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The Study of Method for Complex Processing Turgay Sub-Standard Aluminum-Containing Raw Materials

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ABSTRACT

objects of the research are Kazakhstan's Turgay clay, studied of method for alumina and potassium metasilicate obtaining from Turgay sub-standard aluminum raw materials. Concluded that optimal conditions for the process of Turgay clay: reaction temperature 100°C, original solution K_2O concentration to 300 g/dm^3, reaction time 120 min, liquid-solid ratio of 3:1; optimized the conditions of digestion alumina concentrate: original Na_2O solution concentration of 400 g/dm^3, temperature 280°C, molar ratio CaO : SiO_2 = 1. Recovery is 99.6% of alumina digestion under this condition; crystallized solid phase components as $Na_2O \cdot Al_2O_3 \cdot 6H_2O$ sodium hydroaluminate crystals. Extracted of alumina from solution of sodium hydroaluminate.

Keywords: Potassium Hydroxide Solution; Leaching; Clay; Alumina Concentrate; Sodium Aluminate Solution; Digestion; Desilication; Alumina

1. Introduction

With the reduction of bauxite resources, the application of low-grade aluminum-containing raw materials in aluminum production will be the key question. Kazakhstan is rich of low grade bauxite mine (burnt ash, clay), which were the storage of Turgay clay long enough to provide raw materials for alumina production [1]. For low grade aluminum ore, chemical beneficiation processing – alkali leaching to removal some silicon-containing to extraction silicate mineral, and alumina production by using concentrate can be comprehensive utilization of raw materials, solve problems of production effectively. Study on alkaline leaching of high-silicon aluminum ore, treated by sodium hydroxide solution and extraction silicate widely visible [2,3], not seen on the potassium hydroxide reports. Potassium silicate as main ingredient for high-quality potassium fertilizer of chloride-free is widely used in agricultural production. For the purpose of this study to complex processing Turgay clay, potassium hydroxide solution used for the first time of low grade aluminum-containing raw materials dressing process, by planning central composite second order rotatable experiment and hydrochemical test, to find the best reaction condition of extraction from Turgay clay industrial alumina and chlorine-free potash fertilizer processing methods.

2. Experimental

2.1. Optimization Process of Clay ore Dressing

Potassium silicate solution and alumina concentrate can be obtained by baking clay leaching by potassium hydroxide solution, using central composite second order rotatable test [4] to find the optimal leaching conditions.

Chemical composition of Turgay's baking clay samples for experiments is: SiO_2 37%; Al_2O_3 42.6%; Fe_2O_3 13.8 %; CaO 1.3%; Na_2O 0.8%; other 1.5%; A/S=1.15. The experiments and results: using of the central composite of second order rotatable to develop test, factors influencing the leaching effect of three is made for the variable: X1 as the concentration for K_2O in original solution, g/dm^3; X2 for leaching time, min; X3 for liquid-solid ratio, percentage of SiO_2 into solution (y) selected optimization parameter. Experiment conditions and results of Second order rotatable as shown in **Table 1**.

Table 1 Conditions and results of experiments.

N	Conditions, g/dm^3, min			Solution components, g/dm^3, %			
	X_1	X_2	X_3	K_2O	Al_2O_3	SiO_2	SiO_2
1	100	60	2:1	61.1	1.38	98.0	48.21
2	300	60	2:1	211.5	2.46	243.5	79.00
3	100	180	2:1	56.4	1.63	99.5	48.94
4	300	180	2:1	253.8	2.89	228.5	78.40
5	100	60	4:1	61.1	1.13	70.5	69.32
6	300	60	4:1	282.0	2.64	105.0	79.44
7	100	180	4:1	61.1	1.63	72.5	71.29
8	300	180	4:1	282.0	3.13	97.5	82.84
9	31.8	120	3:1	30.25	2.64	26.5	20.93
10	336.4	120	3:1	235.0	1.13	162.5	80.86
11	200	19	3:1	188.0	1.13	95.0	65.02
12	200	221	3:1	188.0	2.64	112.0	76.66
13	200	120	1,31:1	188.0	2.64	208.0	62.26
14	200	120	4,68:1	188.0	0.12	74.0	79.06
15	200	120	3:1	164.5	1.63	114.3	78.23
16	200	120	3:1	188.0	1.13	114.5	78.37
17	200	120	3:1	164.5	1.13	114.0	78.03
18	200	120	3:1	164.5	1.13	115.0	78.71
19	200	120	3:1	164.5	1.63	113.5	77.69
20	200	120	3:1	88.0	1.63	115.3	78.88

According to the planning of matrix and experimental results the regression equation calculated, the equation is following:

$$У= 78.14 +13.38X1 +1.84X2 +5.61X3 –8.36X12 –1.30X22 –1.36X32 – 4.83X13$$

Analysis equation can reach the following conclusion: the factors influencing the silica into solution of potassium hydroxide, the action of the concentration of alkali solution mostly strong. And compared to other factors, influence of reaction time on the translate silicon dioxide into solution is weak. Combined effects of three factors are more complex; factor values from 0 to 1, optimization parameters (y) grows, factor value is higher than the 1, y value decreases. Three factors affect the complexity can be interpreted as, dissolution and precipitation of silicon dioxide in the system are two processes occur simultaneously, the result of precipitation forming insoluble compounds - hydro aluminum silicate of potassium. When baking clay samples contact with potassium hydroxide solution, silicon dioxide dissolves faster than the hydrated aluminum silicate, β – cristobalite (Beta-SiO_2) in quartz and silicon into solution, this solution (potassium silicate) as the main ingredient for fertilizer production of potassium is chloride-free. At the same time mullite ($3Al_2O_3.2SiO_2$) break down into solution, concentration of alumina in solution at lower relative will exist of aluminum-silicon complexes, where the proportion of aluminium in silicon atoms as a unit, as reaction continues, the complexes with alkali solution combining format insoluble hydrated aluminum silicate of potassium. Experiments concluded that the optimal conditions for the process of Turgay clay: reaction temperature 100°C, original solution concentration K_2O 300 g/dm^3, reaction time 120 min, liquid-solid ratio of 3:1.

2.2. Optimization of Process of Digestion Alumina Concentrate in Sodium Aluminum Solution

The process of extraction alumina products from alumina concentrate carried out by hydrochemical methods [5]. Optimal preparation of alumina under leaching condition concentrate chemical composition as follows: SiO_2 10.4%; CaO 0.2%; K_2O 0.2%; Fe_2O_3 9.55%; Al_2O_3 62.8%; other 5.0%; A/S 6.04. Phase compositions are mainly for Mullite and Hematite, with small amounts of hydrated aluminum silicate of potassium, amorphous SiO_2 content low, shows many SiO_2 in clay in the process is dissolved of solution. Concentrate aluminum oxide digestion in sodium aluminate solution optimizated by hydrochemical process. Test conditions: temperature of 200~280˚C, time 60 min, original Na_2O concentration of sodium aluminate solution 330~450 g/dm^3, a_k 30, CaO: SiO_2 = 1.2~2. Affect of three factors of temperature, concentration and calcium oxide on the aluminum recovery shown in **Figure 1-3**.

Figure 1 test results view, increasing temperatures from 200˚C to 280˚C, percentage of Al_2O_3 into sodium aluminate solution accordingly increased from 90.6 % to 97.63% .

Figure 2 test results, when concentration gradually increased to 450 from 330 g/dm^3, the alumina recovery into solution corresponding increased from 89.3% to 98.56%.

As shown in **Figure 3**, when the calcium oxide content increased from molar ratio CaO: SiO_2 = 1 to 2, alumina recovery solution corresponding decreased from 99.6% to 94 %. Studied the best conditions of digestion: original solution Na_2O concentration of 400 g/dm^3, temperature 280˚C, molar ratio CaO : SiO_2 = 1. Recovery is 99.6% of alumina digestion under this condition.

2.3. Optimization of Process for Desilication of Sodium Aluminate Solution

The chemical composition of sodium aluminate solution from processing alumina concentrate by hydro chemistry method as follows: Na_2O 376.7 g/dm^3; Al_2O_3 65.93 g/dm^3; SiO_2 4.22 g/dm^3, α_k 9.4, α_k high value of this solution, is not conducive to decomposition out $Al(OH)_3$ from aluminate solution, therefore, needed to be decrease values α_k before processing of crystallization. This test uses the crystallization from solution of hydrated sodium aluminate, water soluble crystals method gets the α_k ~1,6 of sodium aluminate solution. When crystallization, small amounts of SiO_2 will affect the crystallization in solution, so before the sodium aluminate hydrate, pre-desilication process of sodium aluminate solution. Take into account the effects of concentration on silicon removal efficiency, Na_2O dilute solution concentration from 376.6 g/dm^3 to 200 g/dm^3, other components as Al_2O_3 35 g/dm^3 accordingly, iO_2 2.2 g/dm^3.

Calcium oxide for desilication agent, add amount CaO : SiO_2 to 1.5, 2 and 3 calculated, reaction time is 30 - 180 min, test conducted in the temperature range of 150-250°C, as shown in **Table 2**.

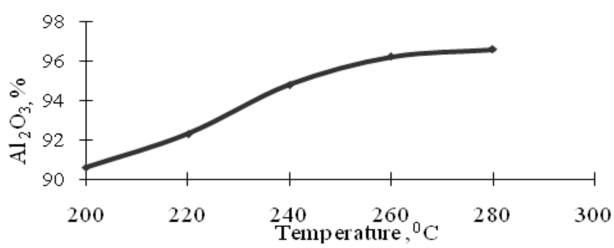

Figure 1. The effect of temperature on the aluminum recovery.

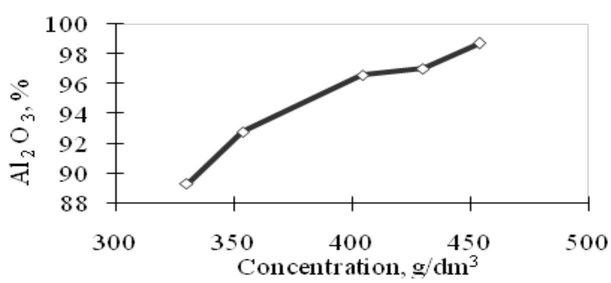

Figure 2. The effect of concentration on the alumina recovery.

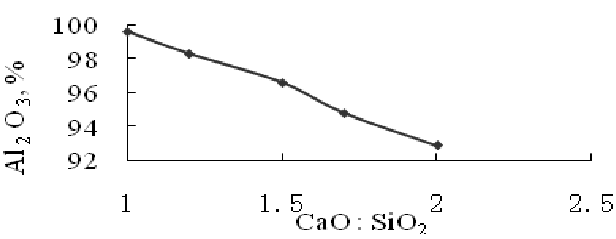

Figure 3. The effect of calcium oxide content on the alumina recov-

ery.

Test results showed highest conditions of sodium aluminate solution desilication rate: reaction temperature 200 °C, CaO added molar ratio CaO : SiO_2 is 3, time for 120 min. Under this condition, sodium aluminate solution desilication rate of 79.5%, solution composition for Na_2O 191 g/dm^3, Al_2O_3 34 g/dm^3, SiO_2 0.594 g/dm^3, α_k 9,2.

2.4. Study on Formation of Alumina from Desilicated Sodium Aluminate Solution

1) Separating out sodium hydroaluminate crystals from desilicated sodium aluminate solution

Consists of sodium aluminate solution crystallization of sodium hydroaluminate - $Na_2O \cdot Al_2O_3 \cdot nH_2O$ test: to evaporation solution concentration of Na_2O for 500-550 g/dm^3, from which separation crystals. Test results are shown in **Table 3**. After enrichment to concentrations of Na_2O 548 g/dm^3, removal of sodium aluminate solution concentration to concentrations of Na_2O after 548 g/dm^3, crystallization temperature of 45°C, under the conditions to quality ratio of 0.05 to join seed crystal, constant stirring. Conditions and results of crystallization shown in **Table 3**.

Table 2. Experimental conditions and results of desilication of sodium aluminate solution.

N	Conditions			Components, g/dm3			Des. rate, %
	t,°C	$CaO:SiO_2$	min	Na_2O	$Al2O_3$	SiO_2	
1	100	3	120	182,4	33,7	1,93	13,4
2	150	3	120	188	33,3	0,6	76,8
3	200	3	120	190,5	34	0,594	79,5
4	250	3	120	196	20,1	0,65	75
5	200	1	120	196	30	0,747	66,7
6	200	1,5	120	190	32	1,00	55,6
7	200	2	120	192	33,5	0,56	75
8	200	3	120	191	34	0,47	79,5
9	200	3	30	196	34	0,725	67,6
10	200	3	60	189	33,3	0,6	77
11	200	3	120	191	34	0,594	79
12	200	3	180	188	34	0,67	69,2

Table 3. Experimental conditions and results of crystallization of sodium hydroaluminate.

T °C	T h	Test conditions, composition, g/dm^3			Test results composition, g/dm^3		
		Al_2O_3	Na_2O	α_k	Al_2O_3	Na_2O	α_κ
45	10	91,89	561,1	10	76,67	550,6	11,8
45	20	91,89	561,1	10	71,44	538,5	12,4
45	30	91,89	561,1	10	31,2	527,3	27,8
45	40	91,89	561,1	10	29,5	502,1	29,0

45	50	91,89	561,1	10	27,33	501,8	30,2

Crystallization rate test results in mixing continuously when 50 h reached the highest value. Chemical composition of separated sodium hydroaluminate crystals: AI_2O_3 25.5%; Na_2O 24.1%; other 34.2%, $\alpha_к$ 1.58 solid phase components as $Na_2O{\cdot}Al_2O_3{\cdot}6H_2O$, sodium hydroaluminate crystal x-ray diffraction curve as shown in **Figure 4**.

2) Extraction of aluminum hydroxide from solution of sodium hydroaluminate

Separate sodium hydroaluminate crystal α_k is 1.58, this water-soluble crystals available α_k value breaks and crystal decomposition conditions of sodium aluminate solution, from which precipitation Al (OH) $_3$ crystal [6]. Test procedures are as follows: dissolving sodium hydroaluminate crystal $Na_2O{\cdot}Al_2O_3{\cdot}6H_2O$, get aqueous chemical composition for, AI_2O_3 100.9 g/dm^3; Na_2O 98.1 g/dm^3; α_k is 1.58, according to the quality of the ratio of 0.3, to join seed – aluminum hydroxide in water solution, temperature of 62ºC - 44ºC conditions, to 70 p/min speed mixing hydrolysis of sodium aluminate solution to 48 h. According to table4 test findings, when 24 h reaction, hydrolysis rate of 41%, when 48 h reaction, hydrolysis rate Increased to 59%.(**Table 4**)

After the complete hydrolysis of sodium aluminate solution, the liquid-solid separation are obtained solid aluminum hydroxide, chemical components for AI_2O_3 61.30%, Na_2O 0.23%, other 35.75%, X ray diffraction curve shows the solid formation is divided into size 20-50 µm of gibbsite, as shown in **Figure 5**.

3) preparation of alumina from aluminum hydroxide

From solid aluminum hydroxide under the condition of temperature of 1050˚C, calcined 1h we are meeting the criteria of production alumina solid products. Analysis and identification of aluminum oxide crystals derived from chemical composition as: AI_2O_3 98.5%, Na_2O 0.78%, SiO_2 0.02%, Ti+V+Gr+Mn 0.01%, ZnO 0.01%, P_2O_5 0.002%, Fe_2O_3 0.026%, weight reduction is 1.2%. X-ray diffraction analysis and its solid groups are divided into: δ Al_2O_3, χ · Al_2O_3,Al_2O_3, θ Al_2O_3, κ Al_2O_3, beta-Al_2O_3, as shown in **Figure 6**.

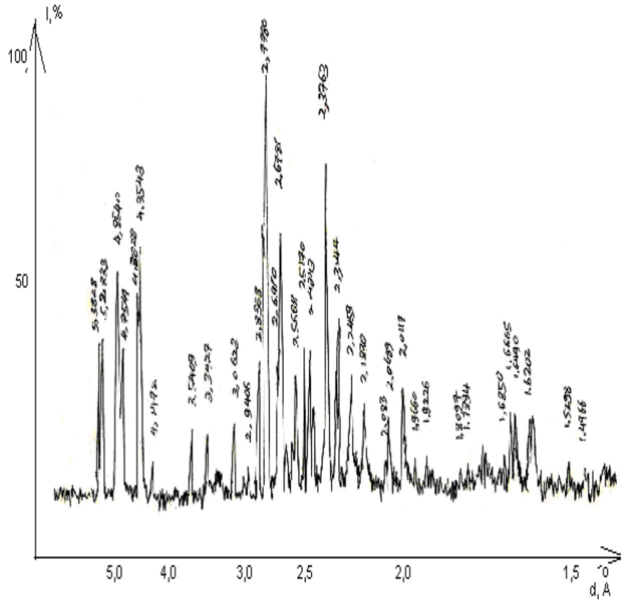

Figure 4. Sodium hydroaluminate crystal x-ray diffraction curve.

Table 4. Hydrolysis of sodium aluminate solution test results.

Seed content	Time, h	Composition, g/dm^3		αк	Hydrolysis rate, %
		Na_2O	AI_2O_3		
0.3	24	100.3	61.5	2.75	41
0.3	48	105.7	42.7	4.1	59

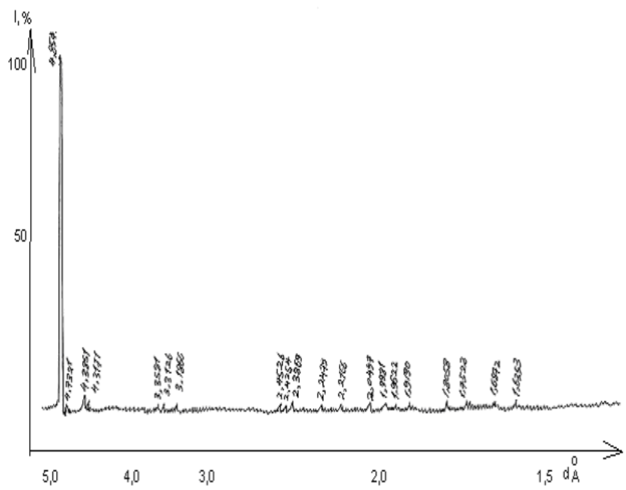

Figure5. Aluminium hydroxide crystals by x-ray diffraction curve.

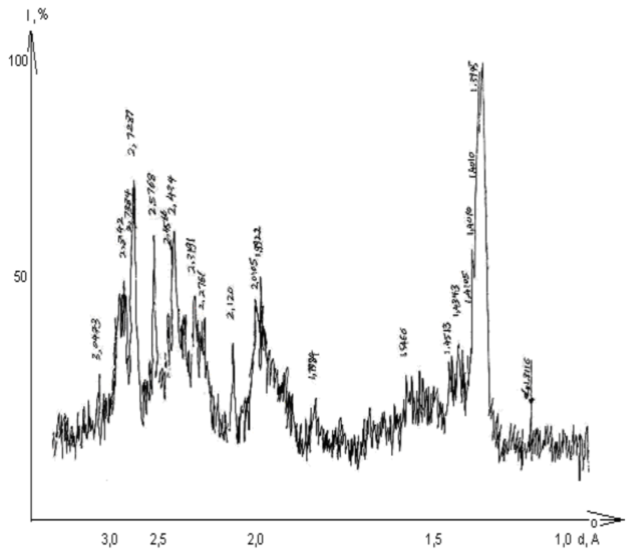

Figure 6 aluminum oxide crystal by x-ray diffraction curve.

3. Summary

By research: summary out KOH solution processing Turgay clay process and sodium aluminate solution digested out alumina concentrate process best reaction conditions; Optimization conditions of dissolving sodium aluminate solution desilication; crystalline solid phase components as $Na_2O{\cdot}Al_2O_3{\cdot}6H_2O$ sodium hydroaluminate crystals; preparation meeting the criteria of production alumina solid products; study for the first time out complex alkaline processing method of clay application of KOH solution.

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