Improvement of Digested Slurry Post-Desilication Efficiency in the Flashing Circuit at Nikolaev Alumina Refinery

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Abstract

To ensure continuous production of G-00 grade alumina at Nikolaev alumina refinery (NGZ) alumina/silica mass ratio (μ_{Si} or A/S ratio) in the pregnant liquor shall be above 210. This limitation is associated with specifics of precipitation process at Nikolaev refinery. Many approaches to μ_{Si} control have been tested in the past: pre-desilication of thick raw slurry; addition of calcium containing additives to the digestion process; post-desilication of digested diluted slurry; seeding with fine fraction of bauxite residue rich in desilication product (DSP) into the raw slurry. These approaches have not delivered the required results as they caused hydrolysis, boehmite reversion, reduction in alumina production rate. The paper discusses the results of test simulation of post-desilication of Guinean bauxite digested slurry by addition of process water to the flashing circuit. Process parameters such as dilution conditions, residence time and temperatures have been determined. The test proved that the proposed solution will provide for increase of μ_{Si} value in the pregnant liquor by 15 - 20 units. Heat calculations showed that post-desilication in the flashing circuit at Nikolaev refinery reduces the specific heat consumption. Based on the obtained results the design data were defined for rearrangement of digestion trains at Nikolaev refinery to perform pilot testing.

Keywords: alumina/silica ratio (μ_{Si}), desilication in flashing circuit, desilication.

1. Introduction

Conditions of bauxite digestion should provide not only the maximum extraction of alumina from raw materials into pregnant liquor, but also the required rate of its desilication to obtain in precipitation aluminum hydroxide of high quality.

In the course of bauxite digestion silica passes into solution in the form of sodium silicate, and then precipitates with red mud as sodium hydroaluminosilicate (if the slurry does not contain Ca^{2+}).

Several flowsheets of aluminate liquor desilication are known in the industry:

- preliminary desilication (under atmospheric pressure, with prolonged residence time of raw slurry at a temperature of 95 - 105 ºC, using various ways of temperature maintenance, including heating through a wall and by live steam) – realized at NGZ;
- post-desilication (under atmospheric pressure, with prolonged residence time of diluted digested slurry at a temperature of 95 - 105 ºC, simultaneously with the process of deep desilication, a process of liquor decomposition occurs releasing aluminum hydroxide that

reduces alumina yield. Refineries operated with high A/C ratio of digested slurry does not use this method due to high losses of alumina) –realized at NGZ.

- feeding DSP seed into the process in the form nozean or cancrinite – requires production of the seed at high temperatures of kaolin processing [1];
- Sumitomo process [2]. The process is based on four key operations (1 - two stream heating and a tubular digestion system. Alumina is completely extracted from bauxite, and reactive silica passes into solution only by half; 2 - high rate settler: digested slurry is separated within several minutes under pressure to prevent further dissolution of silica contained in bauxite; 3 - high rate washers: red mud is rapidly washed by counterflow decantation; 4 – desilication of pregnant liquor under pressure) – the process requires great capital expenditures for implementation. This method is suitable for high silica gibbsite raw material and was realized only at several refineries.

The first two of the considered options of desilication process are realized at the site of NGZ, and the rest ones demand great capital expenditures. Equipment arrangement of the first two options is characterized by a significant amount of tank equipment, the third option - by a large number of pressure vessels.

Our proposal to improve desilication in the flashing (steam separation) circuit of bauxite slurry enables to carry out this process at higher temperatures which promotes more vigorous chemical reactions both in bauxite digestion, and in desilication of pregnant liquor

Unlike the Sumitomo process, the proposed option does not require separation of phases of digested slurry for desilication under pressure [3] that allows to minimize capital expenditures for modernization of the digestion train.

Intensification gives an opportunity to reduce residence time from 8 - 10 to 1 - 2 hours that in case of use live steam as a heat carrier results in dilution of aluminate liquor to a lesser extent.

Increase in desilication temperature of aluminate liquors facilitates reduction in equilibrium content of silica in the liquors and increase in μ_{Si} to 220 units that is much higher than at low-temperature desilication.

2. Experimental

A laboratory autoclave installation was assembled and prepared for investigations that allowed to feed and select samples directly in the course of an experiment.

The laboratory installation (Figure 1) comprises:

- an autoclave, volume 1 dm^3 (p. 1);
- a system for feeding of reagents (spent liquor, sweetening slurry, wash water, etc.) to the reaction zone (p. 2), including a pressure vessel of liquefied nitrogen with reducing gear and measure feeder;
- a system of slurry sampling from the reaction zone (p. 3);
- a system of water cooling of the autoclave to the set temperature (p. 4).

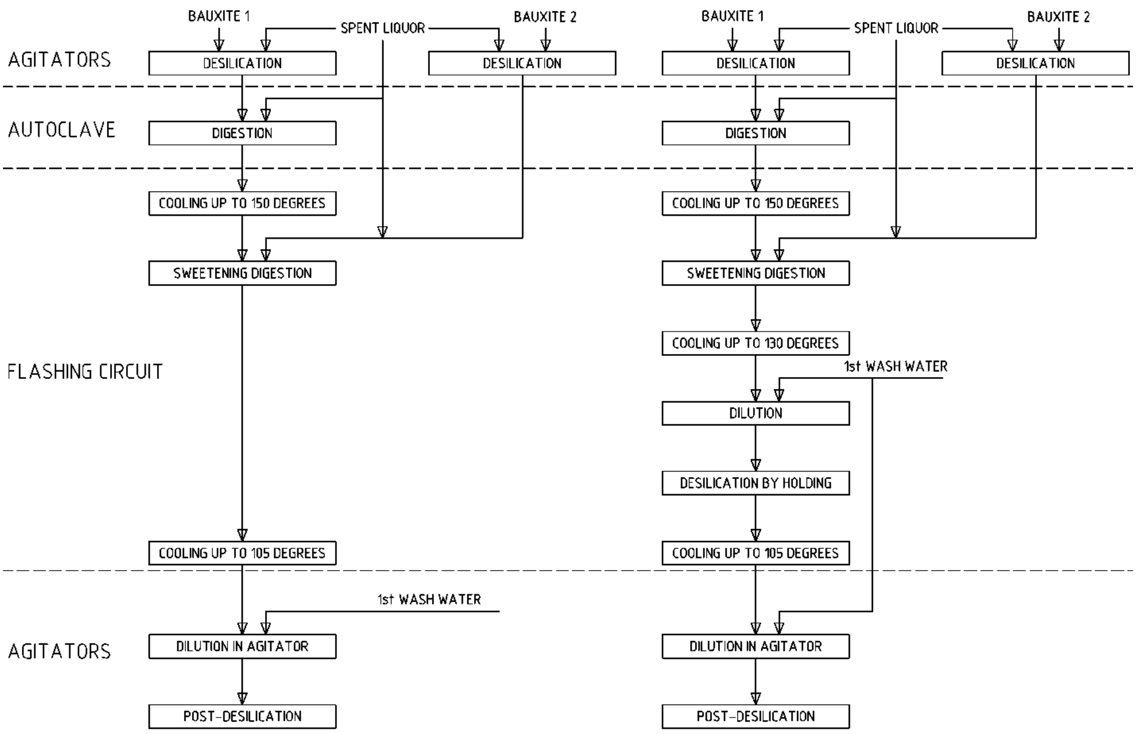
The laboratory autoclave installation simulated the processes taking place at the production site of the NGZ alumina plant on the basis of pre-desilication, digestion and post-desilication.

Figure 2 shows the Block flow diagram of testing in the laboratory:

- a – corresponding to the current alumina production process;
- b – proposed alumina production process.

Input of flows (spent liquor, bauxite pulp 2 after pre-desilication, 1st wash water) into the autoclave was carried out through a pressure vessel created by a vessel with compressed air.

Figure 1. Autoclave installation for simulation of processes of preliminary desilication, digestion, dilution and post-desilication in the flashing circuit.



a b

Figure 2. Block flow diagram of testing in the laboratory.

Due to the impossibility of organizing qualitative modeling of the concentration process of slurry after digestion by flashing, it was decided to supply a more concentrated spent liquor to compensate for the process of evaporation of the pulp liquid phase in the flashing circuit. Thus, it was ensured that the laboratory data were consistent with the modeling of the current scheme for the production of alumina, according to the plant's technical reports.

3. Test Results

As an example for comparison, a series of experiments on physical simulation of the process chain was conducted: preliminary desilication-dilution-post-desilication. Average results of the tests are presented in Figure 3.

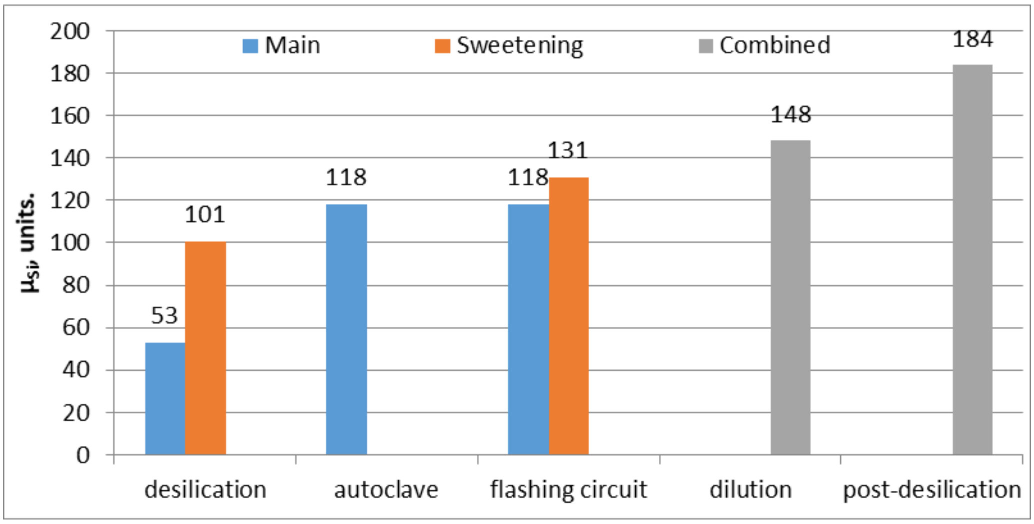


Figure 3. Variation in μ_{Si} of the liquor after each of processes of: preliminary desilication, digestion, dilution and post-desilication.

Analysis of industrial data confirmed convergence of the process simulated in a laboratory with the industrial process.

Since NGZ uses high-temperature digestion with "sweetening" of digested slurry with high-quality gibbsite bauxite, it was decided at first to determine the impact of residence time in the main stream digestion flashing circuit at a temperature of 120 °C on the value of μ_{Si}.

The main objective of this series of experiments was to determine the impact of the obtained μ_{Si} and hydrolysis, of partial dilution of the main stream of the separated digested slurry with the 1st wash water and residence at a temperature of 120 °C within 10, 20, 30 and 40 minutes, and determination of parameters of the slurry fed for deep desilication.

The results of these experiments are presented in Figure 4.

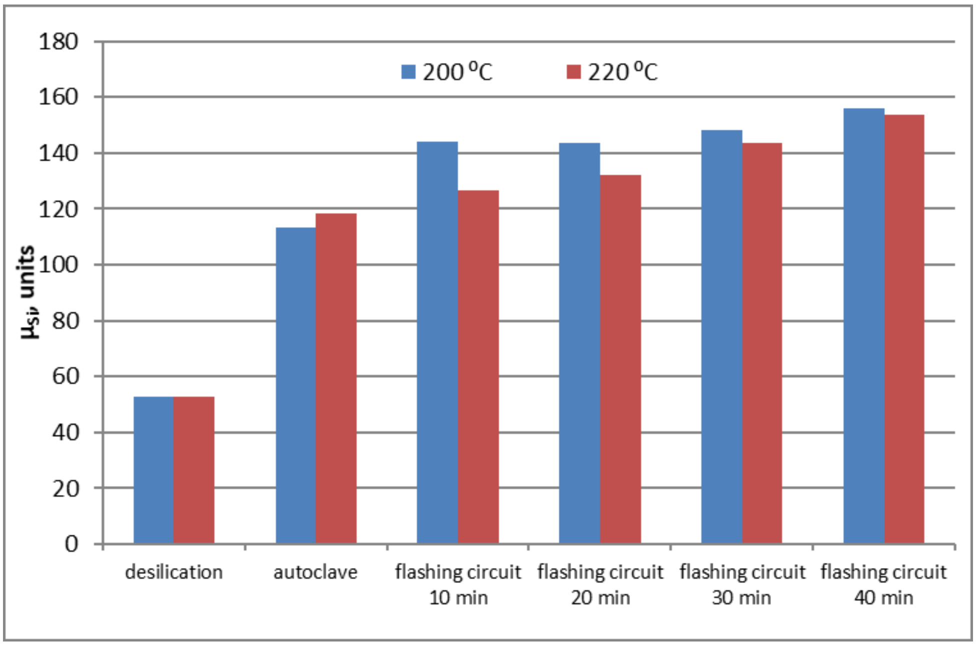


Figure 4. Variation of μ_{Si} of the liquor after each of processes of: predesilication, digestion (200 and 220 °C), dilution and post-desilications in the flashing circuit.

From the obtained experimental data the increase in μ_{Si} exhibits practically a linear dependence on time.

Further dilution allows to obtain μ_{Si} at a level of 176 units, as compared to 154 units in the operating technology, when carrying out the process of desilication in the flashing circuit at a temperature of 120 °C and residence time 40 minutes. Increase in μ_{Si} value is about 20 units.

When favorable results in the main stream of digestion were revealed, the decision was made to check the impact of a sweetening stream on μ_{Si} in the aluminate liquor. To achieve this, a complete process chain was simulated of aluminate liquor production at NGZ.

Average results of a series of experiments of process simulation of the main and sweetening streams are presented in Figure 5.

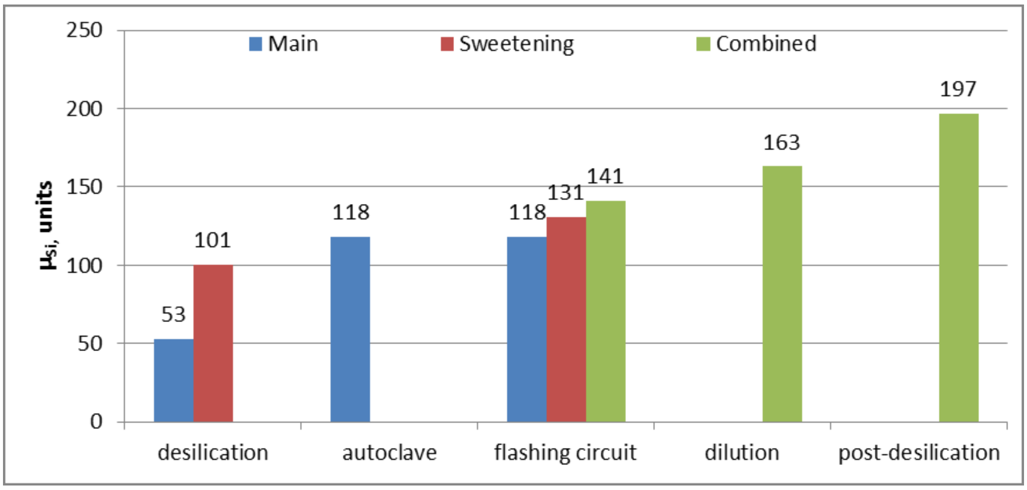


Figure 5. Variation of μ_{Si} of liquor after each of processes of: predesilication, digestion, dilution and post-desilication by the proposed process.

4. Analysis of the Obtained Results

Based on the result of analysis of the obtained experimental data, a Comparative Table 1 of principal quality factors of liquor (by silicon) was composed.

Table 1. Chemical composition of liquid phases and parameters of aluminate liquor preparation by existing and offered technology.

Process stage	Composition of liquid phase after experiment, g/dm^3						
	Current technology				Proposed technology		
	Al_2O_3	SiO_2	μ_{Si}		Al_2O_3	SiO_2	μ_{Si}
Main stream							
Preliminary desilication	223.6	4.23	53		223.6	4.23	53
Preliminary desilication + digestion	235.5	1.99	118		235.5	1.99	118
Sweetening stream							
Preliminary desilication	193.2	1.92	101		193.2	1.92	101
Preliminary desilication + digestion	230.1	1.76	131		230.1	1.76	131
Combined stream							
Preliminary desilication + 2 digestions + dilution 30 % residence time 30 min. (last separator)					204.6	1.45	141
Preliminary desilication + 2 digestions + dilution (beginning of post-desilication)	158.5	1.07	148		162.7	1	163
Preliminary desilication + 2 digestions + dilution + post-desilication (termination of post-desilication)	152.5	0.83	184		153.5	0.78	197

The data presented in Table 1 enable to draw a conclusion on positive impact of additional residence time of partially diluted digested slurry at 120 °C. Increase in μ_{Si} upon termination of the process of post-desilication is 10 - 15 units that allows to obtain aluminate liquor having μ_{Si} 195 - 200 units when using as the main and sweetening raw material bauxite of Guinean deposits with low content of reactive silica.

To obtain higher μ_{Si} it is necessary to consider increase in residence time of partially diluted slurry up to1 hour and dilution to a greater extent – 50 % of the required stream of the 1st wash water

5. Conclusions

The main task of this work was to provide homogeneous quality of produced smelter grade alumina by silica content.

Based on the analysis of alumina production flowsheet at NGZ it was determined that to provide homogeneous quality of alumina by silicon it is necessary to raise the value of μ_{Si} by 15-20 units that will allow to ensure continuous production of G-00 grade alumina not affecting performance of the refinery.

Partial dilution of digested slurry with residence at a temperature of 120 °C for at least 40 minutes allows to increase μ_{Si} of the aluminate liquor fed to precipitation by 20 units, as compared to the current value.

The use of this approach allows to ensure continuous production of alumina by silica content at minimum capital expenditures.

6. References

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