

Management and Control of Sodium Oxalate Precipitation in the Bayer Process

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ABSTRACT

The presence of sodium oxalate in Bayer liquor presents a number of challenges to alumina refinery operators. In many plants, reducing or eliminating the detrimental effects of solid sodium oxalate crystals on process operations requires significant effort and resources. As a result, management of the precipitation of sodium oxalate from solution is integral to efficient operation.

A key to the appropriate management of oxalate is to control where and how it precipitates within the process. Ideally, sodium oxalate precipitation should be prevented in those parts of the process where it is undesirable and enhanced in those areas where solid oxalate is wanted. Control of the crystal form (or morphology) of the oxalate is also critical. Crystal Growth Modifiers (or CGMs) are known to affect oxalate precipitation in various ways, while other chemical additives such as oxalate stabilizers and morphology modifying agents can be readily applied to enhance the control of oxalate.

Recent development of a new range of morphology modifiers (the ORT product range) has enabled more effective production of oxalate in the ball form, instead of the needle-like crystals that typically precipitate. The development and use of these new ORT additives is presented, together with how these products can be used within different operational strategies to enhance management and control of sodium oxalate in the Bayer process.

1. INTRODUCTION

Sodium oxalate is a common contaminant within the Bayer process. Typically, oxalate is formed by the breakdown of soluble organic compounds in the liquor. Organic material within the bauxite, conventionally considered to be composed of a broad variety of compounds including humates, fulvates and lower molecular weight material, is extracted from the bauxite

through digestion. A small amount of soluble organic material can also enter the liquor through contamination (e.g. greases and oils from sumps) or direct addition of process additives (for example, flocculants, antifoams). In general, however, the greatest source of organic content within any Bayer liquor is the bauxite.

The nature and concentration of these organics can vary significantly depending on the bauxite source and quality, the process conditions (particularly digestion temperature and liquor concentration) as well as a range of other refinery specific parameters. Historical work has identified a range of components and classes of organic compounds present in Bayer liquors (Sato and Kazama, 1971; Lever, 1978; Gnyra and Lever, 1979; Lever, 1983; Guthrie *et al*, 1984) with further work continued more recently (Maher, 2015).

Common to all conditions however is the degradation of these soluble organic compounds over time to lower molecular weight compounds (and eventually to carbonate). Specifically, the cyclic nature of Bayer plant operations and the process conditions (time, high caustic, heat) lends itself to degradation of these compounds.

2. OXALATE IN THE BAYER PROCESS

The formation of oxalate (as sodium oxalate) is a critical step in the degradation process. Sodium oxalate in solution is relatively benign in terms of impact on the process. Like other soluble organic components in the liquor, it contributes to overall liquor viscosity. Beyond this however, the presence of soluble oxalate in liquor is not a major problem. In contrast however, the presence of precipitated sodium oxalate crystals (or solid phase oxalate) within the operation can significantly hinder process efficiency across a number of unit operations. As a result, control of sodium oxalate in solution together with management of precipitated sodium oxalate - where it might precipitate, in what form and how to deal with

it once present - is often a significant issue within Bayer plant operations and generally requires the application of substantial resources and time.

As a consequence, over the years there have been a wide variety of methods proposed, developed and utilized to manage and control oxalate in Bayer plants. (Grocott and Harrison, 1996).

2.1 SODIUM OXALATE PRECIPITATION

The precipitation of sodium oxalate is represented in equation (1) below and is a simple combination of sodium ions and oxalate ions.



Given the caustic nature of Bayer process liquor the conjugate acid of oxalate (oxalic acid) is effectively absent. In addition, the high concentration of sodium ions ensures that the solubility limit is generally exceeded. In water at 25°C the solubility of sodium oxalate is listed as 36g/L (Aylward and Findlay, 1981). However, the concentration of oxalate in Bayer liquor is generally limited to less than 10g/L and in most cases is much less than 5g/L.

This can vary with the sodium ion concentration of the liquor and the conditions of the matrix (for example, temperature) which can change within an individual plant. As a result, while a liquor may be under-saturated with respect to oxalate in one part of the plant, downstream in another part of the plant, the same liquor may become supersaturated due to a change in either the process conditions or the liquor chemistry. Effectively then, the solubility of oxalate in Bayer liquor is variable, depending on conditions.

In addition to solubility, perhaps a more important concept is that of oxalate stability. Oxalate dissolved in Bayer liquor can be supersaturated and form a meta-stable solution. In some respects, this is particularly convenient given the transient nature of the conditions in the Bayer circuit. If the oxalate in a liquor is stable in solution for a specific period in a given part of the circuit, then this property can be utilized.

Stability of oxalate in Bayer liquor can vary considerably and is strongly influenced by the presence of organic components in the liquor. As well as being a source of oxalate through degradation, some organic compounds present in Bayer liquors can significantly influence stability of oxalate in solution. Variation of oxalate stability can occur with organic loading as well as the mix of compounds present such that significant changes in oxalate stability from one day to the next are common in many plants. Extensive work has also considered the use of synthetic oxalate stabilizer products (Farquharson *et al*, 1995; Farquharson *et al*, 1996; Sipos *et al*, 1999) and such

products have been extensively used within the industry for many years to help control and manage oxalate.

Stability of oxalate can also be influenced by removal of organic compounds from the liquor (effecting a destabilization of oxalate). A number of organic removal processes are known or have been assessed (Soucy *et al.*, 2004; Maher, 2015) and while the overall aim is generally to remove organic compounds, the removal of those compounds that stabilize oxalate may not be a positive outcome. As a result, the impact of any changes to the organic loading within a liquor needs to be carefully considered.

3. EFFECTS OF SOLID PHASE OXALATE

It is well established that precipitation of sodium oxalate in the early stages of the trihydrate precipitation circuit can cause excessive generation of fine crystals of trihydrate (Lever, 1978; Power and Tichbon, 1990). This can result in product alumina of unacceptable size distribution. In general terms, the solid sodium oxalate crystals act as a secondary seed surface and fine particles of trihydrate nucleate on the surface of the oxalate crystals.

In addition, the presence of solid oxalate with alumina trihydrate can inhibit slurry filtration. The oxalate crystals can form a network within the filter cake which restricts the flow of liquor through the cake and thereby slows the rate of overall filtration

Solid oxalate can also impact on trihydrate classification by preventing appropriate separation of trihydrate in settling tanks. The formation of matted rafts of oxalate needles which settle poorly and capture trihydrate particles is an extreme version of this effect. In the worst instances, such rafts of oxalate and trihydrate can also capture air bubbles within their matrix and this can cause the solids to float and form a scum or foam layer on the top of tanks. In such instances, the associated liquor can often drain (or dry out) leaving a highly undesirable crust as a thick surface of the scum. Formation of such a "blanket" not only prevents appropriate mixing and/or classification, but it may also act as an insulating blanket and restrict the desirable heat loss from the process. In addition, process control can be made more difficult since the presence of a scum layer can make tank level monitoring problematic.

In individual refineries and under different circumstances solid oxalate in the process can also present a number of other challenges (for example, scale formation, soda in product). As a result, the appropriate management and control of oxalate - both in the liquor and solid phase - is an important part of efficient operation of those alumina refineries with substantial organic input.

4. STRATEGIES TO MANAGE OXALATE

In order to reduce or eliminate the detrimental impacts of oxalate on process operations, most refineries where oxalate is present in significant quantity, practice removal and control of oxalate in one way or another.

Efficient removal of oxalate can be used to maintain the concentration of oxalate present in the system within a “manageable band.” This allows process operators to precipitate solid sodium oxalate in appropriate areas of the plant that limit the detrimental effects and allow for appropriate removal from the process. While a number of different removal systems are used or have been proposed, (Grocott and Harrison, 1996) they all require substantial resources and can be expensive and difficult processes to operate.

The corollary of this is to maintain sodium oxalate in solution such that the “manageable concentration” of oxalate in the liquor is elevated. Again, this practice is well established and is often coupled with a separate removal system in order to maintain the status quo and balance the input of oxalate from the bauxite.

While the strategies employed can differ widely in terms of their specific operation, the vast majority of operators focus on one, or all of the following goals:

- (a) Avoid having solid phase oxalate in parts of the process where it is not wanted. (Maintain sodium oxalate in solution in those parts of the plant where it causes issues.)
- (b) Enhance the formation of solid phase oxalate in specific areas to facilitate removal of oxalate from the circuit.
- (c) Precipitation and removal of oxalate from the process to prevent build-up of concentration in the liquor

Control of oxalate in Bayer plants is generally achieved by removal of solid oxalate by one of two means. The first involves co-precipitation of sodium oxalate with trihydrate in the latter sections of the precipitation area where low alumina supersaturation allows the oxalate to precipitate with little or no nucleation of trihydrate generated on the oxalate surface. This minimizes the effect on trihydrate sizing. The oxalate is then usually separated from the filtered trihydrate (for example by water washing). The oxalate rich filtrate is then disposed by some means. This is generally called the co-precipitation process.

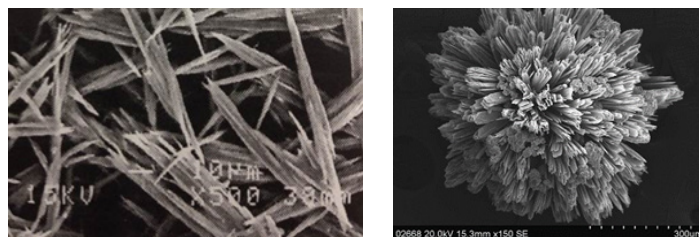
The other method, known as side-stream removal, differs in that the entire trihydrate precipitation process is operated free of solid phase oxalate. A side-stream of spent liquor is treated to precipitate and remove sodium oxalate. The resulting liquor from the side-stream, containing a lower concentration of oxalate, is then returned to the main flow.

It should be recognized that in both cases, stabilization of oxalate in the precipitation slurry is desirable in at least part of the plant, while in a different part, maximum precipitation of oxalate is required. For side-stream removal processes, the oxalate needs to be maintained in solution for the entire duration of the trihydrate precipitation process. In co-precipitation plants oxalate needs to be maintained in solution until the last part of the trihydrate precipitation process. In both processes, co-precipitation of sodium oxalate in the first part of the precipitation process needs to be avoided.

The process parameters that can be used to control precipitation of oxalate (for example, temperature, caustic) are exactly the same parameters that influence precipitation of trihydrate. The unfortunate issue that this presents is that using such parameters to maximize trihydrate precipitation yield can result in highly undesirable precipitation of oxalate, while minimizing oxalate precipitation also restricts trihydrate production. Thus, control and management of oxalate - using conventional means - requires a compromise in trihydrate production efficiency.

In short, while control of oxalate can appropriately mitigate the undesirable issues related to solid oxalate in the process, such control comes at a considerable cost - both directly through the management of the oxalate itself, and indirectly through the “opportunity loss” in production that conditions for control of oxalate entails. As a result, more efficient, alternative ways of controlling oxalate have been considered.

One alternative management process is to precipitate the sodium oxalate in a different morphology - as spherulites or balls. Under the conditions of the Bayer process sodium oxalate most often precipitates in a needle morphology (Figure 1a). Under specific circumstances and in the presence of particular process additives, it is well established that sodium oxalate can be precipitated as “balls” (Figure 1b). (Liu *et al.*, 2007; Fu *et al.*, 2014; Fu *et al.*, 2016).



(a)

(b)

Figure 1. (a) Sodium oxalate needles, and (b) sodium oxalate precipitated in ball morphology (from Liu *et al.*, 2007)

In terms of managing the impacts of solid oxalate on the process, the ball morphology has a number of advantages. Compared to the same amount of oxalate precipitated as needles, formation of oxalate in the ball morphology will result in:

- Enhanced filtration of trihydrate slurry
- Improved classification
- Less solid scum formation
- Less scale formation

In addition to these obvious benefits, the management of oxalate in the ball form also allows greater process flexibility, leading to more efficient trihydrate production (for example, lower back end temperatures).

Nalco Water Crystal Growth Modifier (CGM) products have long been identified as having an influence on oxalate stability and morphology (Liu *et al.*, 2007; Liu *et al.*, 2008; Kouznetsov *et al.*, 2008). A number of plants have used conventional Nalco Water CGM technology to alter the morphology of the precipitated oxalate and deliver process benefits. Recent development by Nalco Water of specific “ORT” reagents has focused specifically on products designed to more effectively change oxalate morphology and allow more extensive use of this management strategy across a broader range of operations and into more general practice.

5. DEVELOPMENT OF ORT REAGENTS

Conventional CGM technology has focused on the impact of these reagents on trihydrate particle coarsening. However, it has long been recognized that a range of other properties including foam control, oxalate stability and oxalate morphology changes may also be produced depending on both the CGM formulation used and the process conditions to which it is applied. Various products have been developed under the Nalco Water CGM range and while they are all effective in coarsening trihydrate, the additional properties can be enhanced or mitigated depending on requirements. As a consequence, there is a range of products available that cover a spectrum of properties. This is schematically represented in Figure 2.

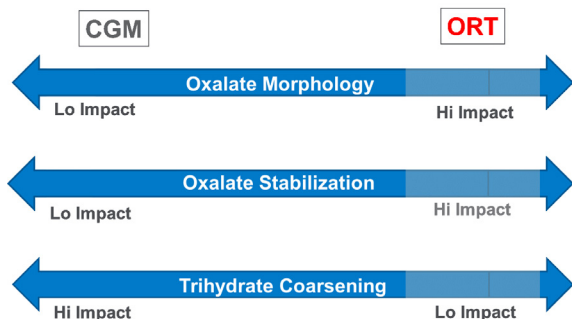


Figure 2. Variation in properties of conventional CGM products showing the difference in ORT within the spectrum

Using the conventional CGM product range as a starting point, Nalco Water undertook a strategy to develop the ORT product range specifically focused on the effects on oxalate morphology. As a result, while some CGM products will be effective, ORT products are specifically designed to change the morphology of precipitated sodium oxalate to balls. The formation of oxalate balls from the treatment of plant slurry with ORT is shown in Figure 3.

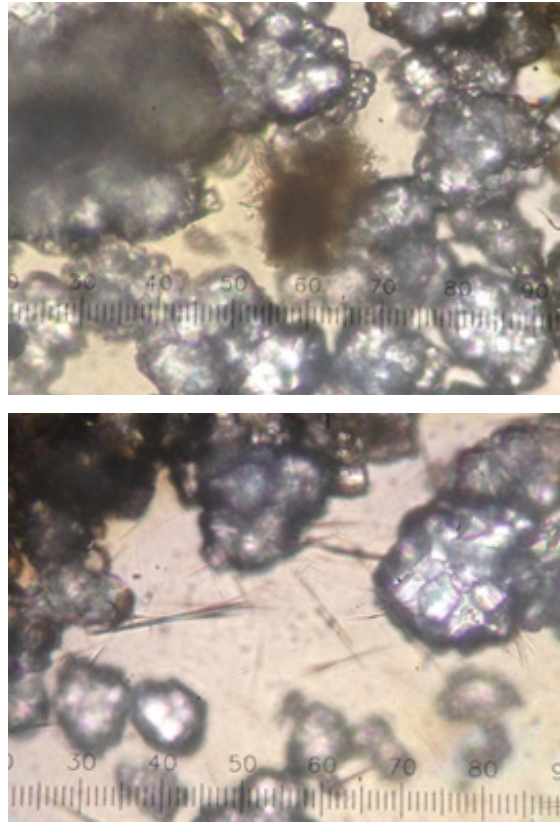


Figure 3. Oxalate balls formed as a result of ORT addition (top) and conventional oxalate needles formed without ORT addition (bottom) under the same conditions

ORT products can be considered to lie at the extreme end of the “oxalate morphology effect” spectrum, with CGM products at the other end (Figure 2).

In developing the ORT range it was important to also benchmark the other known properties of this broad range of products. Specifically, laboratory tests were performed to assess the relative performance of ORT and conventional CGM products across a range of known process conditions. Ideally, the aim was to identify ORT products that were highly effective in changing the morphology of oxalate to balls and in addition, delivered lower coarsening impact on trihydrate, more significant oxalate stability, and controlled foam - or at the very least, did not contribute to foam generation.

5.1 OXALATE STABILITY

Initial tests for oxalate stabilization were conducted by assessing the turbidity of a concentrated aqueous solution of caustic (~200g/L) and sodium oxalate (1g/L) as a concentrated solution of sodium oxalate was added slowly over time. Figure 4 shows the increase in stability (more solution added before crystals form).

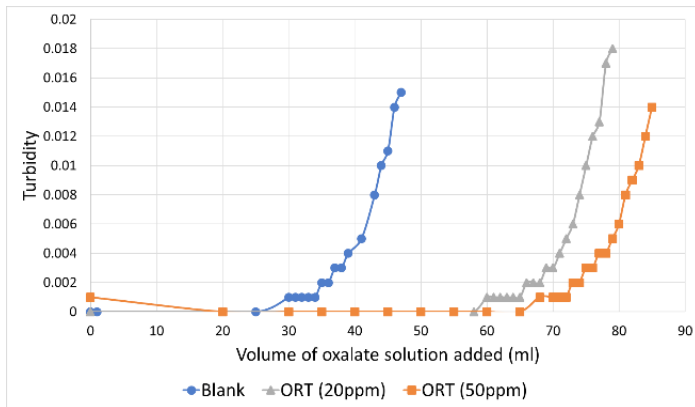


Figure 4. Stability of oxalate in caustic solution as measured by turbidity. Doses of 20ppm and 50ppm of ORT are compared to an undosed sample

To assess the stabilization of oxalate under slurry conditions plant slurries were prepared by combining pregnant liquor and plant seed (~600 g/L). A series of tests were conducted under a range of conditions (temperature, duration). Samples were placed in a rotating waterbath and the oxalate concentration of the liquor was determined after the appropriate period at the required temperature. Three treatments were assessed in duplicate; undosed blank, 50 ppm conventional CGM and 50 ppm ORT. Samples were held at 75 oC for 20 minutes, 60 oC for 17 hours, 52 oC for a further 7 hours, then finally 45oC for 19 hours.

Figure 5 shows the results plotted as the difference between the end oxalate of the individual treated samples and the end oxalate concentration of the corresponding undosed blank sample. In each of the four test conditions across a range of temperatures and duration, the ORT dosed samples were found to be the most stable; maintaining a much higher oxalate concentration in solution.

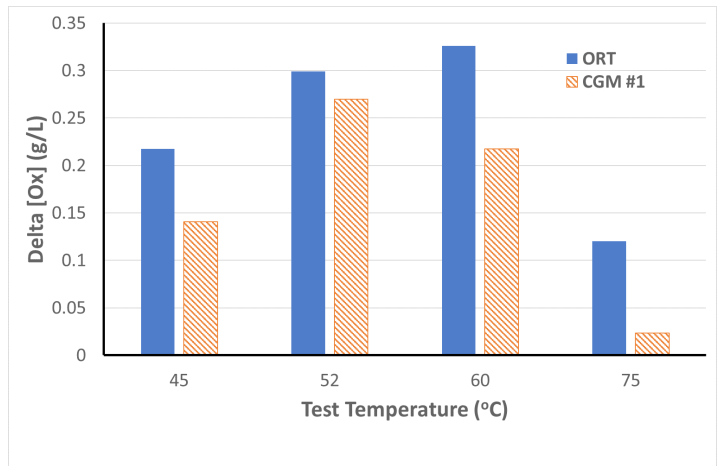


Figure 5. Relative stability of samples dosed with ORT and Conventional CGM. Shown are the difference in final liquor concentration compared to an undosed control samples after holding time under various conditions

5.2 TRIHYDRATE PARTICLE SIZING

While some coarsening of trihydrate may be desirable, it is required that application of an ORT product should not result in excessive coarsening. A simple 4-hour precipitation test using a rotating waterbath, plant liquor and trihydrate seed was completed. Basic test conditions were:

Start liquor A/C = 0.70, Temp = 60 oC, Seed charge = 150 g/L, Seed = DF225 standard seed. Dose rate = 0, 10, 20 ppm.

The resultant outcome of the various treatments (as % -45 µm) is shown in Figure 6 and indicates that the ORT has less coarsening impact than either of the two conventional CGM products used. The limited effect on particle sizing generally makes it more suitable for use in applications focused on changes in oxalate morphology.

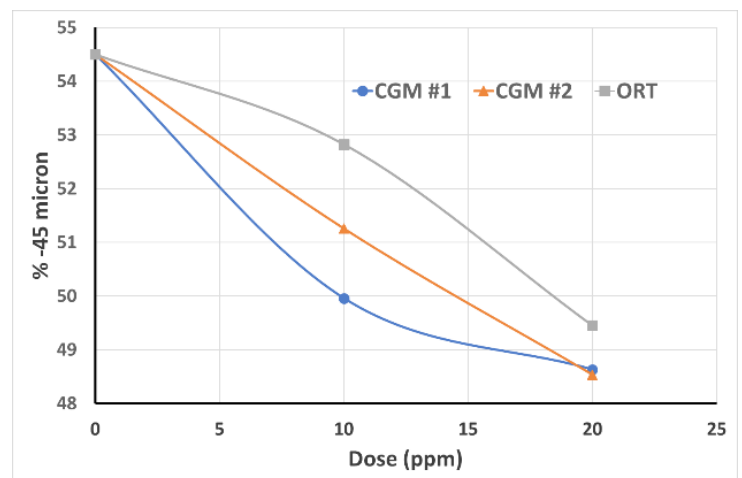


Figure 6. Dose response (as measured by % -45 µm) of bottle test samples treated with two different conventional CGM products (#1) and (#2) and ORT

5.3 FOAM CONTROL

Preliminary foam tests are typically conducted for all liquors when introducing a new additive to the process. Shown in Figure 7 is the outcome from a simple screening test, conducted in two different plant slurries, in which slurry was shaken. Different treatments (untreated and ORT dosed) are shown. Clearly a relatively stable head of foam was formed by the agitation of the untreated slurry in each case, however much less foam is formed in the presence of ORT.

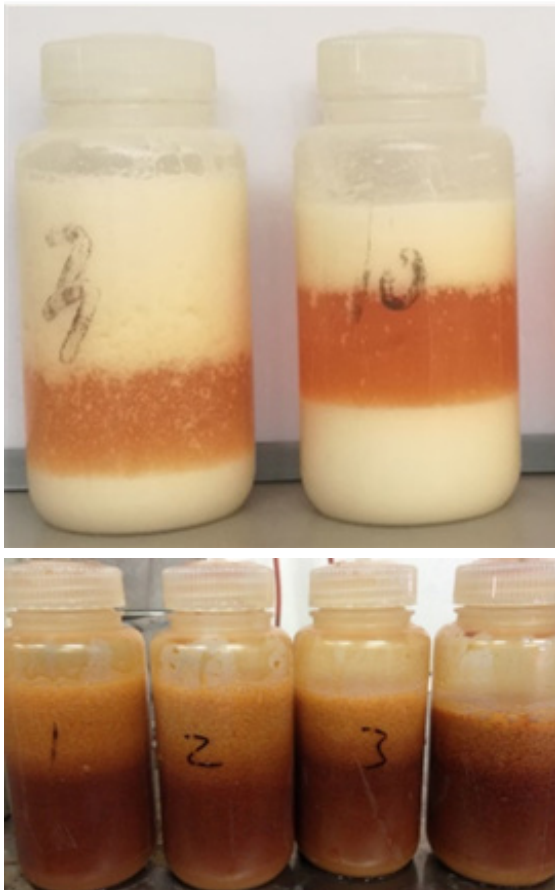


Figure 7. Results from agitation of slurry samples from two separate refineries. Top shows lower organic liquor containing no additional treatment (left) and ORT treated (right). Lower photo shows undosed control sample (left) and doses of 25, 50 and 100 ppm of ORT

6. APPLICATION AND USE OF ORT

ORT changes the morphology of oxalate crystals as they are precipitated. It has no impact on existing crystals that are already present in a slurry. As a result, to achieve a change in morphology of precipitated oxalate ORT must be added prior to the point of crystallization of the oxalate. In the presence of ORT, oxalate will precipitate as balls. However, it is important to note that any existing oxalate present as solid in the needle form will not be affected by the addition of ORT. Existing needles will remain as needles. This determines that the addition point of ORT to the process needs to be early in the precipitation process. Similar to CGM products, addition of

ORT to the incoming pregnant liquor has been found to be a convenient dose point.

ORT can be added directly to the liquor and requires no dilution or separate activation. As long as the product is sufficiently mixed with liquor, dispersion into the slurry is readily achieved.

Plant operating conditions play a substantial role in determining the dose of ORT required with all the factors that affect the formation of solid oxalate considered (e.g. temperature, trihydrate solids, caustic, presence of solid oxalate as seed). In making the transition from precipitating oxalate in the needle form, to precipitation of oxalate balls, these factors need to be considered. Under typical, co-precipitation conditions, sodium oxalate will precipitate as needles. Some of those needles may be recycled with the trihydrate seed and the oxalate needles will themselves act as seed sites to promote the formation of more oxalate as needles. The presence of such needles as seed effectively creates a “path of least resistance” for the oxalate in solution to precipitate as needles. To make the transition, the conditions need to be adjusted to make the formation of balls more favourable. This can be achieved by either increasing the dose of ORT (in the short term) or removing the existing oxalate needles from the process, or a combination of both.

Fortunately, as noted above, ORT has a substantial effect on oxalate stability. As a consequence, addition of ORT to a co-precipitation operation will typically result in less oxalate precipitating as needles in the first instance, due to the stabilization effect. With less oxalate precipitating, more oxalate is recycled in solution so the initial oxalate concentration in the liquor will increase. With a higher starting oxalate in solution, fewer needles present in the seed and ORT present in the liquor, the oxalate will eventually start to precipitate in the ball form. These balls, when recycled with the trihydrate seed will act as seed sites and promote the formation of larger balls (and fewer needles). Overall then this transition phase is a process involving recycle of the inventory of solids within the precipitation circuit.

As a result, the formation of balls is not found to be instantaneous with addition of ORT, but rather the transition is a gradual process that, depending on process flows and turnover, may take days (or weeks) to achieve.

Additionally, the initial stabilization of oxalate (to prevent needle formation) is an important property of ORT and assists in this transition.

It should also be noted that development of the ball morphology can be observed to pass through a number of different “partial” morphology changes before full ball formation is achieved (Figure 8).

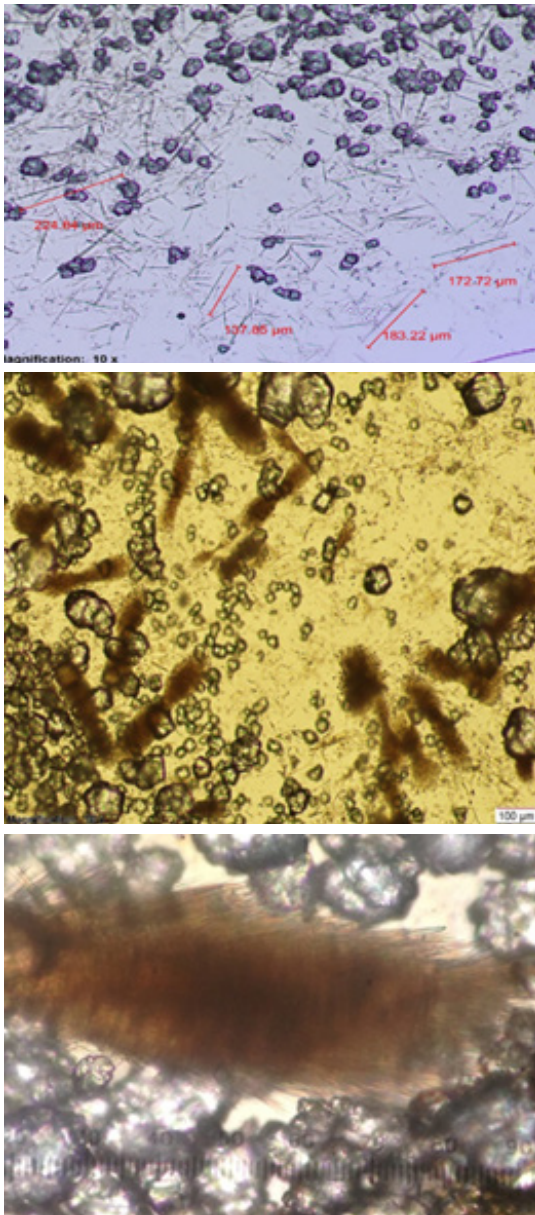


Figure 8. Different morphologies of oxalate needles, fans/bow ties, aggregated balls

Oxalate in the form of so-called fans and bow ties, as well as aggregated balls, which can appear to look like caterpillars under a microscope, have been observed.

Once the transition from precipitation of oxalate as needles to precipitation of oxalate balls is achieved, then the significant process benefits (both immediate and potential) can be extracted. However, it is important to note that while the transition of oxalate to the ball morphology provides such benefits, there is still a requirement to manage and control oxalate within the process.

7. MANAGEMENT OF OXALATE AS BALLS

The input of organic material within bauxite is the major source of oxalate in the Bayer process. As a consequence, under normal operation there is effectively a constant input of oxalate into the circuit. To prevent an uncontrollable build-up of oxalate, there has to be

an equivalent outlet to maintain a stable equilibrium concentration.

In plants where oxalate precipitation as balls has been practiced, a number of removal methods have been utilized. An established method is to use screens to capture the large (mm) balls and separate them from the trihydrate. This provides a relatively simple, well known technology to achieve the separation and the solid oxalate can be readily disposed.

In addition, the production of balls on a consistent basis via the use of ORT enables a broad range of possibilities to now be considered as potential methods for removal and extraction of oxalate from the system.

Unlike needles which tend to report with the fine trihydrate, oxalate balls tend to sink within Bayer process slurry and generally the larger balls report to the bottom of the tanks. Based on this (different) characteristic a number of possible removal processes (perhaps coupled with screening) can be envisaged.

In some circumstances, the oxalate may report with trihydrate product. In calcination the oxalate will be oxidized leaving soda. The increase of soda in product that this delivers may well be tolerable in some circumstances (e.g. existing low soda content) and as such, the removal of oxalate balls with trihydrate product may provide the appropriate outlet required to balance the input of organics with the bauxite.

8. CONCLUSION

Sodium oxalate is a common contaminant within the Bayer process which generally is produced from organic matter present in bauxite. Uncontrolled precipitation of sodium oxalate in the needle morphology causes a number of process issues including restrictions on particle sizing, filtration, trihydrate classification and stabilization of foam/scum. As a consequence, most plants spend considerable time and resources in an effort to manage and control the formation and removal of solid phase oxalate.

The precipitation of sodium oxalate in a different, ball morphology presents opportunities for more effective and efficient management of oxalate. Traditional CGM technology was known to impact oxalate morphology but Nalco Water has now developed the ORT range of products that are specifically designed to influence oxalate precipitation and create oxalate balls.

The use of ORT can enable plants to change the way they manage and control oxalate, making the process simpler and more efficient. In addition, precipitation of oxalate as balls can directly alleviate some of the process bottlenecks associated with the management and processing of oxalate needles. These benefits can include

direct improvements in product filtration, trihydrate seed filtration, classification, temperature control and most significantly production yield.

Use of ORT provides plant operators with the opportunity to change the way that oxalate is managed and controlled within the individual plant operations, leading to improved process efficiency.

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