AUToclave desilication of digested bauxite slurry in the flashing circuit

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Abstract

Bauxite digestion conditions should provide not only the maximum alumina recovery from raw material into liquor, but also the required desilication to produce further alumina of high quality. Several desilication circuits are common for alumina industry, namely low temperature predesilication of crude and digested slurry, Sumitomo process. These processes usually involve extensive process equipment. The proposed option of autoclave desilication in the flashing circuit of the bauxite slurry allows implementation of the process at higher temperature enhancing chemical reactions of green liquor desilication. This improvement gives an opportunity to sharply reduce the retention time, facilitates reduction in the balanced silica content in the liquor. The mud produced after digestion and high temperature desilication has better sedimentation properties as compared to that obtained as a result of low temperature desilication.

1. Introduction

The bauxite processing conditions should provide not only high alumina recovery, but also the required level of green liquor desilication to produce specified product quality. The conventional industrial process and equipment schemes of Bayer liquor desilication include:

- low temperature presdesilication of thick raw slurry and desilication of digested dilute slurry under 95-105°C;
- Sumitomo process based on four key operations (1- double stream heating and tube digestion. Alumina is completely recovered form bauxite and half of reactive silica is transferred to the liquor; 2- high rate thickener: the bauxite residue is rapidly washed by countercurrent decantation; 4- desilication of green liquor in pressure vessels) [1]. For some specific quality bauxites above arrangements are not effective enough.
- at some refineries to achieve required silica concentration the digested diluted slurry / green liquor is treated with lime milk.

At sintering and nepheline processing refineries 4 methods of green liquors desilication are applied [2]:
1. autoclave desilication at 150-170 °C with recycling DSP seed;
2. autoclave desilication with addition of lime or white mud;
3. atmospheric pressure desilication free of any additives;
4. atmospheric pressure desilication with lime or other additive.

The last two methods even with 100 °C required long retention time and this option is not often applied at the plants.

The action of lime under desilication process is based on the cationic exchange with precipitated aluminosilicate where the sodium ions are replaced by calcium ions forming the calcium cencrinite \(CaO\cdot Al_2O_3\cdot 1,7\cdot SiO_2\cdot nH_2O\) and hydrogarnet \(3CaO\cdot Al_2O_3\cdot XSiO_2(6-2X)\cdot H_2O\) depending on process conditions [3]. Lower hydrogarnet dissolution ability in alkaline liquors provides the better desilication level when lime is used. Another effect - \(Na_2O\) losses in the mud reduce and \(Al_2O_3\) losses increase.

It is known that the more low quality (high silica) bauxites are used to produce alumina by Bayer method, the higher are the losses of alumina and sodium oxides with red mud. Formation of DSP in course of autoclave desilication results in the scaling of heating surfaces in heat exchangers and autoclaves, therefore while processing high silica bauxites, to ensure that maximum silica will be bound into DSP before digestion, one tends to complete most of the process at pre-desilication stage, thus applying longer retention time and temperature about 100 °C. Another benefit of accomplishing most or full of desilication process before digestion is obtaining a possibility to avoid conventional atmospheric pressure post-desilication stage, which limits pushing up the A/C ratio due to increased reversion losses. Pre-desilication stage conditions generally allow to dissolve and bound into DSP the silica which is represented by kaolinite.

However, some of monohydrate bauxites have substantial amount of silica in other minerals, i.e. chamosite (chlorite group) that is the case for bauxites of North Urals and Timan (Russia) and North Viet Nam. For these bauxites requiring high-temperature digestion conditions, carrying out most of desilication before digestion is not possible. Similarly, bauxites with high quartz content (some Guinean or Venezuelan) processing leads to quartz being dissolved at high temperature digestion and/or clarification conditions. It is also known that DSP's depending on their formation conditions are subject to solubilisation / restructuring at desilication and digestions stages, sometimes increasing silica level in green liquors [4].

This paper studies the autoclave desilication of digested slurry in the digestion flashing circuit at temperature ~ 150 °C. This technology allows to process the above complicated bauxites with maximum alumina supersaturation (A/C) in green liquor by excluding atmospheric pressure post-desilication.

Due to increase of the desilication temperature comparing with conventional atmospheric pressure process, the slurry autoclave desilication process allows to intensify the chemical reactions for
desilication of the slurry liquid part. This process improvement permits to reduce the slurry retention time down to 30 – 60 min and increase the alumina to silica ratio of the liquor (μS) up to 300 units. The risk of alumina losses due to reversion normally occurring in the post-desilication tanks at lower temperatures is totally excluded.

The mud after digestion process followed by high temperature desilication is characterized by better sedimentation properties to compare with the mud from low temperature post desilication.

This paper is also dealing with the study of further desilication the mud after digestion process followed by high temperature including noselite and cancrinite. Previously the use of sodalite for this purpose was described [5].

2. Study of desilication process for different quality bauxites

In order to compare atmospheric predesilication and autoclave desilication processes efficiency the studies were conducted using low quality boehmite bauxites from North of Russia, diasporic from North Vietnam and predominantly gibbsitic bauxites from Guinea having high quartz content (tables 1, 2).

Table 1

<table>
<thead>
<tr>
<th>Bauxite</th>
<th>Content, mass %</th>
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<tbody>
<tr>
<td></td>
<td>SiO₂</td>
<td>Al₂O₃</td>
</tr>
<tr>
<td>North Russia</td>
<td>6.7</td>
<td>49.4</td>
</tr>
<tr>
<td>North Vietnam</td>
<td>4.2</td>
<td>57.7</td>
</tr>
<tr>
<td>Guinea</td>
<td>3.1</td>
<td>44.1</td>
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Table 2

<table>
<thead>
<tr>
<th>Bauxite</th>
<th>Phase composition of bauxite used, %</th>
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<tbody>
<tr>
<td></td>
<td>Gibbsite</td>
</tr>
<tr>
<td>North Russia</td>
<td></td>
</tr>
<tr>
<td>North Vietnam</td>
<td></td>
</tr>
<tr>
<td>Guinea</td>
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2.1. Experimental

Predesilication: To study the processes of low temperature predesilication the experiments were conducted using steel cups with agitator, fed with spent liquor and held under 95 °C; further bauxite was charged and in some tests lime was added. The slurry was held from 2 to 8 hours with agitation and then the slurry was digested under specified conditions (table 3). After digestion the slurry was filtered in the Bunsen flask using Buchner funnel, then washed to obtain neutral effluent.

Autoclave desilication: The studies were conducted using large scale autoclave unit Parr 4843 (USA) allowing to introduce different additives in the reaction zone under high temperature and pressure and sample liquid and solid phases from the reaction zone during the experiment.

Table 3

<table>
<thead>
<tr>
<th>Bauxite</th>
<th>Content, g/dm³</th>
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<tbody>
<tr>
<td></td>
<td>Na₂O</td>
<td>Al₂O₃</td>
</tr>
<tr>
<td>North Russia</td>
<td>139.9</td>
<td>162.9</td>
</tr>
<tr>
<td>North Vietnam</td>
<td>213.6</td>
<td>244.2</td>
</tr>
<tr>
<td>Guinea</td>
<td>184.8</td>
<td>215.7</td>
</tr>
</tbody>
</table>

Notes: *α* - Na₂O/Al₂O₃ molar ratio, **μS - Al₂O₃/SiO₂ mass ratio

The appropriate amounts of caustic liquor, bauxite and lime were charged in the reactor, agitated with speed 300 RPM and the produced slurry was heated to the reaction temperature following by the retention period.

When the temperature reached 150 °C the time of the beginning of desilication process of diluted slurry was fixed and in pre-set time intervals samples were taken over the period of one and a half – two hours in every 30 minutes to define the process kinetics. Then the samples were filtered on the Buchner funnel. Liquid and solid phases were analyzed. Presented samples of bauxites have different mineral composition (table 2), therefore for their processing it is necessary to maintain different process parameters both of digestion and of desilication (Table 3).

2.2. Results obtained

The study (tables 4 and 5) have shown that for any type of bauxites the autoclave desilication is more effective. It was found that the alumina to silica ratio of the liquor produced after the high temperature desilication is higher than after the preliminary low temperature desilication followed by digestion (fig. 1), the silica content in the liquors after autoclave desilication being 1.5-2.0 times lower than at the low temperature desilication-digestion.

During the study the optimum time of high-temperature autoclave desilication was defined for different types of bauxite: for boehmite bauxite from North of Russia – 30 min. (μS = 275), for diasporic bauxite from North of Vietnam – 90 min. (μS = 247), for gibbsitic bauxite from Guinea – 90 min. (μS = 214).

Table 4

<table>
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