

## MODELLING BAYER PRECIPITATION WITH AGGLOMERATION

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### ABSTRACT

A major impediment to the development of computer models for Bayer precipitators has been the poor understanding of  $\text{Al}(\text{OH})_3$  (gibbsite) agglomeration, which is the major size enlargement process during Bayer precipitation. Recent studies claim to have identified a size-independent mechanism for gibbsite agglomeration in synthetic caustic aluminate liquors. This paper uses these latest findings on the agglomeration kinetics in the development of a population balance model for gibbsite precipitation. The model gave reasonable agreement to experimental data from a 2.8 litre continuous precipitation unit.

### INTRODUCTION

The ability to model Bayer precipitators would greatly improve the design, operation and process optimisation of this part of an alumina refinery. Audet and Larocque [1] give an example of the combined use of Bayer plant precipitation simulations and plant trials to improve plant operation. For this reason, over the years, considerable effort has been directed to the development of such models and a number of research publications have appeared [1-10]. Interest has focused on the development of models that allow simulation of the yield (recovery) and the crystal size distribution (CSD). These models utilise the population balance besides the traditional conservation laws - the mass and energy balances. A major impediment has been the problem of developing a model for  $\text{Al}(\text{OH})_3$  (gibbsite) agglomeration, which is the major size enlargement process during Bayer precipitation. This paper presents the development of a population balance model for continuous gibbsite precipitation and

reports the identification of a mechanism for gibbsite agglomeration. A correlation is presented for agglomeration kinetics with the process conditions. Experimental precipitation data was generated, for comparison with the model predictions, using a continuously operated 2.8 litre, seeded precipitator.

### EXPERIMENTAL

A 2.8 litre crystalliser, fitted with a draft tube and agitated by a marine impeller, was operated as a continuously seeded MSMPR (mixed suspension, mixed product removal) cooling precipitator. A schematic of this continuous precipitation unit is shown as Figure 1. The crystalliser was configured so that the crystals were fully suspended using the minimum power input ( $22 \text{ W m}^{-3}$ ), [11]. The caustic aluminate liquor was prepared from washed industrial grade gibbsite digested in caustic soda and filtered; the supersaturation ranged from 0.25 to 0.5 (as a mass ratio of  $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$ ). The crystalliser was operated at  $79^\circ\text{C}$ , which for the feed used maximised

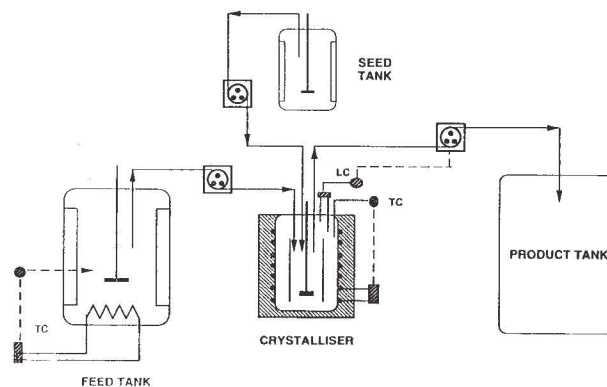


Figure 1: A schematic of the experimental continuous precipitation unit.

crystal growth rates and minimised the induction times, [8, 12].

As the steady state flowrates were low, it was necessary to use periodic, high flowrate product slurry withdrawal to ensure no classification occurred in the crystalliser. The evacuated volume was about 7% of the vessel volume; Randolph and Larson [13] recommend a maximum of 10%.

The feed liquor, at 105°C, was continuously pumped into the draft tube region of the precipitator by a peristaltic pump. A seed slurry (~ 20°C) was also continuously pumped to the precipitator. Tests were conducted to ensure representative delivery of the seed.

The seed crystals were prepared by washing industrial grade gibbsite crystals in warm water and then dividing them into well-defined size fractions using a Warman Cyclosizer. The seed slurry contained less than 5% by weight solids in RO (reverse osmosis) water. The seed slurry flowrate was approximately 9% of the feed stream flowrate.

Prior to continuous operation, the precipitator was operated in batch mode using the same feed liquor and seed. After approximately 90 minutes of batch operation, i.e. the estimated induction time [12] plus one residence time, the unit was switched to the continuous mode. Samples were taken from the precipitator for analysis and later comparison with the model predictions. The CSD determination was done by an Elzone particle sizer and the solution analysis

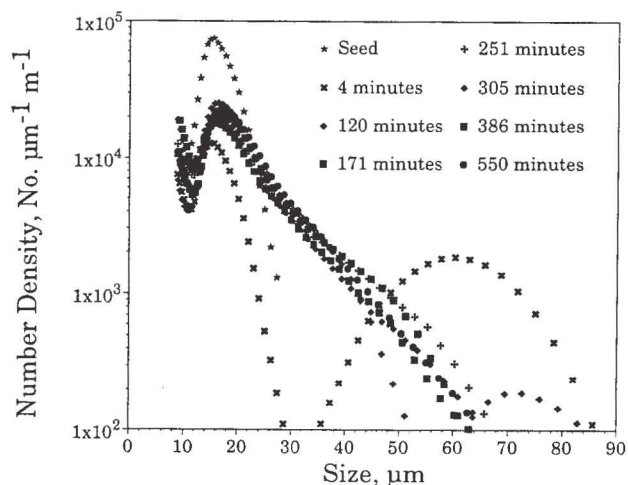


Figure 2: Transient CSD response after switching from batch to continuous operation.

was by AA and titration. Checks had been performed to ensure that the sampling procedures produced representative samples [8]. Steady state was verified by monitoring the CSD transients, as shown in Figure 2. The mass balance was consistent.

### THE PRECIPITATOR MODEL

#### Yield Model

White *et al.* [14] derived the following equation for predicting the exit A/C (as a mass ratio of Al<sub>2</sub>O<sub>3</sub>/Na<sub>2</sub>O) of a well-mixed gibbsite precipitator at steady state in synthetic caustic aluminate solutions, for a given feed concentration,

$$\left(\frac{A}{C}\right)_{exit} = \left(\frac{A}{C}\right)_{eq} - \omega + \sqrt{2\omega \left[ \left(\frac{A}{C}\right)_f - \left(\frac{A}{C}\right)_{eq} \right] + \omega^2} \quad (1)$$

where

$$\frac{1}{\omega} = 1.96 \times 10^{10} \left(\frac{a\tau}{C}\right) (0.645\rho_s - A_f) \exp\left(-\frac{7200}{T}\right)$$

and the equilibrium A/C ratio can be estimated using the solubility correlation of Misra [15],

$$\left(\frac{A}{C}\right)_{eq} = \exp\left(6.211 - \frac{2486.7}{T} + \frac{1.09C}{T}\right) \quad (2)$$

where  $T$  is the temperature in kelvin,  $\tau$  is the precipitator mean residence time in hours,  $A_f$  is the alumina concentration in the feed liquor and  $a$  is the crystal surface area in m<sup>2</sup> m<sup>-3</sup> of slurry.

#### Modelling the Product CSD

The population balance (PB) is now widely used to model precipitators and crystallisers and the book by Randolph and Larson [13] remains the definitive introduction to using PB models. The steady state model for the experimental precipitator described above, shown as Equation (3), is a special case of the general macroscopic PB presented in Randolph and Larson.

$$\frac{\partial n}{\partial t} + G \frac{\partial n}{\partial L} + \left[ \frac{n - n_s}{\tau} \right] = B_{agg} - D_{agg} \quad (3)$$

The terms  $n$  and  $n_s$  are the number density distributions of crystals in the crystalliser and of the seed, respectively; they are functions of crystal size,  $L$ ,

and time,  $t$ . The other terms in Equation (3) are: the mean residence time,  $\tau$ , and the linear growth rate,  $G$ .

The term  $B_{agg}$  represents the birth of particles into the size  $L$  to  $L+dL$  due to the agglomeration of particles sized  $L-\lambda$  with particles sized  $\lambda$ , at time  $t$ , and may be explicitly expressed as:

$$B_{agg} = \frac{L^2}{2} \int_0^L \frac{\beta[t, (L^3 - \lambda^3)^{1/3}, \lambda] n[t, (L^3 - \lambda^3)^{1/3}] n[t, \lambda]}{(L^3 - \lambda^3)^{2/3}} d\lambda$$

$D_{agg}$  is the disappearance of particles from the size  $L$  to  $L+dL$  because they agglomerate with some particle sized  $\lambda$ .

$$D_{agg} = n(t, L) \int_0^\infty \beta(t, L, \lambda) n(t, \lambda) d\lambda$$

The term  $\beta(t, L, \lambda)$  is the agglomeration kernel; it represents the rate with which particle collisions between crystals sized  $L$  and  $\lambda$  will result in the formation of an agglomerate. This term embodies information on the mechanism of agglomeration. The identification of the appropriate kernel for  $\text{Al(OH)}_3$  agglomeration is discussed in a later section.

Ilievski and White [6] present the following correlation for  $G$  ( $\mu\text{m h}^{-1}$ ) in synthetic caustic aluminate liquors,

$$G = \frac{15.0}{\sqrt{C/100}} \exp\left[-7600\left(\frac{1}{T} - \frac{1}{343.16}\right)\right] S^2 \quad (4)$$

where  $C$  is the caustic concentration ( $\text{g l}^{-1} \text{Na}_2\text{O}$ ),  $S$  is the supersaturation ( $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$  by mass) and  $T$  is the temperature in kelvin. The correlation was generated from data compiled from the open literature and from precipitation studies. The 95% confidence limits for the correlation are  $\pm 50\%$ .

#### PB Model Assumptions and Justification

The assumptions made in deriving Equation (3) are: (a) linear size independent growth, i.e. the  $\Delta L$  McCabe law; (b) no growth size dispersion; (c) no nucleation or breakage, i.e. only growth and agglomeration are occurring; (d) constant precipitator volume; (e) well mixed vessel; (f) representative product removal; (g) the liquor and solid phases have the same residence time distribution; (h) the aggregates have no voids; and (i) a dilute particle system, i.e. follows the *free in space* classification of Sastry and Fuerstenau [17].

Clearly there are many assumptions associated with the rigorous use of Equation (3). It is possible to ensure that many of the assumptions are satisfied, or nearly so, for laboratory scale precipitation. However, this is less likely to be the case for industrial scale precipitators and the importance of satisfying the assumptions to the successful application of PB models for precipitators needs to be assessed in each case.

For the current case many of the assumptions above can be justified. Misra and White [18] demonstrated size independent growth and negligible nucleation above  $75^\circ\text{C}$ , for gibbsite precipitation. Brown [19] also reports no nucleation above  $77^\circ\text{C}$ . No nucleation was observed in the current precipitation experiments. Tests showed that particle breakage was negligible and the product (and sample) removal was representative. The presence of agglomeration was verified from SEM micrographs and from the observed decrease in particle numbers. The presence of growth was inferred from the liquor desupersaturation. White and Wright [20] demonstrated negligible growth size dispersion effects for this system and this is consistent with the observations of Ilievski and White [16]. The liquor and solid phase were shown to have the same residence time distribution (exponential, i.e. behaving as a perfectly mixed vessel), using tracer crystals [21] for the solid phase residence time distribution determination and a soluble inorganic [8] for the liquor phase residence time distribution. Steady state operation was verified by observation of the product CSD and the  $A/C$  ratio. The *free in space* assumption is generally acceptable for crystallisation systems as the particles are not constrained only to collide with their neighbours.

#### Identification of an Agglomeration Kernel for Gibbsite Precipitation

Solution of Equation (3) requires knowledge of the agglomeration kernel  $\beta(t, L, \lambda)$ , in particular the functional form of the size dependency. Agglomeration kernels for gibbsite precipitation have been proposed by Low [3], Halfon and Kaliaguine [4] and Groeneweg [6]. Halfon and Kaliaguine propose a size independent mechanism for agglomeration, while the other two present agglomeration kernels that show size dependency. Recent work by Ilievski and White [16] and Ilievski and Hounslow [22] also provides evidence for a size-independent agglomeration mechanism for

gibbsite precipitation in synthetic caustic aluminate liquors. Collectively, they present three independent techniques to identify the effect of particle size on the agglomeration rate and all three techniques clearly showed that agglomeration may be modelled by a size independent mechanism. These three methods are summarised below.

- (1) Approximately 200 CSD curves from batch precipitation experiments were fitted using the PB model for batch precipitation with 5 different agglomeration kernels from the literature. The quality of the fit to the experimental data, i.e. the sum of the squared residuals between the PB model and data (SSE), was used to discriminate between the different kernels. It was shown statistically (i.e. at the 0.00001 level of significance) that the fits with the size-independent kernel resulted in consistently lower values of SSE. This work is reported in [16].
- (2) Ilievski and White [16] reported that the batch agglomeration of gibbsite asymptotically approaches a so-called *Self-Preserving* CSD. This implies that the shape of the CSD does not change after the initial period of agglomeration is over. This *Self-Preserving* CSD, when plotted with volume as the internal co-ordinate, was found to be exponentially distributed. It is a straight forward matter to show theoretically that size-independent agglomeration will result in an exponential *Self-Preserving* CSD [23].
- (3) The third technique [22] employs a two dimensional population balance on precipitation data generated using doped gibbsite "tracer" crystals. The analysis of the experimental tracer response data supported a size-independent model for gibbsite agglomeration.

The values of the size-independent kernel,  $\beta(t)$ , were estimated from batch precipitation experiments, in synthetic caustic aluminate liquors, and were correlated against the supersaturation,  $S$ , and the agitation power,  $\epsilon$ , [24]. It was found that  $\beta(t)$  was very weakly correlated with  $S^4$  and strongly correlated to  $\epsilon$ . The correlation is:

$$\beta(t) = 0.05 S(t)^4 \epsilon^{-14} \quad (5)$$

It must be emphasised that Equation (5) is a weak correlation; i.e. the 85% confidence limits on the correlation are of the order  $\pm 80\%$ .

#### Solution of the PB Model

Establishing a PB model for a system, such as Equation (3), is generally straight forward. The difficulty arises when it comes to solving the resultant equation. Analytical solutions can only be found for a few simple cases. Numerical methods are widely used and the loss in precision, compared to the analytical solutions, is generally lower than the uncertainty in the observed precipitation data. Methods for solving the population balance have received considerable attention in the literature [25-28]. In this work, the discretized population balance (DPB) developed by Hounslow *et al.* [29] was used. This technique, like all DPB techniques, has the advantage of transforming Equation (3), a partial-differential integral equation, into a closed set of ordinary differential equations, a considerable simplification. Unlike the other DPB methods, Hounslow *et al.* formulated their DPB to ensure that the rates of change in the moments determined from the DPB equal the rates of change in the moments described by the continuous PB.

The DPB form for the dynamic continuous precipitator studied in this work may be written, for the  $i$ -th size interval, as:

$$\left(\frac{dN_i}{dt}\right) = \left(\frac{dN_i}{dt}\right)_{agg} + \left(\frac{dN_i}{dt}\right)_{grow} + \frac{N_{i,s} - N_i}{\tau} \quad (6)$$

where  $N_i$  is the number of particles, per unit volume of slurry, in the  $i$ -th size interval leaving the system and  $N_{i,s}$  is the number per unit volume in the same size interval entering the system as seed. The number density corresponding to  $N_i$  can be estimated from

$$n_i = \frac{N_i}{2^{(i-1)/3} \delta L_o}$$

where  $\delta L_o$  is the width of the first size interval. The other two terms in Equation (6) give the rate of change in  $N_i$  due to agglomeration and growth, respectively, and are

$$\left(\frac{dN_i}{dt}\right)_{agg} = N_{i-1} \sum_{j=1}^{i-2} 2^{j-i+1} \beta_{i-1,j} N_j + \frac{1}{2} \beta_{i-1,i-1} N_{i-1}^2 - N_i \sum_{j=1}^{i-1} 2^{j-i} \beta_{i,j} N_j - N_i \sum_{j=i}^{\infty} \beta_{i,j} N_j$$

$$\left(\frac{dN_i}{dt}\right)_{grow} = \begin{cases} \frac{2G}{L_i(1+r)} \left( \left(1 - \frac{r^2}{r^2-1}\right) N_1 - \frac{r}{r^2-1} N_2 \right) & i = 1 \\ \frac{2G}{L_i(1+r)} \left( \frac{r}{r^2-1} N_{i-1} + N_i - \frac{r}{r^2-1} N_{i+1} \right) & i \neq 1 \end{cases}$$

where  $r = L_{i+1} / L_i$ , i.e. the ratio of the upper and lower bounds of a size interval.

The Hounslow discretization ensures the correct prediction of the total particle number and volume for the agglomeration terms (i.e. zeroth and third moments) and correctly predicts the first three moments associated with the growth term. Cresswell *et al.* [10] claim that the Hounslow method slightly under predicts first and second moments for the agglomeration term and over predicts the rate of increase of the third moment for the growth term. The technique is relatively simple, computationally efficient and robust. A disadvantage is that it uses a geometric discretization of the size domain, i.e.  $r = 2^{1/3}$  and assumes a constant number density distribution across each interval. This means that the discretization becomes coarse at the upper end of the size domain and information can be lost. The method of Litster *et al.* [30], a modification of the Hounslow DPB, was developed to improve the resolution of the discretization. However, this method was not available at the time the current work was being conducted. The *finite domain error*, i.e. where particles outside the size domain are wrongly assigned to size intervals within the domain, was minimised by selecting a domain encompassing the entire range of sizes, plus a few extra size intervals.

The DPB was solved using SPEEDUP™, which is an equation based dynamic simulation package. SPEEDUP™ allows the user to supply customised models for unit operations. The elements of Equation (6) were added to the SPEEDUP™ code as FORTRAN procedures. SPEEDUP™ has a facility that, when enabled, will find the steady state solution to the dynamic equation.

**COMPARISON OF EXPERIMENTAL DATA TO THE MODEL**

The agreement was excellent between the exit A/C ratios predicted by Equation (1) and experimentally observed values. The differences between the two ranged from 0 to 3% for the current experiments.

The steady state CSD, conditions corresponding to the experimental precipitation runs, were generated using SPEEDUP™. The CSD were calculated using values of G and  $\beta$  from two sources: (a) from equations (4) and (5) and (b) from matching the moments of the population balance to the experimental CSD. A comparison of the model predictions and the experimental data is given in Figure 3.

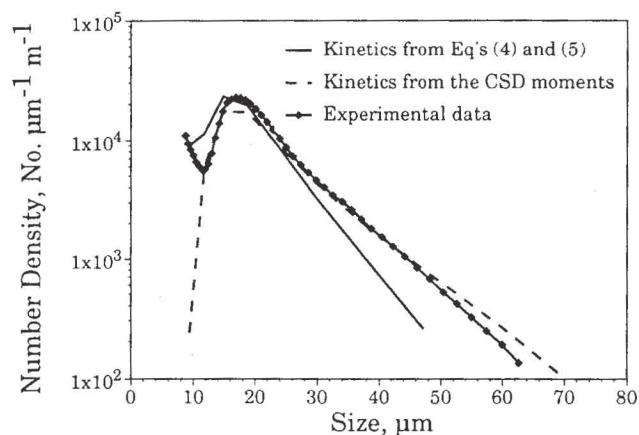


Figure 3: Comparison of experimental steady state CSD and the PB model predictions. Two methods of estimating the agglomeration and growth kinetics were used.

As expected, prediction of the product CSD using the precipitation kinetics estimated from moments of the distribution gives better agreement to the data than when equations (4) and (5) are used. Estimates of agglomeration kinetics,  $\beta$ , from the two sources agreed to within experimental error. However, the estimates of G using the matching of moments were significantly greater than those from Equation (4). A large body of experimentally determined linear growth rates, from a wide range of sources, were used to generate Equation (4) and so is more likely to represent the actual growth kinetics. The discrepancy between the two estimates of G is believed to arise because

Equation (3) does not account for agglomerate voidage. The larger values of  $G$ , estimated by matching the moments to the CSD, may indirectly account for any agglomerate voidage. A publication addressing this issue is being prepared.

### CONCLUSIONS

The yield model of White *et al.* [14] and a population balance model are used to model a continuous gibbsite precipitator. The model predictions were compared with experimental data from a 2.8 litre continuous precipitator at steady state and reasonable agreement was observed. However, the results suggest that the predictions of the product CSD could be improved by correctly accounting for agglomerate voidage in the population balance model.

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### NOTATION

$A$	: aluminium concentration, $g\ l^{-1}\ Al_2O_3$
$a$	: the crystal surface area, $m^2\ m^{-3}$ of slurry.
$B_{agg}$	: birth rate due to agglomeration, No. $\mu m^{-1}\ ml^{-1}\ h^{-1}$
$D_{agg}$	: death rate due to agglomeration, No. $\mu m^{-1}\ ml^{-1}\ h^{-1}$
$C$	: caustic concentration, $g\ l^{-1}\ Na_2O$
CSD	: crystal (or particle) size distribution
$G$	: linear growth rate, $\mu m\ h^{-1}$
$L, \lambda$	: size of a crystal, $\mu m$
$n(t, L)$	: number density function, No. $\mu m^{-1}\ ml^{-1}$
$n_s(t, L)$	: seed number density function, No. $\mu m^{-1}\ ml^{-1}$
$N_i$	: number of particles in $i$ -th discretization interval, No. $ml^{-1}$
PB	: population balance
$r$	: ratio of the lower and upper bounds of a size interval
$S$	: supersaturation, $g\ l^{-1}\ Al_2O_3$
SSE	: sum of squares error between model and experimental data
$T$	: temperature, K
$t$	: time

### Greek

$\beta$	: agglomeration kernel, $ml\ No.^{-1}\ h^{-1}$
$\epsilon$	: energy dissipation rate in a fluid, $W\ m^{-3}$
$\Delta L_o$	: width of the first size interval, $\mu m$
$\tau$	: mean residence time, h.
$\rho_s$	: crystal density, $kg\ m^{-3}$
$\mu_k$	: $k$ -th moment of the CSD, $\mu m^k$

### Subscripts

$agg$	: pertaining to agglomeration
$eq$	: equilibrium condition
$f$	: pertaining to the feed
$grow$	: pertaining to growth mechanism
$i$	: denotes the $i$ -th size interval
$s$	: pertaining to seed

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