AA29 - Gallium Balance in a Greenfield Alumina Refinery

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Abstract

Gallium chemistry and material balance across the Al Taweelah alumina refinery were investigated to identify the influential factors on gallium extraction from a complex chemistry perspective associated with high-temperature digestion, as well as the mechanisms of crystallisation in precipitation. The objective was to understand and improve the quality of produced alumina. In contradiction of the very limited literature on this subject, changes in the parameters of the precipitation circuit directly impacted the uptake of gallium into hydrate (the precursor product to alumina). A modelling approach was proposed to better describe the precipitation conditions that drive the incorporation of gallium into the product. Understanding and knowing how to manipulate this behavior is critical in any gallium management strategy that aims to avoid undesihrable gallium contamination.

Keywords: Gallium extraction, High temperature digestion, Gibbsite precipitation, Alumina quality.

1. Introduction

Al Taweelah alumina refinery successfully started producing alumina in 2019 and has now significant exceeded nameplate capacity. The quality of alumina produced is an important goal of this project and is largely dictated by the bauxite quality, the technology selected, and process conditions employed.

A more rigorous gallium balance investigation became important to understand the impacts of impurities and how to mitigate deviations. Gallium was selected as an in-focus element due to the desire to maintain lower contents in the product.

The aim of this paper is to provide a practical demonstration of gallium balance using plant data, to validate an observation. More fundamental work is required to understand the mechanisms involved in the observations from process information.

2. Gallium Review

The world's most important source of gallium is bauxite. Some authors have reported its main association with Ti minerals [1] and hematite [2,3], while the majority of the literature indicates that gallium is present primarily as a substituted element for aluminium in the aluminous minerals. Gallium is dispersed and associated with the aluminous minerals because of the similarities

between Ga^{3+} and Al^{3+}. The two occupy the same structural positions in minerals, maintaining a nearly constant Al:Ga ratio [4]. Therefore, it is likely that gallium in bauxite is distributed amongst the aluminium bearing mineralogy at a relatively constant Al:Ga ratio (i.e., within gibbsite, boehmite, and kaolin). Importantly, aluminium is also substituted in goethite and to a lesser extent in hematite. It's likely also that gallium is present in the iron mineralogy at a relatively constant ratio with the substituted aluminium. The constant Al:Ga ratio implies that the gallium content in various bauxite ores should also be relatively constant, tied more to the alumina content of the bauxite, rather than the source of the bauxite. In fact, a study of gallium concentrations for bauxite deposits worldwide found that the gallium concentrations in lateritic bauxites was on average 77 ppm Ga_2O_3, and, for karst bauxite deposits, an average of 78 ppm Ga_2O_3, indicating that there are no substantial differences in gallium concentrations between karst- and laterite-type bauxites [5].

Al Taweelah alumina refinery employs a high temperature digestion process (i.e., 280 ºC). During digestion of bauxite for the production of alumina, a fraction of gallium is extracted along with the aluminium species. Within the literature, it is reported that approximately 70 % of the gallium is extracted from the bauxite into the liquor, the remaining 30 % being disposed with bauxite residue [6–8]. There is very limited information available in the literature regarding the digestion process conditions and liquor that would impact gallium extraction yield from the bauxite.

The aqueous chemistry of gallium is very similar to aluminium. In concentrated caustic solution, aluminium forms a monomeric tetrahedral hydroxo complex [$Al(OH)^{4-}$], and in very concentrated aluminate solutions, an oxo-bridged dimer is also formed. Gallium is more soluble in caustic however, and it has been found that only the tetrahedral hydroxo complex [$Ga(OH)^{4-}$] species predominates. Despite the similarities between Al and Ga (very similar atomic/ionic sizes) this differing behavior was attributed to the different chemistry and physics of gallates and aluminates, with the study concluding that gallates are more compact structures than aluminates [6].

In the Bayer process, gallium will dissolve in the caustic liquor and only reaches quite low concentrations, typically between 100 to 500 ppm, far below its solubility. The pregnant Bayer liquor, containing this small amount of gallium will determine the incorporation in the product.

Shaw et all [7] examined the incorporation of gallium into precipitated gibbsite as a function of gallate concentration in batch precipitation tests (96 h, 50–200 g/L seed, alumina to caustic ratio (A/C) 0.5 to 0.7, total caustic level (C) 180g/L, caustic to total alkali ratio (C/S) 0.818, 60 ºC). At these conditions they found that the gallate concentration is linearly related to the gallium content in product (4 % uptake) and that it is likely incorporated by isomorphous substitution. Isomorphous substitution is the substitution of atomic gallium for aluminium in the structure of gibbsite. While the authors claim that temperature also has no impact, the data in this paper is contrary to this finding.

A proper gallium removal system in an alumina refinery brings few benefits. The main direct benefit is the production of gallium metal for commercial application. The indirect benefit is a reduction in gallium in product, hence improving product quality. Understanding the balance well can provide a way to mitigate product quality issues, without necessarily installing a gallium removal process.

3. Gallium Balance in the Refinery

The main input of gallium is bauxite. Major outputs are with bauxite residue, both as physical phase (soluble) and as solid phase and majority of the output reports to the calcined alumina as a trace element. Figure 1 represents a simplified version of the Bayer process with the most relevant points for the balance of gallium. Measurements of bauxite, mud, and alumina were done via X-

ray fluorescence (XRF). Liquor analysis was done via Inductively Coupled Plasma (ICP) spectroscopy. It is also important to perform a gallium balance in digestion, to assess the extraction performance and in precipitation (agglomeration and growth section) to evaluate the gallium uptake from the liquor to hydrate.

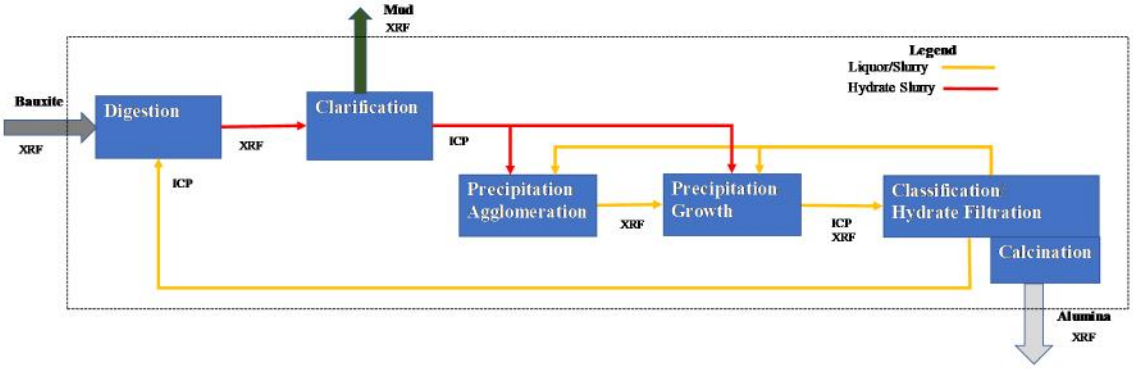

Figure 1. Simplified Bayer circuit with major inputs and outputs for gallium balance.

Table 1 presents all measurements of gallium to perform a proper gallium balance. The sample frequency was daily.

Table 1. Main analysis in the input and output streams of the refinery.

	Analysis
Analysis bauxite feed	XRF
Digestion discharge	XRF
Mud	XRF
PGL liquor	ICP
SPL Liquor	ICP
Hydrate from agglomeration	XRF
Hydrate from last precipitator	XRF
Alumina product	XRF

In a gallium balance, the primary source of gallate in Bayer liquor is from the bauxite and this is the main determinant of gallium in product. However, there are two components of the balance that offer relevance:

1. Bauxite extraction yield, that measures the percentage of gallium in the bauxite that is leached to the caustic liquor. This will influence the mass of gallium extracted from the bauxite, the remainder being disposed with the bauxite residue.
2. The gallium uptake in precipitation, which is the percentage of gallium mass in the Pregnant Liquor (PGL) that will be incorporated into the hydrate and, hence final product. The remainder will recirculate back to digestion.

3.1 Extraction Yield from the Bauxite

The limited information from literature indicates a gallium extraction of 70 % [9–10]. For Al Taweelah alumina refinery, due to the high temperature process (i.e., 280 ºC digestion temperature) and the possibility of gallium extraction from aluminous-goethite, it was assumed initially an extraction value of 80 %. Equation (1) is a simplified calculation of gallium extraction, assuming that iron and titanium oxides are relatively inert and remains as solid after digestion process.

$$E_{Gallium} = \left\{1 - \left(\frac{\%Fe_2O_{3\,Bauxite} + \%TiO_{Bauxite}}{\%Fe_2O_{3\,Mud} + \%TiO_{Mud}}\right).\left(\frac{\%Ga_2O_{3\,Mud}}{\%Ga_2O_{3\,Bauxite}}\right)\right\}.100 \quad (1)$$

Where:

$E_{Gallium}$	Percentage by mass of total gallium extracted from the Bauxite, %
% $Fe_2O_{3\ Bauxite}$	XRF of total iron oxide in Bauxite, %
% $Fe_2O_{3\ Mud}$	XRF of total iron oxide in the filtrated mud after Digestion, %
% $TiO_{\ Bauxite}$	XRF of total titanium oxide in Bauxite, %
% $TiO_{\ Mud}$	XRF of total titanium oxide in the filtered mud after Digestion, %.

Based on Equation 1, gallium extraction was calculated daily using XRF samples. Data for the first 30 months of operation is presented in Figure 2.

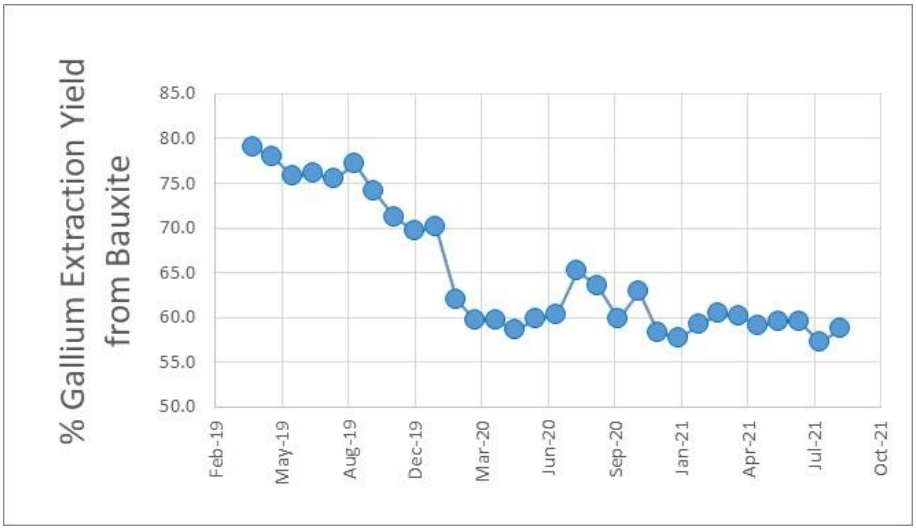

Figure 2. Gallium extraction as a function of time since start-up of Al Taweelah refinery.

As expected, during the early months of refinery operations, the extraction of gallium was marginally below 80 %. Surprisingly, after several months, it quickly declined to values between 60 %. This indicated that something has changed over time and a few hypotheses have been identified. There is a scope of work in progress to further understand this extraction drop effect and other impacts such as lime addition to the holding tube.

For this study, it was assumed a steady state extraction of 62 % but a further study to investigate this extraction drop is in progress.

3.2 Precipitation Uptake from PGL Liquor

Once the baseline for digestion extraction was established, it was necessary to define the behaviour of gallium uptake in the product during the precipitation process, to close the gallium balance.

Analysis of plant data from the early months of operation of Al Taweelah alumina refinery shows that the there is a strong relationship between gallium in liquor and gallium in product (Figure 3), where gallium in product increases with increasing gallium in liquor. The short significant discrepancy at refinery start-up was due to the gallium content of the imported hydrate used to start up the refinery.

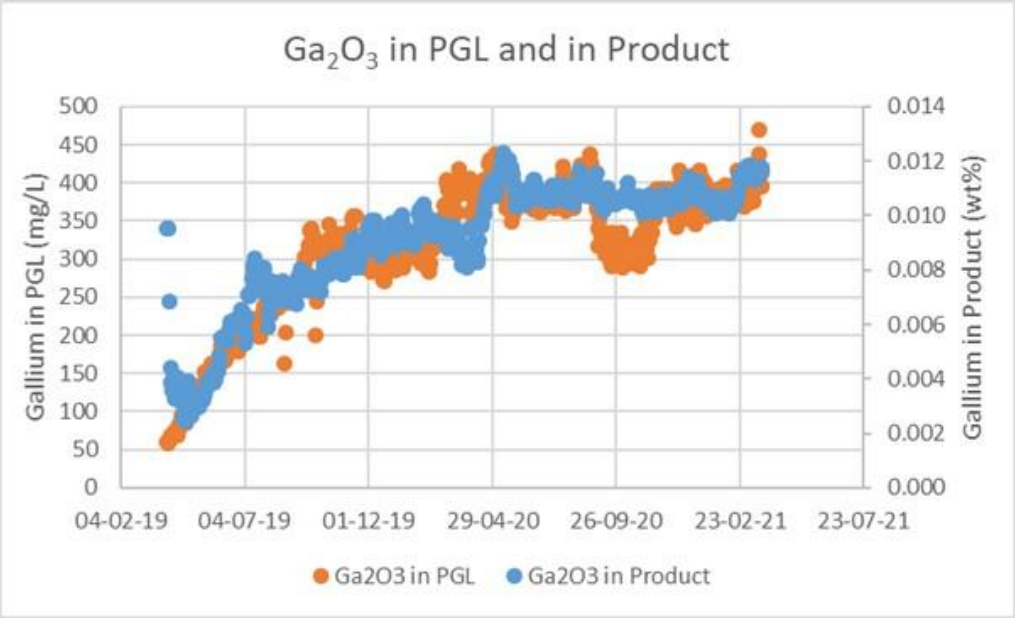

Figure 3. Gallium in PGL and gallium in product.

Al Taweelah alumina refinery's precipitation circuit operates with five agglomeration tanks, arranged in series, and for the growth side, two trains, each with 16 tanks in series. Process data from precipitation, over a relative stable period in the refinery was selected to understand the balance. The data selected was extracted was from the period of 6 May 2021 until 10 July 2021. The flow during this period was stable, with total liquor to precipitation of 3500 m^3/h. There were also very minor variations in precipitation temperature profile and hydrate sizing. A summary of this balance is presented in Table 2.

Table 2. Gallium balance in precipitation.

	Mass of Gallium Precipitated	Gallium Mass Distribution	Mass of Hydrate Precipitated	Hydrate Precipitation Distribution	Gallium Composition of Hydrate Produced
	kg/h of Ga_2O_3	%	tonne/h	%	% Ga_2O_3, alumina basis
Agglomeration	18.3	54	170.7	37.2	0.016
Growth tanks	15.7	46	288.0	62.8	0.008
Total	34.0	100	458.7	100	0.011

According to data from Table 2, the total mass of gallium precipitation in agglomeration and growth sections was determined (as per Figure 1 simplified schematic) to be approximately 50 % of gallium precipitation in agglomeration and 50 % in growth. However, the proportion of hydrate precipitation in agglomeration and growth is 40 % and 60 % respectively. This means the contamination of gallium is significantly higher in agglomeration with fresh crystals reaching almost 160 ppm of gallium, compared to hydrate crystals produced in the growth section at 80 ppm. In agglomeration, precipitation velocity rate is much higher compared to growth and it is known that certain impurities such as sodium, tend to be higher as well.

It is empirically understood that gallium in product correlates with the behavior of bound soda. To validate this observation, direct data of Na_2O (%) and Ga_2O_3 (%) from XRF analysis of

samples collected from last agglomeration slurry (LAPS) and data from last precipitation pump-off slurry (POS) was directly correlated and presented in Figure 4.

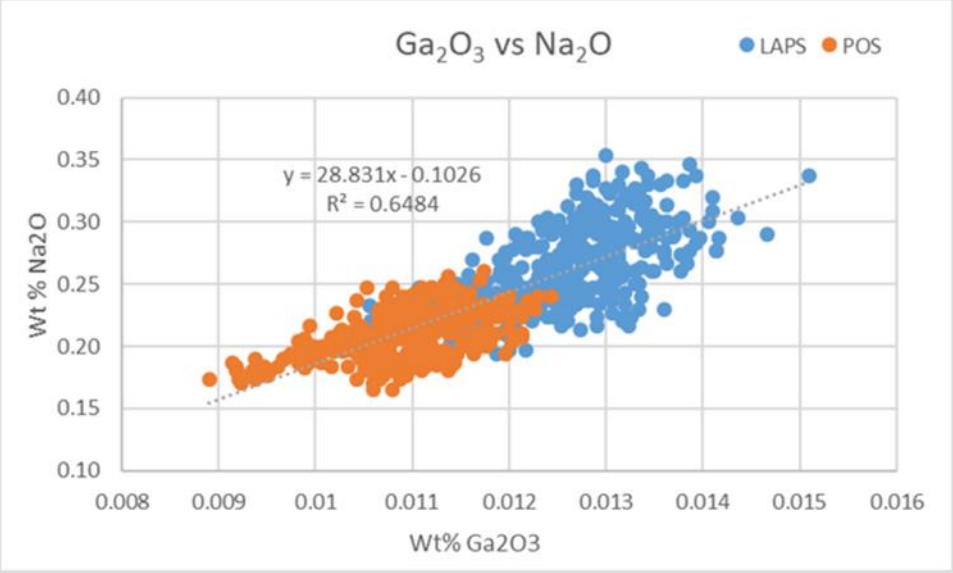

Figure 4. Gallium vs soda in LAPS (Last Agglom Precipitator Slurry) and POS (Pump-off Slurry) for the period where gallium in liquor was relatively constant.

Using this information, it was possible to propose a model of gallium uptake in precipitation based on the correlation with soda uptake and gallium in liquor. This was defined as:

$$Gallium_{LAPS} = K_1 . \frac{Ga_2O_{3PGL}}{Al_2O_{3PGL}} . (0.0278 . Na_2O_{LAPS} + K_2) \quad (2)$$

where:

$Gallium_{LAPS}$	Gallium in hydrate product from agglomeration, % Ga_2O_3 alumina basis
Na_2O	Total Sodium in hydrate product from agglomeration, % Na_2O alumina basis
Ga_2O_3 PGL	Gallium concentration in pregnant liquor, g/L
Al_2O_3 PGL	Alumina concentration in pregnant liquor, g/L
K_1 and K_2	Constants,0.56 and 0.0051 respectively.

Figure 5 shows the correlation between actual Ga_2O_3 in LAPS and predicted. The advantage of being able to correlate gallium uptake with soda is that the mechanism of gallium uptake in hydrate is not yet well understood, but there are several models that describe the uptake of soda in hydrate (e.g., Ohkawa, et al. [11]; Armstrong, et al. [12], Vernon, et al. [13]).

Two of these models are preconfigured in Syscad [11–13]. These were compared and found to show very similar results for soda uptake through each agglomerator and growth precipitation tank. It is empirically well understood that low temperature and high supersaturation both contribute to higher soda levels, as demonstrated by the dependency of SSAT (supersaturation, expressed as (A-A*)/C) and temperature on soda uptake in the following formula from Armstrong, given in Section 3.2.1 as a direct quotation from the Syscad online help [14]. The model of Armstrong, was selected to be used in this study.

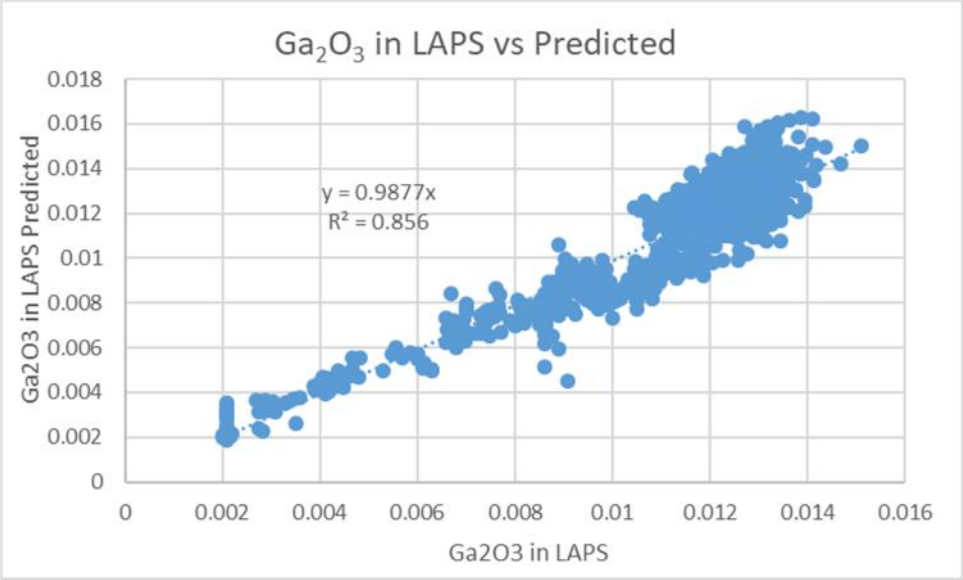

Figure 5. Gallium in LAPS, actual versus predicted.

3.2.1 The Armstrong et al. (1996) Model for Bound Soda [12].

Armstrong, Hunter, McCormick and Warren [12] correlation defines the rate of incorporated soda with the alumina precipitation as:

$$\frac{dS}{dA} = K_{Soda}\left(\frac{A_o - A^*}{C_o}\right)^2$$

where:

K_{Soda} *Overall occluded soda factor,* $K_{Soda} = K_{TuneBS} * K_{Factor}$
K_{TuneBS} *Tuning factor. Default 1.0*

$$K_{Factor} = \left[0.000598C_{25} - 0.00036T_c + 0.019568\left(\frac{TOOC}{C}\right)_{25}\right]$$

dS *Rate of soda precipitation expressed in* Na_2O
dA *Rate of trihydrate alumina precipitation expressed in* Al_2O_3
Ao *Alumina concentration leaving the precipitator expressed in* Al_2O_3 *(g/L)*
*A** *Saturated alumina concentration (g/L)*
Co *Caustic concentration leaving the precipitator expressed in* Na_2CO_3 *(g/L)*
Tc *Temperature in degrees C*
TOOC *Total organic carbon expressed as* Na_2CO_3

3.2.2 Bound Soda / Organic Distribution [12]

The bound soda rate calculated is in terms of $Na_2O(s)$*, however the soda precipitation will be expressed in terms of NaOH*(s) and the bound organics as* $Na_2C_5O_7$**(s).*

BoundSodaFrac:

$$\text{BoundSodaFrac} = \frac{dS}{dA}$$

Bound Soda Precipitation - as NaOH:*

$$\text{BoundSodaFrac} \times dA \times \left(1 - \frac{\text{BoundSoda OrgPart}(\%)}{100}\right) \times \frac{2\mathcal{M}_{NaOH}}{\mathcal{M}_{Na_2O}}$$

Bound Organics Precipitation - as $Na_2C_5O_7$:*

$$\text{BoundSodaFrac} \times dA \times \left(\frac{\text{BoundSoda OrgPart}(\%)}{100}\right) \times \frac{\mathcal{M}_{Na_2C_5O_7}}{\mathcal{M}_{Na_2O}}$$

NOTES:

- *BoundSodaFrac is the (total bound soda + bound organics as Na_2O) per (THA as Al_2O_3)*
- *For User Species models, the bound organic precipitation will be in term of the user-species-model's bound organic species.*

3.3 Modelling Approach and Validation

The dependency of soda uptake with SSAT is also supported by plant data (Figure 6) showing a strong relationship between LAPS %Na_2O and LAPS SSAT (Supersaturation, expressed as A/A*).

Therefore, the inputs and assumptions used in the Syscad model are summarized as follows:

- Ga_2O_3 in bauxite: 75 ppm;
- Extraction in digestion: 62 %.

The model reflected a refinery base case scenario with current digestion flow and production rate. Last washer underflow caustic concentration was 19.1 g/L Na_2CO_3. For each agglomerator and growth precipitator tank, the gallium uptake was determined by Equation 2, with the bound Na_2O defined from the Armstrong et. al bound soda model [12].

The above defined the extent of reaction:

$$2NaGa(OH)_4 = 2NaOH + Ga_2O_3 + 3H_2O \qquad (3)$$

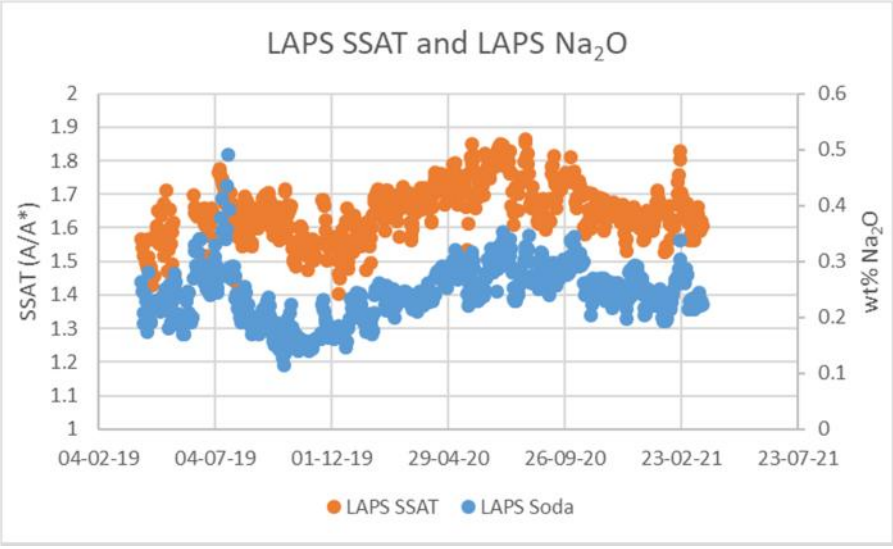

Figure 6. Last Agglomeration Soda and Supersaturation.

The gallium balance from the Syscad model is summarized in Table 3, normalized to tonnes of alumina produced.

Table 3. Gallium balance.

		kg Ga_2O_3/tonne Al_2O_3	%
Input	Ga_2O_3 in Bx	0.179	100
Outputs	Ga_2O_3 with physical soda loss	0.009	5
	Ga_2O_3 within solids residue	0.068	38
	Ga_2O_3 with product	0.102	57
Ga_2O_3 in PGL		375 mg/L	

From Table 3, it can be seen that physical soda takes out 5 % or so of the gallium. Therefore, doubling the physical soda loss would increase this to 10 % and the Ga_2O_3 in product will drop. If the Ga_2O_3 in product were at 120 ppm, then will drop to 110 ppm, a reduction of only 10 ppm. This sensitivity was confirmed with Syscad. If gallium extraction increases to 95 %, gallium in product is expected to increase beyond Smelter specifications, to around 130 ppm.

Table 4 provides a more detailed view of the gallium uptake within precipitation tanks.

Table 4. Gallium balance.

Precipitator Tank	Ga_2O_3 in Al_2O_3 (wt%)	Precipitator Tank	Ga_2O_3 in Al_2O_3 (wt%)
Agglom 10	0.0221	Growth 10	0.0134
Agglom 20	0.0161	Growth 20	0.0129
Agglom 30	0.0109	Growth 30	0.0104
Agglom 40	0.0086	Growth 40	0.0110
Agglom 50	0.0096	Growth 50	0.0096
		Growth 70	0.0105
		Growth 80	0.0095
		Growth 90	0.0098
		Growth 100	0.0091
		Growth 110	0.0085
		Growth 130	0.0081
		Growth 140	0.0077
		Growth 150	0.0074
		Growth 160	0.0074

Like for bound soda in product, the most significant influence on gallium in product is going to be where both yield and SSAT are the highest. These conditions are most significant in the lead agglomerator and it can be clearly seen in Table 4 that the lead agglomerator is a significant driver

for gallium uptake in product. Similar to soda control strategies used elsewhere, this is the logical point of control for gallium management strategies utilizing the manipulation of precipitation conditions.

Therefore, increasing agglomeration temperature and/or reducing lead agglomeration SSAT will act directly to reduce the uptake of gallium into hydrate (product). However, while the lead agglomeration supersaturation is fairly easy to measure, the measured growth rate, taking into account the yield in the tank and the seed specific surface area, is a reflection of not just the thermodynamic driving force (SSAT) of growth, but also takes into account the temperature dependence through an Arrhenius term, exp-(Ea/RT). Therefore, it is plausible that the mechanism of soda and gallium is more closely linked to growth rate rather than SSAT. Hence growth rate is considered to be a more reliable control measure in this study.

While the behavior of bound soda and gallium uptake within precipitation appears to mirror each other, it cannot be concluded that their uptake is controlled by the same mechanism, particularly as the gallate ion is more analogous to the aluminate ion rather than the sodium ion.

At a fixed temperature, both soda and gallium uptake into gibbsite will increase with increasing gibbsite growth rate. For soda, this is thought to be because with increasing growth rate, the ability for the sodium ion associated with the aluminate ion (presumably existing as a polymerised species) has less time to diffuse from the crystal surface before there is subsequent growth, encapsulating the sodium ion within the gibbsite matrix.

At a fixed gibbsite growth rate, both soda and gallium uptake into gibbsite will decrease with increasing temperature. For soda, this is thought to be linked to the increase in diffusion rate of ions, as the temperature increases the diffusion rate of ions also increases, and hence, at higher temperature, the soda would be able to diffuse more rapidly out of the growing gibbsite layer resulting in relatively low soda incorporation [15,16].

In the Growth section, variations in SSAT and temperature (hence linear growth rate) of the first tank can also influence the gallium variations in the final product if conditions of SSAT and temperature in agglomeration are fairly constant. In Al Taweelah alumina refinery, variations in temperature of the lead Growth tank by manipulation of cooling will drive variations in SSA and consequently, in gallium as well. In fact, a direct relation between the percentage of gallium in the final product and the lead growth SSAT (or inverse of temperature vs gallium) is easily observed, as per Figure 7.

The growth of gibbsite is thought to happen as sodium aluminate ions polymerise at the gibbsite surface. With increasing polymerisation, the alumina to soda ratio of the ion pair increases. If the gallate ion does not become incorporated in the aluminate ion polymerisations, then perhaps it remains associated with only the soda ion and as a result, follows a similar mechanism for incorporation, essentially existing in the gibbsite matrix as sodium gallate rather than isomorphous substituted $Ga(OH)_3$.

In this way, the behaviour of the gallate ion (likely present as a contact pair with sodium) is very similar to the behaviour of organics; the surface of the gibbsite is crowded with polymerizing aluminate ions and adsorbed species like organics and gallate, and at high linear growth rates it is unable to diffuse away in time to avoid being captured as part of the growth front. It then gets incorporated isomorphically in the gibbsite structure (along with its attendant sodium ion).

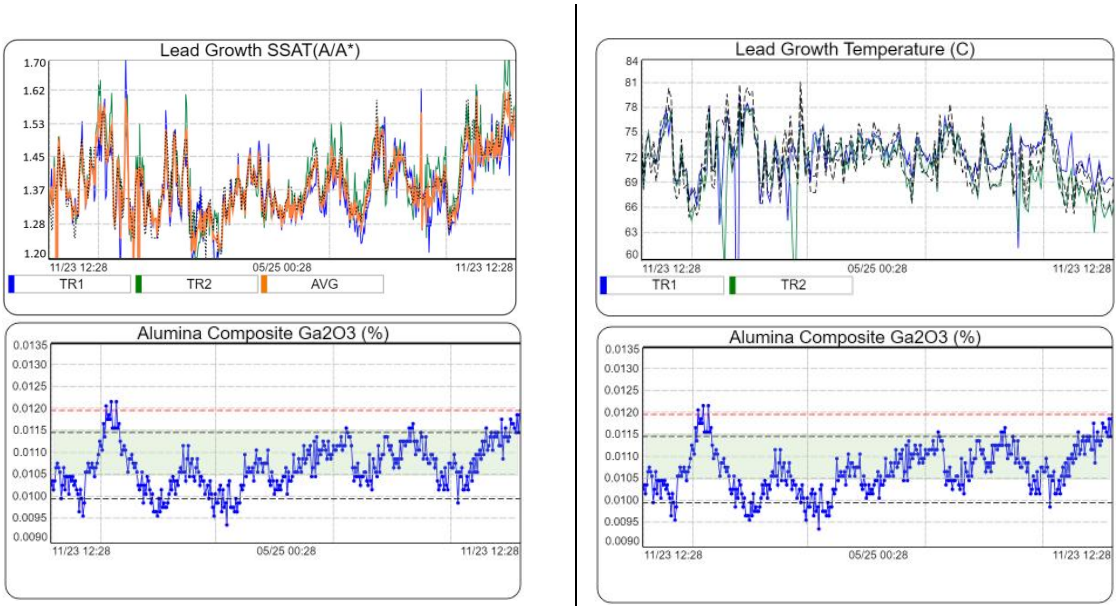

Figure 7. Supersaturation of both trains (Train 1 and Train 2) versus gallium in final product. Also Lead Growth Temperature versus gallium in product.

4. Conclusions

While there are fundamental gaps in the understanding of how gallium is incorporated into gibbsite (hydrate / product), the modelling in this study has assisted in providing a clear understanding of the impacts of precipitation conditions that drive the incorporation of gallium into the product and the gallium balance within the refinery. It is well understood what changes to the precipitation circuit will act directly on the uptake of gallium into hydrate (product). Understanding and knowing how to manipulate the precipitation behaviour is critical in any gallium management strategy and provides a good and aggressive short-term reaction that can assist in avoiding out of spec alumina. However, with lower uptake into the product, this allows the Ga_2O_3 level in liquor to rise.

Around 80 % of the gallium in bauxite was extracted into the liquor during start up but this extraction efficiency quickly depleted over the first 30 months down to 60 %. More scope of work is required to understand this effect.

Changes to the precipitation circuit will act directly on the uptake of gallium into hydrate (product). The modelling has assisted in providing a clear understanding of the impacts of precipitation conditions that drive the incorporation of gallium into the product. Understanding and knowing how to manipulate this behaviour is critical in any gallium management strategy and provides a good and aggressive short-term reaction that can assist in avoiding out of spec alumina.

With lower uptake of gallium into the final product, this allows the Ga_2O_3 level in liquor to rise. The combination of better understanding of gallium extraction as a function of gallium in liquor and gallium uptake in precipitated hydrate will potentially reduce criticality to implement mechanisms for gallium removal from liquor if there is a further drive to reduce this trace element in the final alumina.

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