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ALUMINA YIELD IN THE BAYER PROCESS
PAST, PRESENT AND PROSPECTS

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ABSTRACT

During the past decades a large number of alumina plants were successful in economically increasing the productivity of the Bayer liquor loop. Over a period of 25 years the benchmark precipitation yield has increased from 70 g/l to 90+ g/l, whilst producing sandy alumina. However with the theoretical maximum precipitation yield at 160 g/l there is still a wide gap to be closed. This paper provides an overview of past developments, the present status and the prospects, resulting from increased process knowledge and emerging technologies. It is envisaged that benchmark precipitation yield will soon cross the 100 g/l level.

INTRODUCTION

One of the key objectives of alumina production is to enrich the caustic solution in digestion with a maximum of alumina and to subsequently drive out a maximum of alumina from the solution in precipitation. In other words maximize alumina production per liter of circulated liquor, i.e. maximize liquor productivity or alumina yield. Increasing yield brings advantages in reduced capital cost per annual ton production capacity for digestion, precipitation, decantation, HID, evaporation and power house. Also such unit operating costs as digestion steam, pumping power, labor, maintenance, overheads and other fixed costs decrease as yield increases.

Over the last 25 years the benchmark precipitation yield has increased from 70 g/l to 90+ g/l whilst producing sandy alumina. There is however strong resistance to cross the 100 g/l level. Notably conflicting objectives between extraction and yield in digestion and between product quality and yield in precipitation require a solution.

This paper describes emerging technologies that allow plants to economically operate at plus 100 g/l precipitation yield. Liquor alumina yield can be calculated as follows:

$$Y = \Delta(A/C) \times C \qquad (1)$$

Y   = yield (g/l)
A   = liquor alumina concentration (g/l, expressed as Al_2O_3)
C   = liquor caustic concentration (g/l, expressed as Na_2CO_3)
$\Delta(A/C)$= change in liquor A/C ratio (-)

Because of evaporation and dilution effects, the caustic concentration of the circulating process liquor is not constant over the process cycle. Digestion yield, which is based on the concentrated spent liquor caustic concentration, is higher than precipitation yield, which is based on the caustic concentration of diluted digestion discharge liquor. A paper by Den Hond [1] describes theoretical, maximum yields and related process conditions, which are summarized in Table I below. The data apply to pure solutions and digestion and precipitation of pure gibbsite.

Table I. Theoretical maximum yield and process conditions			
	Digestion	Precipitation	
Yield	675	160	g/l
A/C feed	0.190	0.800	-
A/C discharge	0.800	0.190	-
C feed flow	1110	265	g/l
T	> 150	30	°C

DIGESTION

Alumina dissolution in digestion is, from a thermodynamic point of view, primarily controlled by the following parameters:
– The alumina mineral phase;
– Extraction liquor caustic concentration;
– Digestion temperature.
The relationship between these parameters is shown in Figure 1.
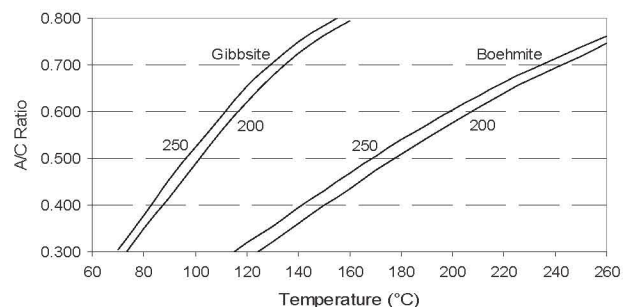

Figure 1. Equilibrium A/C ratios of gibbsite and boehmite for 200 and 250 g/l caustic concentration (as g/l Na_2CO_3).

The alumina mineral phase

To achieve the same A/C ratio in digestion discharge liquor, the digestion of boehmitic bauxite requires a much higher digestion temperature than the digestion of gibbsitic bauxite. Hence from an

economic point of view the processing of gibbsitic bauxites is instrumental to achieve a high digestion A/C ratio.

The efficiency of boehmitic bauxite processing can be improved through sweetening digestion technology. In this technology the high temperature boehmitic bauxite digest is used as a means to increase the extraction liquor A/C ratio with a first increment. As the high temperature digester discharge slurry progresses through flash cooling, gibbsitic bauxite is added when the slurry reaches a temperature in the 180 to 150°C range. This low temperature digestion process for gibbsitic bauxite, called sweetening digestion, raises the A/C ratio of the digestion discharge liquor to the level that is normal for gibbsitic bauxite digestion. A schematic of sweetening digestion is presented in Figure 2 below.
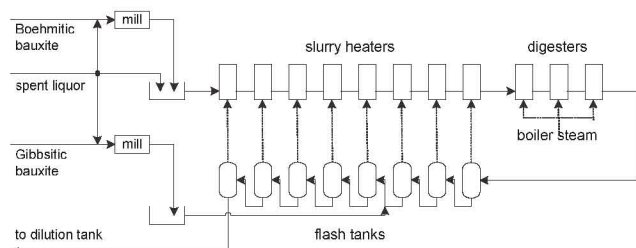

Figure 2. Sweetening digestion schematic.

When mining boehmitic bauxite deposits, it is often possible to separately mine limited quantities (at least 15% of the total) of gibbsitic bauxite, enabling the use of sweetening digestion.

Extraction liquor caustic concentration

The caustic concentration relating to maximum precipitation yield is plant specific, but is below the 300 g/l Na_2CO_3 level. Operating digestion at a higher caustic concentration than precipitation requires the evaporation of water from spent liquor and subsequent dilution of green liquor to precipitation requirements. In the natural course of the Bayer process the main dilution is provided by the bauxite residue wash water passing up the residue wash circuit. An economic solution for evaporation in excess of normal plant water balance requirements for the sake of increasing digestion caustic concentration and hence yield has not yet been found.

Heat transfer in the heat interchange department (HID) by direct means, e.g. using plate heat exchangers, rather than by indirect means, using flash steam, avoids the liquor concentrating effect from flashing. As a result digestion can operate at a higher caustic concentration without changing the precipitation caustic concentration, enabling an increased green liquor A/C ratio.

A constraint in plants with high liquor impurity levels is scale formation on heat transfer areas and other critical equipment surfaces in and downstream of the evaporation area. Said scales result from the precipitation of sodium salts from liquor at elevated caustic concentration (in particular sulphate, carbonate and organics based salts). Development of more efficient methods for removal of the liquor impurities concerned would enable higher liquor caustic concentrations.

The yield potential of digestion systems in which the spent liquor is heated separately from the bauxite slurry is constrained by the liquor free caustic concentration, which is calculated as:

$$FC = C - 1.04 * A \qquad (2)$$

FC = free caustic (g/l, expressed as Na_2CO_3)
C = liquor caustic concentration (g/l, expressed as Na_2CO_3)
A = liquor alumina concentration (g/l, expressed as Al_2O_3)

At elevated liquor free caustic concentrations the risk of stress corrosion cracking (SCC) of steel is prohibitive, whilst the allowable liquor free caustic concentration decreases with increasing liquor temperature. A practical solution is "single streaming", involving the joint heating of liquor and bauxite slurry in a single stream. At increasing temperatures the gibbsite in the bauxite rapidly dissolves, thereby reducing the extracting liquor free caustic concentration.

Digestion temperature

Typically the digestion temperature for gibbsitic bauxite is in the 135 to 150°C range. Increasing digestion temperature implies increasing digestion pressure and such increase is directly associated with operating and capital cost penalties. Temperatures above 150°C would enable an increased green liquor A/C ratio, but in combination with the holding time requirement for sufficient liquor de-silication during the digestion step the risk of excessive gibbsite to boehmite conversion increases, actually reducing the liquor A/C ratio, as described in a paper by Kotte [2]. Hence usually there is little scope to increase the digestion temperature above the 150°C level when processing gibbsitic bauxite.

In the sweetening digestion process gibbsitic bauxite is digested at temperatures as high as 180°C because of a short holding time at the elevated temperature, aiming at fast gibbsite extraction and largely ignoring further green liquor de-silication.

Conflicting objectives in digestion

An economic consideration that limits the green liquor A/C ratio is the so-called digestion dilemma, resulting from two conflicting digestion objectives, which are:
–   Maximize the liquor alumina concentration, which requires overcharging digestion with bauxite.
–   Maximize the alumina extraction form bauxite, which requires undercharging digestion with bauxite.

The practical aspect is that as the green liquor A/C ratio increases, the conditions for gibbsite extraction deteriorate. In addition, with an increasing A/C ratio the premature crystallization of gibbsite on mud in clarification, called reversion, increases rapidly, because the driving force for crystallization as well as the presence of seed, in the form of un-extracted gibbsite in mud, increases. The resulting increase in specific bauxite and caustic soda consumption and specific residue production, as well as scaling problems in polishing filtration will reach a point where a further increase in green liquor A/C ratio becomes uneconomic.

The digestion dilemma can be eliminated by replacing the well-known single step, low temperature digestion process for gibbsitic bauxite, including its sweetening digestion variation, by a two-step, low temperature digestion process. The 1st digestion step aims at maximizing the liquor alumina concentration and the 2nd digestion step aims at maximizing the alumina extraction from the bauxite residue, which results from the first step.

The principal challenge for the 2nd digestion step is to minimize associated capital and operating costs, which is possible once it is appreciated that the 2nd digestion step reduces a refinery's residue production and hence the related residue wash water intake. The resulting reduction in evaporation requirement favors the use of a 2nd digestion step without a conventional "flash vessel-heater" system for heat recovery. Instead a spiral heat exchanger system, especially designed by Alfa Laval to handle thick mud slurries, is used for heat recovery.

Spiral heat exchangers have a much higher heat exchange efficiency between digester feed and discharge flow than the conventional "flash vessel-heater" system because:
- Heat is transferred without the use of flash steam as intermediate energy carrier, avoiding the adverse effect of liquor boiling point rise;
- Counter current heat transfer follows a continuous pathway rather than a discontinuous, staged pathway.

As a result, the external heat supply requirement for the 2nd digest, for example with hot water, is relatively low.

The described apparatus for the two-step digestion of gibbsitic bauxite is elaborated in a patent by Den Hond [3]. The technology is labeled M2M-Technology, which is short for Mud-to-Money Technology, referring to the profitable recovery of alumina from mud. A simplified schematic of the 2nd digestion step of M2M-Technology is shown in Figure 3 below.
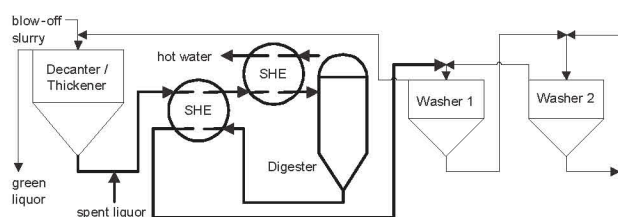

Figure 3. M2M-Technology, schematic of the 2nd digestion step. (SHE is Spiral Heat Exchanger)

Installation of M2M-Technology in an existing refinery is relatively easy, since a spiral heat exchanger system has a much smaller footprint than a conventional "flash vessel-heater" system.

Alumina extraction from residue in the 2nd digestion step does not require bauxite mining, crushing, transport, grinding, slurry holding, liquor decantation, residue washing and evaporation. In addition the residue contains no reactive silica. Hence, the operating cost for the incremental alumina produced from the bauxite residue is limited to energy cost (power, boiler and calcination fuel) and incremental fixed operating cost. Indicative operating cost is around the 35 USD/t level.

The indicative capital cost estimate for installing M2M-Technology at a 2 Mt/y refinery is in the 8 to 14 MUSD range, depending on the unique features of the refinery considered.

Economic evaluations indicate that implementing M2M-Technology provides an economically attractive project. At the same moment bauxite utilization is maximized and residue

generation is minimized, both of which address public demand to reduce the environmental impact of alumina production.

A/C ratio control

Inaccurate A/C ratio control of digestion discharge liquor can result in short periods of overcharging digestion with bauxite. As a result clarification is seeded with un-extracted gibbsite, potentially triggering decanter reversion and polishing filter scaling. Many plants now operate advanced A/C ratio control systems, involving feed forward with feedback trim and utilizing on-line measurement of liquor properties such as electrical conductivity and density. As a result the target green liquor A/C ratio can be set at only a few ratio points below the maximum permitted.

Obviously M2M-Technology will make A/C ratio control more forgiving and installation of the technology in a to-be-commissioned plant would contribute to a successful start-up.

Digestion conclusions

From the above it follows that a high digestion yield requires:
- The use of gibbsitic bauxite and low temperature digestion technology, including sweetening digestion, to maximize green liquor A/C ratio.
- The use of direct means e.g. plate heat exchangers in the heat interchange department (HID) enabling an increased extraction liquor caustic concentration and increased green liquor A/C ratio, without changing precipitation caustic concentration.
- The use of "single streaming" digestion technology, enabling an increased extraction liquor caustic concentration and increased green liquor A/C ratio, whilst avoiding stress corrosion cracking.
- The use of M2M-Technology, combining maximum green liquor A/C ratio with minimum alumina losses with the residue at attractive economics.
- Advanced A/C ratio control, involving feed forward control with feedback trim.

CLARIFICATION

The highly super-saturated green liquor discharging from digestion must safely pass clarification before reaching precipitation. Main concerns in clarification are:
- Reversion in decanters and mud washers;
- Scaling of polishing filter leaves.

Reversion in decanters and mud washers

After passing through sand separation, if required, the digestion discharge slurry is fed to decanters for separation of green liquor and mud. The green liquor is super-saturated with alumina, whilst un-extracted gibbsite in mud acts as seed for premature gibbsite crystallization, also named reversion. It should be noted that minerals such as goethite also exhibit seed activity.

The decanter underflow passes through the mud washers where reversion continues. The use of high rate decanters and washers reduces liquor-to-mud contact time and hence reversion.

Scaling of polishing filter leaves

Reversion in the mud washers can lead to the formation of very fine gibbsite nuclei, moving with the wash liquor towards the main plant liquor circuit, creating scaling problems in the polishing filters. Adding 1st washer overflow to the decanter feed increases the risk of nuclei formation in the decanter. Hence a downward correction to the green liquor A/C ratio is required in case the target green liquor A/C ratio is set on the basis of avoiding nucleation and not on the basis of the more common situation of keeping alumina losses and scaling in the decanters within a particular limit (indicated by delta A/C between decanter overflow and underflow streams).

Adding the 1st washer overflow directly to the polishing filter feed flow would reduce the potential for filter leave scaling, but a penalty is the loss of the decanter as wash stage.

M2M-Technology

Inserting the 2nd digestion step of M2M-Technology in the transfer line between decanter underflow and 1st washer feed (see Figure 3) enables the extraction of gibbsite seed from the decanter underflow slurry, largely eliminating related reversion and nucleation in the mud washers. Both aspects allow the green liquor A/C ratio to be increased.

Clarification conclusions

From the above it follows that safely passing of high A/C ratio green liquor through clarification is facilitated by:
–   The use of high rate settling tanks, reducing the liquor-to-mud contact time and hence reducing reversion.
–   The use of M2M–Technology, largely eliminating alumina losses with mud and reducing the risk of scaling polishing filter leaves.

PRECIPITATION

The recovery of alumina from green liquor is driven by a number of process parameters, including alumina super-saturation in liquor, temperature, seed surface area and holding time. The impact of some of these parameters follows from the kinetic equation describing the growth rate of gibbsite particles in Bayer plant liquors [4]. A generic form of the well established kinetic equation is presented below:

$$LGR = K \times SA \times f(I) \times Exp\left(\frac{-E}{RT}\right) \times \sigma^2 \qquad (3)$$

LGR= linear growth rate of hydrate
K   = pre-exponential constant
SA   = seed surface activity
f(I) = function describing the impact of the ionic strength (caustic and impurity concentrations) of the solution on growth kinetics
E   = activation energy
R   = universal gas constant
T   = absolute temperature
σ   = alumina super-saturation

The process parameters involved in enhancing liquor productivity and the amount of alumina removed from the liquor under steady-state conditions in a precipitator tank can be assessed as follows:

$$P = C \text{ x } LGR \text{ x } SSA \text{ x } SD \text{ x } \tau \qquad (4)$$

P   = Al_2O_3 production in the precipitator tank
C   = constant
LGR= linear growth rate of hydrate in the tank
SSA = specific surface area of the in-tank solids
SD   = in-tank solids density
τ   = solids retention time in the tank

From the above follows that alumina production in a precipitator tank and hence liquor alumina yield can be increased by:
–   Increasing the linear growth rate of hydrate in the tank;
–   Increasing the specific surface area of the in-tank solids;
–   Increasing the in-tank solids density;
–   Increasing the solids holding time in the tank.

The above variables are inter-related, e.g. an increase in SSA lowers the LGR, or reducing τ increases the LGR. A precipitation simulation model is required to iteratively assess the net effect of each of the above variables and their possible combinations.

At the operational level precipitation yield is primarily controlled by the following parameters:
–   Precipitation fill ratio;
–   Specific surface area of solids and the in-tank solids density;
–   Precipitation temperature profile;
–   Precipitation liquor caustic and impurities concentration.

However, precipitation yield should be maximized whilst still meeting alumina product quality specifications, leading to the precipitation dilemma.

Conflicting objectives in precipitation

Certain alumina product quality specifications, such as included soda, attrition index and particle size distribution are controlled in precipitation. Meeting quality specifications is often in conflict with maximization of precipitation yield.

In operating practice, the conditions in the first few tanks, the agglomeration section, are primarily set by product quality considerations and only in the following tanks are conditions set to maximize yield.

Precipitation fill ratio

Precipitation fill ratio is the A/C ratio of the mixture of green liquor, feeding precipitation and spent liquor, recycled with the seed charge. The more spent liquor is recycled, the lower will be the precipitation fill ratio. A reduction in fill ratio reduces the driving force for precipitation and hence reduces yield. In addition, a reduced fill ratio reduces the capacity to agglomerate fines, requiring a coarser hydrate seed, reducing the specific surface area of the in-tank solids, also reducing yield.

Installing seed filters, drastically reducing spent liquor recycle, addresses the above negative effects.

Liquor exposure

To crystallize alumina from liquor requires that the liquor is exposed to a large seed surface area (m[2] seed per liter liquor) for an extended period of time. In first approximation, a doubling of

seed surface area has the same effect as doubling holding time. Liquor exposure, being seed surface area x holding time captures the effects of seed surface area and holding time. Generally speaking it is more cost-efficient to increase seed surface area than holding time, explaining the drive to increase solids concentration in precipitator tanks. Seed surface area is again the combination of SSA and SD.

Typically the solids density in draft tube agitated, flat bottom precipitators is in the range of 400-600 g/l. Benchmark solids density in these type of precipitators is around 750-800 g/l. Solids densities close to 1000 g/l can be sustained with different agitation technology, but not in case spent liquor is recycled with the seed charge. This provides another justification for seed filtration.

It is however the combination of 1000 g/l in-tank solids densities (SD) with increased solid specific surface area (SSA) that has the potential to provide the seed surface area which is needed to break the 100 g/l precipitation yield barrier. The main challenge is to operate with the required finer hydrate inventory whilst producing sandy alumina and maintaining seed balance.

The use of crystal growth modifier allows for a finer hydrate inventory whilst still producing sandy alumina. However, a much more powerful method is fines destruction, which is widely used outside the alumina industry to control crystallization processes.

A fines destruction unit dissolves a controlled amount of excess fine seed in spent liquor. Recovered heat from the calciner alumina coolers could be used for this application. The dissolution of excess fines serves two purposes:
–   Control of the amount of fine seed, to match the capacity of classification and agglomeration systems.
–   The high A/C ratio liquor obtained after dissolution of the fines is utilized to increase the precipitation fill ratio, enhancing capacity to agglomerate fines and thus enabling the use of a finer hydrate inventory.

Application of fines destruction in the Bayer precipitation circuit is elaborated in a patent by Hiralal [5]. A simplified schematic of a fines destruction unit is shown in Figure 4 below.
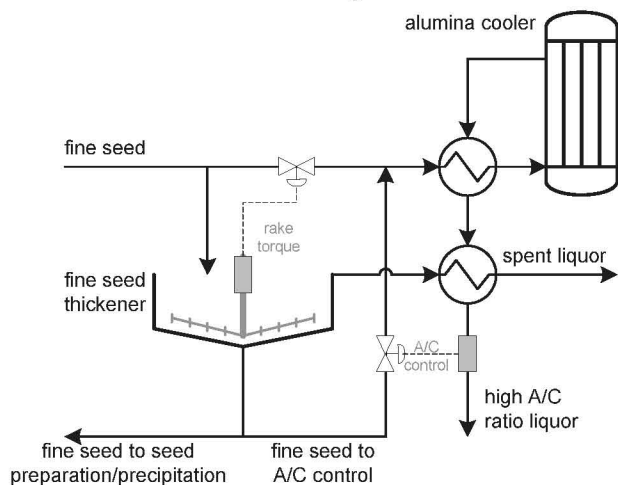

Figure 4. Simplified schematic of a fines destruction unit.

Installation of a fines destruction unit in an existing refinery is relatively easy because of the small footprint of such a unit and the minimal impact on the overall plant heat balance.

Precipitation temperature profile

The liquor temperature associated with the highest crystallization rate depends on the A/C ratio and caustic concentration of the liquor in the precipitator. The matter is a play-off between the Arrhenius factor, which favors higher temperatures in an exponential fashion and σ^2, which favors lower temperatures, i.e. there is an optimum temperature for each tank.

By increasing the number of inter-stage cooling steps it is possible to operate closer to the optimum temperature profile, as is illustrated in Figure 5.
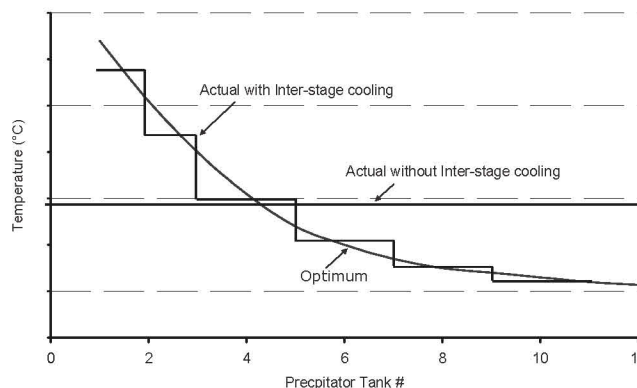

Figure 5. Approaching optimum precipitation temperature profile.

The actual number of inter-stage cooling steps depends on economics. The development of cost-effective heat exchangers, such as Barriquand platular heat exchangers, allows for installation of as much as 5 cooling steps in a precipitator train.

Precipitation fill liquor caustic and impurities concentration

Maximum precipitation yield requires an optimum liquor caustic concentration which is usually in the 260 to 300 g/l range. However, many plants don't operate precipitation at their optimum caustic concentration because of various process restrictions, including:
–   Constraints imposed on digestion extraction liquor caustic concentration, needed to control the risk of SSC of steel.
–   Constraints imposed on precipitation fill liquor caustic and impurities concentration, needed to manage oxalate co-crystallization in precipitation.

As discussed in the digestion section, the risk of stress corrosion cracking (SCC) of steel could put a limit on the spent liquor caustic concentration and hence on the related caustic concentration in precipitation liquor.

Co-crystallization of sodium oxalate with gibbsite usually has a disastrous impact on seed balance and alumina quality, especially if the co-crystallization occurs in the agglomerators. In this case solid phase oxalate (SPO) is included in the freshly formed agglomerates, increasing occluded soda content and resulting in severe particle breakdown in the calciners, adversely impacting on product alumina sizing and dusting behavior.

To keep precipitation free of SPO, often requires that oxalate is removed from the process in a dedicated oxalate removal unit, which processes a spent liquor side stream. The maximum spent liquor oxalate concentration, above which oxalate will co-crystallize is named the critical oxalate concentration (COC) and increasing the COC increases the effectiveness of the oxalate removal unit. The spent liquor COC can be increased by raising temperature and reducing total soda (TS) concentration, which includes soda impurities, such as sodium carbonate, sodium sulphate and sodium chloride.

For increasing oxalate removal requirement a higher COC is needed, requiring precipitation liquor with a higher temperature and a lower TS concentration, both adversely impacting on alumina yield. Hence in plants with a high oxalate removal requirement it is often not economic to combine an oxalate free precipitation circuit with a high yield. Instead oxalate is allowed to co-precipitate with the gibbsite, but only after the agglomeration section. In this situation SPO only binds to the outside of the hydrate crystals. Following hydrate classification, the size fractions are de-liquored on filters and the fine seed filter cake is washed with hot water to dissolve adhering SPO. The wash filtrate is further processed to dispose of the oxalate.

Increasing caustic concentration and hence TS in an oxalate co-precipitation system, moves incipient oxalate crystallization to higher temperature precipitators, which are closer to the front end of the train. The liquor in these precipitators has a higher alumina super-saturation level and the presence of SPO results in excessive nucleation, adversely impacting on precipitation seed balance. The corrective action is to increase precipitation liquor temperature and decrease caustic concentration, adversely impacting on alumina yield.

Following the above the processing of bauxites with low formation rates of oxalate and soda impurities is instrumental for achieving a high precipitation yield.

Removal of liquor soda impurities to increase yield has led to the development of a variety of technologies, targeting carbonate (causticisation), organic carbon (e.g. liquor burning, bauxite roasting, wet oxidation) or a range of impurities (e.g. salting out).

Precipitation process simulation

As described above, maximization of precipitation yield involves complex interactions between temperature, caustic concentration, particle surface area, liquor impurities as well as equipment performance characteristics and capability, etc. These interactions are further complicated by the requirement to meet alumina quality specifications and seed balance. Only an in-depth knowledge of the involved precipitation mechanisms in combination with a sophisticated and credible computer model, featuring the population effects of agglomeration, nucleation and growth and incorporating classification, seed recycle, inter-stage cooling, etc. can lead to successful retrofits/optimization of existing operations and the design of new circuits.

Precipitation conclusions

From the above it follows that a high precipitation yield requires:
– A high precipitation fill ratio, involving the separation of spent liquor from the recycled seed crystals.

– A high liquor exposure, combining seed with a high specific surface area with a high in-tank solids density.
– The use of fines destruction technology, enabling precipitation to operate with a finer hydrate inventory.
– Optimization of the liquor temperature profile involving installation of multiple inter-stage cooling steps.
– The use of bauxites with low rates of oxalate and soda impurities formation.

YIELD INCREASE SUMMARY

A summary of the principal yield increase measures and related capital cost is presented below for a typical 2 Mt/y alumina refinery. Each range of numbers is of an indicative nature only and actual numbers must be defined for each plant on the basis of its unique features.

– Introduction of sweetening digestion technology, using gibbsitic bauxite, in plants which process boehmitic bauxite at high temperature. Indicative yield increase: 6 to 10 g/l. Indicative capital cost estimate: 12 to 16 MUSD.
– Introduction of fines destruction technology, enabling precipitation to operate with a finer hydrate inventory. Indicative yield increase 4 to 8 g/l. Indicative capital cost estimate: 6 to 12 MUSD.
– Introduction of M2M-Technology enabling an increased green liquor A/C ratio whilst recovering alumina from bauxite residue. Indicative yield increase: 4 to 5 g/l. Indicative capital cost estimate: 8 to 14 MUSD.
– Introduction of seed filtration, increasing precipitation fill A/C ratio and fines agglomeration capacity. Indicative yield increase: 5 to 10 g/l. Indicative capital cost estimate: 20 to 30 MUSD.
– Introduction of as much as 5 inter-stage cooling steps, allowing for a precipitation profile which is close to the optimum. Indicative yield increase: 2 to 5 g/l. Indicative capital cost estimate: 6 to 12 MUSD.
– Substitution of evaporative cooling by direct cooling in the heat interchange department (HID). Indicative yield increase: 1 to 3 g/l. Indicative capital cost estimate: 5 to 10 MUSD.
– Introduction of high rate thickening technology for decanters. Indicative yield increase: 1 to 2 g/l. Indicative capital cost estimate: 9 to 12 MUSD (decanters only).

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