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Development of the High-purity Aluminium Oxide Powder Producing Technology on the Ural Region Raw Materials Basis

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Abstract

This article considers an effective way of complex alumina-containing materials of natural or technogenic origin processing with obtaining a whole complex of raw materials: alkali-free highly active aluminum hydroxide, iron hydroxide, aluminum hydroxide, etc. In laboratory conditions the alumina raw materials behavior in the thickening, washing, filtration processes were checked. The optimal parameters of the processes were selected as a result of conducted research.

Keywords: non-conventional alumina raw materials, technogenic materials, hydrochemical processing, ammonium hydrosulfate, environmental friendliness, efficiency.

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In modern conditions, the implementation of the concept of "innovation for economic development - IFED" is increasingly associated with the problems of complex waste-free natural resources processing and the involvement of multi-tonnage industrial waste in environmentally friendly, waste-free innovative technology.

The amount of industrial wastes for the last hundred years grows exponentially. In the world more than 25 billion tons of solid industrial waste is formed annually. Almost one third of this amount - more than 7 billion tons - is accounted for Russia. As of the beginning of 2013, more than 90 billion tons of production and consumption waste have been accumulated on the territory of the Russian Federation. The area occupied by organized waste disposal sites amounted to more than 400 thousand hectares.

One of the promising areas of the innovation process is the complete industrial waste processing within the regional economic complexes framework. It includes extraction of scarce materials (pure oxides, noble, nonferrous, rare, radioactive and other elements) from industrial wastes and creation of structural and functional materials with high operational properties instead of natural, traditional materials and metals. The implementation of this strategy will allow to significantly reduce the consumption of primary natural

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resources by more than 25%, as well as to solve the issues of raw material security of the country, including the materials critical for the ceramic electronic, electrical and metallurgical industries - high alumina and magnesium.

This work is devoted to important technological issues of technogenic and natural alumina raw materials complex use for the purpose of valuable components extraction, while solving environmental problems.

The authors have tested the technology of complex processing of industrial and/or natural aluminosilicate materials production wastes with the use of recoverable leaching sulphuric acid solution - ammonium hydrosulfate solution. The technology includes:

1) Sulphuric acid leaching of raw materials with ammonium hydrosulphate solution with extraction of iron III and aluminum into the solution by the following reactions:

$2\ AlOOH + 6NH_4HSO_4 = Al_2(SO_4)_3 + 3(NH_4)_2SO_4 + 4H_2O$

$Fe_2O_3 + 6NH_4HSO_4 = Fe_2(SO_4)_3 + 3(NH_4)_2SO_4 + 3H_2O$;

2) Filtration of acid-resistant residue containing RO_2, R_2O_5 oxides for subsequent use as raw materials for the production of building materials, refractories, ceramics, abrasives, etc;

3) Iron III deposition from ammonia solution and separation of iron hydroxide III for further use in the production of pigments;

$Fe_2(SO_4)_3 + 6NH_4OH = 2\ Fe(OH)_3\downarrow + 3(NH_4)_2SO_4$;

4) Aluminium precipitation from ammonia solution and separation of aluminium hydroxide for further use as a raw material;

$Al_2(SO_4)_3 + 6NH_4OH = 2Al(OH)_3\downarrow + 3(NH_4)_2SO_4$;

5) Obtaining a pure solution of ammonium sulfate from which crystalline ammonium sulfate is extracted; according to the technology developed and tested at the pilot plant, it is thermally decomposed into ammonium hydrosulfate and ammonia by reaction:

$(NH_4)_2SO_4 = NH_4HSO_4 + NH_3\uparrow$.

6) Return of the obtained ammonium sulfate and ammonia into production.

The raw materials for the study were selected as kaolin waste from the Zhuravlinny Log deposit (Plast, Chelyabinsk Region) and Poletaevskoye deposit (Chelyabinsk) as raw kaolin. Phase composition of initial materials includes, wt. %: kaolinite $Al_4[Si_4O_{10}](OH)_8$ - 95,0-97,0; quartz SiO_2 - 1,0-2,0 and feldspars (microcline $K,Na(AlSi_3O_8)$) – 1,0 – 3,0.

The offered technology of hydrochemical processing, allowing to improve opening of initial raw materials and to increase the target product yield, consists of:

Preliminary grinding of raw materials up to the full passage through the sieve 0,08 mm;

Preparation of H_2SO_4 technical solution with content: 10, 15 и 20 %;

Pelletizing of ground raw materials with addition of H_2SO_4 solution;

Classification of the obtained granules with the separation of the fraction of 3-7 mm and the return of the remaining fractions to the stage of granulation.

Heat treatment of the obtained granules at a temperature of 350-600 ° C.

Phase composition of granulated products after heat treatment at 350 (and 600) °C is presented, wt. %: metakaolinite $Al_2O_3{\cdot}2SiO_2$ - 85,0-94,0 (14,0-66,0); quartz SiO_2 - 1,0-2,0 (13,0-33,0); hydroslides Na,K,$Al_2[AlSi_3O_{10}](OH)_2$ - 3,0-14,0 (43,0-58,0) and aluminum sulfate 0,0-2,0 (15,0-23,0). It has been established that in the process of heat treatment the materials granulated with 20 % sulphuric acid solution and heat-treated at the temperature of not less than 500 °C are subject to the greatest change; at the same time the products have a latent-crystalline, almost amorphous structure of metakaolinite $Al_2O_3{\cdot}2SiO_2$. The leaching process of the target products is based on the reaction:

$$Al_2O_3\ 2SiO_2+3H_2SO_4=Al_2(SO_4)_3+2SiO_2+H_2O \quad (1)$$

in slow boiling mode (5-7 min) and natural cooling of the solution (50-60 min) with further decantation and washing of the sludge. Wash water is collected and directed to obtaining a leaching solution. The results of the leaching process are presented in Table 1.

Table 1: Leaching process results

№ п/п			1	2	3	4	5	6
Process			Leaching					
Reagents, g	Metakaolinite	10 % H_2SO_4	3	-	3	-		
		15 % H_2SO_4	-	3	-	300	450	500
	25 % H_2SO_4 solution		17,5			1748	1966	2185
Conditions	Boiling point,°C		-			90		
	Boiling point, min		50	90		50	60	90
	Excess solution H_2SO_4, %		10 %					
	Δ Drying Point, °C		207	212	258	212	306	297
	Drying Point, min		40	71	110	71	104	120
Filter method			Decantation					
Mass of settled waters (master batch), g			375,6	308,9	301	1137	1949	3818
Mass of undissolved sediment, g		Practical	1,63	1,73	1,77	481	335	303
		Theoretical	1,62	1,62	1,62	270	270	270
Product yield (sludge), %			109	116	110	178	124	112
Additionally		pH initial/final	0,02/ 0,51	0,25/ 0,63	/0,75	/1,06	/094	/1,39

Based on the results of leaching, it can be concluded that the best way to achieve leaching is to use 15% H_2SO_4 as the leaching agent.

The process of deposition of aluminum sulphate solution purified from iron oxides was performed at the temperature of solution 20 - 60 °C by step-by-step introduction of solution NH4OH in the ratio 1: 2 with pH control of the solution every 5-10 minutes to the final pH of the solution 4,0 - Table 2. After sedimentation the sludge settling, decantation and washing is performed. Drying and thermal treatment of hydroxide was carried out in laboratory furnaces at the temperature of 400 - 550 °C until the mass loss was stopped. The obtained product contains, wt. %: Al_2O_3 - 99,2; R_2O - 0,14; RO - 0,01; Fe_2O_3 - 0,05; SiO_2 - 0,08. The specific surface of the material was 5680 m^2/g, the average particle size was 2.7 μm.

Table 2: Precipitation results

№ п/п		1	2	3	4	5	6	7	8
Reagents,g	$Al_2(SO_4)_3$ solution	588	250	500	500	100			
	NH_4OH solution	-	146	268	278	431	548	915	670
	10% solution of NH_4NaSO_3	520	520	1040	1210	2420	2420	1815	1815
Excess 10% solution of NH_4NaSO_3		1,5			2			1,5	
Boiling point,°C		82						74	
Boiling point, min		120							
Δ Drying Point, °C		127-278	162-347	166-296	204-343	194-289	193-320	201-315	199-321
Drying Point, min		124	235	189	180	199	201	200	198
Filter method		Decantation							
Product		$Al(OH)_3$							
Undissolved sediment mass (master batch), g		-	680	-	-	-	-	-	-
Sludge mass actual, g		14	13	33	41	38	140		
Sludge mass theoretical, g		13	13	26	26	26	52		
Product yield, %		108	100	127	158	146	269		
pH after adding technical ammonia		-	4,15	4	-	-	-	-	3,7
pH master batch		-	5,66	-	-	-	-	4,01	-

1. Conclusions

The possibility of developing a technology based on the raw materials of the Ural deposits using metakaolinite to produce pure aluminium oxide and other products from aluminium sulphate, which will make it possible to create an energy- and resource-efficient technology to produce high-purity Al_2O_3 powders with high level of physical and mechanical properties, was demonstrated.

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