DEVELOPMENT OF A LABORATORY SEMI-CONTINUOUS GIBBSITE PRECIPITATOR

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Abstract

The development of a laboratory semi-continuous gibbsite precipitator is described. Lab-scale precipitation represents an essential tool in generating experimental information required for improvements in gibbsite precipitation process design, control, and optimisation. It provides an important means for studying gibbsite precipitation mechanisms and effects of different process parameters on precipitation yield and product quality.

The use of a relatively complex experimental setup, such as semi-continuous precipitation (SCP), may provide experimental data that are easy to interpret and directly relevant to the plant. In the SCP setup developed in this work, four important precipitation variables are controlled: (i) solution supersaturation, (ii) solids concentration, (iii) total volume, and (iv) precipitation temperature. Building this experimental unit required development of customised control software to ensure continuous in-line process monitoring and real-time process control. Data from semi-continuous gibbsite experiments were used to extract gibbsite precipitation kinetics, such as crystal growth, nucleation, and agglomeration.

Notation and units

A	alumina concentration, gL^{-1}
A^*	alumina solubility, gL^{-1}
G	linear crystal growth rate, ms^{-1}
B_n	source term rate, $m^{-4}s^{-1}$
B	birth function, $m^{-4}s^{-1}$
D	death function, $m^{-4}s^{-1}$

1. Introduction

Managing the operation of gibbsite precipitation circuits for optimal performance continues to represent one of most challenging aspects of running a Bayer alumina refinery. Advances in the design and operation of the gibbsite precipitation process can only be achieved through improved understanding of the effect that different operational parameters have on underlying precipitation mechanisms. These relationships have been shown (Misra and White, 1971; Ilievski and Livk, 2006) to be extremely complex in gibbsite precipitating systems as a result of the complex physico-chemical environment in the Bayer process. Although many advances have been achieved in first principle precipitation modelling in recent years based on molecular dynamic simulations (Rohl, 2006), laboratory experiments remain the most reliable source of information to build such quantitative relationships.

Mimicking plant gibbsite precipitation, laboratory precipitation experiments commonly take place in stirred tank precipitators (STP), usually operated in a batch mode. Although information is rich, data from batch experiments may be difficult to analyse and interpret. They typically contain precipitation data under varying process conditions, such as supersaturation, solids concentration, and particle size distribution (PSD). This makes it difficult to deconvolute contributions of individual process parameters on various precipitation mechanisms.

The mixed suspension, mixed product removal (MSMPR) laboratory precipitator (Randolph and Larson, 1988) offers a continuous alternative that can easily overcome issues associated with batch experiments. Unfortunately, MSMPR experiments can be time-consuming and difficult to run. A simpler, constant supersaturation precipitator (Ilievski, 2001) represents a modification, which is easier to run and requires much less feed inventories than an MSMPR. In the constant supersaturation precipitator (CSP), constant concentration of alumina species is maintained by precisely metering the addition of the feed, based on continuous monitoring of alumina concentration in solution. CSP operation, however, is limited to relatively low supersaturations and low solids concentrations.

In order to overcome these limitations, a semi-continuous precipitator (SCP) was developed in this work. In the SCP setup, three main precipitation variables were tightly controlled: (i) solution supersaturation, (ii) solids concentration, and (iii) total volume. In the SCP, a steady operation can be achieved much quicker than in the MSMPR case, while still providing experimental data that are easy to interpret and directly relevant to the plant. Building this experimental setup required development of customised control software to ensure continuous process monitoring and real-time process control.

In the next sections, the experimental setup used to run SCP experiments is described together with the resulting experimental data and extracted gibbsite precipitation kinetics.

2. Experimental

The main objective of the SCP experimental setup is to ensure consistent gibbsite precipitation for a considerable period of time while minimising the requirement on the process feed, both liquor and seed. In order to achieve a consistent operation, four process variables were controlled continuously: (i) solution supersaturation, (ii) solids concentration, (iii) total volume, and (iv) precipitation temperature. As the temperature control of a jacketed vessel is not specific to gibbsite precipitation, the discussion in this paper only focuses on the first three process parameters.

The SCP experimental configuration, as shown schematically in Figure 1, consists of a draft-tube precipitator and two auxiliary mixed tanks containing feed liquor and seed. Peristaltic pumps are used to deliver feed liquor and seed and to maintain the outlet flow rate. The four process parameters are measured continuously via monitoring turbidity, temperature, conductivity and level. The data are collected by a computer through a data acquisitioning interface, which at the same time converts digital computer outputs into appropriate control signals.
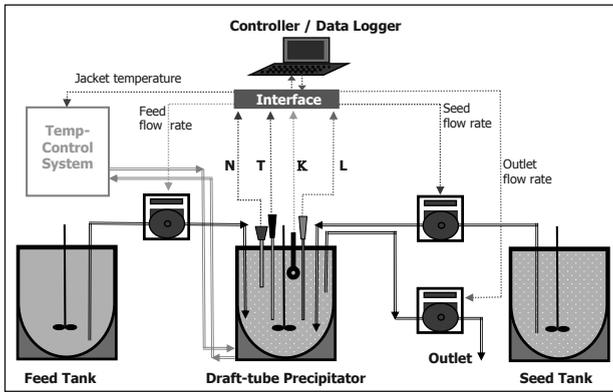
Figure 1. Schematic of the semi-continuous precipitation setup. The labels in the figure have the following meaning: (i) N turbidity, (ii) T temperature, (iii) K conductivity, and (iv) L level.

A feedback control structure, which was applied to stabilise the three main process variables is illustrated by a block diagram shown in Figure 2. In the presented control strategy, three single input-output feedback loops were configured, namely:

- Feed rate (liquor) – Al_2O_3,
- Seed rate – Solids concentration, and
- Outlet rate – Volume.

Three independent feedback controllers regulate these feedback loops based on the corresponding process measurements, such as conductivity, turbidity, and level. Although the three controllers are independent, they do affect process variables other than the one assigned to that controller. This is due to the interactive nature of gibbsite precipitation. It means, for example, that an increase in the feed flow rate, required to regulate alumina concentration, will also affect solids concentration and level in the tank. Likewise, a change in the seed flow rate will not only affect the solids concentration, but also the alumina concentration and level. Similarly to the situation in the plant, the maximum value of supersaturation achievable in the SCP system depends on a number of parameters, including:

- alumina concentration in the liquor feed,
- alumina and solids concentrations in the seed feed,
- solids concentration in the tank, and
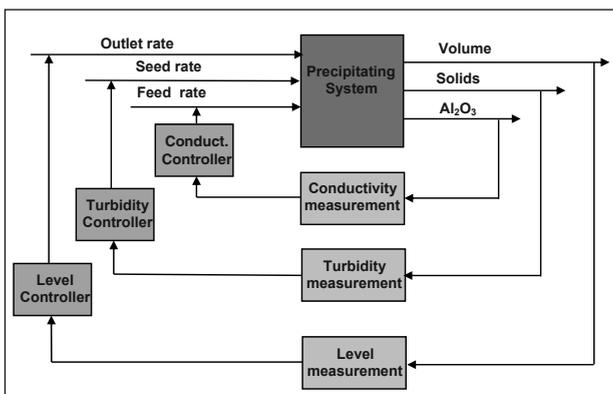- gibbsite nucleation and crystal growth kinetics.

Figure 2. Block diagram of the control strategy implemented in the laboratory SCP setup.

The control strategy shown in Figure 2, has been implemented in a Labview programming environment and integrated with the experimental hardware via a data acquisition interface. The software, which has been developed in-house, features a multi-tab graphical user interface (GUI), which enables both automatic and user-interacted operation of SCP experiments. A snapshot of responses of measured variables from one of SCP experiments is shown in Figure 3.

The precipitation process starts when seed is added to the system. At that moment, the conductivity of the system drops, but the turbidity raises due to increased solids concentration. This triggers a response from the controllers, which regulate the system inputs such that conductivity, turbidity and level are maintained at desired values. A stable SCP operation is reflected in consistent alumina and caustic concentrations, and solids concentration, as presented in Figure 4 and Figure 5, respectively. Note that these concentrations have been measured off-line by titration.
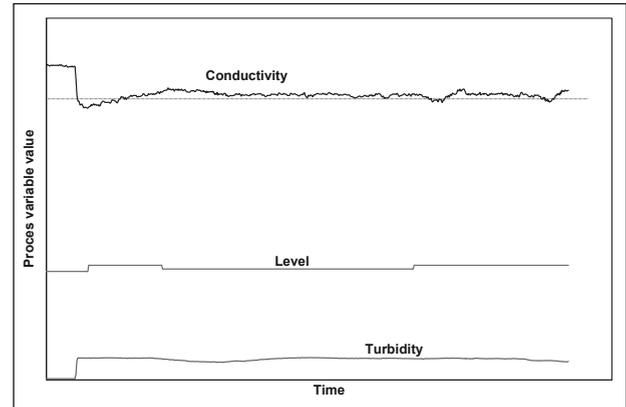

Figure 3. Transients of controlled process variables as measured during a semi-continuous precipitation run.
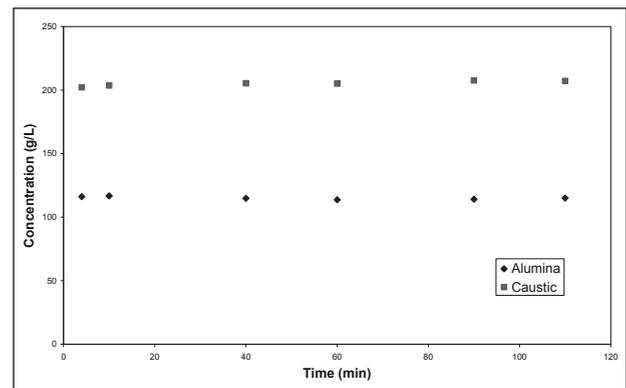

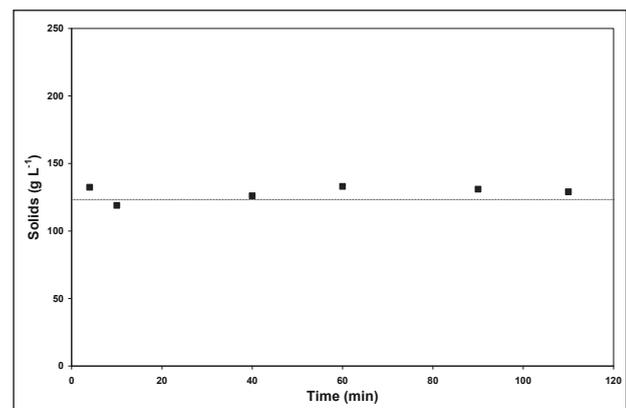Figure 4. Alumina and caustic concentrations measured at different times.

Figure 5. Solids concentration measured at different times.

Shown in Figure 6, is the evolution of PSD in the precipitator. After 90 minutes of precipitation under the given conditions, the amount of fines is significantly reduced compared to the initial seed. Furthermore, very similar PSDs can be observed for two consecutive measurement times, at 90 and 110 minutes. This indicates that at this stage in the process a steady operation was not only achieved in terms of the liquor concentrations, but also in terms of the PSD of the product. The mean residence time in this case was estimated to be around 40 minutes.

In order to ensure that the analysis of experimental data is well-defined, the precipitation system was set up to operate as a representative solids removal (RSR) precipitator. This condition was monitored by comparing PSDs in the precipitator and in the outlet stream. As shown in Figure 7, a very good agreement between both PSDs was observed confirming that the RSR condition was valid for our system.
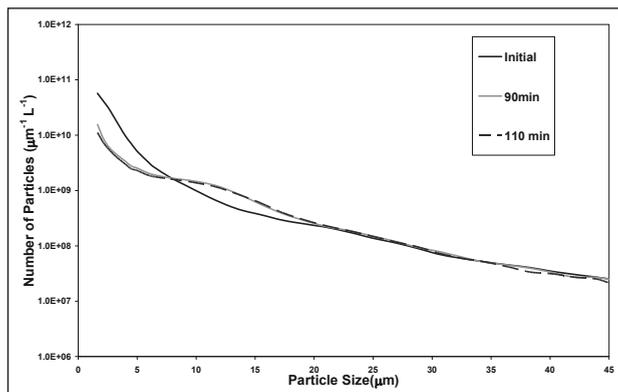

Figure 6. Comparison of PSDs measured at different times during a semi-continuous precipitation run.
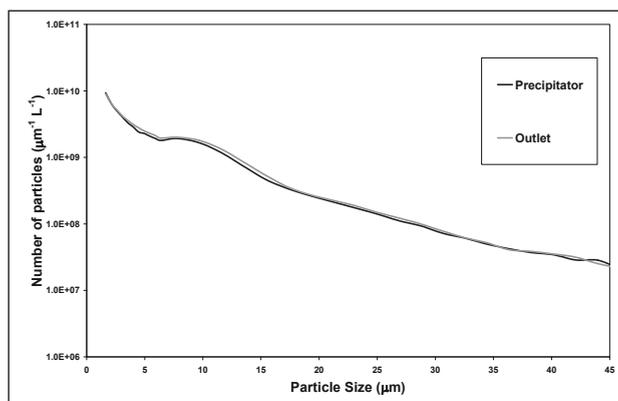

Figure 7. Comparison of PSDs measured in the precipitator and in the outlet from the precipitator.

3. Validation of a Semi-Continuous Precipitation System

Evolution of the PSD in a laboratory continuous precipitation process can be modelled using a population balance (PB) mathematical framework (Randolph and Larson, 1988). The PB model of a continuous well-mixed constant-volume precipitator can be stated as

$$\frac{\partial n(L,t)}{\partial t} + G\frac{\partial n(L,t)}{\partial L} = B_n \, \mathrm{d}(L-L_1) + B(L,t) - D(L,t) + \frac{1}{V}\left(Q_{in}\, n(L,t)_{in} - Q_{out}\, n(L,t)_{out}\right) \qquad (1)$$

where $n(L,t)$ is the number density of crystals, a function of crystal size, L, and time t. G is size-independent linear growth rate. The first three terms on the RHS of equation (1) represent the rate of change in crystal numbers due to the secondary nucleation, and crystal agglomeration. The last term accounts for the net change of crystal numbers in and out of the precipitation system, where Q_{in} and Q_{out} are the volumetric flow rates *in* and *out*, and V is the total volume. As indicated in equation (1) in the SCP case, crystal number density is affected not only by the precipitation kinetics, but also by the feed and outlet flow rates.

Based on the above precipitation model, a methodology for estimating gibbsite precipitation kinetics from SCP data was developed. More details on the adopted estimation technique can be found in Li *et al.* (2001). In order to validate the SCP experimental setup, gibbsite precipitation kinetics were estimated from SCP experimental data and their values compared to the kinetics estimates obtained from batch precipitation data.

In Figure 8, gibbsite crystal growth rates estimated from batch and SCP data under similar operating conditions are shown. The batch data lead to the estimation of growth rates at different supersaturations, as measured during a batch precipitation run. In the SCP case, a single point is obtained, since the entire experiment took place at constant supersaturation. The growth rate estimated from the SCP data matches very well with the value obtained at the same supersaturation from the batch data. Similarly good agreement between the two cases can also be observed for the estimated gibbsite nucleation rates, as shown in Figure 9.

A slightly lower nucleation rate estimated in the SCP case can be ascribed to a slight difference in solids concentrations between both cases at that point. A larger difference is observed when comparing the corresponding agglomeration rates, presented in Figure 10. A higher agglomeration kernel, which is a measure of the agglomeration rate, was estimated in the SCP case. This difference is also ascribed to a slight difference in solids concentrations between the two cases. The error bars in the plots indicate uncertainties in the kinetics estimates. The finding of a close match between the kinetics estimated in the two different precipitation systems is in accordance with the agreement between the batch and constant supersaturation precipitation kinetics reported by Li *et al.* (2001).
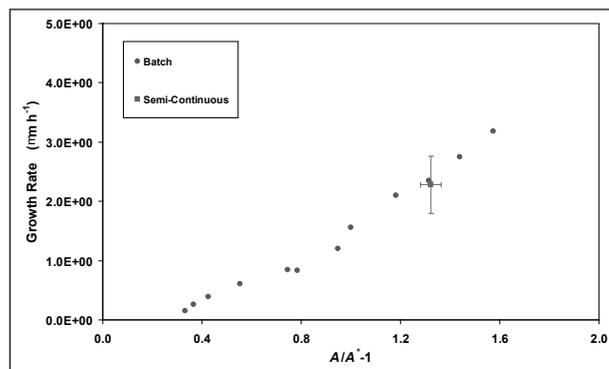

Figure 8. Comparison of semi-continuous and batch precipitation system crystal growth rate estimates.
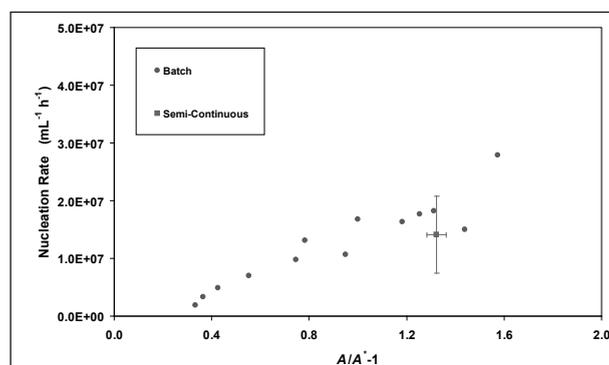

Figure 9. Comparison of semi-continuous and batch precipitation system nucleation rate estimates.
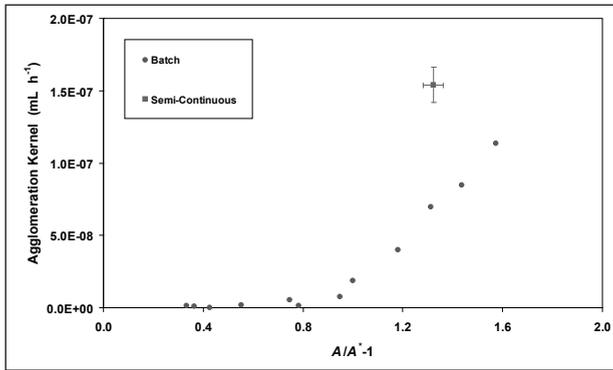
Figure 10. Comparison of semi-continuous and batch precipitation system agglomeration rate estimates.

4. Conclusions

The development of a laboratory semi-continuous gibbsite precipitation (SCP) setup has been described. The developed SCP experimental unit has an advantage over the conventional laboratory batch precipitation due to its ability to maintain gibbsite precipitation under consistent operational conditions for an extended period of time. In order to achieve this functionality, four main operational variables, supersaturation, solids concentration, volume, and temperature were controlled continuously. It was demonstrated that in terms of solution and solids concentrations, and PSD this precipitation setup can reach a steady operation in a time period equivalent to around 2.5 mean residence times. Gibbsite precipitation kinetics, such as crystal growth, nucleation, and agglomeration, estimated from SCP data were shown to match reasonably well kinetics estimated from batch precipitation data.

The presented advanced experimental precipitation setup can greatly contribute to enhancing understanding of gibbsite precipitation mechanisms required to facilitate further improvements in the design and optimisation of industrial gibbsite precipitation circuits.

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