



Technology Of Hydrochloric Acid Processing Of Aluminosilicates

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Article History	Abstract
Received: 12 September 2023 Revised: 17 December 2023 Accepted: 26 December 2023	<i>In order to develop a technology for obtaining a coagulant - aluminum sulfate from Gavasai clay, the decomposition of aluminosilicate clay, previously moistened with concentrated hydrochloric acid, was studied, and a technological scheme for obtaining the product was developed.</i>
CC License CC-BY-NC-SA 4.0	Keywords: Aluminum Silicates, Kaolin, Bentonite, Alumina, Alumina, Coagulant, Aluminum Sulphate, Iron (III), Water Treatment, Paper Manufacture, Hydrochloric Acid Processing Of Aluminosilicates.

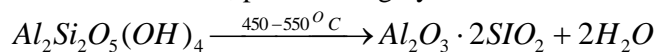
1. Introduction

Aluminosilicates, i.e. kaolinite, montmorillonite clays, alunites and other minerals are very valuable raw materials in the production of refractories, ceramics and are used to produce paper, aluminum sulfate, alumina and other chemicals [1,2,3]. By acid, in particular sulfuric acid treatment of aluminosilicates, aluminum sulfate is obtained from them, which is used as a coagulant in water treatment, paper and other enterprises [4,5,6]. For such a process, there were fewer combinations of iron compounds (especially Fe⁺²) in the composition; with a low content of compounds, the main component is required - aluminosilicates rich in aluminum oxide [7]. On alumina, which contains many iron compounds, as well as those components that are considered important for other manufacturing industries (for example, potassium compounds are the most necessary mineral fertilizer for agriculture), it is important to process poor aluminosilicates and turn them into effective products [8].

2. Methods and Materials

The presence of aluminosilicates, the composition and properties of which are different in the conditions of the territory of the Namangan region, their properties, reserves and possibilities of use have been studied in detail and continue to be studied. The work on the transformation of such gomashes into productive products for the national economy through processing is one of the important tasks facing chemists and chemists-technologists [9,10]. The purpose of this study is the acid processing of Gavasai montmorillonite into target

products [11,12,13]. For this purpose, we studied hydrochloric acid processing of montmorillonite from the Gavasay deposit, composition (wt.%): $Al_2O_3 = 19,56$; $SiO_2 = 61,35$; $Fe_2O_3 = 2,70$; $FeO = 1,87$; $K_2O = 4,21$; $MgO = 1,46$; $TiO_2 = 1,29$; $CaO = 0,02$ and etc. After grinding natural rocks, they were subjected to heat treatment at a temperature of 400-800°C. At the same time, kaolinite, which is contained in the composition of montmorillonite, passes to highly active metakaolinite:



This compound under the action of acids passes into the corresponding salts.

Table 1 shows that, unlike kaolin, during heat treatment, up to 4,24% of volatile compounds are released into the gas phase (in kaolin over 8%, in rich rocks they reach up to 10-12%). This means that the main amount of SiO_2 in the composition of Gavasai clays is in the free form. So this silica can be isolated by hydroenrichment and enrich the raw materials with useful components. After heat treatment of montmorillonite for 60 minutes, it was treated with hydrochloric acid at a concentration of 25%, and sulfuric acid at a concentration of 30,91% was used to regenerate the anion exchanger to extract aluminum on aluminum sulfate. Acid processing was subjected to calcined at a temperature of 600°C for 1 hour, the composition (wt.%): $Al_2O_3 = 20,41$; $SiO_2 = 64,01$; $Fe_2O_3 = 2,82$; $FeO = 1,95$; $K_2O = 4,38$; $MgO = 1,41$; $TiO_2 = 1,34$; $CaO = 0,02$.

Table 1 Influence of technological indicators of heat treatment of mineral clays Gavasai on the chemical composition

Firing temperature, °C	God time, minutes	Mass loss		Chemical composition, mass, %						
		г	%	Al_2O_3	Fe_2O_3	FeO	SiO_2	K_2O	TiO_2	MgO
400	60	2,18	2,18	20,00	2,76	1,91	62,71	4,30	1,31	1,39
400	120	2,64	2,64	20,09	2,77	1,92	63,01	4,32	1,32	1,40
500	60	3,36	3,36	20,24	2,79	1,94	63,48	4,36	1,34	1,41
500	120	3,70	3,70	20,31	2,80	1,94	63,71	4,38	1,34	1,41
600	60	4,16	4,16	20,41	2,82	1,95	64,01	4,38	1,34	1,41
700	60	4,24	4,24	20,43	2,82	1,95	64,07	4,40	1,35	1,42
800	60	4,24	4,24	20,43	2,82	1,95	64,07	4,40	1,35	1,42

The main indicators of technological parameters are: the influence of the stoichiometric norm of hydrochloric acid on the content of components (Al_2O_3 , Fe_2O_3 , FeO, K_2O and MgO) in the raw material and the acid concentration (i.e. L:T) on the yield of Al_2O_3 and Fe_2O_3 . During hydrochloric acid processing of clays, hydrochloric acid salts of the corresponding components contained in the composition of clays are formed. The process of decomposition of calcined montmorillonite with hydrochloric acid was carried out under thermostatically controlled conditions at a temperature of 80°C for one hour in a three-necked flask, one of which was equipped with a thermometer, and the other with a reflux condenser. An agitator connected to an electric motor is installed in the main throat. The concentration of hydrochloric acid was varied in the range of 10-25%, the stoichiometric norm was taken as 90-105% in relation to the masses of the components (Al_2O_3 , Fe_2O_3 , FeO, K_2O and MgO) in the raw material. Acid is first poured into the flask, and then clay is dosed. After the decomposition process, the suspension was filtered, and the contents of the components in the filtrate were determined (Table 2). Based on the obtained data, the yields of Al_2O_3 and Fe_2O_3 were calculated.

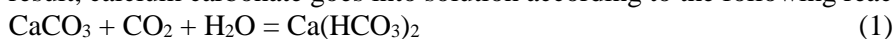
3. Results And Discussion

The results showed that at a stoichiometric rate of acid of 100% and its concentration of 10% (L:T = 5,81:1), 47,5% Al_2O_3 and 42,2% Fe_2O_3 pass into the solution. At this rate and an increase in the acid concentration to 25% (L:T = 2,32:1), the yield of Al_2O_3 increases to 83,0% and Fe_2O_3 to 59,3%. Reducing the rate of acid to 90% and concentration to 20% leads to a decrease in the yield of Al_2O_3 to 72,9% and Fe_2O_3 to 40,5%. Increasing the acid rate to 105% at the same concentration leads to an increase in the yield of Al_2O_3 up to 83,9% and Fe_2O_3 up to 57,8%. These results are close to those obtained using 25% acid at a rate of 100% of stoichiometry. In the case of using an acid rate of 105% of stoichiometry, excess acidity must be neutralized.

Table 2 Influence of technological parameters of hydrochloric acid processing of local (Gavasay, Namangan region) aluminosilicate clays on the chemical composition of intermediate solutions and product yield

Acid rates, %	Hydrochloric acid concentration, %	W:T	The composition of the filtrate, wt. %						Product yield, %	
			Al ₂ O ₃	Fe ₂ O ₃	FeO	K ₂ O	TiO ₂	MgO	By Al ₂ O ₃	By Fe ₂ O ₃
1	2	3	4	5	6	7	8	9	10	11
100	10	5,81:1	1,67	0,20	0,06	0,04	-	0,05	47,5	42,2
100	15	3,87:1	3,79	0,36	0,09	0,06	-	0,08	71,8	49,2
100	20	2,90:1	5,79	0,54	0,13	0,09	0,01	0,10	82,3	55,1
100	25	2,32:1	7,30	0,72	0,17	0,11	0,01	0,12	83,0	59,3
90	20	2,61:1	5,70	0,44	0,14	-	-	0,11	72,9	40,5
105	20	3,05:1	5,61	0,53	0,13	0,08	-	0,09	83,9	57,8

Therefore, the optimal rate of hydrochloric acid can be considered 100% and a concentration of 20-25%. In this case, the yield of the main components is 82.3-83.0% for Al₂O₃ and 55.1-59.3% for Fe₂O₃. After filtering the suspension, the sludge was washed with water, and the filtrate was returned to dilute the original acid. Sludge can be used to obtain building materials. To replace the chloride compounds contained in the filtrate with sulfate salts, the filtrate was passed through an anion exchange resin with sulfate anions. Neutralization of excess acidity and precipitation of iron compounds was carried out by introducing into the solution, after ion-exchange purification, calcium carbonate to a pH 4.5-5.0 of. In this case, 90-95% of iron and 4-6% of aluminum precipitate. During neutralization, the solution was saturated with carbon dioxide (CO₂). As a result, calcium carbonate goes into solution according to the following reaction:



Iron is precipitated with calcium bicarbonate by the following reaction:



The iron contained in the sludge can be recovered by acid processing, or the sludge can be used as a flux in the iron and steel industry.

Increasing the pH of the solution above 5.0 leads to an increase in the degree of aluminum precipitation into the solid phase, and when the pH of the medium is below 4.0, the degree of iron precipitation into the solid phase decreases and the product, aluminum sulfate, is contaminated with iron compounds.

By evaporation of the resulting solution, high-quality aluminum sulfate was obtained, composition (wt.%): Al₂O₃ = 14,4; Fe₂O₃ = 0,04; FeO = 0,02; H₂SO₄ (free) = 0,07; But. = 0,1, etc. In addition to the pulp and paper industry, you can use crystallized aluminum sulfate with an admixture of iron (III) sulphate, obtained without precipitation of iron.

The basic technological scheme and the material balance of the process of hydrochloric acid processing of aluminosilicates are shown in Fig.1.

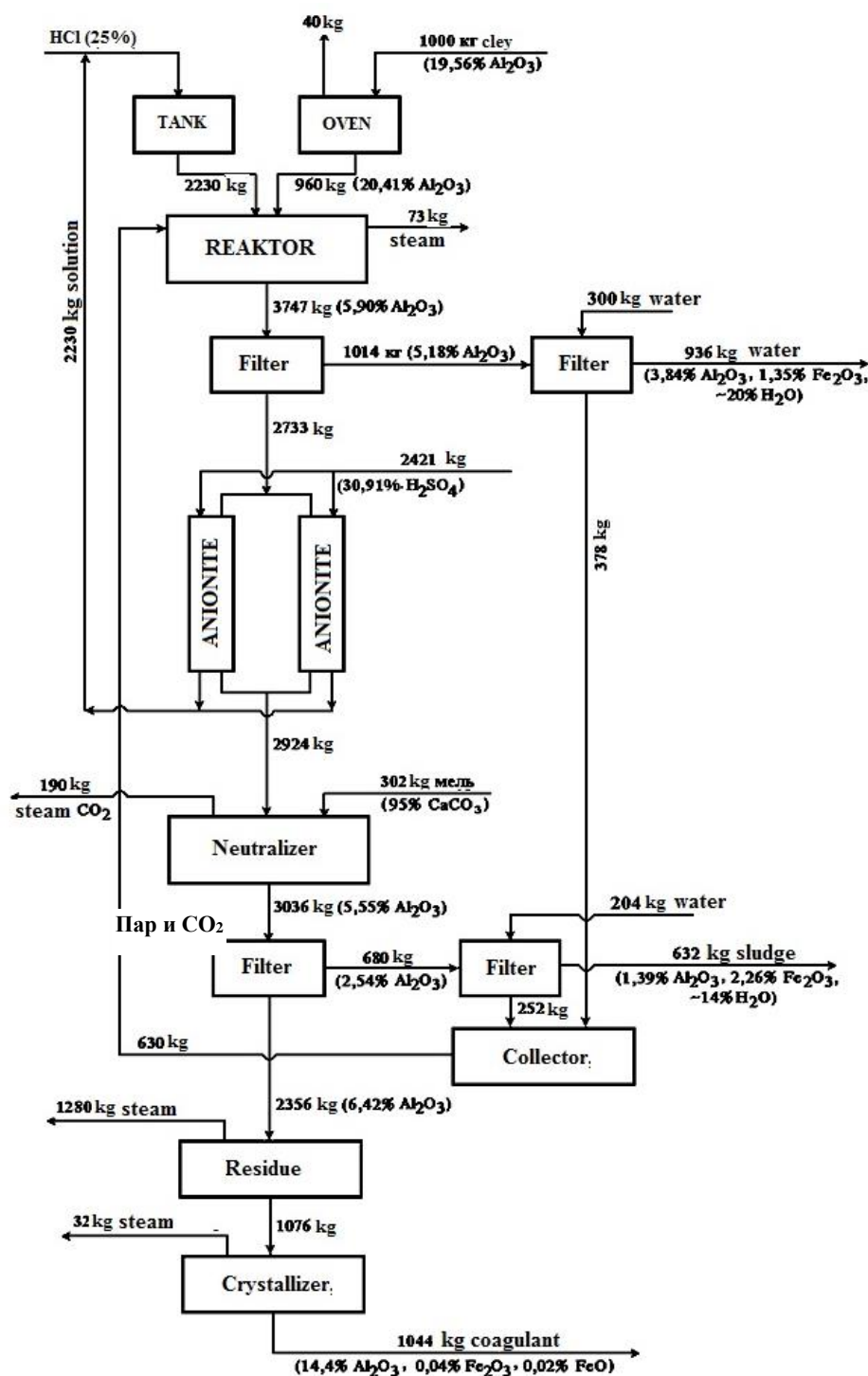


Fig.1. Principal technological scheme and material balance of the process of hydrochloric acid processing of Gavasai aluminosilicates.

To obtain 1044 kg of coagulant, 1000 kg of crushed (particle size 2-4 microns) Gavasay mineral clay (19,56% Al_2O_3) enters the furnace (preferably an electric furnace) and is fired at a temperature of 600 ° C for 1 hour. About 40 kg of the volatile fraction is released into the gas phase. 960 kg (20,41% Al_2O_3) of fired clay is obtained which is fed into a reactor which is pre-filled with 2230 kg of hydrochloric acid at a concentration of 25%. It also receives 630 kg of circulating solution after washing the sludge to establish the ratio L:T = 2.90:1. The concentration of hydrochloric acid in the liquid phase is 20%. In the reactor, the process proceeds at a temperature of 80°C for 1 hour with continuous stirring. The 73 kg gas-vapor mixture released (vapors of water and hydrochloric acid) is sent to the absorber and the purified phase is released into the atmosphere.

The resulting 3747 kg suspension (5,90% Al_2O_3) is sent to a filter device to separate the liquid (2733 kg) and solid (1014 кг, 5,18% Al_2O_3) phases. The solid phase is washed with 300 kg of water and the washed (930 кг, 3,84% Al_2O_3 , 1,35% Fe_2O_3 and 20% H_2O) sludge is thrown into the dump. The resulting filtrate is sent through the collector to the cycle. The solution obtained by filtration is sent to an ion exchange column SO_4^- form, where chloride ions are replaced by SO_4^- ions. A solution containing sulfate salts of aluminum, iron and other cations is sent to the neutralizer, neutralized with calcium carbonate (chalk containing 95% CaCO_3) and saturated with carbon dioxide. In this case, 190 kg of the gas-vapor mixture is released into the gas phase. The suspension is fed to a filter to separate the liquid (2356 kg, 6,42% Al_2O_3) and solid (680 kg, 2,54% Al_2O_3) phases. The solid phase is washed off with 204 kg of water, and the resulting sludge (632 kg, 1,39% Al_2O_3 , 2,26% Fe_2O_3 , and about 14% H_2O) is thrown into the dump. The filtrate is fed through the collector to the cycle. The main filtrate in the amount of 2356 kg (6.42% Al_2O_3) is fed to the evaporation and evaporated to a state of melt (1076 kg). In this case, 1280 kg of water is released into the gas phase. The melt is crystallized on a drum crystallizer (32 kg of water evaporates) to obtain 1044 kg of a coagulant product containing 14.4% Al_2O_3 , 0.04% Fe_2O_3 , 0.02% FeO , etc.

The anion exchanger is regenerated with sulfuric acid at a concentration of 30.91%, and the regenerate containing hydrochloric acid is used in the process of raw material decomposition.

4. Conclusion

Thus, the optimal conditions for the hydrochloric acid processing of aluminosilicates with a relatively low content of Al_2O_3 and a high content of iron compounds have been established. The optimal conditions are: firing temperature 600°C, firing time 60 minutes, hydrochloric acid concentration 25%, acid rate 100% of the stoichiometry (L:T = 2.9), decomposition temperature 80°C, time 60 minutes. As a result, 82% aluminum and 55% iron pass from montmorillonite clays into solution.

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