

**INTERNATIONAL COMMITTEE FOR STUDY OF
BAUXITE, ALUMINA AND ALUMINIUM
ICSOBA
NEWSLETTER**



View of the 1.07 MTPY Achinsk Alumina Refinery of RUSAL, processing nepheline feedstock

A biannual publication

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In case you consider publishing in this forum, please contact the editor before writing your article.

Deadlines for a June issue is 10th of June and for a December issue 10th of December.

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FOREWORD



Dear ICSOBA Members!

This year, ICSOBA reached a new milestone: 50 years of existence. Half a century of vocation for an organization such as ICSOBA is quite an achievement by all means. Many entities created around the same time no longer exist today, but ICSOBA is alive and doing well. 50 years of service dedicated to the Al-community is a wonderful achievement. In the last 50 years, 32 ICSOBA events - symposiums, congresses and seminars were organized in 15 countries in different parts of the world. Most events took place in Europe in the first 25 years with bauxite and alumina being the predominant subjects. The next 25 years witnessed ICSOBA travel to other parts of the world such as Brazil, Iran, Jamaica, Russia, India, Canada and China. Consequently, ICSOBA has become a well-recognized and truly international organization. A TRAVAUX volume documenting conference proceedings was published following each event.

ICSOBA is genuinely a dynamic, healthy international organization and it is a pleasure to be a part of the team. With the quality of people that attend ICSOBA events and their commitment to excel, we all may be assured that next 50 years will be just as successful as the first. Congratulations and thanks to all of those who maintained the integrity and values that were fundamental to our association from its beginning. If you come to Krasnoyarsk in September, you will feel lucky, honored, and excited to participate in this historical event. Commemorative 50-anniversary pins will be distributed among all ICSOBA participants.

As the preparations for the conference continue, more and more delegates register and confirm their papers. In mid-May there were approximately 120 submitted papers. There were also 30 exhibitors already registered for the Krasnoyarsk conference.

We trust that they all will take opportunity that the event offers to increase their international visibility and grow networking. More about the conference program you may read in ICSOBA website www.ICSOBA.info.

There are several field trips, which have been announced. Among the field trips two will be dedicated to smelters. One will be a local visit to Krasnoyarsk smelter, which is of Söderberg type. The other smelter possible to visit is the Khakas smelter, which is not that far from Krasnoyarsk.

The first five Rusal's RA-300 pots began operating there in 1997. In 2007 eight RA-400 pots were mounted at the smelter. Another interesting trip will be to Achinsk Alumina Refinery, which is Russia's largest producer of alumina. The plant produces alumina from nepheline ore and lime using a unique technology developed by Rusal.

Later, when you forego walks along the Yenisei River or visit Stolby national park you will appreciate the part of Siberia nature, which is so inaccessible to most.

I want to personally thank you for being part of our community. Your journey is part of our journey, and we're delighted and humbled when we hear stories of how our members are using ICSOBA to connect, learn, and find opportunity. Please register for the Krasnoyarsk conference if you have not done so yet. The registered delegates will remain ICSOBA individual members until July 2014.

All of us come to work each day focused on our shared mission of connecting the world's professionals to make them better informed, and consequently become more productive and successful. As we close the first half century of activity we already think about future events. We're excited to show you what's next.

I look forward to meeting you in Krasnoyarsk and to working with each of you to sustain and grow our professional society.

Thank you for this great opportunity.

Best regards

Frank R. Feret
President, ICSOBA

NEWS AND EVENTS

Financial aspects of operation of ICSOBA

Following the financial audit carried out by accounting office of Paul Bussiere of Saint Jerome, Quebec and corresponding tax declarations, both Canada Revenue Agency and Quebec Revenue Agency have confirmed as of May 16, 2013 that ICSOBA does not owe any taxes for the 2012 financial year.

ICSOBA-2013, the 31st Conference in Krasnoyarsk, Russia

The International Committee for Study of Bauxite, Alumina & Aluminium (ICSOBA) has great honour to announce the 31st International Conference and Exhibition of ICSOBA. The event will be held in the Siberia hotel of Krasnoyarsk in Russia (Siberia) from 3 to 6 September, 2012 in cooperation with UC RUSAL and Non-Ferrous Metals (NFM), in conjunction with the V International Congress & Exhibition "NON-FERROUS METALS – 2013.

Objectives of the Conference are:

1. to review the status of bauxite, alumina and aluminium industries in the world with emphasis on Russia;
2. to discuss promising research developments aimed at production, productivity and cost improvements;
3. to highlight proposed Greenfield and Brownfield activities in the aluminium industry;
4. to discuss developments in the field of environment and safety;
5. to update market aspects of bauxite, alumina and aluminium and their products;
6. to provide an excellent opportunity to interact with international experts, scientists, engineers, technology suppliers, equipment manufacturers and representatives of aluminium industries the world over.

There will be technical excursions hosted by UC RUSAL to the Krasnoyarsk Aluminium Smelter and Hydro dam or Krasnoyarsk Non-Ferrous Metals Plant included in the program and optionally also to the Achinsk Alumina Refinery and the Khakass Aluminium Smelter in the Krasnoyarsk region.

We look forward to seeing you in September 2013 at the ICSOBA-2013.

Dr. Frank Feret, President ICSOBA

Peter Polyakov, president NFM

For more information please, visit the ICSOBA website www.icsoba.info

ICSOBA's policy for reimbursement of cost for participating in the ICSOBA conference

ICSOBA has a formally adopted Payment Policy that governs the payment of fees, stipends, allowances and the provision of reimbursements to members. Below text describes when the cost of participating in an ICSOBA conference may be on account of ICSOBA.

In order to qualify for total or partial reimbursement of costs without special decision of the directors all items below must be true:

1. Person is an individual member or one of the nominated employees of a corporate member;

2. Person has assumed an official function, such as Organizing committee member, Director, Newsletter editor, Council member;
3. Person's presence is required and key to the success of the conference (presenting a paper or chairing a session is not sufficient);
4. the costs for participating in the event are not absorbed by a 3rd party employer (retired, self-employed, or employer does not want to support ICSOBA);
5. total reimbursement only as long as total amount of reimbursements remains within event budget, otherwise partial reimbursement .

In order to qualify for a grant from ICSOBA's Subsidy Fund:

- the member has to demonstrate that he/she does not have sufficient means to participate in the Event;
- the member makes (or has made) a contribution that is considered important for the functioning or continuity of ICSOBA;
- there are sufficient monies in the Subsidy Fund;
- the approval of at least 3 directors has been obtained.

The maximum amount of the grant varies, in accordance with the member's contribution, from free registration fee to reimbursement of reasonable accommodation and / or travel expenses.

TECHNICAL PAPERS

Bauxite resources of Venezuela and their commercial potential

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Abstract

Through detailed current studies, it has been determined that bauxite resources in the Venezuelan Guayana (Guiana) Shield may reach 2.621 Gt. Venezuela might become third in the worldwide ranking, only behind Guinea and Australia, after reserves certification. The largest potential area is called La Cerbatana, and is located in the northern area of the *Cedeño District Bauxite Quadrangle*, with more than 680 Mt. It has been suggested to accomplish a detailed geological prospection campaign to get reserves certification, as a target in middle term, in order to get enough raw materials for future aluminum projects.

Keywords: Bauxite resources, Guayana Shield, worldwide ranking, La Cerbatana, reserves certification.

1. Introduction

The Venezuelan Guayana (Guiana) Shield bauxite deposits have been subdivided in two classes, according to their parental rocks (modified from Mariño et al., 2007; 2010), which vary from the weathering of quartz-syenite with *rapakivi* texture, to orogenic granitoids and diabase sills, all of Mesoproterozoic age, resulting in bauxite of:

- a) Highest potential: granitic bed rock, with higher percentages of alumina (Al_2O_3), up to 52%; and
- b) Lowest potential: Mafic bed rocks, with high iron content ($Fe_2O_3 > 30\%$).

The geographical location of all of these bauxite resources in granite bed rock in the Venezuelan Guayana Region is shown on Figure 1, updated in May 2012.

In order to locate and outline possible bauxite resources nearby Los Pijiguaos area, *Cedeño District Bauxite Quadrangle (CDBQ)* is proposed as a new name (modified from Mariño et al., 2007; 2010) with 1.478 Gt inferred mineral resources (Fig. 1).

2. Planation surfaces and bauxite resources in the Venezuelan Guayana (Guiana) Shield

Systematic exploration was first carried out by the Ministerio de Minas e Hidrocarburos (MMH) and the Corporación Venezolana de Guayana (CVG) later on, since the early 1940's. Target areas were selected mainly on the basis of geological and geomorphological criteria, guided by correlation with known deposits elsewhere in the Guayana Shield (Menendez and Sarmentero, 1984). The Venezuelan Guayana Shield has been subdivided into four geological provinces north of parallel 6° by Menendez (1968): Imataca, Pastora, Cuchivero and Roraima. Of these the Cuchivero Geological Province has the best prospect for bauxite exploration and it is well known that the planation surfaces are the key to find bauxite deposits in the area (Fig. 2). The

geomorphology of Los Pijiguaos area is characterized by an uplifted and deeply dissected, regional-scale plateau at an elevation between 600 and 700 m above sea level (m.a.s.l.), termed the Nuria surface (Meyer et al., 2002) (Fig. 2), which represents the final stage of an intense erosional period that took place in the Venezuelan Guayana Shield during Late Cretaceous to Early Tertiary times (McConnell, 1968 in Meyer, et al., 2002)

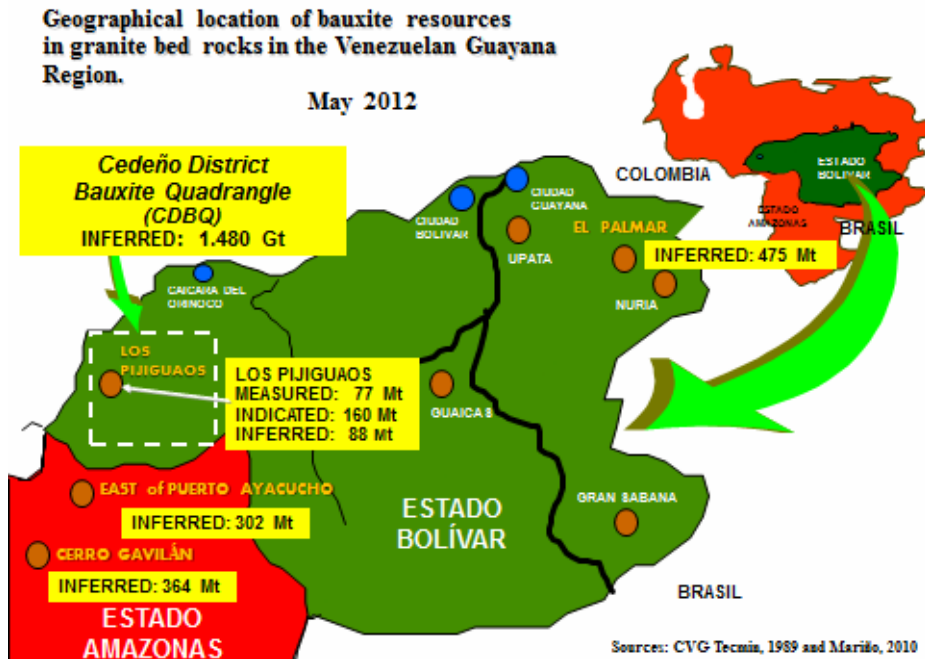


Figure 1: Geographical location of bauxite resources in granite bed rock in the Venezuelan Guayana (Guiana) Region.

The bauxite reserves in Venezuela were estimated by the US Bureau of Mines (Baumgardner and McCawley, 1983) to be 235 Mt and these same authors suggested inferred resources of 6 – 8 Gt in Los Pijiguaos area. Menendez and Sarmentero (1984: 404) pointed out that the remnants of plateau surface in which bauxite occurs extend over a total of 350 km², suggesting that the Pijiguaos Mountains contain 5.8 Gt of possible bauxite reserves. Yanez (1995) indicated 1Gt and Mendoza (1972, 2000, 2012: 303) around 4 Gt. And finally, Robb (2005: 226) indicated that “one of the largest bauxite deposits in the world is at Los Pijiguaos in Venezuela... a total resource that could be as much as 6 billion tons”.

A detailed geomatic study of the adjacent bauxite deposits of Los Pijiguaos has been conducted since 2003 and according to Mariño et al. (2007; 2010), and update in this report, the remnants of Nuria surface in CDBQ bauxite resources may reach 1.48 Gt (Fig. 3). The total of high potential granite bed rock bauxite resources in Guayana Shield, with higher percentages of alumina (Al₂O₃) up to 52%, may arise 2.621 Gt (Fig. 3). An increment of 405 Mt has been determined since last updated in 2010.

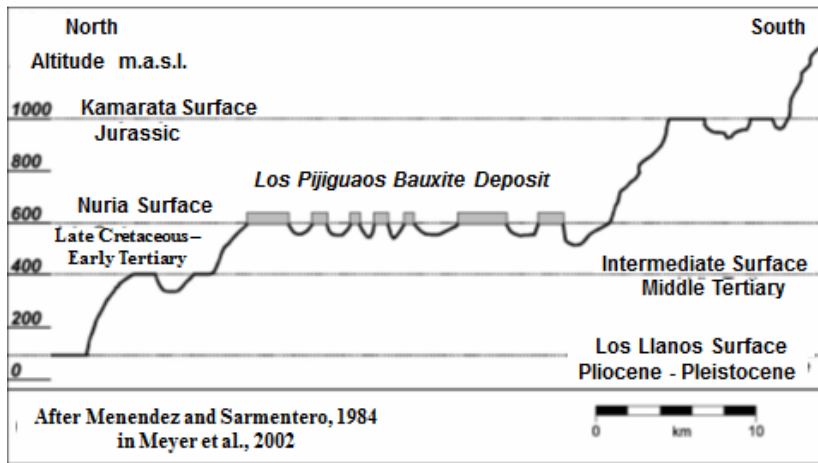


Figure 2: Schematic N-S profile of Los Pijiguaos region showing the position of planation surfaces (After Menendez and Sarmentero, 1984 in Meyer et al., 2002).

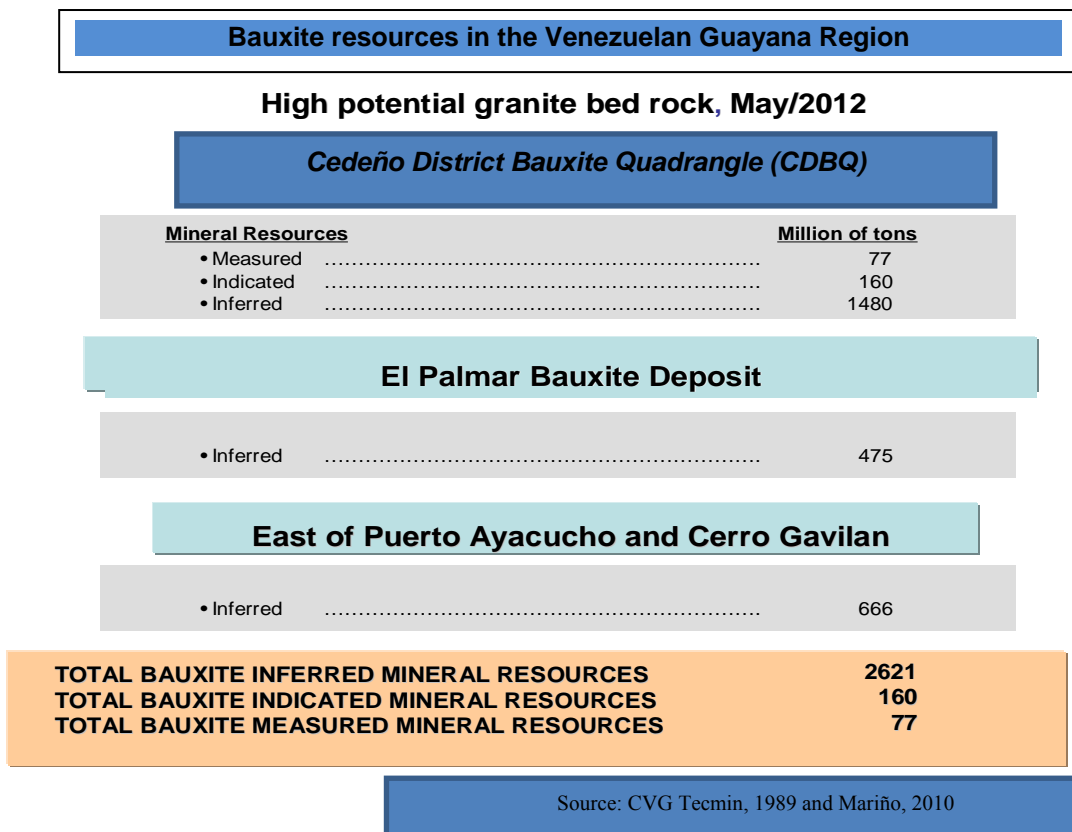


Figure 3: Bauxite resources in granite bed rock in the Venezuelan Guayana (Guiana) Region.

3. Discussion

Although previous authors point out a great volume of bauxite resources in Venezuelan Guayana Shield, different points of view on the definite quantity of this volume were appreciated. Meyer et al. (2002) referred that the figure of 6 Gt of probable reserves was estimated from Menendez and Sarmentero (1984), who considered the area of plateau remnants of Nuria surface, on which bauxite occurs, to extend over a total area of 350 km² and consider the current estimation shown at Fig. 3 “*much more reliable*”, (Meyer, pers. comm., 2012)

According to what has been described, Venezuela ascends to the third place in the worldwide ranking among countries with larger bauxite resources and first position in the Americas (Fig. 4), but only has 77 Mt of proven reserves at Los Pijiguaos CVG Bauxilum Mine, by December 2011. As an economic geology consideration, this mean only 16 years of production at 4.8 Mt per year. A long-term plan does not exist. That is a great limiting factor and it is a very low figure to guarantee the service and life of the only bauxite mine of the country, including also the survival of the Venezuelan Aluminum Sector.

| Countries | Bauxite Resources Mt |
|--------------|-------------------------|
| Guinea | 7,400 |
| Australia | 6,200 |
| Venezuela | 2,621 (*) |
| Vietnam | 2,100 |
| Jamaica | 2,000 |
| Brasil | 1,900 |
| China | 750 |
| India | 770 |
| Guyana | 700 |
| Greece | 600 |
| Suriname | 580 |
| Kazhakastan | 380 |
| Russia | 200 |
| Others | 3,200 |
| Total | 29,381 |

(*) Total Bauxite Inferred Resources

Source: Mibam, 2010

Figure 4: Worldwide bauxite reserves in Mt (Mibam, 2009) and position of Venezuela.

As a positive assessment of the commercial potential of world bauxite deposits, Bárdossy and Bourke (1993) ranked Los Pijiguaos among the top three candidates. Their ranking was based on mineralogical and chemical parameters that control the efficacy of the Bayer process and non-ore related criteria such as ease of mining, environmental aspects, and infrastructure (Meyer et al., 2002).

A prospecting plan to evaluate all mineral bauxite resources in western Bolívar State has been proposed to local authorities. The biggest potential area is called La Cerbatana with 96 km² and it is located in northern *CDBQ*, with more than 680 Mt of bauxite resources in two different

deposits: north area with 325 Mt (CVG Tecmin, 1989) and south area with 355 Mt (Ruiz and Villegas, 2008). (Fig. 5). The goal will be to measure and to certify mineral resources in all *CDBQ* in a suggested plan between three to five years, always depending on weather factors and the productivity of the drilling crew. An initial investment of US \$ 15,000,000 has been estimated in a proposed plan of 250 km² drilling program (modified from CVG Tecmin, 2006). No road exists yet to get La Cerbatana bauxite deposits neither in other areas of *CDBQ*.

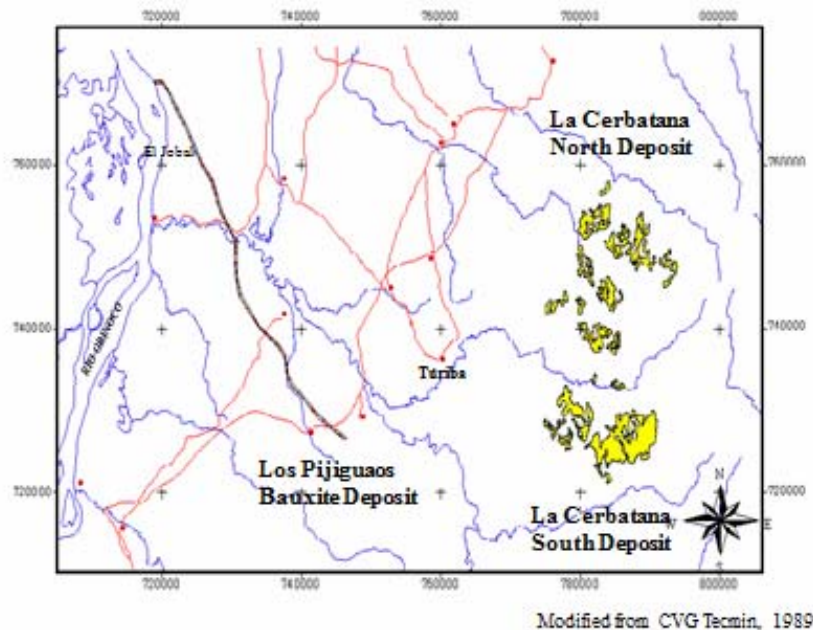


Figure 5: Relative position of La Cerbatana Bauxite Deposits with Los Pijiguaos Bauxite Deposits.

4. Conclusions

- ✓ Los Pijiguaos could be considered in a middle term as one of the world's main bauxite deposits, with huge inferred resources, but many areas remain without detailed geology studies.
- ✓ The total of high potential granite bed rock bauxite resources in the Venezuelan Guayana Shield, with high percentages of Al₂O₃ (up to 52%), may reach up to 2.621 Gt.
- ✓ It is considered that this value of inferred resources of bauxite is "*much more reliable*" for future studies and estimations, than other much larger estimates.
- ✓ Venezuela may become third producer in the worldwide ranking, only behind Guinea and Australia, after reserves certification.
- ✓ The largest potential area is called La Cerbatana, which could host more than 680 Mt.
- ✓ A prospecting plan to evaluate all bauxite resources in western Bolívar State has been proposed to local authorities and it could cost US \$15,000,000 in 250 km² drilling program.

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Tendencies in the design of bauxite processing plants and alumina refineries

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ABSTRACT

Recently designed preparation plants and refineries introduced new technical options to reach higher operational efficiency and mitigation of environmental impacts. This paper reviews these new tendencies as compared to the classical washing flow sheet for the beneficiation plant (scrubbing plus size separation) and the classical Bayer process plant.

These alternatives are the beneficiation of bauxite ores via proper classification, density separation, magnetic separation and froth flotation (either direct flotation of boehmite or reverse flotation of quartz). The dewatering of tailings demands special attention today. In some plants a homogenization stockyard for the ore, before the beneficiation plant may be interesting and important.

In the refinery, main concerns are the proper dewatering of the bauxite residue (red mud) via paste thickening or pressure filtration, the classification of aluminate hydrate seeds via cycloning and calcination via flash or fluidized bed reactors.

PREPARATION PLANT

In the practice of aluminium metallurgy industry, the beneficiation of the bauxite to be fed to the refinery is not usual. This reflects the fact that high quality bauxite ores are common and abundant all over the world. In these orebodies the ore is just mined and fed to the refinery.

The only problem with this configuration is that the refinery has little flexibility in relation to the quality of the ore it will receive: It is tailor-made designed for such an ore. If a new ore is fed to it or if the quality parameters change, problems will arise.

Some considerations about the ore quality demands are an interesting reflection:

1. The available alumina grade must provide a minimum value for the process economy. Depending on the silica/alumina ratio, poorer ores can be fed to especially designed refineries at the cost of huge soda losses.
2. Reactive silica content – the SiO_2 contained in clay minerals reacts with the sodium hydroxide during refining, increasing soda consumption, and forms a complex compound with alumina. So, its “reactive silica” content must be kept to a minimum.
3. Zinc and phosphorus are contaminants.
4. Iron content - a minimum amount is necessary for proper processing. The excess dilutes available alumina and increases the volume of generated red mud.
5. Insoluble silica content The insoluble silica increases the volume of final red mud. Otherwise, quartz is a very abrasive mineral consuming high energy in grinding, thus increasing preparation costs.
6. Organic carbon content is noxious due to the humic acids.
7. Size distribution - it must be not so coarse as to make the chemical reaction difficult or bring bauxite losses in digestion nor so fine as to hinder settling.

This shows that there are a number of reasons to make bauxite beneficiation attractive.

The classic circuit for the “washing plants”, when it exists, consists of a disaggregation operation, made in scrubbers, followed by crushing plus size separation done in classifiers or screens. This reflects the fact that clay minerals are by nature built in fine particles which disaggregate rather than demand crushing and grinding.

The classic crusher, until recent years, was the horizontal shaft impact crusher. The sticky characteristics of the bauxite ores demand a series of adaptations to the basic model as moving shoes, heated walls or reversible hammers. In the last years the sizer crusher (Figure 1) has been introduced with great success and is gradually replacing the impact crushers.



Figure 1 Sizer

The scrubber is the universal choice due to its good performance and high capacity of production. The only alternative presented is the HydroClean™ equipment, which uses a high pressure water jet to clean the surface of the coarser particles, thus demanding less space and decreasing water demand. Doubts remain about its ability to deal with greater capacities.

To take up the finest fractions, screens and classifiers, especially cyclones, are used, the choice among them depending on the separation size. A great progress has been achieved in the last decades regarding the sizes that the screens can separate. High frequency linear movement screens are the current choice for fine screening.

At CBA’s Itamarati de Minas plant, MG, an amphibolytic ore had a lot of iron and titanium ores in the finest fractions. The cut size for proper recovery of bauxite grades was 28# (0.59 mm). The undersize (-28#) had a lot of bauxite, but it was diluted by these ores. An additional installation of concentrating spirals plus a wet high intensity magnetic separator (Figure 2) was introduced to get additional recovery (1).



Figure 2. Itamarati de Minas' preparation plant

At CBA's Miraf, MG, plant a reverse froth flotation plant is in erection. The mother rock of the ore at this site is a gnaiss and the finest fractions have a lot of quartz. The cut size at this plant is 48# (0.297 mm) and a process for reverse flotation of the quartz was developed and demonstrated in bench (Figure 3) and pilot plant scale. The quartz particles are floated and the bauxite and the iron-titanium minerals are depressed, thus demanding an auxiliary magnetic separation (2, 3).



Figure 3. Reverse flotation – pilot plant tests

In China, direct flotation of bauxite is performed. The bauxite ore, however, is mainly diasporic, different from Brazilian bauxites, of which the mineral ore is mainly gibbsite.

At Paragominas, PA, a pipeline was built to transport the bauxite to the refinery, 250 km away. Hydrotransportation demands a finer grinding, so an installation of a SAG mill complemented by ball mill is being used. The concept then changed, and the bauxite is ground, not to fit Bayer process requirements, but to fit hydrotransportation demands. The proper mill to deal with the ore, not with washed bauxite, is not the ball mill anymore, but the SAG mill.

In Brazil, there are two types of laterization, the one which occurs in the plateaus of Northern Brazil (Porto Trombetas, Juruti, Paragominas and Rondon do Pará) and the one that occurs in the Southeast (Zona da Mata) and the central part of the country (Barro Alto, Goiás State). See Figure 4. In the last geophysical feature, bauxite occurs on the top and sides of insulated mountains (“half orange mounts”). The intensity of the weathering changes in the horizontal and vertical directions and as a consequence of the nature of the mother rock. So, the quality changes from orebody to orebody and inside the orebody itself. The feed to the preparation plant is erratic in terms of its quality (chemical and particle size). More sensible preparation processes can demand a preceding homogenization of the feed. This is a very expensive installation in terms of both capital and operational costs and a proper economic evaluation must be carried out about its feasibility.

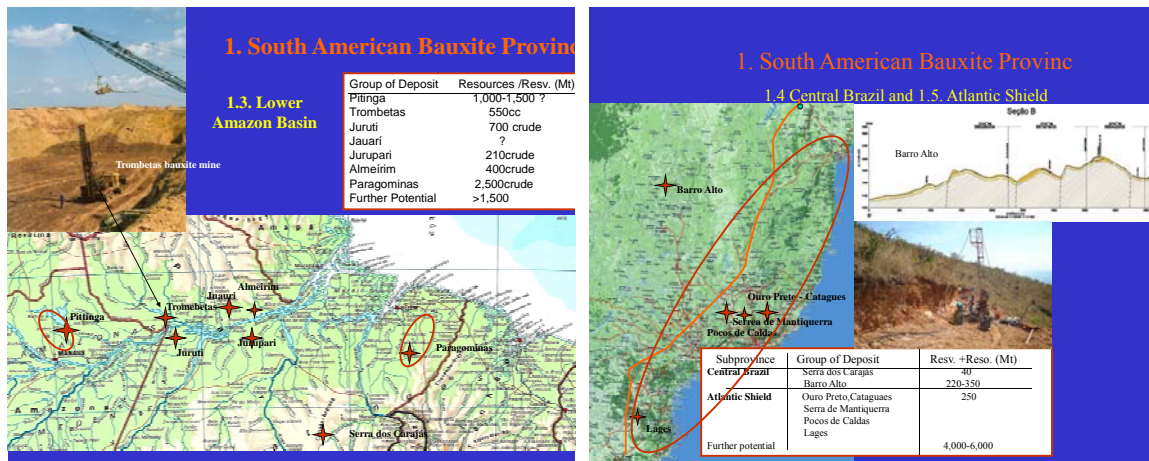


Figure 4. Main Brazilian bauxite deposits (4)

Finally, tailings disposal is a very important concern in terms of the disposed volumes and on the environmental impact. MRN’s tailings are very difficult to settle. Beneficiation plants are built close to mine sites, on the plateaus. The tailings are pumped to tailing dams where some settling happens. The slurry from the bottom of the dams is then pumped to the bottom of the strips of the mine and covered with the overburden. Environmental impact is thus practically eliminated.

Alternative tendencies to the classic tailings dam are the thickening of the tailings and disposal of the thickened slurry in the tailing dam or dewatering the thickener underflow in vacuum or press filters and stacking the cake.

REFINERY

The classic Bayer Process flow-sheet remains essentially the same. However, important modifications in the involved unit operations must be mentioned.

The initial operation is the fine grinding, classically performed in rod/ball mills in open circuit. This equipment is being gradually displaced by shorter greater diameter ball mills in closed circuit with significant savings in energy consumption. This change is important as open

operation, generates a greater amount of fines which will hinder settling and a greater amount of coarser particles that present a risk of not being completely digested, causing losses of alumina.

The reactive silica in the ore reacts with soda yielding an expressive soda losses. Sometimes this can be interesting regarding the conversion of reactive silica to sodium aluminium hydrosilicate prior to the digestion (“predesilication”), as the residence time in the autoclaves is decreased.

After digestion, to separate the solids in suspension in the aluminate solution, polymeric flocculants are becoming more and more efficient and specialized. Some companies design tailor-made polymers for that specific application.

The pregnant liquor is cooled and alumina hydrate seeds are used in the precipitators. Classifying cyclones are used to size the alumina hydrate particles that are ready to go to the next step (precipitation and product filtration) and those that must return to the process and act as nuclei for agglomeration or crystals growth (seeds).

Next steps are filtration and washing operation followed by calcination of the hydrate to yield alumina. The filtration and washing operation is usually done by horizontal pan or drum filters.

The calcination is classically performed in rotary kilns, which are being displaced by fluid bed calciners, with gains in capital and operational costs.

Finally, the red mud disposal increasingly demands special attention. The red mud or bauxite residue as it is currently named, carries a lot of aluminate and soda in solution. The best is the dewatering, i.e. the recovery of dissolved alumina and soda. Associated to them are the benefits to the environment, as a lot of dissolved soda and alumina are not disposed into the dams.

The filtering of this residue, especially in pressure filters, is becoming mandatory. The cake results with very low moisture content (means increased recovery of alumina and soda) and can be stacked by trucks. The inclination of the mud piles is so great that the rain will flow over it, instead of percolating it. It is a much safer operation.

An alternative solution is the paste thickeners, used mainly in arid sites, where the final drying by sun exposure is possible. They seem to be technically feasible, but more expensive than the filtration process.

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CFD application for mixing study in industrials thickeners

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Abstract

In mineral industry, thickeners usage is a key operation for solid-liquid separation process. In Brazil, most part of those equipments were installed before 1990's decade and, therefore, before the modern computational optimization concepts. Faced the continuously increase of competition, chemical engineers are challenged to work on cost reduction using old equipments. Further to that and in most cases, new investments for equipment substitution are not considered as an option. Taking this as a target, the present paper work shows an assessment of new possibilities to operate 30 years old equipment without any financial budget associated. Through numerical simulations using computational fluid dynamics, the aim was to raise opportunities for process improvement in synthetic flocculent mixing inside the thickener. There is no chemical reaction involved. With this approach, was possible to identify process gains around the potential order of USD 1 million/year throughout mixing improvements of polymer injection points.

Keywords: CFD; thickener; mixer; flocculent; simulation.

1. Introduction

Economics business evaluation associated to new desires from society for sustainability raises a new challenge on the horizon for mining companies. In that way, the role of chemical engineers should be "laser focused" on plant optimization. Keeping the business attractive for stakeholders and society, reducing costs and increase profits bring new challenges

that can be matched using advanced computational tools.

This challenge is enhanced by the desire of stakeholders to have that profit increased with as minor as possible additional investment. Furthermore, plant engineers need to optimize equipments with 10 – 30 years. Additionally, any investment to be approved must be exhaustively proven from feasibility point of view. Computational tools are important and have been used to justify new investments [1].

In this context, plant engineers must adopt an innovation approach for classic processes. For that, this study has the intention to address the treatment of a specific problem, the branch of the mining industry, related to the mixture of flocculent with the liquor of the hydrometallurgical plant working to leverage the profit.

In the branch of the aluminum industry, the mud thickeners are equipment used during the refining of bauxite, responsible for the separation of the solid waste from liquor work. Therefore, its optimization is crucial to minimize the loss of plant liquor, which implies the loss of caustic soda and sodium aluminate to the residue pond as to the recovery of those species from the lake, through the reintroduction of water in the process.

The use of chemical additives to accelerate sedimentation of the solids is essential for allowing the increase of flow with minor modifications or equipment investments. In this line, the homogeneous mixture between the mud and the flocculent is crucial to maximize gains, whose economy can reach USD 1 million / year.

2. Problem statement and target situation

2.1. Paper title and authors

The existence of large thickeners is a reality in plants which its process is based on hydrometallurgy, playing a key role in the solid-liquid separation. In those, typically are two inlets: the liquid mixture, typically known as slurry, and synthetic flocculent, whose function is to accelerate the sedimentation of solids.

It is easily found old equipment with major efficiency opportunities, related to the speed of sedimentation. Often, there is variation in flocculent flow rate dosage that may reach 500% in less than 24 hours. It is important to point out the mean residence time for solids usually are days. Usually, a field inspection does not identify any problem to justify the higher dosage, associated to its further reduction without the record of any problem. The variability in the dosage of flocculent presents itself as a major point of improvement, capable of saving money from a raw material which usually figures among the five most expensive to this type of industry.

In the presented case, one old equipment is assessed. It has more than 20 years, therefore there is low flexibility for operational adjustments. The feeding and slurry outlet are fixed, and the work flow rate must match production targets. The maintenance planning is very tight so that there is little room for testing the process plant. Because of this, any plant test for improvements must be well grounded. And it is exactly on that point that computer simulation comes as a subterfuge to justify a possible test.

In fact for that case, there is only one degree of freedom to work available to chemical engineer: once the best flocculent is selected from market, ensure the mixture of flocculent is conducted as efficiently as possible. The points of flocculent dosage in the thickener feedwell are fixed and there is no proven work to guide how deep this injection should be to ensure best mixing. Or even if the points available are sufficient to ensure a good mix.

Thus, this work has 2 aims: the problem of injecting flocculent in a thickener feedwell, i.e.

mixer, which is a cylinder with baffles that must ensure a mix between flocculent and slurry in a final solution as homogeneous as possible. The other aim is to raise a step guide for computational approaches in industries.

With regards to savings, it is linked to cost reduction by optimizing the mixing process. The estimated gain is based on economic optimization of flocculent, since the settling effectiveness of the product is directly proportional to a good mix process. Basically, the gain can be stratified in three: good flocculation enables the increase of solids concentration in the bottom of the thickener. Hence, less plant liquor is lost to lakes, reducing the loss of soluble caustic soda and alumina. The second gain is to reduce the flow of flocculent, which impacts on a smaller unwelcome dilution, reduce cost with the purchase of the additive and less expenditure of energy to heat the extra dilution. The third is the gain from the reduction of variability, critical to optimize any installed advanced control loops, what allows the raise of production.

3. Industrial application of CFD

This work was conducted in a hydrometallurgical plant in Brazil. The industrial process involved is a caustic leaching, known as Bayer Process, used to produce alumina [2,3].

The study of mixing in the industry is critical in various businesses. The effective contact between solvent and solute is fundamental and basic to any specific kinetic considerations [4]. This is no different in the case of the flocculation process, where the better the mix the greater the contact between flocculating and solids in the slurry. Hence, this will lead to a better interaction inherent in the flocculation process.

Parameters such as residence time and dispersion of the material within reactors can be evaluated. Mostly, in computational simulations, the aim is to reduce operating costs modifying the geometry of the equipment to minimize waste of raw materials by raising the efficiency of the reaction [5]. Where agitators are used to improve the homogeneity of the

mixture, the CFD can be used to size accurately even for viscous agitators [6].

Specifically in relation to thickeners, since the invention of the Dorr thickener, this equipment has not stopped evolving and meets the most diverse industries, to meet a growing need for solid-liquid separations [7]. According to [8], it is possible to provide significant gains in both the design and optimization improvements in the mixing processes.

4. Simulation methodology

Computational simulation helped to understand how the dispersion of flocculent happened inside the mixer. With this understanding, process engineer should be able to identify the best approach for plant optimization.

It was important to see how homogeneous the mix flocculent-slurry was. As mentioned before, the contact between them is crucial to good settling process. It follows the step by step methodology that was followed, which can be adjusted to various situations:

1. Determine the problem and justify the use of CFD
2. Determine current status and target status
3. Determine the volume control to be considered
4. Raise all available physical data
5. List all process variables that would be relevant for the computational simulations (variables that are controlled in the system, the ones that are changed to keep control and the ones that causes disturbances and are not controlled)
6. In CFD software package, create the geometry, defining the areas with all inherent characteristics
7. Create the mesh in the software. Refine the mesh created in key areas of analysis:
 - 7.1. Close to walls.
 - 7.2. Pipes.
 - 7.3. Any other particular area relevant to have the mass, heat and/or momentum evaluation.
8. Feed CFD software all physical properties of the fluids, for each phase.
9. Feed CFD software with the initial and boundary conditions of the system.
10. Based on the system characteristics, set the expected error that must be met.

11. Use the software solver.
12. See the results and make critical analysis:
 - 12.1. Preliminary results: do they have physical relevance?
 - 12.2. Final results after any adjustments to the model and/or simulation.
13. Analyze the results, comparing it to the state target.
14. Verify the simulation output, feeding the model with one known situation for all inputs and outputs (as close as possible to real situation).
15. For optimization purpose, do a step test plan varying one variable at once and collecting all output results. Thus, collect all residence times for responses (when possible).
16. Each simulation has one particular input, which will deliver to a specific output. With the residence time listed, it is possible to evaluate in advance this output scenario when a plant trial is needed to validate the results, based on real data. This is helpful especially when it is needed to have people following the test in shifts, 24 hours around the clock.

Physical considerations must be observed in order to both ensure correct information to feed the model and the interpretation of results in comparing with the real case. For the present study, key points listed below should be observed:

- The pressure in the flocculent feed line is constant. This approach is valid for the case of an injection system with positive displacement pumps.
- Flocculent concentration is constant, coming from the same batch for each simulation.
- The fluids are incompressible.
- The flow is isothermal.
- The given solids concentration does not change significantly.
- No relevant change is observed in pumping system, so that the mass flow is constant.
- Turbulent flow is observed only inside the thickener and stationary within the thickener (speed is minimal due to the large tank volume).

- There is no heat transfer, given that a change in temperature in the field does not occur. So all properties was fed according to the tank temperature.
- Both mud thickener and its mixer start the operation filled with the working fluid.
- Flocculent injection points are fixed and are available to operate.

Important input details for simulation are shown below in Figures 1, 2, 3 and operational conditions that shall be observed in Table 1:

4.1. With regards to modeled geometry and mesh detail

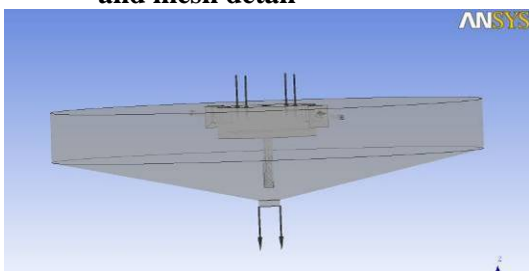


Fig. 1. Equipment geometry detail (side view)

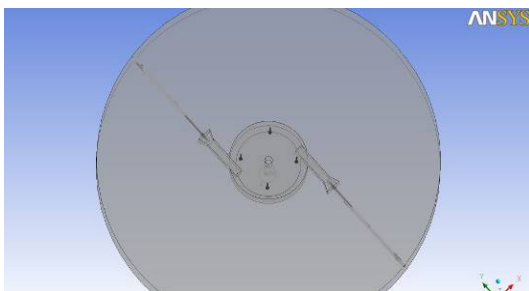


Fig. 2. Equipment geometry detail (top view)

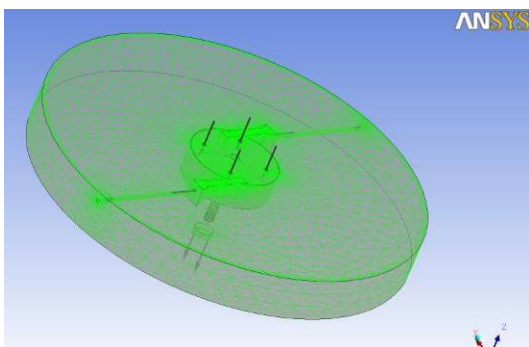


Fig. 3. Mesh

4.2. Operational conditions

Just as a reference, once the solution can be run and applied to various flows.

Table 1: operational parameters

| Inlets | Typical value |
|--------------------------|---------------|
| Total mass flow (t/h) | up to 1500 |
| Solids mass fraction (%) | 0.4 to 0.5 |
| Temperature (°C) | 50 to 55 |

5. Computational simulation

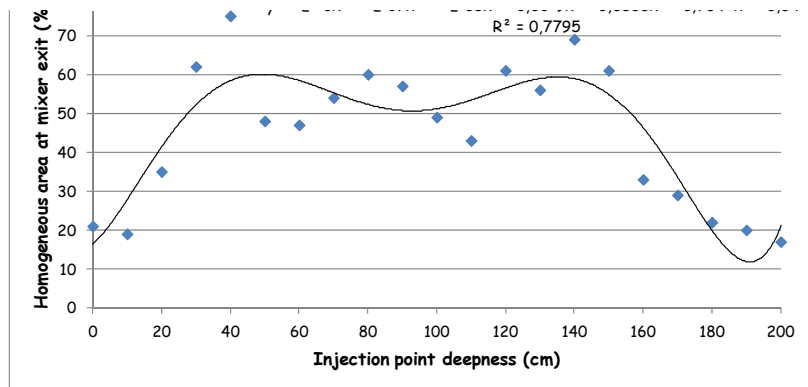
Using the proposed methodology, one thickener operation was simulated using the software package CFX 12.0, from ANSYS. A solid was generated with 929,897 elements 229,787 nodes.

The contours of circular pipes inside the mixer, thickener and walls were refined in order to enable the study of all intersections, interactions and impacts of the wall effect on fluid flow.

In each performed simulation, a change the flocculent injection depth in every 10 cm. 20 simulations were conducted, and from them was possible to identify 6 conditions that would be tested in plant.

How to identify desired conditions to be tested, once the mixer is closed and the access is restricted? To answer this questions, the process engineer must list process variables that would be impacted and, indirectly, evaluate the simulation outputs. After this, it is important to assess the homogeneity for the mix. The best scenario, taken as a theoretical reference, is to have 100% of the circular area in the mixer exit at the target mass fraction for flocculent concentration (which can be calculated, once the target concentration is known from laboratory trials and the process variables are controlled).

Once the higher the area covered with homogeneous mixing the better, it is important to have a real base case. For that purpose, a reference based on operational expertise was taken. That reference says that the best injection depth is around 80cm. In software, this condition was simulated and the covered area calculated. All simulations output can be found in Graph 1. It is also shown trough Figures 4 to 6 output figures that supported the evaluation.



Graph 1 – simulation output summary

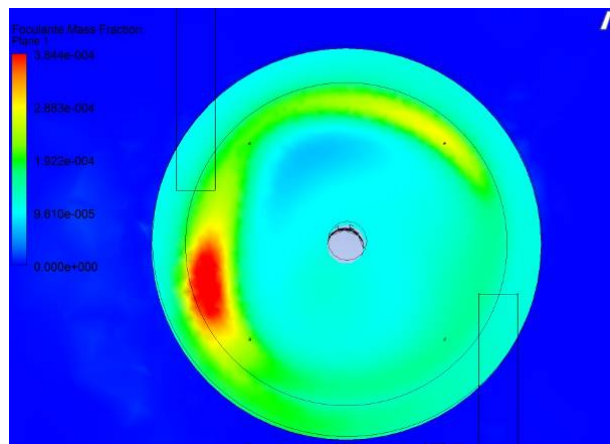


Fig. 4 80 cm deep, which corresponds to 60% area covered with mixture at target condition. This was the reference for process trial.

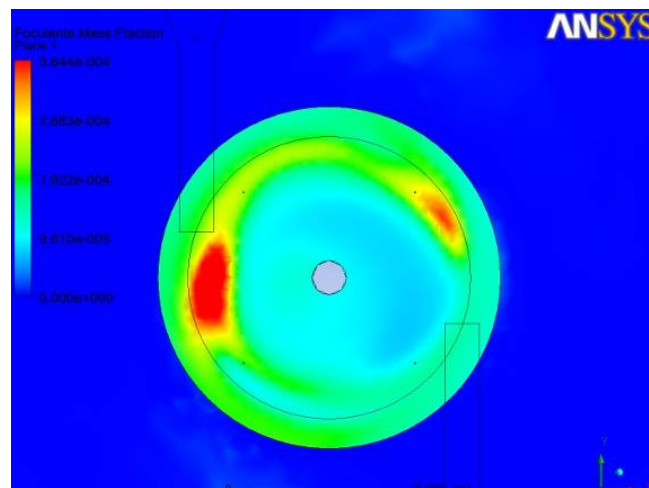


Fig. 5 140 cm deep, which corresponds to 69% area covered with mixture at target condition. This was the best output case.

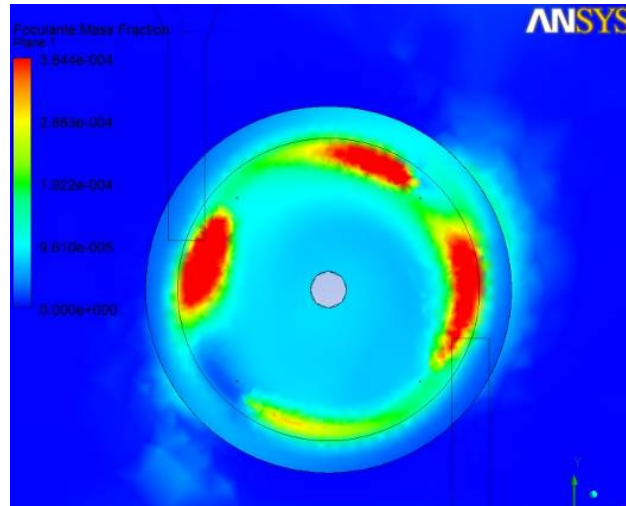


Fig. 6 200 cm deep, which corresponds to 17% area covered with mixture at target condition. This was the worse output case.

6. Conclusions

It was possible to simulate the mixing process for a typical industrial mixer. From those simulations, it was possible to extract results with good physical meaning and feasible for immediate application.

The first important observation was to identify areas of injection of the product that should be avoided with the current design of the equipment. Conversely, it was listed which depths must be avoided, once a poor mixing result is achieved.

As next step, a plant trial must be conducted to confirm theoretical financial gains.

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Selected Applications of X-ray Diffraction Quantitative Analysis for Raw Materials of the Aluminum Industry

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Abstract

In the last few decades, X-ray diffraction (XRD) systems have been paramount and irreplaceable in controlling bauxite exploration, as well as the Bayer and reduction processes. XRD quantitative phase analysis in the aluminum industry witnessed a steady deployment of the Rietveld method, which at present progressively replaces existing methodologies in research and plant laboratories.

Rietveld analysis not only helped to surpass traditional XRD calibration methods, it also opened the door for new applications previously not possible. The use of the Rietveld method for characterization of selected materials unique to the aluminum industry, such as bauxite, red mud and alumina is demonstrated and discussed. This paper also presents how synchrotron based diffractograms obtained for bauxite and red mud samples allowed a much better understanding of mineralogical representation, and made it possible to leverage their Rietveld quantification. Despite clear advantages, the Rietveld method also has important limitations that are revealed.

For phase quantification of each material a dedicated Rietveld analytical program was built with dedicated structure data of corresponding minerals. For example, for alumina 8 mineralogical phases: alpha, beta ($\beta\text{-Al}_2\text{O}_3 = \text{Na}_2\text{O}\cdot 11\text{Al}_2\text{O}_3$), delta, gamma (2), kappa, sigma and theta are used. The paper gives unique examples of phase quantification in aluminas of various origins and phase composition. Shortcomings in the analysis of bauxite are discussed.

Keywords: bauxite, bauxite residue, alumina, XRD, Rietveld analysis, phase quantification

Introduction

Rietveld X-ray diffraction analysis has been increasingly used in the aluminum industry since the beginning of the 1990's. Initial applications involved predominantly raw materials and selected products. During the last decades its use expanded to every stage of aluminum production and presently Rietveld analysis has become a recognized analytical technique, applied routinely in bauxite exploration, reduction and fabrication processes. Typical Rietveld applications in the aluminum industry at present are listed in Table 1. The number of crystalline phases determined represents usual industrial requirements, and may vary between laboratories. The conventional cavity slide sample preparation technique is the most commonly used for the applications. The success rate of Rietveld application, understood as accuracy and reliability of obtained information, obviously depends on the matrix.

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Table 1 Typical Rietveld applications in the aluminum industry

| MATERIAL | No OF PHASES | SAMPLE TYPE | SUCCESS RATE |
|-------------------|--------------|-------------------|----------------|
| Bauxite | 14 - 24 | cavity slide | limited |
| Red Mud | 30 - 60 | cavity slide | limited |
| Alumina | 8 | cavity slide | limited - high |
| Electrolytic Bath | 9 - 16 | briquette, cavity | high |
| Spent Potlining | 30 - 60 | cavity slide | limited - high |
| Dross | 10 - 20 | cavity slide | high |
| Intermetallics | > 20 | cavity slide | high |

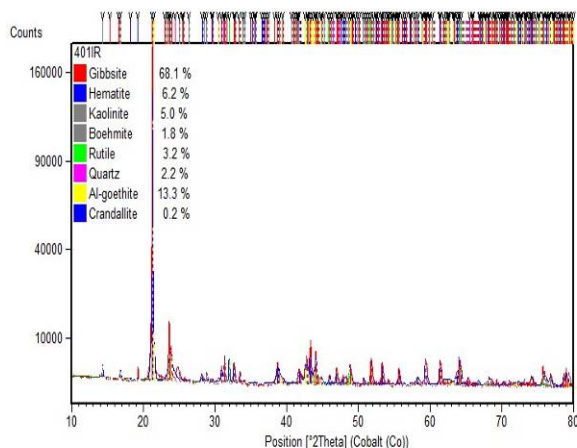
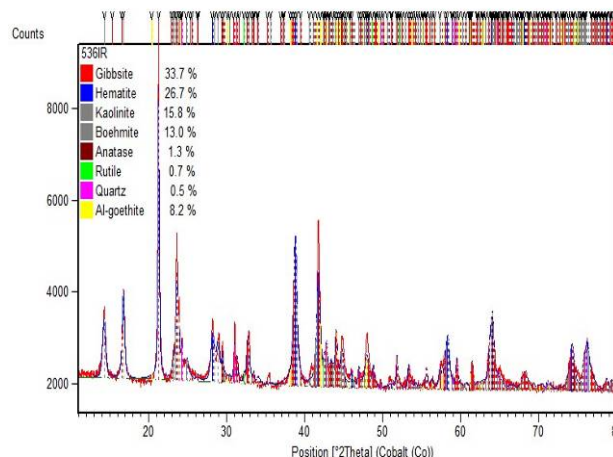
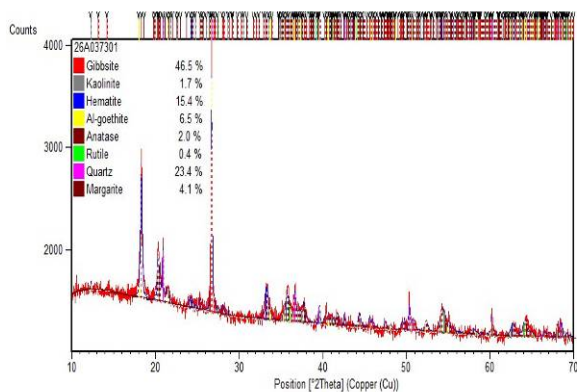
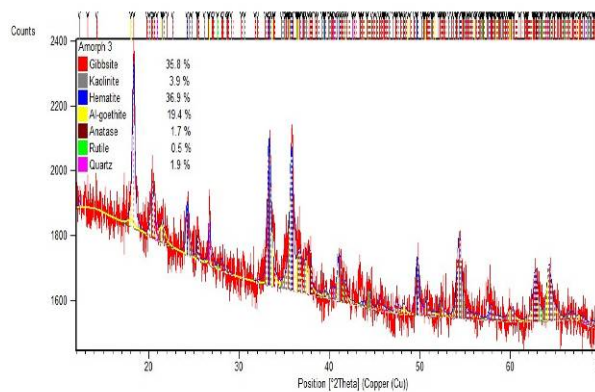
Rietveld analysis not only helped to replace traditional XRD calibration methods, it also opened the door for new applications previously not possible. The use of the Rietveld method to characterize selected materials unique to the aluminum industry (for example bauxite, red mud, alumina, electrolytic bath, spent potlining and dross) became routine at present. As there is too much to cover in one paper the content was divided into two parts. This paper deals with industry raw materials (bauxite, red mud and alumina). The paper will also present how synchrotron based diffractograms obtained for bauxite and red mud samples allowed a much better understanding of mineralogical representation, and made it possible to leverage their Rietveld quantification. The second part will appear at a later time and will cover Rietveld characterization of materials such as electrolytic bath, spent potlining and dross.

Bauxite

Aluminum bearing minerals constitute a group of raw materials vital to the aluminum industry. Bauxite, a hydrated aluminum material is the primary raw material used in the Bayer process. Other materials of concern, such as clay, red mud and sand are waste products. Clay is obtained during bauxite washing, whereas red mud and sand appear during the bauxite digestion process.

An accurate estimate of bauxite quality being surveyed, mined and supplied is important to efficient operation of a mine and a Bayer plant. Traditional methods used to determine bauxite phase composition are based on wet chemistry. They are relatively slow and require large supply of chemicals as well as considerable space and manpower. Compared with wet chemistry, X-ray diffraction (XRD) offers speed and much lower cost of analysis, but is less accurate. Conventional X-ray diffraction (equipped with an X-ray tube) is known to be seriously affected by the sample's amorphous content (Fig 1-4). In Fig. 1 gibbsite is underestimated by approximately 8% and in Fig. 2 it is overestimated by the same amount with respect to WCh and mass balance calculations. High degree of bauxite amorphous content is frequently encountered during bauxite exploration (Fig. 3-4). In Fig 3 and Fig. 4 the estimated amount of the amorphous content is 32% and 49%, respectively. In consequence, concentrations of the major phases are largely overestimated.

As a result of low X-ray power and material crystallinity, the limit of detection (LOD) of phase constituents is relatively high [1]. Known applications of Rietveld analysis to bauxite are very limited [2]. It was believed that synchrotron patterns with much better signal-to-noise ratios would help provide new information that cannot be obtained with X-ray tube instrumentation [3]. It was also expected that the diffractograms would largely overcome the amorphous content drawback, thus seriously plaguing analysis of bauxite and red mud material using conventional XRD.


Fig. 1 Example 1 of Rietveld data for bauxite

Fig. 2 Example 2 of Rietveld data for bauxite

Fig. 3 Example 3 of Rietveld data for bauxite

Fig. 4 Example 4 of Rietveld data for bauxite

The samples for the synchrotron experiments were ground to -325 mesh and packed into a thin wall glass capillary [3]. Diffractograms were recorded at the Japan Synchrotron Radiation Research Institute (JASRI) at wavelengths of 1.002 Å and 0.202 Å and covered 5-75 °2θ and 0-15 °2θ ranges, respectively. The effect of the power of synchrotron radiation on bauxite diffractogram can be appreciated in Fig. 5 that gives a comparison of diffractograms of BXT-12 (Rio Tinto Alcan bauxite standard) from the 1 Å, 0.2 Å synchrotron and 1.54 Å Cu Kα radiation sources. The synchrotron diffractograms are not only much more intense, but the background is much less noisy and individual peaks much better defined than in the case of the 1.54 Å radiation. The 0.2 Å source diffractogram simply dwarfs two other diffractograms by a large margin. The first two peaks from kaolinite and boehmite are very well defined on the 0.2 Å source diffractogram.

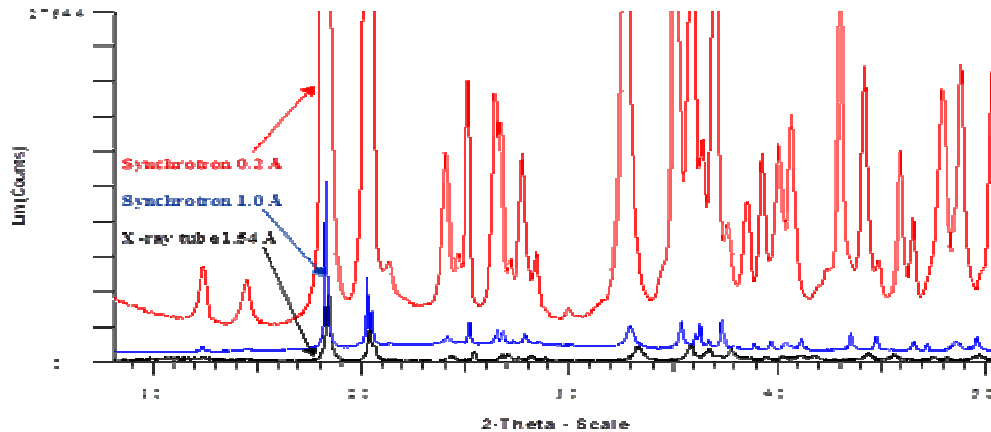


Fig. 5 Comparison of diffractograms of BXT-12 from three sources

In order to characterize all bauxite samples involved in the project, wet chemistry (WCh), BQuant [4], XDB Hungalu [5], XRF [6] and TGA determinations were carried out, in addition to Rietveld-XRD investigations.

Rietveld-XRD quantification results of RTA (Rio Tinto Alcan) bauxite reference materials using the 0.2 Å source diffractogram are presented in Figures 6-7. In Table 2 major phase concentrations obtained for BXT-12 are compared. Gibbsite (GIBB) is to some extent overestimated and boehmite (BOEH) concentration is underestimated using synchrotron and the X-ray tube diffractogram in connection with the Rietveld method. Boehmite concentration is lower from the 0.2 Å source diffractogram than from other sources. By contrast, Al-goethite (GOET at 3.2%) is clearly confirmed in BXT-12 bauxite contrary to conclusions from a conventional X-ray tube-based diffractograms. Anatase (ANAT) in Table 2 represents the sum of anatase and rutile.

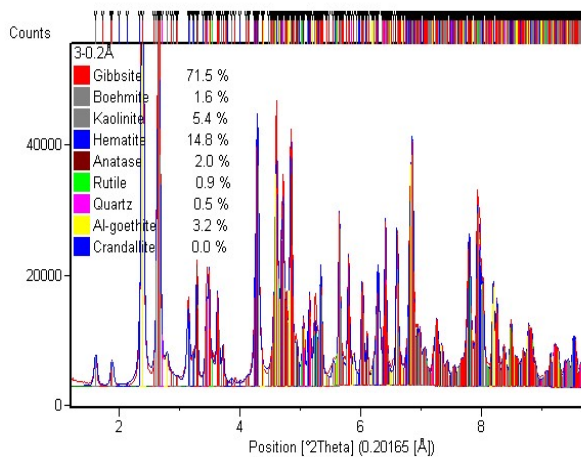


Fig. 6 Rietveld data for BXT-12

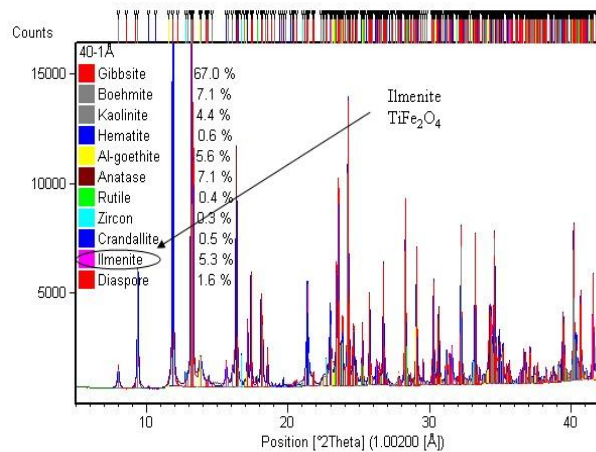


Fig. 7 Rietveld data for BXT-02

Figures 7 and 8 illustrate Rietveld-XRD results for BXT 02 and BXT-08 and confirm diaspore and ilmenite. Those phases are impossible to quantify at low content using an X-ray tube diffractogram.

Table 2. Comparison of phase concentrations for BXT-12

| | GIBB | BOEH | KAOL | GOET | HEMA | ANAT | QUAR |
|---------------------------|------|------|------|------|------|------|------|
| Wet Chemistry | 69.5 | 3.0 | 5.9 | | | | |
| Mass Balance [4] | 68.7 | 3.2 | 6.9 | 3.0 | 13.5 | 3.2 | 0.3 |
| Rietveld-Synchrotron 0.2Å | 71.5 | 1.6 | 5.4 | 3.2 | 14.8 | 2.9 | 0.5 |
| Rietveld Cu X-ray Tube | 71.2 | 2.4 | 5.9 | 1.2 | 15.7 | 3.2 | 0.4 |

Besides the phases appearing in Figures 6-8, additional phases such as calcite, dolomite, magnetite, magnesite, chamosite, illite, muscovite, hausmanite, manganite, chabazite, and other may also appear in small quantities, depending on the bauxite deposit.

A concentration correlation for gibbsite reveals that the X-ray tube - Rietveld concentrations of gibbsite are mostly overestimated, sometimes largely, with respect to WCh concentrations (Fig. 9). The main reason is most of the time due to underestimated boehmite, kaolinite and goethite. Because the Rietveld-XRD analysis normalizes all concentrations to 100%, increasing content of the major constituent (gibbsite) compensates for whichever part of the matrix is missing. The correlation curve also demonstrates that the synchrotron diffractograms and XDB interpretations help obtain the best correlation with WCh.

Rietveld-XRD determinations employing X-ray tube diffractograms tend to strongly underestimate kaolinite. This is evident from the XRF-WCh mass balance. The underestimation convincingly suggests that a part of kaolinite must be X-ray amorphous. By contrast, the synchrotron data is much less affected by the kaolinite's amorphous content effect. BQuant's $k \cdot \text{SiO}_2$ correlation appears good as this determination was modeled on Rio Tinto Alcan standards and they form a majority in the studied group. The XDB mass balance calculations of the $\% \text{SiO}_2$ in kaolinite ($\% \text{SiO}_2 - \% \text{quartz}$) provide the best fit with the WCh data given low SiO_2 content.

The synchrotron study helped validate the analytical methods employed for bauxite phase quantification and has changed our understanding of the bauxite matrix. Kaolinite and goethite, in particular, were already believed to be partially amorphous. However, contrary to past beliefs, even gibbsite and hematite may appear partially amorphous. The problem of bauxite amorphous content, although anticipated, was shown to have a profound effect on quantification of selected phases using Rietveld and XDB methods and X-ray tube diffractograms. Simply, if a part of the bauxite matrix is X-ray amorphous, it does not contribute to a diffractogram. If several mineral contributions are missing on diffractogram simultaneously, they can be quantified as a group, but not individually. The bauxite amorphous content, believed to be at the level of a few percent, appears to be much higher than that.

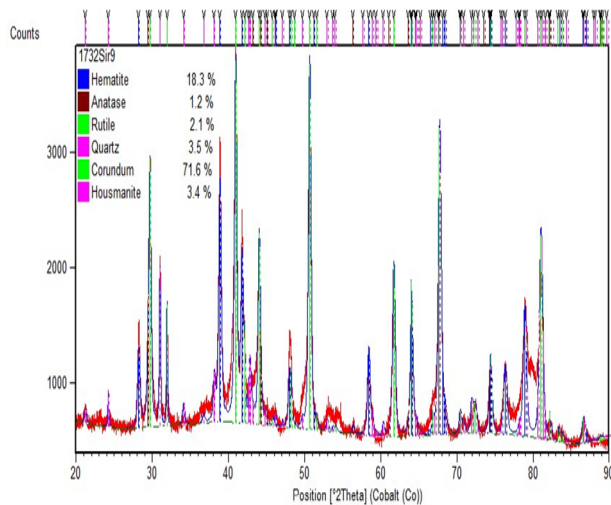


Fig. 10 Rietveld quantification of calcined bauxite

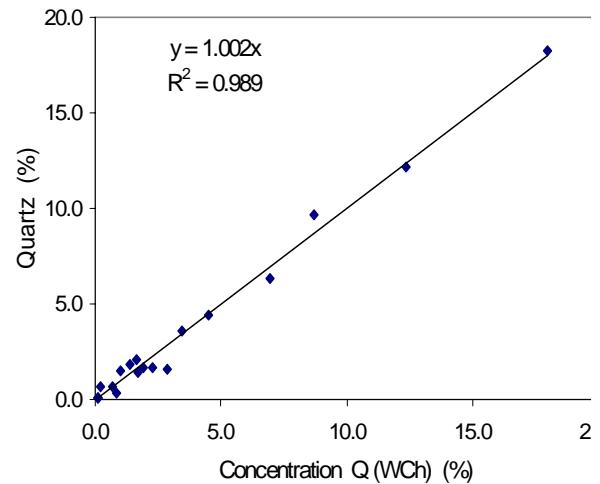


Fig. 11 Comparison of quartz (%) from WCh and Rietveld methods

In order to determine quartz concentration corresponding to the natural basis, the quartz content from Rietveld analysis, Quartz (R), needs to be corrected for various dilution effects in one single step. Employing one simple correction equation the corrected quartz concentrations, % Quartz are:

$$\% \text{ Quartz} = \text{Quartz (R)} \frac{t.TiO_2}{\text{Rutile(R)}} \quad (1)$$

where $t.TiO_2$ is total TiO_2 known from XRF, and Rutile (R) is the rutile content from Rietveld estimate. All of the Ti detected by bulk analysis is present as rutile.

Material mass balance applies well to the quartz quantification problem. Accuracy of quartz determination with the new method corresponds to that obtained by the conventional “by difference” approach (Fig. 11). This approach is based on total SiO_2 determined by XRF and kaolinitic silica (Si_{150}) determined by WCh. The study also proves that SiO_2 determined in the low temperature digestion process (Si_{150}) is due to kaolinite and illite and not just due to kaolinite as previously believed. This fact has important implication for plants processing bauxite that contains illite.

Occurrence and Characterization of Zn and Mn in Bauxite

Zinc is one of the secondary elemental constituents occurring in Caribbean bauxite and the associated non-bauxitic material. It is generally believed that Zn in bauxite could either occur in gahnite or sphalerite, or substitute for Fe in goethite. Manganese represents an appreciable impurity in Caribbean bauxites and is identified on diffractograms as lithiophorite $(Li,Al)MnO_2(OH)_2$. As Zn has been observed to increase with the MnO content, the objective of the work was to better understand the mineralogical nature of Zn and Mn compounds. Data representative of 340 bauxite samples of different origin was assembled [8]. It was found that Zn in bauxite could not possibly substitute for Fe in goethite or hematite (Fig. 12). Strong evidence was obtained that Zn occurs in the same compound as Mn. Application of the Rietveld method to characterization of diffractograms and other data suggest an aggregate, which we may call “zincophorite” $Al(Zn_xMn_{1-x})O_2(OH)_2$. Based on the obtained records, the x parameter could vary from 0.02 to 0.24. The concentration data corresponding to the investigated group of samples is given in Fig. 13.

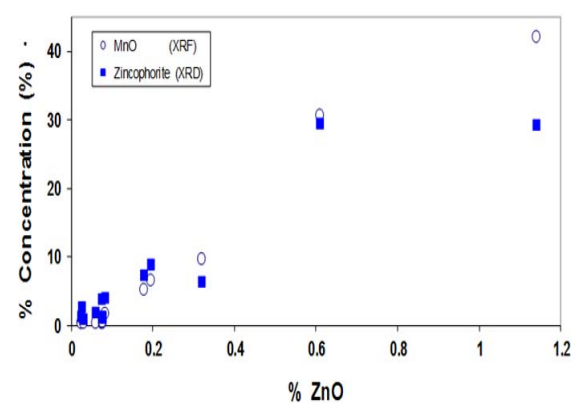
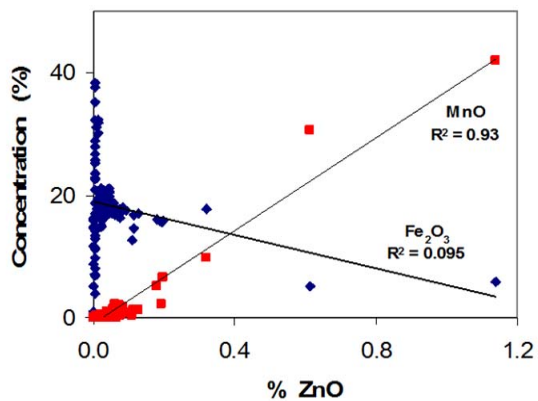


Fig. 12 Correlation of ZnO versus MnO and Fe₂O₃ in bauxite

Fig.13 Concentration graph obtained for Jamaica “zincophorite”

In Fig. 13 concentrations of ZnO are plotted against the concentrations of MnO and zincophorite. The correlation between the ZnO and MnO content is strong. Some points representative of zincophorite are placed much below those of MnO at high ZnO concentrations. This is highly suggestive that a portion of lithiophorite might be X-ray amorphous.

Red Mud (Bauxite Residue)

Certain red mud samples were also analyzed with synchrotron experiments [9]. A dedicated control file containing all necessary crystallographic parameters was developed for quantification of 45 mineralogical phases in red mud. The maximum number of crystalline phases allowed during Rietveld refinement is limited on most commercial programs. Hence, the refinement was a tedious and time-consuming process beginning with phase confirmation and rejection, followed by Rietveld trials for a selected group of minerals. If a particular mineral was confirmed absent during the trial, it was replaced by another mineral. Given a large number of potential choices most minerals determined with a concentration below 0.5% were rejected as unreliable. Composition (%) of 28 different mineralogical phases in red mud matrix was determined: gibbsite, bayerite, boehmite, kaolinite, anatase, rutile, quartz, hematite, Al-goethite, sodalite, carnegieite, calcite, katoite-Si, brookite, ilmenite, portlandite, nepheline, perovskite, cancrinite-H₂O, cancrinite-CO₃, cancrinite-NO₃, cancrisilite, carbonate-hydroxyl-apatite, zircon, nosean, diaspore, lawsonite and schaeferite.

The phase identification process followed by Rietveld refinement involved four different Al-Na silicates: sodalite (Na_{8.08}Al₆Si₆O_{28.88}S_{0.98}), cancrinite (Na_{7.14}Al₆Si₆O_{31.6}), cancrinite NO₃ (Na_{7.92}Al₆Si₆O_{31.56}N_{1.74}) and nosean (Na₈Al₆Si₆O₂₈S).

The concentrations in Fig. 14-15 correspond to as obtained basis and are overestimated due to the X-ray amorphous portion of the sample material. In order to express content of the crystalline phases more realistically, the sample amorphous content needed to be determined first.

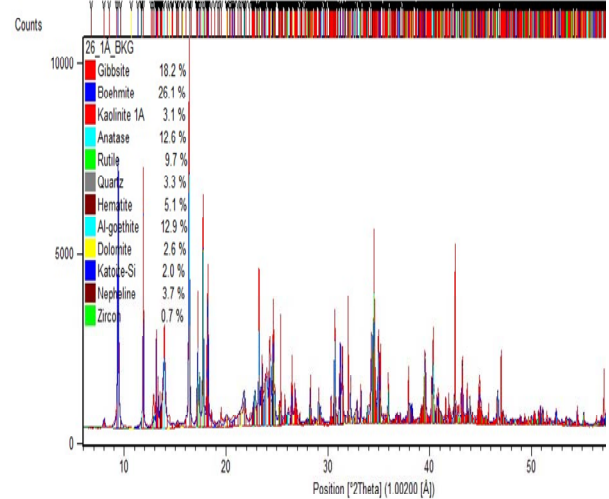
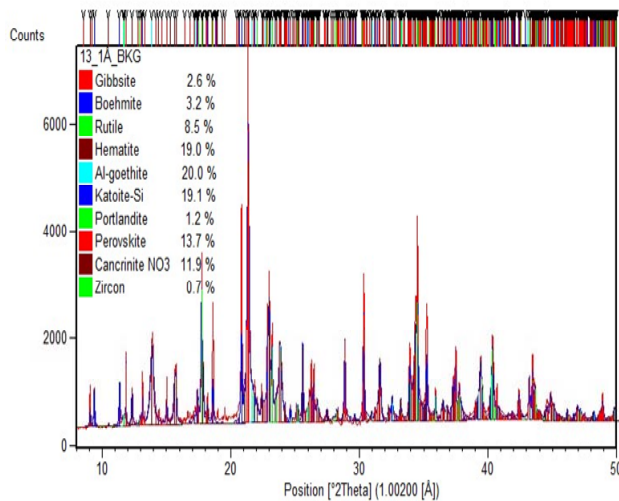


Fig. 14 Example 1 of Rietveld data for red mud **Fig. 15 Example 2 of Rietveld data for red mud**

Red mud is known to be partially amorphous. Selected phases such as hematite, quartz, rutile and anatase are considered to be crystalline in the red mud matrix. However, phases such as sodalite or cancrinite are represented on diffractograms only to some extent. Hence, one of the objectives of the study was to verify whether the synchrotron radiation could overcome the amorphous content problem. Initial Rietveld concentrations of the crystalline phases allowed calculation of concentrations for major element oxides: Fe_2O_3 , CaO and TiO_2 . Then, estimation of the sample amorphous content was made using concentrations obtained from Rietveld estimated phases and determined by XRF. For some samples, the corresponding mass balances between XRF and Rietveld data suggest the occurrence of important quantity of the amorphous material. Figure 16 illustrates a correlation between XRF and Rietveld data for Fe_2O_3 constituent. The Fe_2O_3 concentrations from Rietveld analysis are clearly overestimated.

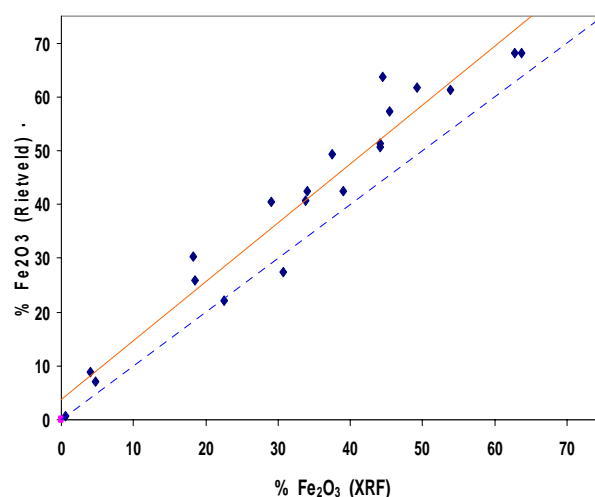


Fig. 16. Correlation between XRF and Rietveld data for Fe_2O_3

Table 3. Summary of the phase composition

| MINERAL | CHEMICAL FORMULA | MAX (%) | MIN (%) |
|------------------------------|---|---------|---------|
| Gibbsite | Al(OH) ₃ | 24.4 | 0.7 |
| Bayerite | Al(OH) ₃ | 4.0 | 3.6 |
| Boehmite | AlO(OH) | 41.3 | 3.6 |
| Kaolinite | Al ₂ [Si ₂ O ₅ (OH) ₄] | 4.7 | 2.2 |
| Anatase | TiO ₂ | 13.5 | 2.5 |
| Rutile | TiO ₂ | 10.3 | 0.6 |
| Quartz | SiO ₂ | 4.9 | 0.2 |
| Hematite | Fe ₂ O ₃ | 66.2 | 0.6 |
| Al-goethite | (Fe,Al) ₂ O ₃ ·nH ₂ O | 57.5 | 1.7 |
| Sodalite | Na _{8,08} Al ₆ Si ₆ H _{1,92} O _{28,88} S _{0,98} | 22.5 | 1.8 |
| Carnegieite | Si ₄ Al ₄ Na ₄ O ₁₆ | 2.5 | 1.3 |
| Calcite | CaCO ₃ | 35.0 | 1.0 |
| Dolomite | CaMg(CO ₃) ₂ | 2.5 | 1.5 |
| Katoite-Si | Ca ₃ Al ₂ (SiO ₄ ,CO ₃ ,OH) ₃ | 8.3 | 2.3 |
| Brookite | TiO ₂ | 5.4 | 3.1 |
| Ilmenite | FeTiO ₃ | 6.9 | 6.9 |
| Portlandite | Ca(OH) ₂ | 2.9 | 1.4 |
| Nepheline | NaAlSiO ₄ | 2.0 | 0.4 |
| Perovskite | CaTiO ₃ | 9.5 | 1.5 |
| Cancrinite H ₂ O | Na ₈ (Al,Si) ₁₂ O ₂₄ (OH) ₂ ·2H ₂ O | 38.8 | 3.8 |
| Cancrinite CO ₃ | (Na,Ca) ₈ (Al,Si) ₁₂ O ₂₄ (CO ₃)·4H ₂ O | 9.1 | 8.6 |
| Cancrinite NO ₃ | Na _{7,92} Si ₆ Al ₆ O _{31,56} N _{1,74} | 42.8 | 0.6 |
| Cancrisilite CO ₃ | Na _{7,86} (AlSiO ₄) ₆ (CO ₃)(H ₂ O) _{3,3} | 4.6 | 4.6 |
| Carbonate-hydroxyl-apatite | Na _{0,8} Ca _{8,4} C _{2,4} P _{3,6} O _{23,6} | 4.8 | 4.8 |
| Zircon | ZrSiO ₄ | 1.0 | 0.0 |
| Nosean | Na ₈ Al ₆ Si ₆ O ₂₄ (SO ₄) | 8.9 | 0.0 |
| Diaspore | AlO(OH) | 3.7 | 3.7 |
| Lawsonite | CaAl ₂ (Si ₂ O ₇)(OH) ₂ (H ₂ O) | 13.0 | 4.3 |
| Schaeferite | (Na _{0,7} Ca _{2,3})(Mg _{1,85} Mn _{0,15})(VO ₄) _{2,88} (PO ₄) _{0,12}) | 17.2 | 17.2 |
| X-ray amorphous part | | 33 | 0 |

In red mud samples the average amount of the amorphous content was 18%, whereas the maximum was 33%. With the sample amorphous content increasing, the mass balance deficit for Na, Al, Si and H₂O also increases. This indicates that a hydrated sodium aluminum silicate (resembling sodalite) makes up most of the amorphous content. Moreover, red mud is very complex in terms of the number and type of residual and/or neo-formed possible crystallographic phases that might occur. The cycles of phase identification, Rietveld phase quantification and mass balance calculations were carried out for all samples several times. Mass balance calculations provided a feedback triggering the beginning of a new cycle. In Table 3 summary of the phase composition range is given.

In spite of the time and effort given into the evaluation stage, the study clearly indicates that using high quality synchrotron diffractograms the sample X-ray amorphous content is not overcome and the true

sample composition is not known. Differences among mass balance estimates of the amorphous content for individual samples reflect the limited accuracy of the method.

Characterization of Alumina

Like red mud, alumina is also produced in the Bayer process, in which crushed bauxite is digested with hot sodium hydroxide solution. One of the products is aluminum hydroxide which is then calcined. Depending on the degree of calcination, metallurgical or ceramic grade aluminas are obtained. They differ with respect to the alpha alumina content, grindability, particle shape and size, impurity content, etc.

Application of XRD for the analysis of alumina in the aluminum industry dates back to the sixties. Special aluminas (non-metallurgical grades) have always presented a major challenge [10, 11]. Aluminas may have a variety of morphological forms (Fig. 17) for which there are marked differences in relative intensities of their major XRD reflections (the preferred orientation effect), Fig. 18. Comparing intensities (whether peak height or integrated) of selected reflections from a sample with those of a reference material may cause a serious analytical error. If a reference standard is used in measurement of intensities for a selected reflection followed by quantification, then morphology of this standard must be of the same morphology as the unknowns.

Estimation of occurrence of so called sub-alphas (sub-alphas are phases other than alpha) is only possible with Rietveld method. For quantification of metallurgical and special aluminas a dedicated Rietveld analytical program was built with structure data for 8 alumina mineralogical phases: alpha, beta ($\beta\text{-Al}_2\text{O}_3 = \text{Na}_2\text{O} \cdot 11\text{Al}_2\text{O}_3$), delta, gamma (2), kappa, sigma and theta, also gibbsite. Figures 19-21 give unique examples of phase quantification in aluminas of various origin and phase composition using the Rietveld method. Some of alumina samples composed of sub-alpha phases are partially amorphous.

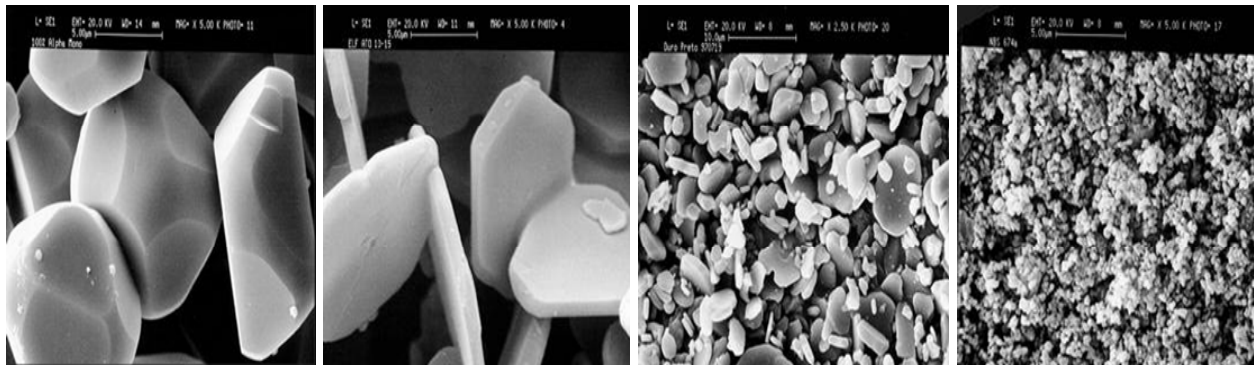


Fig. 17 Examples of morphological forms of ceramic alumina

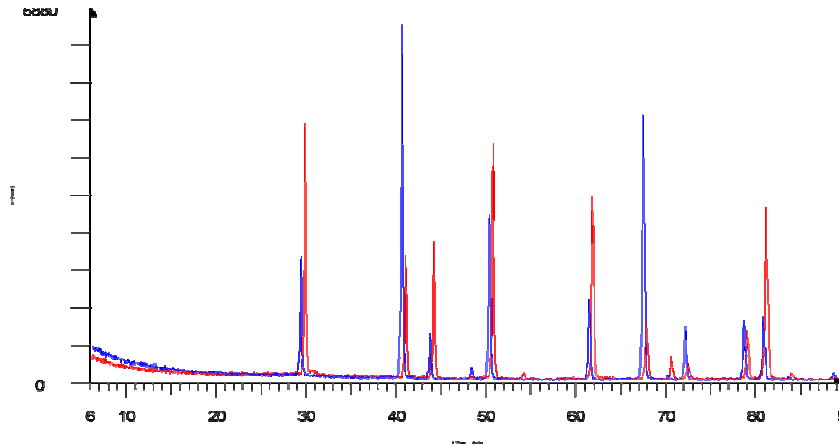


Fig. 18 Superposition of two diffractograms from two ceramic aluminas of different morphology

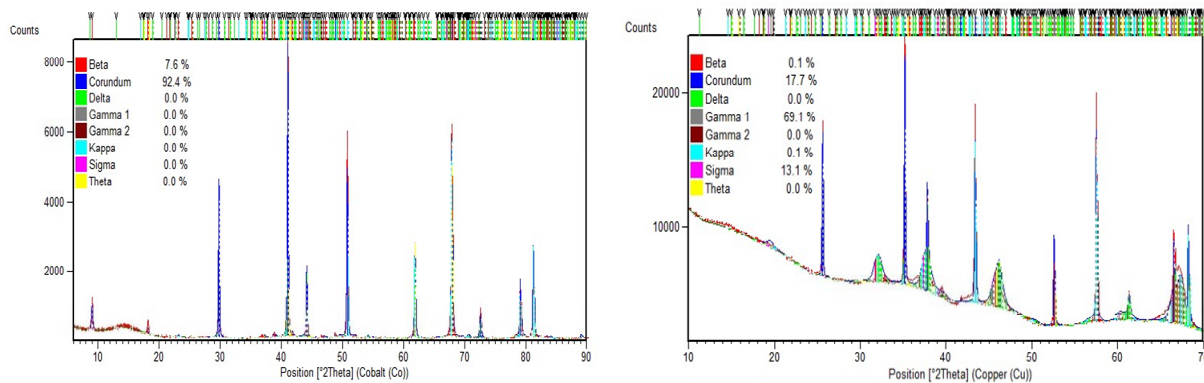


Fig. 19 Example of Rietveld data for ceramic alumina

Fig. 20 Example 1 of Rietveld data for sub-alpha specimen

Scrubber Alumina

Dry scrubber is the most modern and efficient system generally used to prevent all discharge of pollutants from the aluminum plant. Gases from each pot are exhausted into the dry scrubber in which alumina is used to clean fluorine compounds from the pot gas and the dust is collected in filter bags. Up to a total of 99.8% of the overall fluorine is removed from the emissions.

Alumina from the dry scrubber, including bound fluorine mixed with dust, is called reacted alumina and is used in the pots as raw material. This recycling of fluorine in the dry scrubber reduces the need to purchase fluorine materials necessary to operate the pots. Although total F is typically analyzed by XRF or a table-top NMR, XRD is called upon periodically to verify unusual content of reacted alumina or a mixture of alumina and AlF_3 . Fig. 22 gives an example of such analysis. Because most of the sample material is X-ray amorphous the Rietveld analysis gives an indication of distribution of AlF_3 and crystalline alumina phases.

Discussion

As the aluminum industry requirements for better process and quality control become increasingly important, Rietveld-XRD applications have been steadily increasing. From the technological beginning of aluminum manufacture (bauxite) to final manufacturing processes there is and will be a place for Rietveld analysis. Choice of a suitable X-ray instrument will always depend on the price and performance ratio and will be dictated by a particular application. The advantage of XRD as an analytical instrument lies in the variety of characteristic parameters it can measure, simplicity of sample preparation and the ability to run analysis of a series of samples in an automatic mode. Hence, XRD is

capable of responding to today's laboratory demand for even greater flexibility and task multiplicity. Given the fact that future Rietveld applications will be extended to materials such as new ceramics, environmental samples and composites, it is obvious that XRD will remain universal and a very appealing analytical tool for the aluminum industry.

Accurate determination of Al_2O_3 distributed among various minerals is critically important for the industry as it affects material's commercial value and its process performance. It is certainly not realistic to expect Rietveld analysis using an X-ray tube diffractogram to become an accurate tool in the phase quantification of a majority of bauxite. The Rietveld software cannot consistently provide reliable concentrations of gibbsite, boehmite, Al-goethite or kaolinite (which are considered strategic) for most bauxite cases studied over the years. Hence, application of the Rietveld method to bauxite exploration and exploitation is unquestionably limited. The major obstacle in the quantification process is the bauxite amorphous content, which originates not from one but from several sources simultaneously. There are numerous examples of bauxite diffractograms with missing representation of boehmite or kaolinite. Yet occurrence of these phases is confirmed by other methods. The total amorphous content of the bauxite matrix can obviously be quantified using the internal standard method, but assigning its parts to individual mineralogical constituents is impossible employing XRD alone. The synchrotron radiation cannot entirely overcome material's amorphicity either. The best proof of the synchrotron radiation sensitivity to the amorphous material is the contribution to diffractogram from the glass capillary which is obviously amorphous. Synchrotron diffractograms offer substantial advantages over diffractograms from an X-ray tube. Nevertheless, whether from synchrotron or from an X-ray tube, a diffractogram is seldom a sufficiently complete representation of the mineralogical content of bauxite sample. Gibbsite, being the most abundant phase and occurring partially amorphous is the major victim of the Rietveld-XRD analysis of bauxite. Only very well crystallized bauxite from selected deposits can be successfully analyzed, at least for certain mineralogical constituents. Diasporic bauxite shows a better chance for successful phase quantification [Liangqin Nong et al, 2007]. Already for some time new non-XRD methods emerged (mathematical modeling) [4], which have certainly eroded traditional WCh and some Rietveld applications

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ICSOBA MATTERS

ICSOBA Bylaws

During the first 48 year of its existence, ICSOBA was an informal association and its statutes were an internal document only. As a consequence of its formal registration as a Not-for-profit organization, ICSOBA has to comply with the Canadian Not-for-profit Act, including annual reporting and submission of bylaws. The latter were prepared with assistance of a law firm, adopted by the Annual Meeting of Members in Belém and submitted to Corporations Canada by the end of 2012. The full text of ICSOBA's bylaws can be downloaded from <http://www.icsoba.info/about-us>, and a summary is given below.

The bylaws relate to the conduct of the affairs of ICSOBA by specifying rights and obligations of its members, including council members, officers and directors.

Members, Annual Meeting and Financial Statement

Membership is open to natural persons (as individual member) and to corporations (as corporate member) that are interested in furthering ICSOBA's purposes. The purposes are stated in ICSOBA's Articles of Association as:

- promote and organize international and regional events,
- collect and publish documentation for its members, and
- promote the collaboration in the field of bauxite, alumina and aluminium production.

Members may terminate their membership via a letter to Board, they can be expelled or membership can automatically expire if membership fee is not paid within twelve calendar months of the membership renewal date.

Members enjoy reduced event registration fee. In case this is not possible due to a temporary cooperation with a local organization, membership fee payment for that year is included in the event fee. Being a not-for-profit corporation, ICSOBA is not allowed to distribute profits to its members. Expenses incurred by members for organizing ICSOBA activities are reimbursed and directors or officers are allowed to receive a reasonable remuneration for their work. A separately adopted Payment Policy describes who qualifies for allowances, reimbursements and subsidy. ICSOBA's Annual Financial Statement is prepared by ICSOBA's public accountant and available to members.

Members are invited for and have voting rights in members' meetings, which are held in conjunction with an ICSOBA event. Each individual member has one vote and each corporate member can be represented by two employees who have one vote each. A quorum at a meeting of the members is 10% of the members entitled to vote at the meeting (unless a greater number of members is required to be present by the Act). At any meeting of members every question shall (unless otherwise provided by the Articles or bylaws or by the Act) be determined by a majority of the votes cast on the questions. In case of an equality of votes the chair of the meeting shall have a second or casting vote.

Directors and officers

Directors supervise the management of the activities and affairs of ICSOBA. Directors have to be members and are elected by members at the members' meeting, to hold office for two years from the date of election or until their successors are elected or appointed in their stead.

The Board of directors may appoint a committee or advisory body. Such committee may formulate its own rules of procedure, subject to such regulations or directions as made by the Board. Any committee member may be removed by resolution of the Board of directors.

By resolution the Board of directors can appoint officers. A director can hold any office and any two or more offices may be held by the same person. Except in case of President, Vice-President and CEO an officer doesn't need to be a director. The board of officers manages the business and property of ICSOBA. Officers hold office for two years from the date of appointment or until their successors are appointed in their stead. The Board of directors may from time to time and subject to the Act, vary, add to or limit the powers and duties of any officer. The following officers and their duties are described in the bylaws:

- President
- Vice-President
- Chief Executive Officer (CEO)
- Secretary
- Chair of the Council
- Treasurer

Council

The Council is the advisory board of ICSOBA and is composed of the Chair of the Council and 15 to 25 Council members who jointly represent the following areas:

- The technical areas: bauxite, alumina, aluminium, fabrication, environment, etc.
- Scientific areas such as academies of sciences, universities, scientific institutions, etc.
- International societies such as TMS, GDMB, AIM, EAA, etc.
- The regional areas: Asia, Australia, Americas, Middle East, Europe, Africa. A formula is used to ensure adequate regional representation.

The Council members' duties are to advise the board of officers on the course of action, to actively contribute to the organization of activities, such as Events, Newsletters etc., and to promote ICSOBA's wellbeing and standing.

The Council and the board of officers may propose members as new Council member for appointment by the Board of directors, and their appointment is approved in the meeting of members by simple majority. The members of the Council are appointed for the period of two years and they can be re-appointed.

For further information on the bylaws, you can contact the treasurer, Marja Brouwer at m.brouwer@alcortechology.com

Public relations and Communication

Website

Printed proceedings of past ICSOBA events, the so-called Travaux volumes, have been scanned to separate searchable pdf files. There are a few exceptions, these are being searched and scanned as soon as possible. The Tables of Contents of the scanned Travaux volumes have been made public on the website <http://www.icsoba.info/downloads/proceedings-of-past-events>. ICSOBA members can obtain digital versions up to 20 papers each year at no cost by sending an email request to Dipa icsoba@icsoba.info. Additional papers are charged for \$ 20 each.

Your feedback to make the website more attractive is welcome.

ICSOBA's executive office



Not only requests for past proceedings, but all inquiries sent to ICSOBA, whether by email to icsoba@icsoba.info or by phone to + 91 982 328 98 17, are addressed by Ms. Sudipta (Dipa) Chaudhuri in Nagpur, India.

Also mailings and the underlying database of ICSOBA's contacts are taken care of by Ms Dipa Chaudhuri in the executive office.

Corporate members

Currently ICSOBA has the following Corporate Members. For more details including links to the company's website, please refer to the member section of the website: <http://www.icsoba.info/about-us/corporate-members>

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| AMBER DEVELOPMENT | www.amber-development.com |
| BOKELA GmbH | www.bokela.com |
| COLT International BV | www.coltsmelters.com |
| CYTEC | www.cytec.com |
| DUBAL Aluminium Co Ltd. | www.dubal.ae |
| FLSmidth | www.FLSmidth.com |
| Hangzhou New Time Valve Co Ltd | www.hzntfm.com |
| HATCH Associates | www.hatch.ca |
| HINDALCO Innovation Centre | www.hindalco.com |
| NALCO Ecolab | www.nalco.com |
| OUTOTEC Pty Ltd | www.outotec.com |
| RIO TINTO ALCAN | www.riotintoalcan.com |
| Shandong Jingjin Filter Press | www.dmxs-com@263.net |
| STC Engineering GmbH | www.stc-engineering.de |
| WesTech Process Equipment India P.Ltd | www.westech-inc.com |