M106 Seeded batch crystallization of ammonium phosphates by electrical conductivity measurement

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Introduction Batch crystallizers are used extensively in the chemical industry for manufacture of fine chemicals in small-scale operations. However, batch crystallizer is difficult to operate at constant supersaturation and can be operated over a wide range of sizes for very slow growth rate. Operationally, it has been used in different modes such as seeding, without seeding, forced cooling, and programmed cooling mode. In this study, essential information such as supersaturation and CSD, is introduced for modeling of MAP (Mono Ammonium Phosphates) crystallization process at various operating conditions.

Experimental Seeded batch experiments were conducted to study the effect of seed sizes, seed loading amount and initial supersaturation. Experimental condition is shown in Table.1 [1].

Modeling First, conductivity data history is converted to supersaturation concerning temperature data. By using supersaturation balance and kinetic model, initial kinetics parameters are estimated. Secondly, kinetics are recalculated for population balance from final crystal size distribution data. During this calculation, total numbers and averaged particles size are verified. Finally, simulated size distribution is validated against data [2].

Results and Discussion Table 2 shows final results of nucleation and growth rate kinetics of MAP for various experimental conditions, these parameters were firstly estimated from supersaturation data by using non-linear regression method. The nucleation and growth exponents were 2.2 and 1.4 respectively.

The supersaturation profiles are compared in Fig.1. The resulted kinetics could provide a good validation for MAP batch crystallization.

Conclusions The seed-loading ratio has the most significant effect on the CSD profiles and the degree of supersaturation influences not only the kinetics but also the crystal shape. Moreover, kinetic data such as nucleation and growth parameter obtained in this study are very useful for a MAP crystallisation process, and this simulation tool by analysis of conductivity measurements can be applied for crystalliser design, performance testing and other modelling tool.

References [1] J.Utomo, Y.Asakuma, K.Maeda, K.Fukui, N.Balliu, M.O.Tade, Chemical Engineering Journal, in press (2009) [2] J.Utomo, Y.Asakuma, K.Maeda, K.Fukui, N.Maynard, M.O.Tade, Chemeca (2009)

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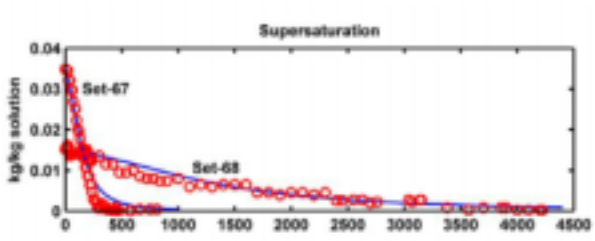

Fig.1Validation and comparison of supersaturation between experiment and simulation

Table 1 Experimental condition for seeded batch crystallization

Set	ΔC-initial [g/100 g solution]	Cs [%]	Ls [μm]
67	4.55	5.0	82.5
68	2.21	5.0	82.5
72	2.21	5.0	165.0
73	2.21	20.0	165.0
78	2.21	30.0	165.0
77	2.21	5.0	137.5
80	2.21	18.0	196.0
79	2.21	50.0	275.0

Table 2 Nucleation and growth parameters for seeded batch crystallization

Set	k_b no/[kg s (kg/kg)$^{b+1}$]	k_g m/[s(kg/kg)^g]
67	6.832E+08	9.357E-05
68	6.562E+08	3.089E-05
72	1.274E+08	3.971E-05
73	1.272E+08	4.770E-05
78	1.310E+08	4.171E-05
77	1.477E+08	3.672E-05
80	1.038E+08	5.036E-05
79	2.563E+07	5.408E-05