



# Crystallisation of tricalcium aluminate from sodium aluminate solution using slaked lime



Reza Salimi, James Vaughan \*

The University of Queensland, School of Chemical Engineering Building (74), College road, St. Lucia, Queensland 4072, Australia

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## ABSTRACT

Crystallisation of tricalcium aluminate (TCA) hydrate was investigated by reacting slaked lime with sodium aluminate solution at 368 K in an agitated baffled batch and continuous reactors. Using an x-ray diffraction calibration established by the intensity ratio of characteristic peaks of lime-TCA admixtures; it was determined that >90% of the slaked lime was converted to TCA within 5 min in the batch reactor. From particle counting measurements, TCA nucleation and growth was observed within the first 30 s by an increase in 2–5  $\mu\text{m}$  particles, whereas finer particle counts decreased due to lime dissolution. The mean particle size based on particle counting increased up to 10  $\mu\text{m}$  due to growth dominated by agglomeration of sub-particles over the course of 2 h. The TCA particle morphology initiated as hexagonal platelets which became arranged as “rosettes” then filled in to become spherical. Reaction seeding with 10 wt.% of the total final solid TCA mass resulted in a narrower particle size distribution. Seed loading did not significantly influence the particle morphology, however slight growth of TCA sub particles was evident from the micrographs. The effect of the key operating parameters, residence time and agitation intensity, were then assessed in the context of Bayer liquor filter aid production in an agitated continuous reactor. As the residence time was increased, the fine particle number concentration decreased and particle growth increased which was dominated by agglomeration. With a longer residence times, the particles become highly agglomerated leaving little void space which is undesirable in terms of filter aid quality. An agitation intensity of 600 RPM which exerts a maximum Reynolds number of 9200 on the fluid phase based on tip speed and residence time of 30 min generated the best filter aid in terms of minimising the fine particle fraction and minimising large agglomerates.

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## 1. Introduction

In the Bayer process for producing alumina from bauxite ore, aluminium is leached into caustic solution in the digestion stage as aluminate [1]. The pregnant liquor is isolated and clarified through settling and liquor filtration. Aluminium trihydroxide (gibbsite;  $\text{Al}(\text{OH})_3$ ) is then precipitated from the liquor. This is achieved by cooling the solution and seeding with gibbsite, essentially reversing the initial dissolution process. Gibbsite is then either recycled as seed or converted to alumina by high temperature calcination [2,3].

In the liquor filtration stage of the process, tricalcium aluminate (TCA) is used as a filter aid. This filter aid slurry is combined with settler overflow pregnant liquor and the resultant mixture fed to the security filter where the solids build up on the cloth. Kelly, vertical pressure or backflush filters are the types of equipment used. TCA is produced on site by adding slaked lime to either spent liquor or pregnant liquor [4, 5]. It is important that the filter aid is chemically inert, cheap and the porous coating maintains a void space that is sufficiently small to trap the

fine mud and colloidal silica, but not so small that flow is restricted or the filter cloth is blinded [6]. In terms of the particle size, a mono-modal distribution with higher mean size is preferred for this application.

Previous research on batch crystallised TCA is summarised in Table 1. TCA morphology and particle size distribution are of particular interest for filter optimisation as these particle properties determine the filtration unit productivity in terms of throughput and time in between cleaning cycles [7]. Studies into the effect of reactor operating conditions concluded that elevated temperatures (368–373 K) and high alkalinity in spent liquor favour crystalline TCA with a low proportion of fine particles which is beneficial to filtration performance [4]. It has also been reported that, crystallising TCA using spent as opposed to pregnant liquor improved the uniformity of the filter coating as there were no cracks and a satisfactory level of dissolved calcium in the filtrate which can potentially contaminate the alumina product [8]. Unfortunately, the spent liquor generated coating exhibited a reduced filtration rate. There is evidence that the filtration rate can be improved by controlling how slaked lime is introduced to the reactor [9] and maintaining a low liquor to lime ratio. An increase in TCA batch precipitation time from 30 to 120 min was shown to improve filtration efficiency in

\* Corresponding author.

E-mail address: [james.vaughan@uq.edu.au](mailto:james.vaughan@uq.edu.au) (J. Vaughan).

**Table 1**  
Summary of batch TCA crystallisation studies.

Solution phase: (g/L)	T (K)	Lime added as:	Time (min)	Key points	Ref.
Al(OH) <sub>3</sub> = 20 NaOH = (50, 78, 100) Ca(OH) <sub>2</sub> = (60, 65, 70) (plant liquor)	320–355	Ca(OH) <sub>2</sub>	180	Reaction of Al(OH) <sub>4</sub> <sup>-</sup> (aq) on the hydrated surface of the Ca(OH) <sub>2</sub> forms a hydrated calcium aluminate precursors (metastable species), which then convert to TCA.	[13]
Al(OH) <sub>3</sub> = 6.5 NaOH = 2.6 (synthetic liquor)	323	CaO (dry)	5–60	1 - As the reaction time is increased, the degree of crystallinity of the TCA also increases. 2 - The formation of calcium aluminate at the lime surface is sensitive to the stirring rate where the reaction rate was controlled by the slow diffusion of aluminate ions to the lime surface.	[22]
Al(OH) <sub>3</sub> = 91 NaOH = 150 (synthetic liquor)	343–498	CaO (60 g/L)	30	The preparation of TCA in sodium aluminate solution is optimal at a temperature of 373 K in a highly caustic solution in the absence of CO <sub>3</sub> <sup>2-</sup> (aq).	[14]
Al(OH) <sub>3</sub> = 71–230 NaOH = 91–272 (plant liquor)	369	CaO (dry) <sup>a</sup>	30	Higher slaking temperatures and high alkalinity result in the formation of large, highly crystalline TCA due to the additional conversion of the quicklime to TCA thereby resulting in increased solid content which possess good filtration properties.	[4]
Al(OH) <sub>3</sub> = 191 NaOH = 304 (plant liquor)	363	CaO (200 g/L water)	30	1 - Structure of TCA improves with increased reaction time 2 - It is important to evaluate the Al <sub>2</sub> O <sub>3</sub> /CaO ratio as closer as possible to the stoichiometric, possibly having aluminate solution in excess, aiming to avoid the excess of Ca(OH) <sub>2</sub> in the reaction.	[10]
Al(OH) <sub>3</sub> = (57,92,115) NaOH = (150,200)	373	CaO (60 g/L liquor)	80–240	CaO/Al <sub>2</sub> O <sub>3</sub> molar ratio between 1.8 and 3 forms rhombic dodecahedron morphology of TCA, while a molar CaO/Al <sub>2</sub> O <sub>3</sub> ratio less than 1.8 forms TCA of a less well defined morphology.	[7]
Unspecified plant liquor	373	CaO (dry)	120	The faster the lime is introduced, the finer the solid, and the wider the particle size distribution.	[9]
Al(OH) <sub>3</sub> = 132 NaOH = 100 Na <sub>2</sub> CO <sub>3</sub> = 42	368	Ca(OH) <sub>2</sub> <sup>b</sup> (100 g/L water)	120	1 - The mean particle size increased to a maximum value of 10 μm due to growth dominated by agglomeration. 2 - The particle morphology shifted from an irregular shape to more spherical throughout the course of the reaction.	[23]
Al(OH) <sub>3</sub> = 191 NaOH = 304 (plant liquor)	363	Ca(OH) <sub>2</sub>	30–120	Increase in residence time from 30 to 120 min resulted in an increase in the average particle diameter.	[10]

Slaking temperature: (a) = 369 K, (b) = 343 K.

terms of higher impurity removal and decreased filter cake resistance. The improvement in filtration rate was attributed to a decrease in fine particles and an increase in the average particle diameter. The need for a deeper study in to this phenomenon was noted [10].

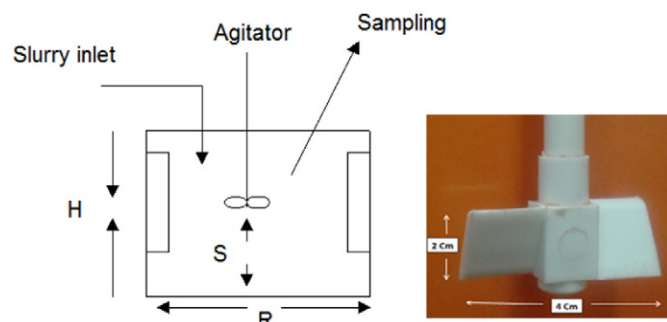
TCA properties are influenced by the lime quality and slaking conditions. Research has shown that the lime to water ratio has a significant role in determining the particle size and settling rate for slaked lime. In a study by Boynton (1980), the time for slaked lime to settle to 50% of the slurry volume was recorded to demonstrate how the slaked lime particle size is strongly influenced by the ratio of lime to water [11]. It was also reported that slaking temperature influences the particle size distribution and the specific surface area of the hydrated lime particles. As the slaking temperature was increased to 371 K, a finer particle size distribution and greater specific surface area was apparent [12].

It was suggested that the TCA crystallisation pathway proceeds through the formation of highly hydrated calcium aluminate precursor on the surface of the lime particles and that the early stages of the reaction may be mass transfer limited by availability of aluminate [13]. This reaction mechanism would explain the observed improvement in TCA crystallinity in spent liquor as the aluminate concentration is lower providing more time for the crystals to become well-formed and also indicates that the reactor hydrodynamics should have a significant influence on particle morphology. It is also understood that under certain conditions, the reaction of aluminate at the hydrated lime surface may be impeded by the formation of a calcium carbonate or gypsum coating on the lime particles if carbonate or sulphate are present at sufficiently high concentrations [14].

While there are industrial examples of both batch and continuous TCA crystallisation reactor configurations, modern refineries tend to operate continuously. Practical advantages of the continuous reactor

configuration are that they do not need to be filled and emptied which improves infrastructure availability and can translate to a smaller overall reactor volume as redundant tanks are not required. Furthermore, with appropriate process control, a uniform product is more easily obtained at steady-state operation. For applications involving relatively small amounts of material, it is often convenient to use a batch crystalliser as they are relatively easy to shut down for cleaning and servicing [15,16].

Even though continuous reactors are more commonly employed for industrial production of TCA filter aid, most reported studies of this process have been conducted in batch reactors. Experimentally, studying the batch reaction is faster and simpler than in continuous mode as it is necessary to ensure that steady state conditions are achieved which takes additional time and the set-up involves continuous dosing of two slurries at constant rates instead of combining reagent once at the onset of the test. In terms of the crystallisation mechanism and



**Fig. 1.** Schematic diagram of the batch crystalliser and image of the impeller (R = 7.2 cm, S = 2 cm, H = 12.2 cm).

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