AA03 - Further Development of RUSAL’s Alumochloride Technology for Alumina Production from Non-Bauxite Resources

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Abstract

RUSAL is currently developing an alumochloride process for the production of smelter grade alumina using kaolin clays from domestic Russian deposits. The outcome of this project will be significant cost reductions for the transportation of bauxites and alumina. The chemistry and a mass balance of the process, and the quality of smelter grade alumina produced were confirmed at mini-plant scale, and the design of a pilot plant has been completed. The proposed process is characterized by low operating costs, flexibility in the raw materials it can use, and the production of high-margin products. In the course of development, several technology directions and process flowsheets have been tested to reduce heat consumption and the concentration of product impurities such as iron and phosphorus, and including the closed cycles of intermediate products: calcium chloride, aluminum oxychlorides, hematite, etc. The most profound modernization is the replacement of the thermal decomposition of aluminum chloride hexahydrate by its neutralization with ammonia. Further development and integration of technology options will enable further improvements to the existing benefits of the process and enhance the cost-effectiveness of the alumochloride technology.

Keywords: Alumochloride process, smelter grade alumina, ammonium chloride, boehmite.

1. Introduction

Some aspects of the hydrochloric acid (alumochloride) process for alumina production from kaolin developed by UC RUSAL’s Engineering & Technology Center have already been presented at previous ICSOBA conferences. A detailed description of the technology is documented in the relevant Russian patent [1]. The technology has been successfully tested at the specialized mini-plant, and as of today, the design of an industrial pilot plant has been completed. The new technology, named the ‘Lainer’ process (after Yuri A. Lainer - distinguished Soviet and Russian metallurgy researcher – who made valuable contributions to the development of an acid process for alumina production [2]), can be related to a number of hydrochloric acid (chloride) methods for the processing of aluminum non-bauxite raw materials [3].

In the almost forty years since the publication of this work, the following studies on this topic have been published: US Bureau of Mines [4], Orbite (Canada) [5], Nordic Mining (Norway) [6], Altech Chemicals (Australia) [7]. Shenhua Group (China’s largest mining and power company), in partnership with the GAMI Institute, have achieved notable success in establishing an industrial pilot operation in Inner Mongolia [8]. The company has successfully carried out pilot tests of their proprietary hydrochloric acid process for coal fly ash processing and is planning to build an alumina refinery with capacity of 125 000 tpa using coal fly ash as a raw material.

Based on the analysis of all known conceptual solutions, this study has concluded that despite the lack of rigorous process testing, the paper by Australian researchers from the Commonwealth Scientific and Industrial Research Organisation (CSIRO), describes the technology that most closely corresponds to the "conventional chloride process for alumina production" [9].

Unlike all the above-mentioned technologies, RUSAL's method [1] comprises acidic and alkaline processes that are characterized by a fine balance of flows/streams and accumulated impurities resulting in lower heat consumption and lower operating costs compared to traditional sintering and similar alumochloride processes. This method enables the production of sandy smelter grade alumina (SGA) that complies with all current requirements of primary aluminum producers.

In the course of RUSAL's technology development, several variants to the main process have been identified which can provide additional energy savings and allow simplification of acid-resistant equipment.

2. Reducing energy consumption at ACH calcination

First of all, this study focused on the calcination of aluminum chloride hexahydrate (ACH), the most energy intense process area. Estimated heat consumption amounts to 2300 kCal/kg Al_2O_3 but when operating heat losses are accounted for, it increases to around 3250 kCal/kg Al_2O_3 [10]. As per the known process solutions, crystallized ACH was calcined in two stages; firstly at a temperature of 200–400 °C, and in a second stage, at a temperature of 900–1100 °C. Water vapor may be optionally injected to the second stage. Calcination was performed either under standard or under highly hydrothermal conditions. This results either in high residual chlorine in the product (~0.1 %), or increased alpha-phase content in alumina (> 70 %) when water vapor was fed into ACH calcination at 1000 °C. Such alumina is not suitable for smelting.

For this reason, an alternative solution was proposed [11], where water vapor is fed to the second ACH calcination stage at a comparatively low temperature. Laboratory tests showed that at temperatures between 150–450 °C with continuous water vapor supplied, and provided that the ratio between the total mass of the supplied vapor and the mass of the alumina product is between 0.2 and 5.7, the second ACH calcination stage allows the production of alumina with low chlorine and alpha-phase contents.

Thermal decomposition of aluminum chloride hexahydrate is a variant of its thermal hydrolysis but in the technical literature this process is usually referred to as calcination and represented by the following overall equation:

$$2AlCl_3{\cdot}6H_2O = Al_2O_3 + 6HCl + 9H_2O \tag{1}$$

In fact this process comprises a number of intermediate transformations of basic aluminum chlorides (oxychlorides) of different basicity and hydration degrees. Some of these intermediate are not necessarily sequential but can be presented as follows:

$$AlCl_3{\cdot}6H_2O \rightarrow Al(OH)Cl_2 + 5H_2O + HCl \tag{2}$$

$$Al(OH)Cl_2 + H_2O \rightarrow Al(OH)_2Cl + HCl \tag{3}$$

$$2Al(OH)_2Cl + H_2O \rightarrow Al_2(OH)_5Cl + HCl \tag{4}$$

$$Al_2(OH)_5Cl + H_2O \rightarrow 2Al(OH)_3 + HCl \tag{5}$$

Reaction (2) generates sufficient water to stoichiometrically complete the overall reaction (1). However, the reference data and the results of the research conducted show that at temperatures between 100 and 500 °C the reactions stop when the material becomes a mix of aluminum oxychlorides because waters leaves the reaction volume as vapor and does not react with the intermediate decomposition products as indicated in reactions 3–5. To enable further transformations, the temperature is increased to 900–1000 °C in the presence of flue gases (as implemented in the prototype). The water produced by organic fuel combustion is important, but not sufficient for fast and efficient alumina production.

The result is very different if after aluminum oxychloride generation, water vapor is added to the process. Then reactions (3)–(5) proceed smoothly and aluminum oxide is formed by the hydrothermal mechanism as described by the literature. With excess water, hydrolysis is extensive and irreversible, the residual chlorine in the end product is minimal as is the alpha-phase content due to conditions not favoring its generation. Consequently, the second high-temperature calcination stage is not required, and as a result, energy consumption is approximately halved.

This process solution is then as follows; Al-bearing raw material is leached with hydrochloric acid before solids are removed and the clarified chloride solution is evaporated or bubbled with hydrogen chloride gas to precipitate ACH crystals. The crystals obtained are heated in a kiln at a temperature of 110–250 °C to form aluminum oxychloride, inhibiting further chemical reactions due to exhaustion of water generated during the hydration of initial product (the first stage). In the second stage, a temperature of 150–450 °C is sufficient, reducing heat losses. The nominated ratio between the total mass of the supplied vapor and the mass of the alumina product of 0.2–5.7 provides for minimum heat consumption for vapor superheating, depending on the selected kiln technology, as conditions for the reaction of solids and gaseous (vapor) phase differ.

As water vapor is supplied only to the second stage of ACH decomposition, overall vapor consumption decreases while maintaining high quality alumina product and process performance. Preliminary estimates suggest that total heat consumption for ACH decomposition can be reduced by up to 50 %.

The impact of mildly hydrothermal conditions on ACH calcination was studied. A 100 g kaolin sample (36.4 % Al_2O_3; 45.3 % SiO_2; 0.78 % Fe_2O_3; 0.51 % TiO_2; 0.96 % CaO; 0.49 % MgO) was mixed with 25 % hydrochloric acid at a liquid to solid ratio of 4 to 1, then poured into the PTFE digester and held for 3 hours with constant stirring. The slurry obtained was filtered and the clarified chloride solution was bubbled with hydrogen chloride gas to precipitate ACH crystals. The crystals were washed with 38 % hydrochloric acid on the filter. The process yield was 151.3 g $AlCl_3 \cdot 6H_2O$. The ACH crystals were placed into the laboratory tube furnace at 200 °C for 0.5 hrs for the first decomposition stage. The second decomposition stage was performed by supplying water vapor to the furnace from a vessel boiling water. Vapor consumption was determined by the mass water boiled-away during the test period. The ratio of the mass of supplied vapor to the mass of alumina product was maintained in the range of 0.2–5.7. The residence time of the second stage did not exceed 0.5 hrs.

Other tests were carried out under various second stage conditions. Table 1 shows the results of the tests which indicate that the products obtained in tests 1, 2 and 12 did not comply with current requirements for smelter grade alumina (alpha-phase content of 10 % or less, chlorine content of 0.01 % or less). The other tests met the SGA specification.

Optimum process parameters and residence time need to be determined for each type of kiln, along with the amount of water vapor added to the process to ensure acceptable alumina quality, process performance and the minimum consumption of heat energy. It should be noted that the conditions for reaction of the vapor with solids are less favorable in the laboratory tube furnace compared to industrial kilns where solids are mobilized or move in a fluidized bed. However, the advantages of the process as compared with the prototype are obvious.

For industrial-scale implementation two consecutive kilns are recommended. This enables carrying out two stages of ACH decomposition in a continuous mode, while supplying water vapor to the second kiln only.

Table 1. Results of experiments on ACH decomposition at soft hydrothermal conditions.

Test #	**Second Stage of ACH Decomposition**		**Alumina Phase Composition (XRD)**	**Chlorine (Cl^-) Concentration in Alumina, %**
	Temperature, °C	**Residence time, min**		
1	150	20	gamma	0.052
2	150	40	gamma	0.023
3	150	60	gamma	0.009
4	250	20	gamma	0.010
5	250	40	gamma (trace of alpha)	0.008
6	250	60	gamma (trace of alpha)	0.004
7	350	20	gamma (trace of alpha)	0.004
8	350	40	gamma (trace of alpha)	0.0035
9	350	60	gamma and <3 % alpha	0.0032
10	450	20	gamma and 3 % alpha	0.0028
11	450	40	gamma and 6 % alpha	0.0023
12	450	60	gamma and 12 % alpha	0.0023

The process described in the 2013 UC Rusal patent [12], can be considered a variant of the above method, enabling the recycling of the intermediate product of partial ACH decomposition. In this case, the first stage of ACH thermal decomposition is carried out to form oxychlorides and amorphous aluminum hydroxide, part of which is gradually added to the clarified solution before ACH crystallization to reach a pH of 1.6–2.2. Iron hydroxide precipitates is separated and mixed with mother liquor, and a part of the hematite obtained is added as seed to the clarified solution at a pH of 1.6–2.2.

The results indicate that a neutralizing agent can be obtained using the hydrochloric acid (chloride) method at the first stage of ACH thermal decomposition from the generation of aluminum oxychlorides of various basicity and X-ray amorphous reactive aluminum hydroxide by reactions (2)–(5).

These aluminum compounds are readily soluble in hydrochloric acid solutions and can increase the liquid pH up to 1.6–2.2. Such neutralizing agents can be easily regenerated by repeated thermal decomposition. So balanced recycling of such agents can be arranged without addition of alkaline reagents to the acidic circuit. This allows improved alumina quality, decreasing product Fe_2O_3 by 0.003–0.007 % (absolute) without any additional costs.

3. Introduction and Circulation of $CaCl_2$

Another variant of the conventional process is the injection of calcium to the process circuit in form of lime [13] (Figure 1).

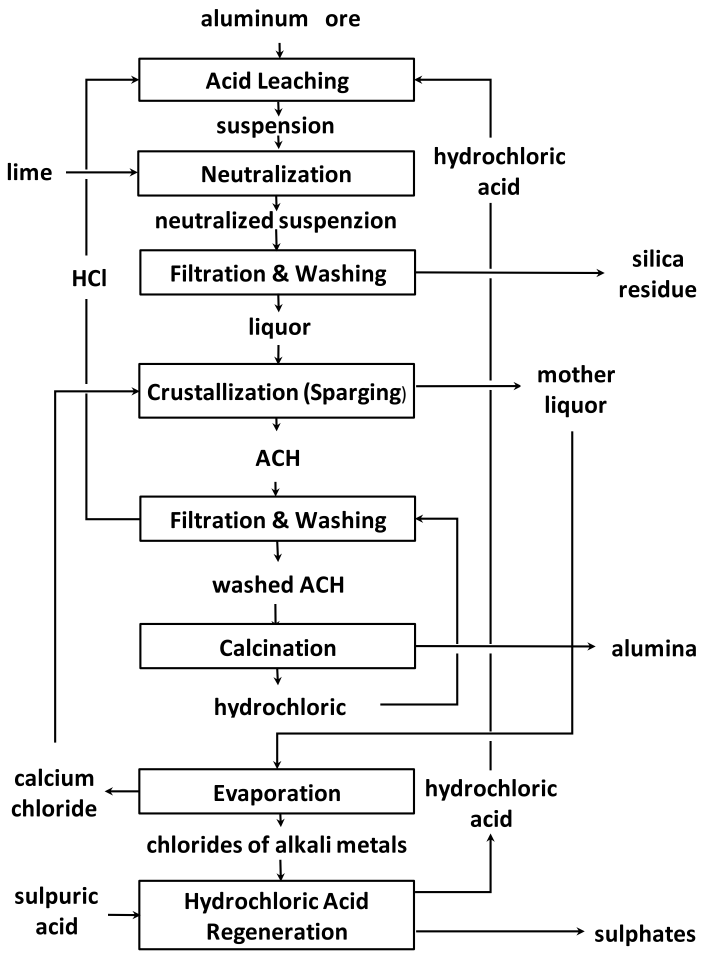

Figure 1. Hydrochloric acid (chloride) process with calcium chloride recycling.

After digestion of the Al-bearing raw material with hydrochloric acid, the chloride slurry is neutralized with calcium oxide to a pH of 4.6–2.2. In this case, ferric iron precipitates as hematite and phosphorus precipitates as calcium phosphate. The insoluble precipitate (Si-residue) is separated. The pH range is selected to ensure the complete removal of iron from solution but without auto-precipitation of aluminum chloride, which would result in losses of product with Si-residue. Calcium chloride (as a saturated or supersaturated solution, or crystals) is added to the clarified chloride solution obtained to crystallize aluminum chloride hexahydrate.

Aluminum chloride hexahydrate crystals are washed with >30 % pure hydrochloric acid to remove the residual mother liquor. The hydrochloric acid wash is returned to the initial acid digest of incoming Al-bearing raw material. Washed ACH crystals are thermally decomposed to produce alumina. Hydrogen chloride recovered in the process in the form of hydrochloric acid is returned to wash ACH crystals. If only a part of the recovered acid is needed for washing, the remaining part is used in the acid digest of Al-bearing raw material. The mother liquor is evaporated in stages for selective crystallization of alkali metal chlorides, and separation of calcium chloride crystals or concentrated solution, or a mixture thereof (i.e. supersaturated solution); wherein a part of calcium chloride is returned to ACH crystallization.

If calcium chloride crystals are fed to ACH crystallization, they are thermally treated at a temperature of > 45 °C to transform crystalline hydrates into dihydrate form, improving their crystallizing properties.

As the method stipulates addition of calcium as oxide, an excess of calcium chloride is generated in the circuit, so the remaining portion is directed to regeneration with alkali metal chlorides to obtain hydrochloric acid which is supplied to acid digest of Al-bearing raw material.

Regeneration is carried out by treating the remaining calcium chloride and alkali metal chlorides with sulfuric acid. Sulfates generated are removed from the process.

Laboratory tests were carried where kaolin (Al_2O_3 36.4 %, SiO_2 45.3 %, Fe_2O_3 0.78%, TiO_2 0.51 %, Na_2O 0.05 %, K_2O 1.74 %; CaO 0.96 %, MgO 0.31 %, P_2O_5 0.12 %) was treated with 20 % hydrochloric acid at 170 °C for 3 hours with constant stirring in laboratory digesters at a stoichiometric ratio of 1.05, providing for the transformation of total Al, Fe, Ca, Mg, Na and K oxides into chlorides. Calcium oxide was added to the slurry obtained at 100 °C with constant stirring to reach a pH of 1.6–2.2 and stirred for one hour.

Upon completion of the process, the slurry was filtered. The clarified solution obtained was poured into the rotary evaporator at a temperature of 80 °C. The solution or crystals of calcium chloride obtained in the previous tests were gradually added to complete ACH crystallization. ACH crystals were separated and washed on the filter with two portions of pure 38 % hydrochloric acid. The washed crystals were placed into the laboratory tube furnace, heated to 1100 °C and held for 3 hours. The alumina product was analyzed for Fe_2O_3 and P_2O_5 content.

The remaining mother liquor was evaporated in the rotary evaporator under vacuum to crystalize alkali metal chlorides and part of the calcium chloride. The evaporated liquor was used in further tests for ACH crystallization.

Crystals of alkali metal chlorides and calcium chloride were treated with concentrated sulfuric acid at a temperature of 60 °C. When the solution obtained was cooled, the crystallized sulfates were separated and regenerated hydrochloric acid made up to 20 % concentration and used in further tests.

In some tests calcium chloride crystals obtained from mother liquor evaporation were dried at a temperature of >45 °C to transform all crystalline hydrates into dihydrate form. Apart for improving their crystallizing properties it also facilitates the circuit's water balance without compromising the quality of alumina product.

In all tests which used lime, the required Fe_2O_3 (≤ 0.015 %) and P_2O_5 (≤ 0.001 %) were achieved, whereas without lime addition, these values were much higher at 0.03 % and 0.003 % respectively.

In addition to obtaining product of higher quality, the evaporation of liquors containing calcium chloride required less energy, as the presence of calcium shifts the azeotrope of the system. Calcium chloride containing liquors are also less aggressive, so the corrosion resistance requirement for equipment is lower.

4. Chlorammonium Variant

An even more unique variation of the hydrochloric acid process is an ammonium chloride version which can exclude the use of complex ACH [14]. Figure 2 shows the process flow diagram of this method. It comprises two closed circuits: hydrogen chloride and ammonia. Both circuits are interconnected by ammonium chloride.

The alumina production process starts with the Al-bearing raw material being mixed with ammonium chloride and then roasted, where the raw material is partially activated, and ammonium chloride decomposes. Hydrogen chloride reacts with the raw material's oxides and free ammonia is dissolved in water to form aqueous ammonia. These reactions constitute the roasting and chlorination stage of raw material processing.

To completely dissolve the valuable components, the roasted raw material is treated with hydrochloric acid, with further separation (e.g. by filtration) of the solids (mainly Si-residue) and the aluminum containing chloride solution. The solution is bubbled with hydrogen chloride gas to crystallize and precipitate ACH. These crystals are neutralized with aqueous ammonia (from the roasting stage) to form partially dehydrated aluminum hydroxide (boehmite) and an ammonium chloride solution. Boehmite is fed to calcination to obtain an alumina product. Mother liquor from hexaqua aluminum chloride crystallization is directed to pyrohydrolysis. Impurity metal hydroxides and oxides, mainly hematite, are formed and hydrochloric acid is regenerated as both aqueous solution and hydrogen chloride gas are used for hexaqua aluminum chloride crystallization. Ammonium chloride solution is evaporated in stages with repeated use of the heating steam.

Multiple stage evaporation reduces heat consumption compared with conventional ACH calcination, where all water generated during crystalline hydrates decomposition is evaporated in one stage [9]. Water of crystallization is approximately 80 % of ACH mass.

In total, it reduces specific heat consumption for alumina production from kaolin from 34.6–37.1 kJ/kg Al_2O_3 using conventional alumochloride technology [8], to 32.23 kJ/kg Al_2O_3. Moreover, boehmite calcination consumes only 2.15 kJ/t. One process issue that remains unsolved is the design of a chlorination vessel, as for the laboratory tests a small acid-resistant reactor equipped with a throttle valve to remove excessive ammonia was used.

Alumina produced by this method complies with standard Russian requirements for G-0 smelter grade alumina in terms of chemical composition and particle size distribution. The product particles are brittle however, and have a high attrition index, and a bulk density of 0.3–0.6 kg/dm^3 (Table 2). Alumina produced from this raw material by the CSIRO method contained unacceptably high amount of phosphorus that is probably an issue that is impossible to solve. The technology does not provide any ways to remove phosphorus and it passes from the raw materials to the product.

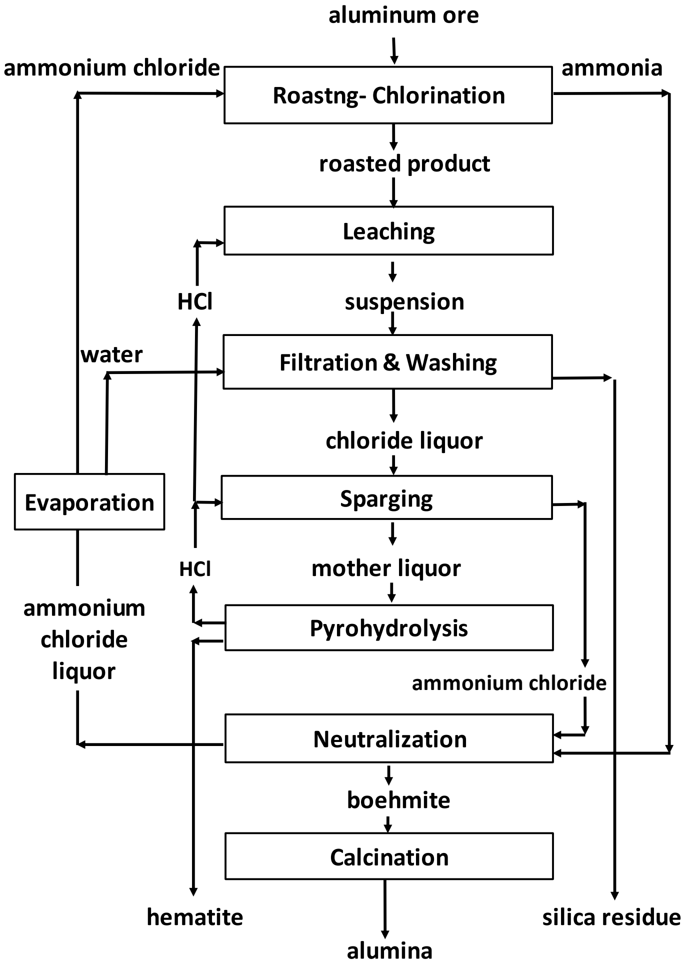

Figure 2. Process flow diagram of a ammonium chloride technology for alumina production.

Table 2. Quality of alumina obtained by ammonium chloride technology in comparison with other products.

SGA	Concentration of impurities, %							LOI (300-1100 °C), %	-45 µm, %
	SiO_2	Fe_2O_3	TiO_2+ V_2O_5+ Cr_2O_3+ MnO	ZnO	P_2O_5	Na_2O+ K_2O (as Na_2O)	Cl^-		
RUSAL	0.01	0.021	0.015	0.01	0.001	0.3	0.008	0.8	12
Russian Standart (G-0)	≤ 0.03	≤ 0.05	≤ 0.02	≤ 0.02	≤ 0.002	≤ 0.5	-	≤ 1.2	≤ 25
CSIRO [8]	-	0.023	-	-	0.03	-	0.03	0.14	12

The method can be improved by implementation of water leaching of the ore after chlorination [15]. The viability of this stage is obvious, taking into account that during the kaolin roasting iron chlorides are mainly formed. For that reason, it was decided to extract them from the roasted material by preliminary water leaching to prevent their ingress into the aluminum chloride solution by acid leaching. To minimize supply of additional water to the process circuit wash water from other areas, i.e. from Si-residue washing, can be used for water leaching of the roasted material. The iron chloride solution is directed to pyrohydrolysis. Thus, iron content in roasted alumina can be reduced to 0.006 %.

Ammoium chloride technology produces alumina with gradient porous structure of a cell-type [16] that can be used as a catalyst carrier, or sorbing agent with specific surface area of approx. 250 m^2/g or a dehumidifier with high moisture retention capacity especially as condensed water.

5. Conclusions

In the course of the development of a competitive alternative technology for alumina production from high-silica raw materials, RUSAL has developed and proven an effective basic method for production of smelter grade alumina at mini-plant scale. To improve its performance the following variants of the processes of this technology have been tested at laboratory scale.

1) To enhance Lainer technology, aluminum chloride hexahydrate is decomposed in the presence of water vapor (under hydrothermal conditions). This reduces fuel consumption and the process temperature to between 150-450 °C, while obtaining a product with low chlorine and alpha-phase contents.

2) Laboratory tests show that if the first stage of thermal decomposition is carried out until oxychlorides and amorphous aluminum hydroxide are formed, then part of these ACH incomplete decomposition products can be added to the alumochloride solution as seed before ACH crystallization to reach a pH of 1.6–2.2, preventing iron hydroxide precipitate from ingress to the crystallized product.

3) Experiments have shown that addition of calcium chloride to the Lainer circuit reduces heat consumption for rectification of recycled hydrochloric acid. This method also provides for significant (down to 0.0023 %) reduction of phosphorous pentoxide in alumina. Phosphorous pentoxide is a difficult impurity that easily passes from the raw material into the product in acid processes.

4) A variant of the alumochloride technology was proposed where the ACH thermal decomposition is replaced with treatment of this intermediate product with an aqueous ammonia solution. As a result, aluminum oxyhydroxide (boehmite) is formed and directed to production of calcined alumina. This ammonium chloride technology comprises two interconnected circuits, (hydrochloric acid and ammonium), and reduces energy consumption and improves alumina quality. The ammonium chloride process provides opportunities for further fine-tuning and obtaining value-added products.

All the above variants of upgraded alumochloride technology have potential to further improve the current version of the process. Further extensive lab and mini-plant scale tests are required before they can be integrated into more developed processes.

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