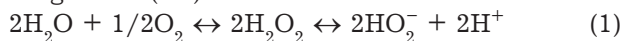
ars have used a real aluminate solution obtained by leaching the alunite ore.

At the same time, the precipitation of aluminium hydroxide by H_2O_2 from diluted solutions obtained from alunite has not yet been investigated. The crystallization of aluminium hydroxide during the precipitation by H_2O_2 is also poorly studied. These data are scattered and in some cases contradictory, which is most likely due to the conditions of precipitation, the properties of the selected precipitator, or raw material.

Hydrogen peroxide is one of the most important inorganic reagents and products of chemical industry and has some advantages over other precipitators due to its eco-friendliness. Because of its eco-friendliness, ease of use, low cost, the absence of impurities in the composition and during processing, instantaneous deposition, low process temperature, the possibility of alkali recovery at the end of the process, precipitation of aluminium hydroxide by H_2O_2 favourably differs from other methods. The use of hydrogen peroxide (as a green oxidizer) as a relatively affordable raw material ensures the high yield of aluminium hydroxide.

The decomposition of H_2O_2 into free radicals in the liquid phase occurs at room temperature. Hydrogen peroxide decomposes into water and oxygen to form intermediate active centres ($\text{OH}^\bullet$ and $\text{HO}_2^\bullet$ radicals) in the system, and these active centres selectively cause different-purpose reactions [16].

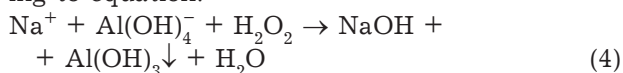
As mentioned above, when the alkali concentration in aluminate solutions is 1–6 mol/L, the aluminium ion is in full coordination with the hydroxide ion to form $\text{Al}(\text{OH})_4^-$ ion. To precipitate aluminium, it is necessary to separate the hydroxide ion (OH^-) from the $\text{Al}(\text{OH})_4^-$ ion, and this can occur as a result of the separation of the water molecule by the condensation reaction. Therefore, the solution containing the aluminate ion must be neutralized with the H^+ ion. When H_2O_2 is added to a sodium hydroxide solution, H_2O_2 decomposes into hydroperoxide (HO_2^-) and hydrogen ion (H^+):



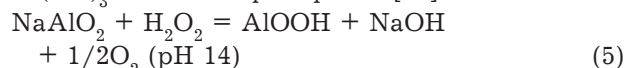
On the other hand, hydroperoxide ions can be disproportionately decomposed into hydroxide ions (OH^-) and oxygen gas by reaction:



The H^+ ion interacts with a solution of sodium aluminate to form an $\text{Al}(\text{OH})_3$ precipitate according to equation:



While H_2O_2 is stable in neutral or acidic medium, it is unstable in alkaline solutions. Depending on the concentration of alkali, the H^+ ion interacts with the aluminate ion to form either $\text{Al}(\text{OH})_3$ or AlOOH precipitate [13].



Unlike other precipitators, when aluminium in aluminate solution is precipitated by H_2O_2 , it is possible to ensure the recovery of alkali (equations (4) and (5)).

In the presented article, the precipitation of aluminium hydroxide by H_2O_2 from aluminate solutions obtained from the alkaline leaching of alunite, the recovery of alkali (sodium hydroxide) and its recycling to the leaching stage of alunite ore has been studied, several difficulties encountered during the precipitation of aluminium hydroxide by other methods have been eliminated.

EXPERIMENTAL

Materials

Aluminate solution obtained from alkaline leaching of alunite ore (Zaglik deposit in Azerbaijan). Alunite ($\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 2\text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$) ore is an alkaline aluminosilicate ore consisting of the following main components, mass %: Al_2O_3 18.2–27.11; Fe_2O_3 2.53–5.95; SiO_2 44–56; $\text{Na}_2\text{O}-\text{K}_2\text{O}$ 2.58–4.9; CaO 0.78–4.60; SO_3 5.4–19.0; V_2O_5 0.028–0.057; Ga_2O_3 0.001–0.0044.

Hydrogen peroxide 30 %.

Alkali NaOH (GOST 4328-77).

0.1 M hydrochloric acid (GOST 3118-77).

Methods of analysis

X-ray phase analysis (XRD) of the samples was performed using Bruker AC200 (Germany).

Scanning electron microscopy and energy-dispersive X-ray spectroscopy (SEM/EDS) analysis were carried out using Jeol JSM6610LV (Japan).

pH and temperature control were monitored by the HI2211 Hanna Instruments automatic regulator and thermometer (Romania).

The concentration of aluminium in the filtrate was determined by the photocalorimetric method (FEC-3) and calculated using the equation of precipitation of aluminium:

$$\varepsilon = \frac{C_0 - C_1}{C_0} \cdot 100 \% \quad (6)$$

where C_0 and C_1 are the concentrations of aluminium in the aluminate solution before and af-

ter the experiment, respectively. The concentration of sodium oxide in sodium aluminate solution was determined by titration with 0.1 M HCl .

Aluminium hydroxide precipitation device

In the laboratory, the precipitation of aluminium hydroxide was carried out by continuous stirring in a 400 cm^3 container with alkali (with concentration, calculated for Na_2O , 14–50.37 g/L), pH ~13 and temperature control (in the range of 20–80 °C); the ratio of the H_2O_2 volume to volume of aluminate solution ($\bar{V}_{\text{H}_2\text{O}_2}/V_{\text{sol}}$) is equal 2–20 (Fig. 1).

The addition of H_2O_2 as a liquid precipitator to the reaction zone (aluminate solution) has been carried out by pouring slowly for a certain time. Different concentrations of H_2O_2 have been added to the aluminate solution by the drip method.

The precipitate was separated from the parent solution in a Büchner funnel and washed with hot distilled water until there was no sulphate ion in the filtrate (washing was controlled by checking with BaCl_2 solution), the wet sediment was dried at 60 °C for 24 h. Precipitation of aluminium hydroxide from solutions with a pH of more than 13 and an alkali concentration of more than 10 % was observed with the intensive release of O_2 gas.

Preliminary experiments had firstly been carried out on synthetic aluminate solutions ob-

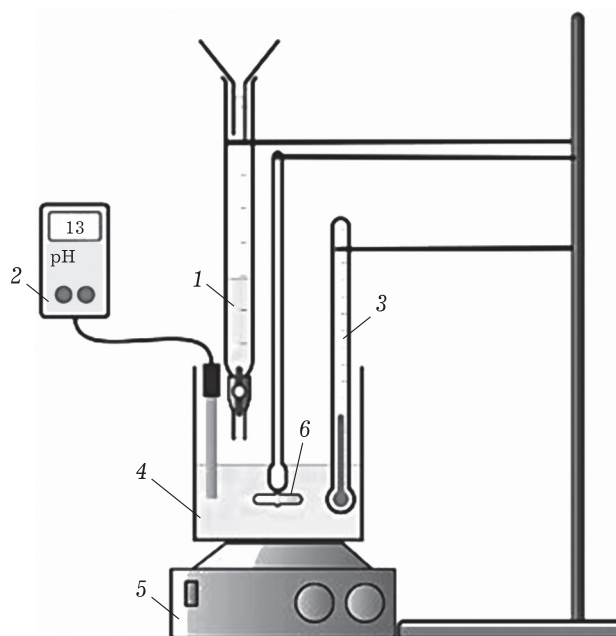


Fig. 1. Device for aluminium hydroxide precipitation: 1 – precipitating solution (H_2O_2); 2 – pH meter; 3 – thermometer; 4 – container with aluminate solution; 5 – heater; 6 – mechanical stirrer.

tained by dissolving $\text{Al}(\text{OH})_3$ in NaOH solution at 90°C , and the aluminate solutions were diluted with water to obtain the required consistency. The caustic modulus of the sodium aluminate solution (ratio of the molar amount of sodium oxide ($C_{\text{Na}_2\text{O}}$) to the molar amount of alumina ($C_{\text{Al}_2\text{O}_3}$), $K_\alpha = (C_{\text{Na}_2\text{O}}/C_{\text{Al}_2\text{O}_3}) \cdot 1.645$) had been taken as 1.45–1.65. The results of these experiments were tested in solutions obtained from the leaching of alunite in 5 % NaOH (42 g/L for Na_2O): 40.53 g/L Na_2O ; 40.8 g/L Al_2O_3 ; 1.55 g/L K_2O ; 1 g/L SO_2 ; 0.39 g/L SiO_2 ; $K_\alpha = (40.53/40.80) \cdot 1.645 = 1.634$.

RESULTS AND DISCUSSION

Two factors have been followed during precipitating aluminium hydroxide from sodium aluminate solutions:

- 1) The rate of deposition of aluminium hydroxide.
- 2) The concentration of alkali after precipitating the aluminium phase at the moment of equilibrium.

Factors affecting the precipitation of aluminium hydroxide

The factors affecting the precipitation of aluminium hydroxide by H_2O_2 from aluminate solutions have been investigated: the concentration of H_2O_2 and alkali, amount of H_2O_2 ($V_{\text{H}_2\text{O}_2}$), reaction time, temperature, the ratio of the volume of H_2O_2 to volume of aluminate solution ($V_{\text{H}_2\text{O}_2}/V_{\text{sol}}$), caustic modulus of solution (K_α).

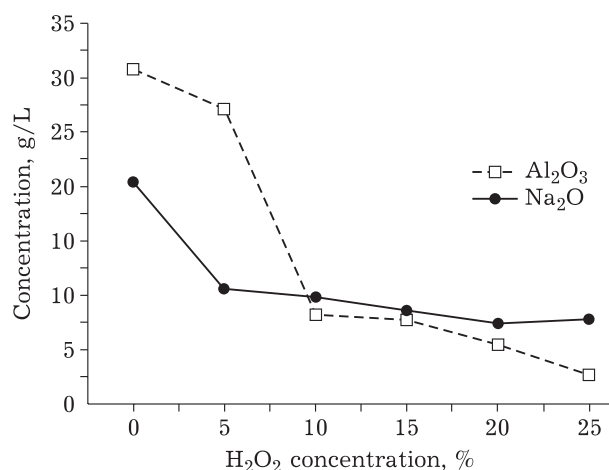


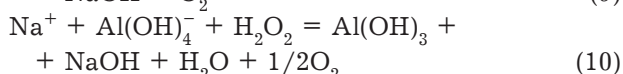
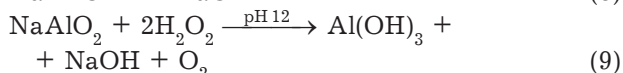
Fig. 2. The effect of concentration of H_2O_2 on the concentrations of Na_2O and Al_2O_3 ($T = 20^\circ\text{C}$, $C_{\text{Na}_2\text{O}}^{\text{initial}} = 20.47$ g/L, $C_{\text{Al}_2\text{O}_3}^{\text{initial}} = 30.76$ g/L, $V_{\text{H}_2\text{O}_2}/V_{\text{sol}} = 20 : 20$ mL, mixing speed – 300 rpm).

The effect of hydrogen peroxide concentration on the precipitation of aluminium hydroxide and the concentration of Na_2O in solution

The results of the studies of H_2O_2 concentration effect on the precipitation of aluminium hydroxide and the change in the concentration of Na_2O are given in Fig. 2.

The experiments were performed with 5 % NaOH at room temperature (20.47 g/L Na_2O) with $V_{\text{H}_2\text{O}_2}/V_{\text{sol}} = 20 : 20$ mL, varying the concentration of H_2O_2 between 5–25 %. As can be seen from Fig. 2, the initial concentrations of Al_2O_3 and Na_2O decrease rapidly until the concentration of H_2O_2 is 10 %.

As the concentration of H_2O_2 increases, the number of active centres (radicals) in the system increases, at low concentration of hydrogen peroxide, the reaction of H_2O_2 decomposition in liquid solutions is directed to the formation of H^+ ions (reaction (1), right), and H^+ ions cause the precipitation of aluminium hydroxide by breaking down the OH^- ions in the $[\text{Al}(\text{OH})_4]^-$ complex (reaction (7)).



At the high concentration of hydrogen peroxide (more than 10 %), the number of OH^- ions increases, the reaction of H_2O_2 decomposition is directed to the formation of O_2 , as a result of obtaining alkali, the value of the caustic modulus ($\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$) increases. Thus, the aluminium hydroxide precipitate begins to dissolve in the alkali solution again, and the amount of sediment decreases.

In this case, the concentration of Al_2O_3 decreases from 30.76 to 8.21 g/L, and the concentration of Na_2O decreases from 20.47 to 12.81 g/L. It is estimated that 73.26 % of aluminium precipitates with 10 % H_2O_2 . Increasing the concentration of H_2O_2 to 25 % leads to a decrease in the concentration of Al_2O_3 to 2.66 g/L. In this case, 91.34 % of the aluminium in the solution precipitates, and the concentration of Na_2O is reduced by 2 times compared to the initial concentration. It shows that while part of the alkali is spent on the precipitation of aluminium, part is spent on the recovery of alkali.

Changes in the concentration of Na_2O and Al_2O_3 depending on changes in the volume ratios of H_2O_2 (30 %) and sodium aluminate solutions

One of the most important factors in monitoring changes in Na_2O and Al_2O_3 concentrations is finding the dose of H_2O_2 added to the solution. In this research, the ratio of H_2O_2 volume to the solution volume was changed from 0.1 to 1.0, and changes in the concentrations of Na_2O and Al_2O_3 in the solution and the values of the caustic modulus were observed under the influence of these factors. It is shown in Fig. 3.

As can be seen from the results, the precipitation of Al_2O_3 or the decrease in the concentration of Al_2O_3 and Na_2O in the aluminate solution also depends on the amount of H_2O_2 added. When the volume ratio of hydrogen peroxide and aluminate solution is 1 : 4, the precipitation of Al_2O_3 begins rapidly, and as the volume ratio increases, the rate of deposition raises. When this ratio is 1 : 2, Al_2O_3 precipitates by 69.85 %, and when it is 1 : 1, it peaks at 95 %. The change in ratios increases the concentration of Na_2O from 48.42 g/L (5.84 %) to 23.675 g/L (3.05 %). Since a sharp increase in the caustic modulus ($\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratio) at a ratio of 1 : 1 ($K_a = 16.819$) leads to a decrease in Al_2O_3 precipitation, it is recommended to conduct the precipitation at a value of K_a 3.0 ($V_{\text{H}_2\text{O}_2}/V_{\text{sol}} \sim 10 : 20$).

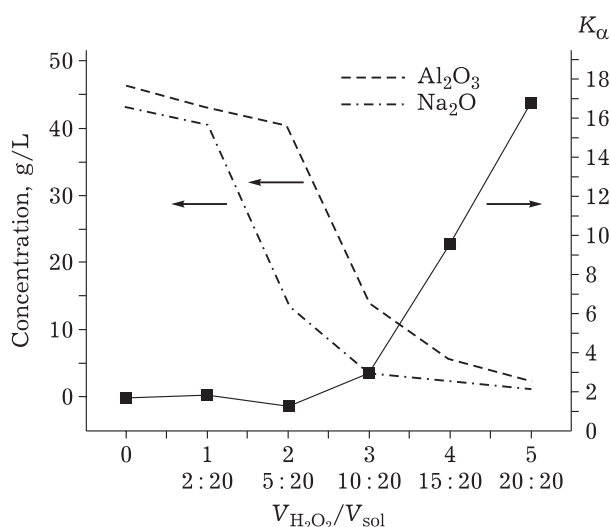


Fig. 3. Effect of changes in the volume ratio of H_2O_2 and sodium aluminate solution ($V_{\text{H}_2\text{O}_2}/V_{\text{sol}}$) on changes in the concentrations of Na_2O and Al_2O_3 ($T = 20^\circ\text{C}$, $C_{\text{Na}_2\text{O}}^{\text{initial}} = 48.42$ g/L, $C_{\text{Al}_2\text{O}_3}^{\text{initial}} = 46.42$ g/L, $K_a = 1.71$, $C_{\text{H}_2\text{O}_2} = 30\%$ (9.81 g/L); mixing speed – 300 rpm).

The effect of temperature and time on the precipitation of aluminium hydroxide by H_2O_2 from sodium aluminate solutions

The temperature and time dependence of the precipitation of aluminium hydroxide from sodium aluminate solutions is shown in Fig. 4. It can be seen that the intensive deposition of $\text{Al}(\text{OH})_3$ occurs in the first minutes and reaches a maximum in 30 min for 4 studied temperatures. A comparison of temperatures showed that the best results were observed at room temperature ($\sim 20^\circ\text{C}$) and 40°C . At 40°C , 74.5 % of aluminium in solution precipitates in 30 min and 90.1 % in 3 h. Further increase in temperature (60 and 80°C) reduces the percentage of precipitation of aluminium from the solution. The reason for this decrease is the thermal decomposition of H_2O_2 as the temperature increases, and the rapid reaction with alkali in the aluminate solution. Another reason is that reaction (5) is exothermic. It is known that the increase in temperature in exothermic reactions directs the process towards the endothermic reaction.

As shown in Fig. 4, at a H_2O_2 concentration of 15 %, the reaction time in the ratio $V_{\text{H}_2\text{O}_2}/V_{\text{sol}} = 15 : 20$ is not the main factor for the precipitation of aluminium.

The main focus of the study was to determine how the concentration of NaOH (Na_2O) changes during the return to the circuit after precipitation. The results of studies on the effect of the initial concentration of alkali on the precipitation of aluminium from sodium aluminate solutions by H_2O_2 are given in Table 1 and Fig. 5.

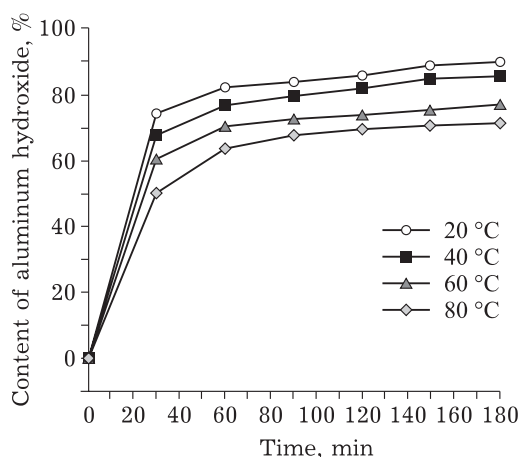


Fig. 4. Effect of time on precipitation rate of aluminium hydroxide at the different temperatures ($C_{\text{Na}_2\text{O}}^{\text{initial}} = 40.53$ g/L, $C_{\text{Al}_2\text{O}_3}^{\text{initial}} = 40.8$ g/L, $C_{\text{H}_2\text{O}_2} = 15\%$, $K_a = 1.634$, $V_{\text{H}_2\text{O}_2}/V_{\text{sol}} = 15 : 20$; mixing speed – 300 rpm).

TABLE 1

Precipitation of aluminium from sodium aluminate solutions by H_2O_2 in different concentrations of alkali ($C_{\text{H}_2\text{O}_2} = 10 \%$)

Parameter	Experiment					
	1	2	3	4	5	6
Concentration:						
NaOH initial, g/L (%)	18.07 (1.78)	36.97 (3.56)	41.54 (4.00)	54.62 (5.20)	60.23 (5.80)	65.00 (6.50)
NaOH equilibrium, g/L (%)	14.90 (1.47)	26.87 (2.61)	31.65 (3.07)	48.88 (4.60)	47.82 (4.55)	58.80 (5.60)
Na_2O initial, g/L	14.00	28.65	32.27	42.33	46.67	50.37
Na_2O equilibrium, g/L	11.54	20.82	24.53	37.88	37.00	45.60
Al_2O_3 initial, g/L	14.64	15.89	16.25	16.96	15.87	16.96
Al_2O_3 precipitated, g/L	12.00	14.14	14.78	16.11	14.11	13.96
Al_2O_3 in solution, g/L	2.64	1.74	1.46	0.84	1.76	3.00
Precipitation rate, %	82.00	89.00	91.00	95.00	88.90	82.30
Caustic modulus	7.20	19.60	27.58	73.48	34.54	9.30

As can be seen, 82 % of aluminium in the solution precipitates when the initial concentration of alkali (according to Na_2O) is 14 g/L, and the subsequent concentration of alkali at 42.33 g/L ensures that up to 95 % of aluminium hydroxide in the solution precipitates. An increase in the subsequent concentration of alkali to 50.37 % reduces the precipitation rate to 82.3 % as it causes the precipitated aluminium in the solution to dissolve.

The concentration of aluminium in the solution decreases from 14.64 to 2.64 g/L when the concentration of alkali is 11.54 g/L. An interesting fact is that the caustic modulus of the solution reaches 73.48 g/L when the alkali concentration is 42.33 g/L, *i. e.* 95 % of aluminium in it precipitates, although the initial concentration of NaOH decreases from 5.2 to 4.6 %. After the sedi-

ment is filtered and washed, the alkaline solution can be sent back to the leaching of a new batch of alunite.

The aluminium hydroxide precipitate is separated from the solution by vacuum filtration and washed several times by hot distilled water. The washing process is carried out until obtaining neutral colour of litmus paper. The precipitate is dried at 100 °C for 24 h.

Figure 6 presents the results of the X-ray phase analysis of $\text{Al}(\text{OH})_3$ precipitated at room temperature (a) and the precipitate obtained at 80 °C for 48 h (b) ($C_{\text{H}_2\text{O}_2} = 10 \%$; $V_{\text{H}_2\text{O}_2}/V_{\text{sol}} = 10 : 20$; alkali concentration 42.33 g/L (according to Na_2O)). X-ray diffractograms show that while aluminium hydroxide precipitated at room temperature is an amorphous structure, as a result of low crystallization rate, small peaks of bayerite are observed in the sediment at 80 °C along with 2 precise peaks of gibbsite.

The morphology of the sediment is clarified in Fig. 7 based on the results of the SEM/EDS analysis. The results of EDS analysis of the sediment obtained at 80 °C show that the material is composed of aluminium hydroxide particles (aluminium – 52.70 %, oxygen – 46.98 %). In addition, the sample powders contain traces of impurities such as Na – 0.31 %.

Scanning electron microscopy image of the sample of $\text{Al}(\text{OH})_3$ precipitated by hydrogen peroxide and aged at 80 °C shows that the precipitate is composed of agglomerates of hexagonal plates. It is also known from the literature that gibbsite from aluminate solutions crystallizes in the form of agglomerates consisting of hexagonal plates.

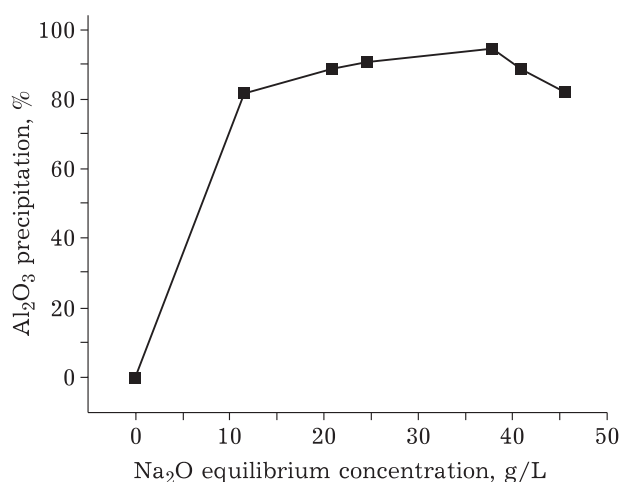


Fig. 5. Effect of concentration of Na_2O on Al_2O_3 precipitation rate ($C_{\text{initial}} = 14.64\text{--}16.96$ g/L, $V_{\text{H}_2\text{O}_2}/V_{\text{sol}} = 1 : 2.8$; room temperature 21 °C, $C_{\text{H}_2\text{O}_2} = 10 \%$).

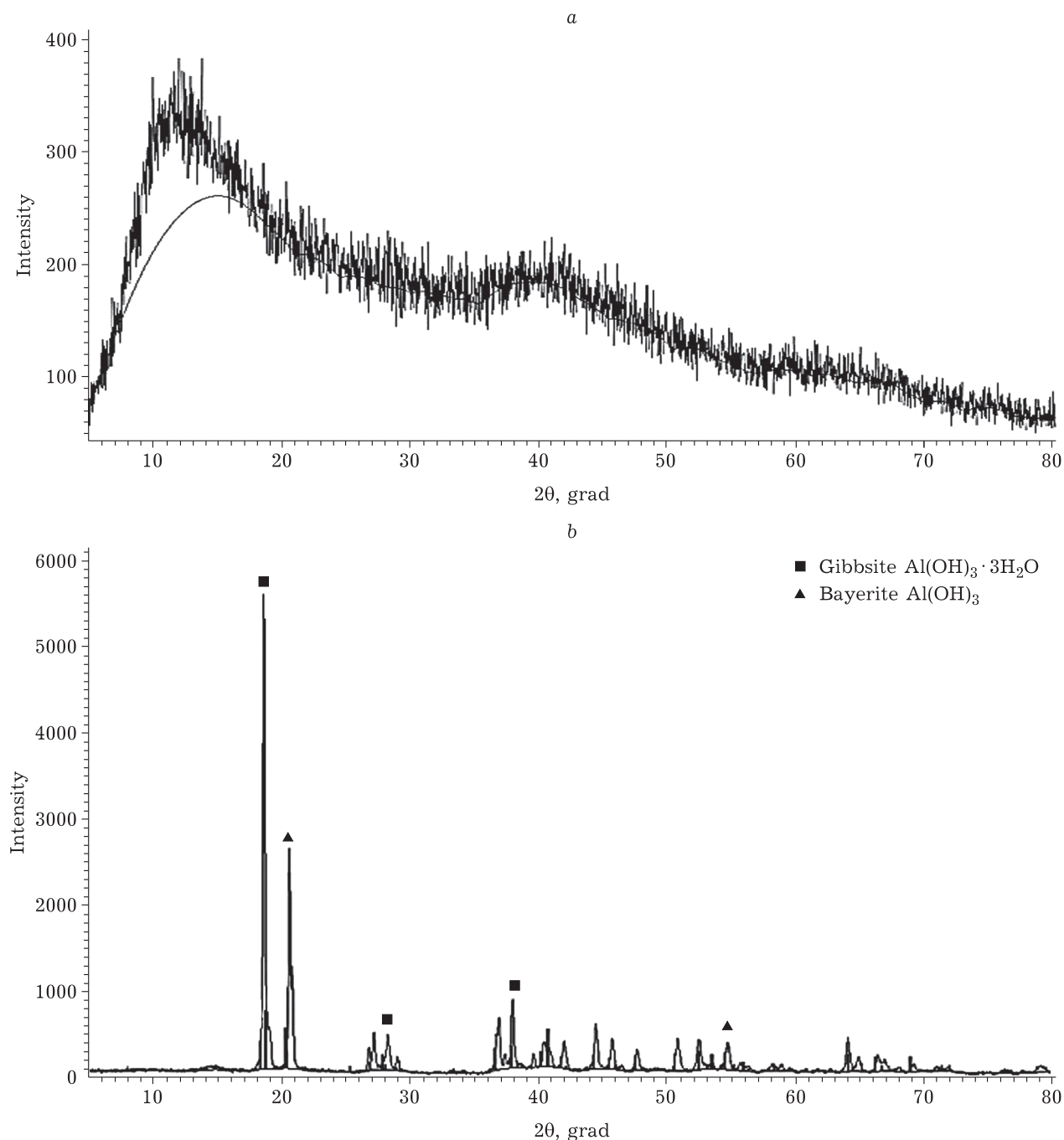


Fig. 6. XRD analysis of a sample obtained from sodium aluminate solutions at room temperature (*a* – amorphous aluminium hydroxide) and the precipitate obtained at 80 °C for 48 h (*b* – gibbsite and bayerite phases).

CONCLUSION

Based on conducted experiments, under optimal conditions (initial concentration of hydrogen peroxide >10 %, temperature 20–40 °C, $V_{\text{H}_2\text{O}_2}/V_{\text{sol}} = 10 : 20$, alkali concentration 42.33 g/L (according to Na_2O)) precipitation of ~95 % of aluminium in aluminate solution is achieved.

The results of the X-ray phase analysis show that the precipitate obtained from exper-

iments with hydrogen peroxide at room temperature is amorphous aluminium hydroxide, the sample obtained at 80 °C for 48 h is a mixture of gibbsite and bayerite phases. According to electron microphotography (SEM) analysis, this sediment consists of small hexagonal plate agglomerates.

In addition, the concentration of the alkaline solution (sodium hydroxide) obtained after filtration of aluminium precipitate is approximately

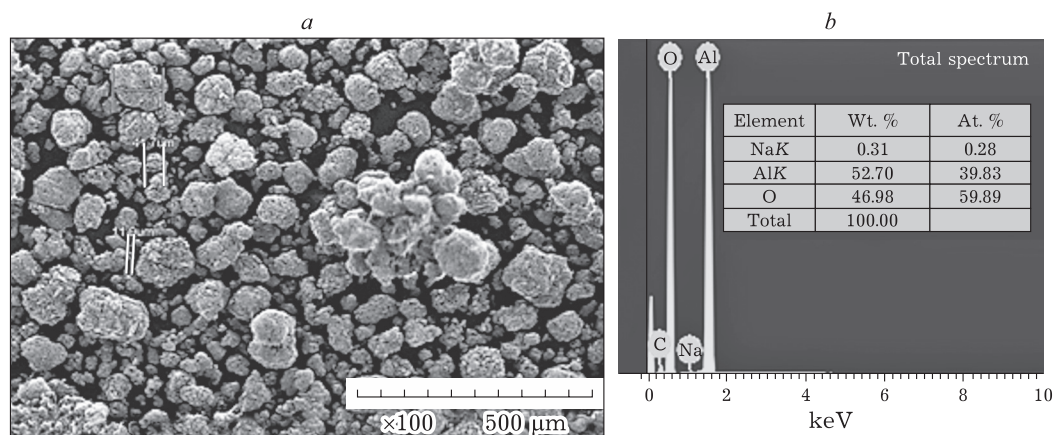


Fig. 7. SEM image (a) and EDS analysis (b) of the sample of $\text{Al}(\text{OH})_3$ (gibbsite and bayerite phases).

2–3 %, thus it allows its recycling to the leaching stage of alunite ore after thickening.

As a result, reducing the consumption of alkali used in the processing of alunite, the absence of additional by-products proves the suitability of the proposed method.

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