FILTRATION OF PREGNANT HOT SLURRIES WITH HIBAR FILTRATION - A KEY FOR INNOVATING THE DIGESTION PROCESS

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ABSTRACT

Alumina refineries are permanently looking to increase the extraction yield of the digestion process but also to optimize and to reduce the operational costs. The focus of the experts is also on the sustainability of the operation of alumina refineries, and as a consequence the long term disposal and liability costs of the bauxite residue (in most cases considered to be hazardous waste). The extraction yield of the digestion process has been a major focus for decades and is coming again to the foreground. During the downstream thickening and washing process. some percentages of alumina are lost due to the long residence time, the lower operating temperature and the lower caustic concentration of the liquid phase and mud. If it was possible to separate the very hot, highly caustic and over-saturated digestion suspension just after the autoclave or tube digestion reactor, a lot of extractable alumina could be saved. More advanced digestion processes like ILTD (Improved Low Temperature Digestion) Process offer attractive options.

1. INTRODUCTION

The continuous pressure filtration (HiBar Filtration) in combination with steam pressure filtration offers the chance to separate and to effectively wash the hot bauxite residue just after digestion within a very short, controllable residence time. The same process demands are evident when hot slurries have to be separated from acid leaching processes of alternative alumina minerals (such as Orbite process). It would be preferable to filter those residues without an initial cooling of the slurry after leaching in order to avoid the precipitation/ crystallization of dissolved matters in the hot brine. The precipitated materials can be seen as a loss or form very much unliked scale. The continuous pressure filtration (HiBar Filtration) allows separation and washing of the brine suspension without cooling affects. The equipment for the filtration plants needs special know-how and expertise for the filtration process and plant design for applications under such conditions.

2. HIBAR FILTRATION TECHNOLOGY

HiBar Filtration of BOKELA is an advanced continuous pressure filtration technology (also known as Hyperbaric Filtration) that includes also the patented steam pressure filtration process and HiBar counterpressure filtration. The first hyperbaric filter started operation in 1986 for dewatering of coal concentrate. Nowadays, this economic high performance technology is well established and wide spread in the market. Typical applications are found in coal preparation, in copper, zinc and iron ore beneficiation, in starch production, in the fine chemicals industry and for filtration and purification of PTA (Pure Terephthalic Acid) with the HiBar Oyster Filter.

2.1 Plant and Process Design of HiBar Filtration

HiBar Filtration is realized on rotary drum or disc filters which are installed in a pressure vessel (Figure 1). Thus, process pressure values of up to 15 bar, process temperatures up to T = 200℃ and filtration pressure differences up to 6 bar and even higher have been realized. The filtrate pipes of the rotary filter are usally connected to the environment and the suspension feed is pumped by an appropriate pump into the pressurized vessel. The filter cake is removed from the filter cloth by compressed air blowback and discharged from the pressurized vessel through a sluice system. The vacuum pumps used with a conventional vacuum filter are replaced by a compressor that supplies the necessary compressed air to the vessel and for the compressed air blowback. The compressed air from cake blowback also serves as process air to

maintain the overpressure in the vessel for the filtration process. The application of overpressure instead of vacuum, where filtration pressure difference Δp under technical conditions is restricted to about maximum 0.8 bar, allows a high filtration pressure difference Δp, which ensures a high specific throughput and dewatering capability even with filter cakes of fine particles such as filter cakes of bauxite or bauxite residue where high capillary forces in the cake exist i.e. a high cake resistance have to be overcome.

2.2 HiBar Steam Pressure Filtration

HiBar Steam Pressure Filtration is an innovative hybrid process which leads to extremely dry and pure filter cakes. Contrary to the conventional steam filtration the HiBar steam pressure filtration uses pure steam instead of compressed air for dewatering, washing and cake drying. A filter cake which is formed at the low temperature of the feed slurry enters a specially designed steam cabin immediately after emerging from the slurry in the filter trough. Here, a superheated steam atmosphere exists and the following phenomenon takes place which can be described by the model of the "condensate front" (Figure

The residual cake moisture content of a product reduces if higher differential pressures are applied which is described by the capillary pressure curve of the product. The higher the differential pressure, the smaller capillaries of a filter cake can be overcome by the compressed air. Particularly for fine solids such as bauxite residue which do not dewater on vacuum filters or only to a limited extent, high pressure differences are necessary for effective cake dewatering because such cakes have small-sized cake pores with a high capillary pressure that has to be overcome.

2.)The steam condenses on the cold cake surface and a homogeneous condensate layer is formed, moving through the cake in a plug flow ("condensate front").

- The moving "condensate front" replaces of the mother liquor to the maximum extent (in some cakes to nearly 100%)
- When the "condensate front" reaches the filter cloth, the filter cake is heated up completely to steam temperature. At this point the cake leaves the steam cabin.

  Now compressed air passes the pre-dewatered and hot filter cake causing a very effective thermal drying which leads to extreme low cake moisture contents.

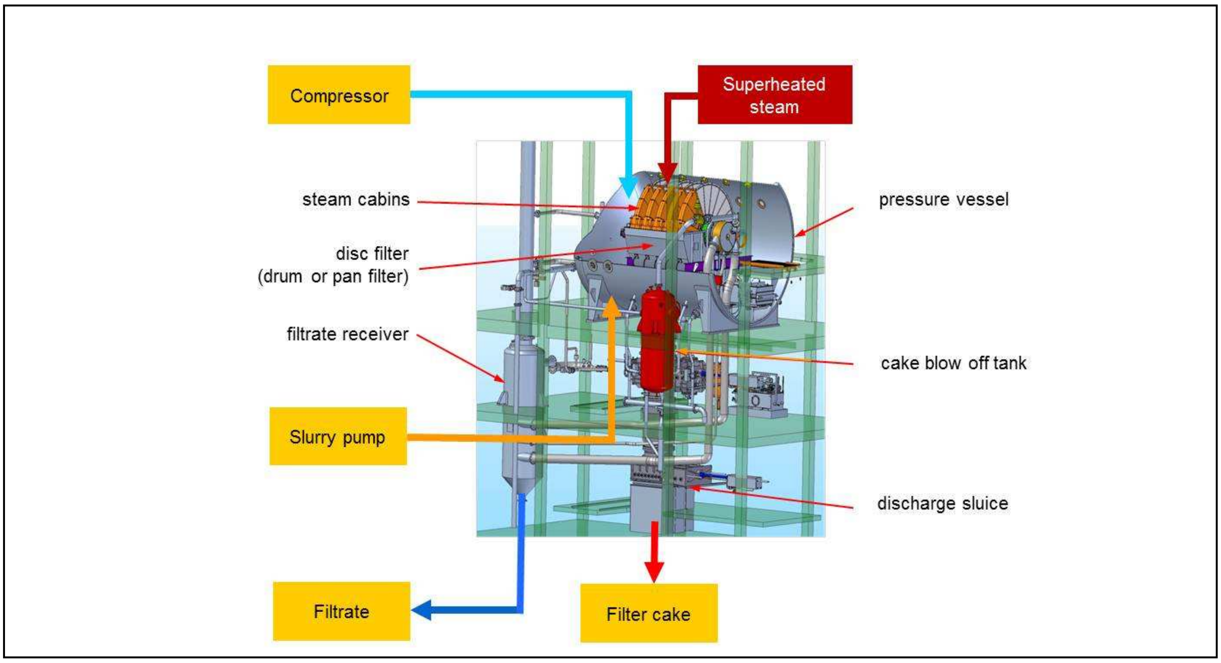

Figure 1. Schematic view of HiBar Filtration with steam cabins for steam pressure filtration

These thermal/mechanical phenomenon inside the filter cake leads to a nearly homogeneous cake and therefore to highly intensive cake washing and cake demoisturing without pressure and energy loss by a "fingering". Thus, extremely low moisture contents and a highly efficient cake washing is attained. Typical steam consumption rates range between 60 and 100 kg/t (dry substance) and depend on the properties of the solid material.

Differences in the design of a steam pressure filter and a standard pressure filter are additional steam cabins and pipes necessary for steam supply (Figure 1). All other components, e.g. the pressure vessel, filter with feed trough or sluice with discharge system, match those of a continuous pressure filter. Moreover, it is important that steam does not fill the whole pressure vessel but is only fed into the steam cabins covering the filter discs.

3. DRY BAUXITE RESIDUE WITH HIBAR FILTRATION

HiBar Filtration has shown its capability to produce a non-thixotropic, very dry and easy to handle, bulk-like bauxite residue which meets future demands and opens new possibilities for re-usage of this residue. Dry bauxite residue (DBR) with lowest moisture contents and soda contents are achieved with steam pressure filtration (Schepers, B., 1973). The treatment of the bauxite residue cake with steam immediately after cake formation leads to moisture contents in the region of 25 wt-% and below.

Filtration results for bauxite residue from various refineries with bauxites from different sources are presented and discussed by Bott et al. 2012 including results from running vacuum drum filters, results for HiBar pressure filtration and HiBar steam pressure filtration. Generally, it can be seen from this survey that vacuum drum filters achieve moisture contents in the region of 35 – 50 wt-%, continuous pressure filtration achieves moisture contents in the range of 25 – 30 wt-% while HiBar steam pressure filtration achieves moisture contents below 25 wt-% in the range of 22 – 25 wt-%. A critical phenomenon which sometimes constrains bauxite residue filtration is the formation of cracks in the cake.

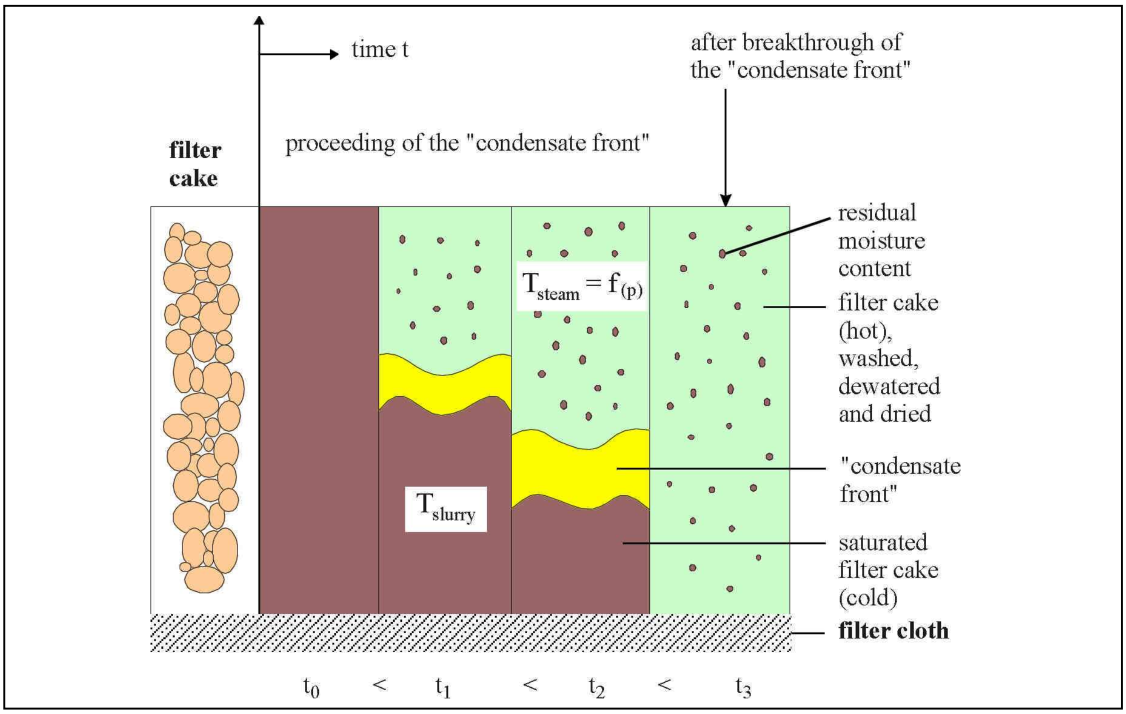

Figure 2. Steam Pressure Filtration: model of the "condensate front"

Currently, BOKELA are working on a comprehensive R&D project along with academic partners to investigate very promising approaches to prevent or to handle the formation of cracks in filter cakes which may occur with some bauxite residues or other fine products.

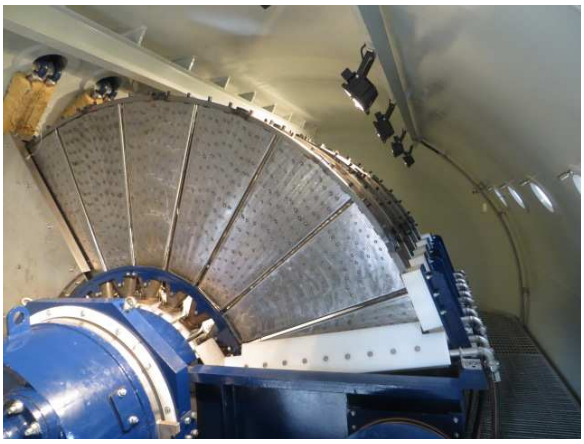

Figure 3. HiBar disc filter (70 m²) with steam cabins in the vessel for steam pressure filtration of bauxite residue

Figure 3 and 4 show the first HiBar steam pressure filtration plant for the dewatering of a fine bauxite residue (x_{50} = 1.9 µm). Two HiBar disc filters of 70 m² each are installed with one unit in operation and one filter as stand-by unit.

Figure 4. Filter building with both units (right)

The refinery operator decided in favour of HiBar Steam Pressure Filtration in order to achieve dry bauxite residue (DBR) with lowest moisture content. Required value of moisture content is mc ≤ 28 wt.% to go below the thixotropic point for improving residue handling and ensure high yield stress of the bulk. The dry HiBar filter cake will both improve residue handling and residue disposal. The dryness and high yield stress of the filter cake ensure steeper dumping slopes i.e. reduced disposal area and improved safety of the disposal site.

Table 1. Operational results of HiBar disc filter (70 m²) during commissioning – steam pressure filtration of dry bauxite residue

HiBar Steam Pressure Filtration of Dry Bauxite Residue Operational results during commissioning		
Moisture content	≤ 28 wt% ≤ 25 wt%	with operation in pressure filtration mode during commissioning with steam pressure filtration for hot operation later
Solids throughput	10 t/h 32 t/h	with restricted speed and restricted filter area (only 3 discs in operation) due to limited feed slurry later
Filter speed	0.3 rpm (up to 1.2 rpm)	
Cake height:	9.5 mm	
Pressure difference	Δp = 4 - 6 bar	

Commissioning of this first HiBar Steam Pressure Filtration plant for bauxite residue dewatering was in September 2014. First operational results are shown in table 1.

4. FILTRATION AT HIGH TEMPERATURES AND HIGH PRESSURES

4.1 HiBar Process and Plant Design for Hot Slurry Filtration

Many production processes such as the Bayer process run under high temperatures and high pressures to enable or improve specific reactions, to keep wanted or unwanted components in solution or to improve the process efficiency. Suspensions from such processes have to be handled in a careful way and a drop of pressure and temperature are associated with implications which should be avoided such as boiling and evaporation of the liquid or product contamination due to unwanted reactions and/or crystallisation of unwanted by-products etc.

Filtration of such slurries should ideally be performed at high temperatures and pressures and the filtration process/technology should meet the following challenging requirements:

- maintaining the temperature and pressure at the lower pressure side (filtrate side) above a certain level to prevent boiling (T_b, p_b) of the filtrate
- continuous filtration as a one-step process with short residence time
- prevention of unwanted reactions and/or crystallisation/scalings in the filter cloth and filtrate pipe system
- recovery of mother liquor and/or pure solids by counter-current cake wash with low wash liquor consumption and sharp separation of mother liquor and wash liquor
- applicable in corrosive, toxic or explosive atmosphere
- withstand high material stress
- filter sizes for small, medium and large (e.g. in alumina refineries) production rates
- availability of different sluice technologies for high temperature applications
- reliable operation control with different sequences, for instance; for heating procedure, start of operation, continuous operation and maintenance mode

Therefore, solid/liquid separation of such slurries is often performed as a complex multi-stage separation process with a series of single steps to evade said implications since appropriate filtration technologies which meet the above mentioned requirements have not been available. Now, HiBar Filtration enables filtration of hot slurries at high temperatures of up to $T > 200°C$ and high pressures of up to $p_{abs} = 15$ bar and has shown its capability to operate in a reliable way under such challenging conditions in many reference applications.

The HiBar Filtration process can be performed with a high filtration pressure difference $\Delta p = p_1 - p_2$ (p_2 = atmospheric pressure) and as a counterpressure filtration process (Figure 5) with high vessel pressure p_1 and a filtration pressure difference $\Delta p = p_1 - p_2$ with a pressure p_2 at the filtrate side above the boiling point T_b, p_b of the flash curve ($p_{atm} < p_b < p_2 < p_1$). This enables draining off the filtrate at a high temperature and high pressure without boiling and crystallisation effects and it allows processing of the hot filtrate in hot downstream process steps.

4.2 Direct Filtration of Bauxite Residue in the Bayer Process from the Highly Caustic, Hot Slurry

For alumina refineries the extraction yield of the bauxite processing has been a major focus for decades and accordingly the loss of extractable alumina in the process chain has to be minimized as far as possible. Loss occurs in the digestion reactor where extractable alumina (gibbsite) stays undigested, in the settlers and in the bauxite residue washers which sums up to a loss of about 6% and is therefore a topic of economical relevance /den Hond 2005 and 2006/. As a rule of thumb (den Hond, R., 2005) in the settlers 2% gibbsite reversion is lost and 2 more percent is lost during bauxite residue washing mainly due to the following reasons (Figure 6)

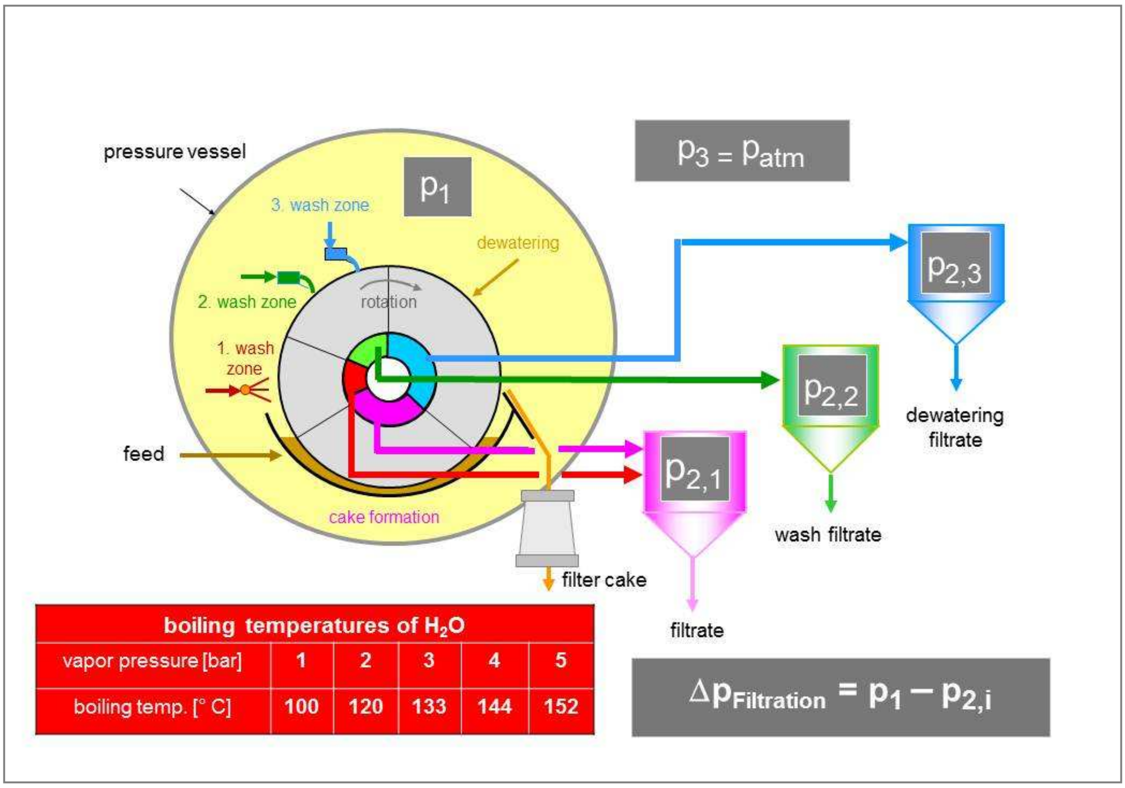

Figure 5 Principle of HiBar counterpressure filtration (with 3-stage cake wash – optional if required), p_1 = vessel pressure, $p_{2,i}$ = pressure at filtrate side

- cooling down of the liquor to ≤ 100℃ together with long residence time in the settler and bauxite residue washers with contact of the pregnant liquor with the bauxite residue (the liquid phase becomes oversaturated for aluminiumhydroxide (gibbsite))
- precipitation of aluminium hydroxide (gibbsite) enhanced by the presence of seed
- decreasing of the alumina solubility along with the decreasing caustic soda concentration due to the more diluted conditions in the bauxite residue washers

If it was possible to separate the bauxite residue and wash it from the highly caustic and hot slurry quickly and directly just after digestion without cooling and with a short residence time, a lot of extractable alumina could be saved.

In the early years of the Bayer process often plate and frame filter presses were used for the direct filtration of the blow off slurry and the cake was washed with condensate. Operational and maintenance cost, however, were immense especially due to cost for filter cloth wash water consumption and manual efforts for cake discharge. In some refineries they were replaced by Kelly filters which showed a better separation performance and reduced maintenance and labour cost. However Kelly filters were not suitable for direct filtration due to the high loss of soluble soda and alumina. In 1973 B. Schepers performed pilot scale testwork on direct filtration of the bauxite residue directly after the digestion reactor with continuous pressure filters in the Guilini Werk in Ludwigshafen, Germany (Schepers, B., 1973). The testwork was performed with an old vintage rotary pressure drum filter of 0.75 m² filter area. Maximum pressure that could be realized with this pilot filter was 3 bar, i.e. the filtration pressure difference was Δp = 2 bar. Feed temperature of the slurry was 100℃ while the filtrate was drained off with a temperature of T = 60 -70℃. Solids concentration of slurry feed was 47 g/l, particle sizes were not recorded but it can be assumed that they were in the typical range of $x_{50} \leq 10 \mu m$.

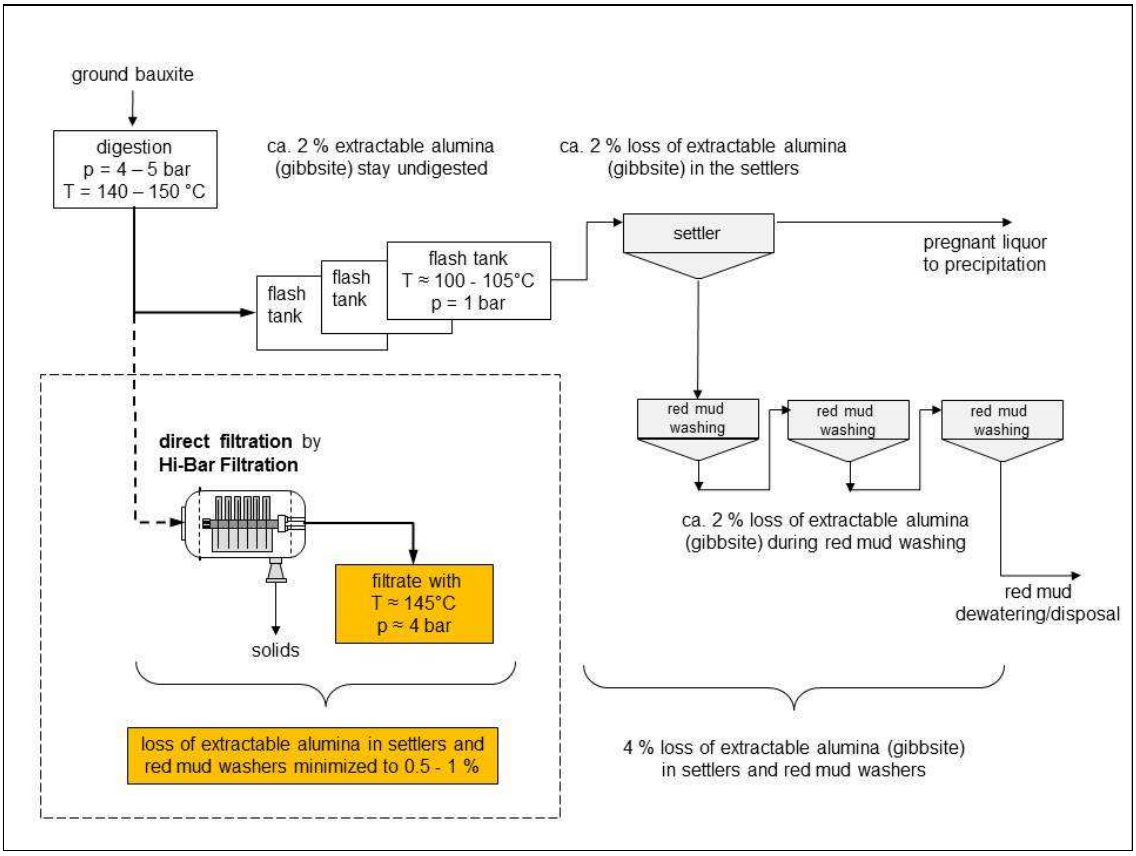

Figure 6. Simplified scheme of the “red side” of the Bayer process with a process option of bauxite residue direct filtration with HiBar Filtration

The filter cake was washed with boiler feed water of 100℃ and discharged with a discharge roller. The filtrate performance was in the range of 0.5 – 0.6 m^3/m^2h, the washed bauxite residue filter cake was nearly free of soluble soda and had a low moisture content of mc = 22 – 33% (Schepers, B., 1973). Even though this testwork was performed on an old fashioned, crude pressure filtration technology both the technical and the economical feasibility of this direct filtration option could have been demonstrated.

With HiBar Filtration, a most modern technology for continuous pressure filtration is available which is well proven in reference applications in several industries. HiBar Filtration is also applied for hot slurry filtration in many reference applications at high temperatures and high pressures with a specially and individually adapted process and plant design (see Para 4.1). HiBar Filtration is therefore a technology which has the capability for direct filtration of the hot pregnant liquor slurry within a very short residence time of some minutes or less at or very close to the digestion temperature of T ≈ 145-150℃ and the digestion pressure of p ≈ 4-5 bar. The filtrate can be drained off with somewhat lower but still fairly high temperature of e.g. 140 - 145℃. This way the loss of extractable alumina in the settlers and in the bauxite residue washers, which sums up to about 4 %, could be minimized to approximately 0.5 – 1 % (Figure 5).

4.3 Direct Filtration of Hot Slurries in the ILTD Process

The ILTD (Improved Low Temperature Digestion) Process is a modification of the Bayer process that has been developed by G. Bánvölgyi to retrofit existing refineries or to be implemented in new alumina refineries.

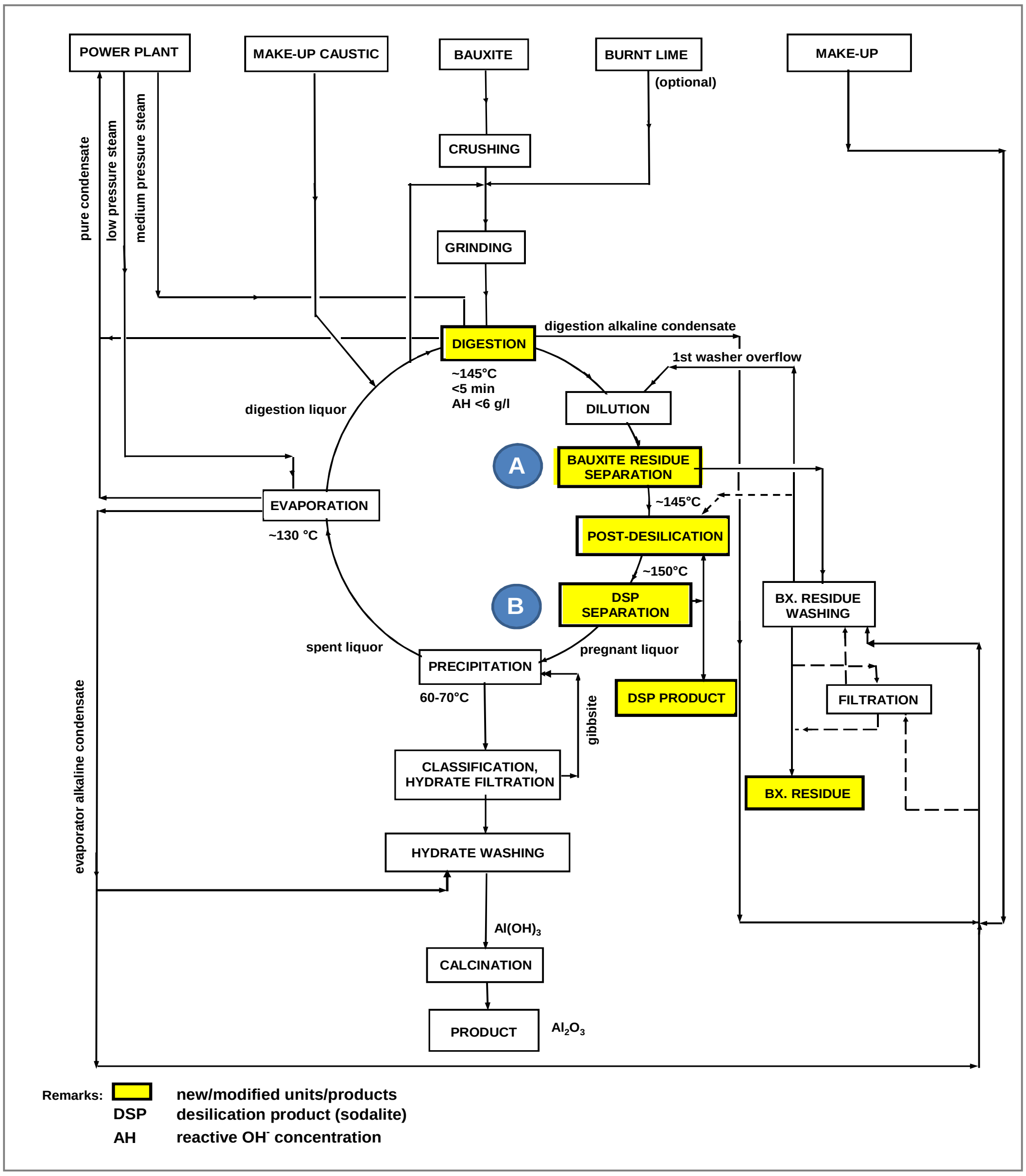

Figure 7 Simplified scheme of the ILTD process (Courtsey of G. Bánvölgyi); A,B: process steps of possible hot slurry filtration with HiBar filters

This process is assigned for Al-hydroxide production from gibbsitic bauxites. It has been tested and proven on a laboratory scale with various bauxites (Bánvölgyi. G, Siklósi, P., 1998). In contrast to the conventional Bayer process, in the ILTD process the digestion is carried out in a way that the total dissolving capacity of the digestion liquor is utilized with maximum extraction of the gibbsite. The consequences and main benefits are reported as follows /den Hond 2005, ILTD prospectus 2008/:

- reduced material and energy cost per ton of alumina by 9-13%
- 30 – 90% savings in the chemically combined NaOH losses
- 1 – 7% less bauxite consumption
- 5 - 20% higher digestion capacity

    - purer product (less silica, iron contaminants)
- desilication product (DSP) of high purity as a new by-product
- bauxite residue with extremely low soda content

The ILTD process contains two decisive separation steps where separation technologies such as HiBar Filtration which are capable to process hot, fine particulate slurries under extreme conditions with respect to temperature and pressure and within a short time are of crucial importance (Figure 7):

- After digestion the bauxite residue has to be quickly separated from the liquor preferably at the temperature and pressure of digestion: T = 145-150℃ , p = 4-5 bar (pos. A in Figure 7).
- The precipitated desilication product (DSP) has to be separated from the pregnant liquor at the temperature T = 145 - 150℃ and the pressure p = 4 - 5 bar (pos. B in Figure 7).

BOKELA and Bán-Völgy Bt Ltd. have formed an alliance to promote the implementation of the ILTD Process.

4.4 Direct Filtration of Hot Slurries in the Orbite Process

The Orbite process is a new process developed by Orbite Aluminae Inc. to extract alumina, contrary to the Bayer process, through hydrochloric acid leaching that takes place under high temperature and high pressure. Therefore, this process contains separation steps where separation technologies are needed such as HiBar Filtration which are capable to continuously filter hot slurries under extreme conditions with respect to temperature, pressure and with high demands for the materials of construction. This patented process is reported to enable the production of alumina (suitable for use in aluminium smelters) and other valuable by-products without generating bauxite residue, from a variety of feedstock materials such as aluminous clay, bauxite (high grade and low grade), kaolin, fly ash and other aluminium containing ores / Primeau, D.; Gilbert, M., 2012. The Orbite Process, Primeau, D.; Gilbert, M., 2012. Remediation of red mud using an innovative approach/. It is also capable of remediating bauxite residue which would allow the treatment of existing stocks of bauxite residue by separating its individual components such as Al_2O_3, SiO_2, Fe_2O_3, TiO_2 etc. as valuable products (Primeau, D.; Gilbert, M., 2012. Remediation of red mud using an innovative approach). It is reported that this process has been tested and proven in pilot plant scale with different raw materials (Primeau, D.; Gilbert, M., 2012. The Orbite Process).

The Orbite process consists of several plant sections as described in detail in /Primeau, D, 2012, 8/ which have to be adapted to the used feedstock material. In the first step, the raw material is crushed and ground to small particles (85% < 63 microns). In the next phase, the particles are acid leached with hydrochloric acid at a high of temperature of 150 – 195℃ and a high pressure which is specified (Primeau, D.; Gilbert, M., 2012. The Orbite Process) as "the reactor's maximum allowable working pressure". During acid leaching aluminium chloride ($AlCl_3$) is formed which is then transformed to alumina through calcination. Iron dissolves and forms ferric chloride ($FeCl_3$) from which the pure ferric oxide is precipitated. By acid regeneration the hydrochloric acid is recovered from the mother liquor while in parallel other components which remained in the leachate as metallic solutions including magnesium, gallium, alkaline and rare earths are converted to oxides and extracted as valuable by-products. Only silica and titanium remain undissolved during acid leaching and need to be removed by filtration with maximum recovery of the mother liquor containing the dissolved valuable components.

Expected requirements on a separation technology for filtration tasks in the Orbite process can be more or less summarized as: high slurry temperature of 150 – 195℃, supposedly high pressure (as mentioned above), intensive cake washing for maximum recovery of the mother liquor, capability to filter, wash and demoisture slurries with fine to very fine particles with high specific throughput rates, and large filter sizes per unit in order to handle large production rates. This task breakdown coincides with the special capabilities of HiBar Filtration. The acid atmosphere, however, requires a special plant and apparatus design by using construction material withstanding the strain of such an application.

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