IMPROVEMENT OF PROCESSING CHARACTERISTICS OF HIGH CARBONATE AND HIGH SILICA DIASPORIC BAUXITE BY ENRICHING ROASTING

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Abstract

Behavior of rock forming minerals of Ural bauxite (diaspore, pyrite, carbonates, chamosite) was investigated in a wide range of temperatures depending on different conditions (size, temperature, holding time, atmosphere). Conditions were selected allowing to transfer diasporic material into an easier recoverable form and to reduce significantly the content of detrimental impurities (sulfur, carbonates, organic matter, moisture, ferrous iron).

Investigation was made of process characteristics of roasted bauxite and feasibility is shown of its processing by a simplified Bayer cycle eliminating the necessity of soda, sulfates and other impurities removal from the process which brings about reduction in the cost as compared with processing of raw bauxite at Ural refineries.

Introduction

Alumina production from diasporic bauxites of North Ural Bauxite Mine (SUBR) with high content of carbonates, sulfides, ferrous iron, and organics requires rigid temperature and concentration modes at main process stages (digestion, evaporation), which leads to alkali losses with brown soda and berkeite salt ($Na_2CO_3 \cdot 2Na_2SO_4$). All these result in increased heat consumption, inefficient alkali efficiency in Bayer cycle, etc.

The aim of the work was investigation on an experimental scale of SUBR bauxite roasting allowing 1) to decrease concentration of CO_2 to below 1 mass. %; 2) to convert $S^{2-} \rightarrow SO_4^{2-}$ with partial desulfurization by at least 30 %; 3) to burn out organics, 4) to transform $Fe^{2+} \rightarrow Fe^{3+}$; 5) to decrease moisture content from 7-8 % to near 0 mass %; 6) to convert all boehmite and diaspore alumina into a more active form of Al_2O_3 in terms of dissolution behaviour in digestion compared to initial boehmite and diaspore.

Technological advantage of beneficiation roasting of bauxites with increased content of harmful impurities including sulfur, silica, carbonates, and organics was studied in numerous works [1-3]. As a rule, implementation of this technology is restricted by high heat consumption for roasting comparable to that for alumina production from gibbsitic bauxites by Bayer process. Therefore, conditioning is not widely applied for gibbsitic bauxites.

Another situation is observed at conditioning of diaspore bauxites. In 1950-1970's as a result of numerous studies conducted by VAMI, Urals Politechnical Institute, Kamensk (UAZ) and Bogoslovsk (BAZ) aluminum plants it was established that bauxite crushed to -30 mm can be roasted in stationary fluidized bed furnaces or in rotary kilns at 600-750 ºC within 10-15 min without overcalcining of fine particles [4-5]. In 1976 pilot selection of optimal roasting mode for coarse SUBR material (lump size 2 - 20 mm) in 0.3 t/h fluid bed unit demonstrated that

up to 55 % of sulfur in the form of pyrite (FeS_2) or marcasite (FeS_2) was removed [6]. Carbonates present in the form of calcite ($CaCO_3$) (80%) and siderite ($FeCO_3$) (20%) are removed mainly due to decomposition of siderite, which is thermally dissociated already at ~ 500 °C.

Based on the bauxite roasting of results, the following conclusions were made:

1) The main phase of roasted bauxites is α-Al_2O_3, which is formed from diaspore. The mechanism of thermal decomposition of the main alumina-containing mineral in gibbsite, boehmite and diaspore was studied in depth by Stumpf [7] and confirmed by Brown, Clark and Elliott [8]. As shown by their studies and works of Russian scientists [4 - 6, 9], the principal distinction between diasporic vs gibbsitic and boehmitic bauxites lies in the fact that upon thermal decomposition of gibbsite, bayerite and boehmite, a set of easily digestible alumina oxides is formed (κ, η, θ, γ, etc) while diaspore straight converts into low temperature modification of alpha alumina without the above intermediate phases (fig. 1).
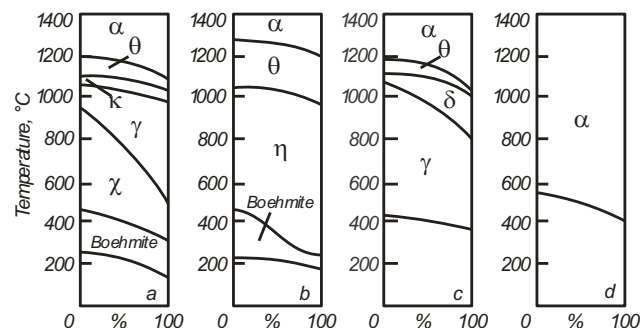

Fig. 1. Temperature fields of stability of transition forms of alumina hydroxides and oxide: a) gibbsite, b) bayerite, c) boehmite, d) diaspore [9]. The percentage of individual phases, depending on temperature is determined by the intersection points of temperature horizontal lines with border curves of existence of adjacent phases.

Low temperature α-modification can be digested at lower temperature and alkali concentration as compared to diaspore. Improvement of digestion conditions is due to the fact that at these roasting temperatures initial phase already ceased to exist and non-equilibrium particles of α-Al_2O_3 are formed that are very fine and porous due to release of water and corundum crystalline lattice is not stabilized, thus this phase is not only better amenable to digestion compared to natural corundum, but also is more reactive at alkaline digestion compared to the initial diaspore [9].

At higher temperature of roasting the crystalline structure becomes more ordered and thus α-Al_2O_3 form becomes more rigid to alkaline digestion having process characteristics close to those of diaspore.

2) Chamosite (an ideal formula: $4FeO-Al_2O_3-3SiO_2-4H_2O$). Upon heating FeO contained in chamosite, is oxidized step-by-step to Fe_2O_3, crystallohydrate moisture is removed in two stages and at ~740 °C olivine-type structure $[(Mg, Fe) SiO_4]$ is formed, which is sufficiently resistant to digestion [10 and 11];

3) Pyrite and marcasite under conditions of moderate oxygen access at 350-500 °C are dissociated by stages into S and FeS. Further, at 650-700 °C, sulfur is oxidized to sulfurous anhydride (SO_3) and removed with exhaust gases, and FeS is oxidized to $FeSO_4$ or $Fe_2(SO_4)_3$, and with partial sulfur removal to Fe_3O_4 or Fe_2O_3.

The selected roasting modes (650-750 °C, holding time 10-15 minutes) did not solve one of the main problems of SUBR bauxite: decrease in carbonate content to the acceptable level of ≤ 1 mass % CO_2.

In our work the following target parameters were selected: bauxite roasting to CO_2 content ≤ 1 mass.% and possibility of significant improvement of digestion parameters in terms of final A/C ratio, digestion temperature or concentration of digesting liquor.

Experimental

Studies on determination of optimal roasting conditions were carried out with two SUBR bauxite samples: HCB grade (high carbonate bauxite) and GB-1 (hydrochemical bauxite) grade (Tables 1 and 2). Digestion was performed with plant spent liquor. Roasting was performed in a laboratory kiln with silicon carbide heaters and non-adjusted environment.

Table 1 - Chemical composition of SUBR bauxite samples

Element	Al_2O_3	SiO_2	Fe_2O_3	TiO_2	FeO	CaO	LOI, incl.	C_{total}	C_{org}	CO_2	S	μ_{si}
HCB	42.2	3.0	18.2	1.8	3.7	12.8	18.6	2.94	0.32	10.1	0.44	14.1
GB-1	50.2	3.3	18.8	1.9	3.7	7.2	15.8	1.75	0.3	6.0	0.68	15.2

HCB bauxite is characterized by very high CO_2 content up to 10.1 mass.%. According to the data of XRD analysis, alumina in the samples is presented mainly by diaspore with minor amount of boehmite, carbonates are in the form of calcite, silica is in the form of chamosite (Table 2).

Table 2 - Mineral composition of SUBR bauxites samples

Bauxite*	Rock-forming and impurity minerals,				
	Diaspore 3.99 A	Boehmite 6.10 A	$CaCO_3$ 3.035 A	Hematite 2.69 A	Chamosite 7.02 A
HCB 1)	375	55	450	160	40
HCB 2)	42.0	6.1	22.9	10.0	10.72
GB-1 1)	750	35	370	240	65
GB-1 2)	54.8	2.71	13.2	10.6	10.72

*Where: 1) peak intensity, imp/sec., 2) mineral content, mass %.

Sulfur-containing mineral exists in amount below the level of method sensitivity (< 1.5 mass %).

In order to determine optimal roasting conditions for SUBR bauxites under which decrease or removal of harmful impurities is provided, the following tests were performed in the following temperature range:

- HCB sample from 500 to 900 °C in increment of 50 degrees, holding time: 0.5 and 1 hour;

- GB-1 sample from 700 to 900 °C in increment of 50 degrees, holding time: 0.5 and 1 hour.

Results and discussion

Based on results of X-ray phase analysis it is possible to conclude that for HCB bauxite at 600 °C diaspore is completely transformed into $\alpha-Al_2O_3$. At 700-750 °C dissociation of $CaCO_3$ starts, and increase in roasting time to 1 h promotes completeness of this process. At 800 °C calcite is decomposed completely (Table 3, figure 2).

For GB-1 bauxite the behavior of minerals contained in bauxite upon roasting is similar to that of minerals contained in HCB (Table 4):

- In HCB bauxite at 750°C and holding time 0.5 h calcite content decreased significantly (~ by 5 times), at holding time 1 h only trace amounts of calcite remained, at 800°C and holding in 30 min calcite was completely thermally dissociated.

- For GB-1 bauxite optimal roasting temperature is 750-800 °C.

Table 3 - Semiquantative XRD analysis of roasted HCB (imp/sec)

Roasting Temp., °C	Time, hour.	Diaspore, 3.99 Å	$\alpha–Al_2O_3$, 2.085 Å	$CaCO_3$, 3.035 Å	CaO, 2.40 Å	Hematite, 2.69 Å	Boehmite, 6.10 Å	Chamosite, 7.02 Å
500	0,5	80	1555	5430	-	950	120	70
600	0,5	traces	1535	3815	-	1000	-	-
700	0,5	-	1440	4190	traces	1095	-	-
700	1	-	1415	1435	690	1180	-	-
750	0,5	-	1390	1440	930	1160		
750	1	-	1315	70	1210	1175	-	-
800	0,5	-	1260	-	1520	1220	-	-
800	1	-	1340	-	1470	1255	-	-
850	0,5	-	1350	-	1230	1290	-	-
850	1	-	1380	-	1625	1265	-	-
900	0,5	-	1370	-	1540	1250	-	-
900	1	-	1420	-	1550	1294	-	-

Table 4 - Semiquantative XRD analysis of roasted GB-1 (imp/sec)

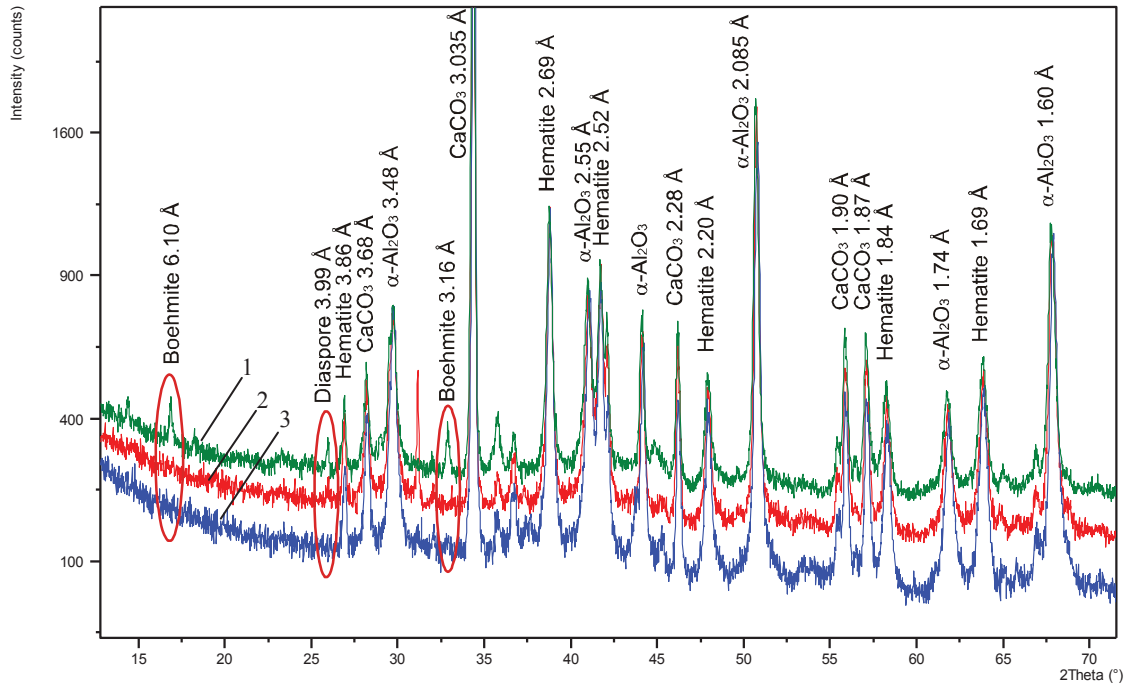| Roasting Temp., °C	Time, hour.	Diaspore, 3.99 Å	$\alpha–Al_2O_3$, 2.085 Å	$CaCO_3$, 3.035 Å	CaO, 2.40 Å	Hematite, 2.69 Å	Boehmite, 6.10 Å	Chamosite, 7.02 Å
700	0.5	-	1965	2126	traces	1380	-	-
700	1	-	1830	890	460	1425	-	-
750	0.5	-	1870	760	370	1425	-	-
750	1	-	1525	180	365	1175	-	-
800	0.5	-	1820	230	555	1500	-	-
800	1	-	1755	-	625	1480	-	-
850	0.5	-	1870	-	770	1540	-	-
850	1	-	1810	-	740	1535	-	-
900	0.5	-	1535	-	620	1300	-	-
900	1	-	1645	-	695	1460	-	-

Fig. 2. XRD patterns of HCB samples roasted at different conditions as per positions no. 1,2,3 at Table 3: 1- 500 C, 30 min, 2- 600 C, 30 min, 3- 700 C, 30 min. Elimination of characteristic peaks of diapore and boehmite as temperature increases and typical alpha alumina phase peaks are visible.

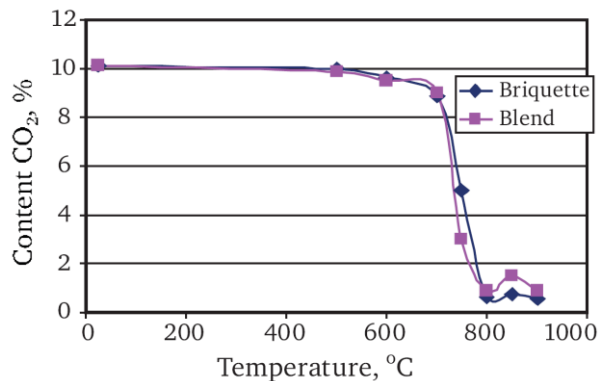Figures 3 - 5 illustrate CO_2 removal from bauxite as a function of temperature and holding time.

Fig. 3. CO_2 removal as a function of temperature at holding within 0.5 h and 1 h at roasting of HCB bauxite
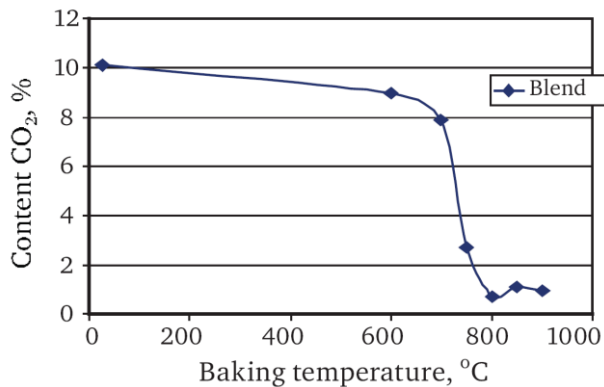*a) Roasting of HCB bauxite, holding time 0.5 h*

Fig. 4. CO_2 removal as a function of temperature at holding within 0.5 h and 1 h at roasting of HCB bauxite
b) roasting of HCB bauxite, holding time 1.0 h

Sulfur in the studied samples of SUBR bauxite is contained in the form of pyrite mineral. It is known that upon heating of pure pyrite two thermal effects are observed in thermogram:

- at 300-560 °C there occurs dissociation into FeS and S, oxidation to Fe_2O_3 or $Fe(SO)_4$, or $Fe_2(SO_4)_3$ depending on sample fineness and air access;

- at 650-670°C the formed compounds are decomposed.

In the course of heating to 500-600 °C oxidation of bivalent sulfur is observed and at further heating sulfur is removed from the process as SO_3.

In case of HCB sulfur was removed by 20%, in case of GB-1 - by 30%. Low percentage of sulfur removal is probably caused by the presence in the samples of active CaO and formation of $CaSO_4$ in the roasted bauxite.

Investigations of digestion of roasted SUBR bauxite were conducted with the aim to determine feasibility of processing within Bayer cycle.
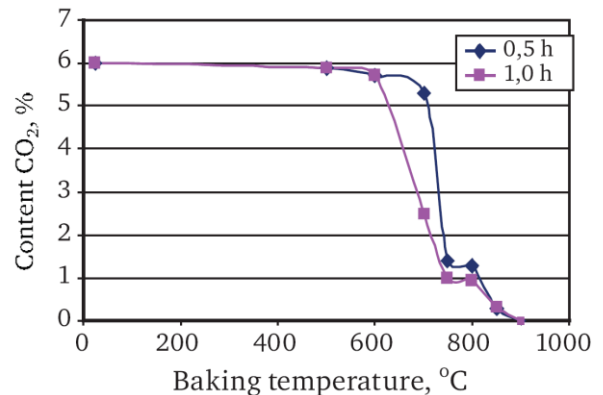

Fig. 5. CO_2 removal as a function of temperature at holding within 0.5 h and 1 h at roasting of GB-1 bauxite

Investigations were conducted relating to the conditions of BAZ: temperature 235 °C, holding time 1.5 h, molar ratio $\alpha_\kappa = 1.6$. Then, possibility of digestion temperature reduction to 225 °C was investigated.

BAZ plant spent liquor was used for digestion, it was diluted considering moisture content of natural bauxite and live steam directed for heating of the following composition, g/dm^3: Na_2O_{total}=253.9; Na_2O_{caust}=235.87; Al_2O_3=136.7, SiO_2 = 0.81.

The results of HCB digestion are presented in Table 5.

Table 5 - Alumina recovery from HCB bauxite

Test #	Bauxite	Condition of digestion		Molar ratio (A/C) of liquid part of digested slurry		Al_2O recovery (from total), %		
		T, °C	Time , h.	Target	Actual	Theoretical	Actual	After desilication
1	SUBR (HCB) not roasted	235	1.5	1.6 (0.601)	1.66 (0.580)	92.89	86.41	86.47
2	SUBR (HCB) 700 °C (0.5 h.)				1.64 (0.587)	93.89	89.1	89.37
3	SUBR (HCB) 800 °C (0.5 h.)				1.66 (0.580)	92.97	87.08	86.83
4	SUBR (HCB) not roasted	225	1.5		1.69 (0.569)	92.89	86.46	86.62
5	SUBR (HCB) 700 °C (0.5 h.)				1.67 (0.576)	93.89	89.26	89.46
6	SUBR (HCB) 800 °C (0.5 h.)				1.65 (0.583)	92.97	86.85	86.45
7	SUBR (HCB) 700 °C (1 h.)	235	1.5		1.60 (0.601)	93.91	87.08	87.39
8	SUBR (HCB) 800 °C (1 h.)				1.66 (0.580)	93.49	85.42	86.48

It should be noted that all muds after digestion of HCB bauxites contain high amount of hydrogarnet due to high content of calcium oxide in initial HCB bauxite and increase in its activity during roasting. As a consequence, A/S ratio of the aluminate liquor after digestion of HCB bauxite roasted at 800 °C is 318, and the overall desilication was performed via calcium hydrogarnet; sodium hydroaluminosilicate in the mud is contained in trace amounts, and sufficiently low sodium to A/S ratio is observed.

In addition, further studies of roasting and subsequent digestion of SUBR bauxite, Bayer grade, with increased content of carbonates were performed.

Laboratory tests were aimed at the following performance:

- roasting aimed at the mode providing removal of at least 90% CO_2 from bauxite;

- digestion aimed at lower temperature to the value, at which alumina extraction decreases by the value not exceeding 10 % of theoretically possible one;

- increase of target A/C ratio (for which bauxite is dosed) to the minimally possible value, at which alumina extraction by the value not exceeding 10 % of theoretically possible one.

On the basis on the samples supplied by Department of Alumina Production technology (BAZ) and the data obtained from SUBR Geology Directorate, a mechanical mixture was prepared consisting of GB-1 and HCB bauxites with the ratio of GB : HCB = 3 : 1 (by mass). Composition of the mixture is presented in Table 6. The obtained mixture approximately corresponds to bauxite mined at Cheremukhovskaya Glubokaya mine without selective separation.

Table 6 - Chemical composition of bauxite mixture GB : HCB = 3 : 1 (by mass)

Chemical composition, mass. %														
SiO_2	Al_2O_3	Fe_2O_3	TiO_2	P_2O_5	CaO	MgO	Na_2O	K_2O	V_2O_5	Cr_2O_3	MnO	SO_3	L.O.I.	Incl.CO_2
3.0	47.0	19.3	1.8	0.5	8.5	0.63	0.2	<0.15	0.02	0.2	0.02	3.3	16.1	7.8

In order to examine the impact of particle sizes the initial sample was comminuted to:

- 100% less than 1000 μm;
- 100%- 600 μm;
- 100% less than 160 μm.

Roasting was carried out in the temperature range from 700 to 850 °C with increment of 50 degrees. The obtained data showed that within a narrow range under conditions of stationary kiln coarsening of particle size does not influence thermal dissociation of calcite ($CaCO_3$). At roasting temperature of 750°C, irrespective of holding time and particle sizes, CO_2 is removed by more than 90 %. Consequently, this temperature

was selected as a basis in the course of preparation of samples for digestion.

At all roasting temperatures dehydration of boehmite and diaspore was accompanied by formation of low temperature modification of α-Al_2O_3.

The whole content of FeS_2 in bauxite under selected roasting modes in a stationary kiln with non-adjusted oxidizing environment is decomposed with formation of Fe_3O_4. Herewith, oxidation $S^{2-} \rightarrow SO_4^{2-}$ takes place together with partial desulfurization due to evolution of gaseous SO_2. Sulfur is removed in amount of ~ 25-31 %. The remained portion of sulfur reacts with CaO with formation of $CaSO_4$.

Digestion was carried out with BAZ spent liquor, which was diluted with consideration for moisture of natural bauxite and live steam directed for heating.

Digestion modes were varied as follows:

- digestion temperature was tested in the range of 220-180°C with increment of 20 degrees;
- target molar ratio was varied from 1.6 to 1.35 with increment of 0.05;
- lime dosage was not done, since after thermal dissociation of calcite intrinsic active lime was at the level of 9.5 mass.%;
- holding time was not varied and was 1.5 h which corresponds to actual exposure of slurry in autoclaves at a refinery at the reaction temperature.

The results obtained in the tests are presented in Table 7.

XRD analysis of mud after digestion of roasted bauxite demonstrated that underextraction of alumna is related to incomplete decomposition of low temperature α-Al_2O_3 and secondary losses of alumina due to formation of significant amounts of hydrogarnet.

The obtained results show that after bauxite roasting at 750 °C low-temperature α-Al_2O_3 is produced from diaspore with properties similar to those of boehmite. This is the background which allow performing the process for lower ratio or at digestion temperature of 200-210 °C.

Estimation of heat consumption for conditioning of 1 t of bauxite for BAZ

The calculations were based on the following initial data:

- Fuel - fuel oil with calorific value of 9600 kcal/kg;
- Air excess factor - 1.5 and 2.0;
- Bauxite moisture content- 7 %;
- CO_2 - 10.1%;
- Fuel oil temperature - 95°C;
- Primary air temperature -20°C;
- Exhaust gas temperature - 200°C;
- Bauxite temperature - 20°C;
- Roasting temperature - 700, 750 and 800°C;
- Heat losses via kiln shell - 5 % of heat input;
- Heat loss due to incomplete fuel burning - 2 % of heat input;
- Air temperature at cooler exit - 200°C.

As can be seen, heat consumption for conditioning of 1 t of HCB bauxite is comparable to heat consumption for calcination of 1 t of alumina.

Table 7 - Results of alumina recovery from the mixture of bauxites

No. of test	Condition of digestion		Molar ratio (A/C) of liquid part of digested slurry		Al_2O recovery (from total), %		
	t, °C	Time, h	Target	Actual	Theoretical	Actual	After desilication
1	220	1.5	1.60 (0.601)	1.64 (0.587)	93.06	88.06	87.38
2			1.55 (0.621)	1.55 (0.621)		88.06	87.48
3			1.50 (0.641)	1.54 (0.625)		87.43	86.82
4			1.45 (0.663)	1.44 (0.668)		87.08	86.40
5			1.40 (0.687)	1.42 (0.677)		86.42	85.69
6			1.35 (0.713)	1.37 (0.702)		85.83	85.09
7	200	1.5	1.60 (0.601)	1.7 (0.566)	93.36	76.06	76.51
8			1.55 (0.621)	1.72 (0.559)		73.56	73.64
9			1.50 (0.641)	1.67 (0.576)		72.41	71.86
10			1.45 (0.663)	1.6 (0.601)		71.42	71.33
11			1.40 (0.687)	1.61 (0.598)		65.11	65.05
12			1.35 (0.713)	1.58 (0.609)		65.60	65.55
13	180	1.5	1.60 (0.601)	2.55 (0.377)	93.62	21.74	21.98
14			1.55 (0.621)	2.43 (0.396)		18.82	19.09
15			1.50 (0.641)	2.53 (0.380)		17.97	18.13
16			1.45 (0.663)	2.43 (0.396)		20.13	20.46
17			1.40 (0.687)	2.37 (0.406)		17.48	17.61
18			1.35 (0.713)	2.19 (0.439)		20.49	20.78

Table 8 - Specific fuel consumption for conditioning of 1 t of HCB bauxite

Air excess factor	Bauxite roasting temperature, °C	Specific fuel consumption		
		kg of conv. fuel /kg	kcal/kg	kJ/kg
Without heat recovery of roasted bauxite				
1.5	700	0.059	413.18	1730.00
	750	0.063	438.31	1835.19
	800	0.065	457.09	1913.84
2.0	700	0.056	390.86	1636.54
	750	0.060	417.09	1746.35
	800	0.062	436.70	1828.46

Air excess factor	Bauxite roasting temperature, °C	Specific fuel consumption		
		kg of conv. fuel /kg	kcal/kg	kJ/kg
With heat recovery of roasted bauxite				
1.5	700	0.036	254.60	1066.00
	750	0.040	279.72	1171.19
	800	0.043	298.51	1249.85
2.0	700	0.024	170.13	712.32
	750	0.028	196.35	822.13
	800	0.031	215.96	904.24

Conclusions

1. Preliminary studies on roasting of high-carbonate SUBR bauxites and processing of roasted bauxites by Bayer process revealed the following advantages as compared to non-roasted bauxite:

   - due to removal of up to 90 % CO_2 caustic alkali losses are reduced by 50-60%;

   - due to desulfurization of bauxite by ~ 30% amount of soda - sulfate mixture decreases by 50-60 % (for processing of 100 % roasted bauxite);

   - purity of aluminate liquor increases due to burning-out of bitumen organic impurities at 550-600 ºC;

   - due to removal of 7-8 % of free and up to 15 % of crystallohydrate moisture amount of evaporated water in Bayer process decreases by 10-12 %;

   - with CaO content in bauxite higher than 8 mass % chemical losses of alkali are reduced by 8-10 kg/t Al_2O_3.

   Among disadvantages is increased bauxite consumption in case of processing of HCB bauxite by Bayer process due to high secondary losses of Al_2O_3 upon formation of hydrogarnets.

2. The study of roasting process demonstrated that up to 400 °C diaspore remains in bauxites, at 500 °C thermal transformations of diaspore are taking place according to diaspore $\rightarrow$ α-Al_2O_3 route; in the temperature range from 600 to 1100 °C alumina is only in the form of α-Al_2O_3.

3. Bauxite decarbonation rate was 70-90 % at 800 °C, CO_2 content in HCB bauxite decreased from 10.1 to 0.88 %, and in GB-1 - from 6.0 to 0.95 %.

4. Digestion of roasted bauxite with regard to Bogoslovsk Alumina Refinery (BAZ) conditions (digestion temperature 235 °C, digestion period 1.5 h) was performed which showed better technological performance compared to uncalcined bauxite: alumina extraction is 97-98 % of theoretically possible value as compared to current BAZ value of 92.5 %, alkali consumption and mud factor are reduced. Lowering of digestion temperature of roasted bauxite to 225 °C demonstrated that there was no decrease of alumina extraction and of A/C ratio of digested slurry.

5. Calculation of heat consumption for conditioning of 1 t of HCB bauxite with regard to BAZ conditions demonstrated that it is comparable to specific heat consumption for calcination of 1 t of hydrate in fluidized bed kilns.

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