



## A new model and a design procedure for an Oslo-Krystal cooling crystallizer



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### ABSTRACT

The operation of the industrial Oslo-Krystal cooling crystallizer is simulated in this work as a continuous fluidized bed crystallizer with liquor recycling, where seed crystals are added at the top of the fluidized bed and product crystals are withdrawn at the bottom of the fluidized bed. It is assumed that the liquid phase moves upward through the bed in plug flow and the solid phase in the fluidized bed is perfectly classified. A model is developed to describe the variations of crystal size, bed voidage, crystal residence time and crystal production rate with the vertical position within the crystallizer. The effects of seed feed rate, feed solute concentration, recycle ratio and height/diameter ratio on the performance of such continuous fluidized bed crystallizer can be elucidated. A procedure based on the model can be used to rapidly design the crystallizer.

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### 1. Introduction

The continuous fluidized bed crystallizers have long been used in industry for the production of large uniform crystals because less nucleation rate than that in an agitated crystallizer can result [1]. The seed crystals added at the top of the crystallizer settle downward slowly through upward fluid. The solution velocity is chosen so that the crystals are uniformly suspended in the fluidized state. As the supersaturated solution flows upward, the liquor contacting the bed relieves its supersaturation on the growing crystals and subsequently the supersaturation decreases along the upward direction. The crystals are continuously withdrawn from the bottom of the crystallizer as product.

The opposing effects of classification and mixing interact with each other to determine the overall behavior of a fluidized bed crystallizer when the bed is composed of particles of different sizes [2–7]. By measuring the particle size distribution at various heights within the bed, Al-Dibouni and Garside [2] concluded that, for values of ratio of largest to smallest particle size present in bed greater than about 2.2, classification dominates the behavior of the fluidized bed and the variation of size distribution with height could be accurately predicted by assuming perfect classification. As the crystal size in a fluidized bed crystallizer varies as a function of bed height, the variation of bed

voidage with height also exists [2,8]. Information on the variation of voidage with bed height is important, because it determines the size of the crystallizer, which can be a significant factor affecting the total cost of the process. In the bed expansion experiments [2,8,9], the relationship between crystal size and bed voidage was found to be in agreement with the Richardson–Zaki equation [10].

Mullin and Nyvlt [11] proposed a design method to examine the important parameters in fluidized bed crystallizer operation. The variation of bed voidage with height was not rigorously taken into account in their model. Instead, they suggested that the overall mean values of bed voidage in the range 0.8–0.9 can usually be used in practice. Frances et al. [8] studied crystal size distribution during the fluidized bed crystallization and concluded that the fluidization behavior of crystals in the bed can be described by a cascade of perfect mixed crystallizers. By dividing the crystallizer into 30 layers considered as subsequent cells, Belcu and Turtoi [12,13] developed a computer program for the simulation of a fluidized-bed crystallizer and pointed out the strong influence of the equipment geometry. Shiau and coworkers [14,15] modeled the behavior of a fluidized bed crystallizer operated in a batch mode, in which the variation of local bed voidage along the bed was taken into account. However, the liquor recycling in a fluidized bed crystallizer was not considered.

In practice, a continuous fluidized bed crystallizer with liquor recycling would more reflect the actual behavior of an industrial Oslo-Krystal cooling crystallizer. The objective of this study is to develop a model to simulate the variations of crystal size and solute concentration with the vertical position in a continuous fluidized bed

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### Nomenclature

|                      |  |
|----------------------|--|
| $A$                  | cross section area of the bed, m   |
| $C$                  | solute concentration of the solution, kg solute/kg free water                        |
| $C_f$                | fresh inlet solute concentration of the solution at point A, kg solute/kg free water |
| $C_H$                | effluent solute concentration at the top of the bed, kg solute/kg free water         |
| $C^*$                | saturated concentration of the solution, kg solute/kg free water                     |
| $D$                  | diameter of the bed, m   |
| $g$                  | gravity acceleration, 9.8 m/s <sup>2</sup>   |
| $G$                  | growth rate of crystal, m/s  |
| $H$                  | total height of the bed, m   |
| $k_a$                | surface shape factor of the crystal, dimensionless                                   |
| $k_v$                | volume shape factor of the crystal, dimensionless                                    |
| $k_g$                | constant in Eq. (5), (kg free water/kg) <sup>n</sup> m/s                             |
| $L$                  | crystal size, m  |
| $L_0$                | seed size, m   |
| $L_p$                | product crystal size, m  |
| $n$                  | constant in Eq. (5)  |
| $N$                  | seed feed rate, 1/s  |
| $P$                  | crystal production rate, kg/s  |
| $Q_0$                | flow rate of the fresh solution at point A, kg free water/s                          |
| $R$                  | recycle ratio, dimensionless   |
| $u_{mf}$             | minimum fluidization velocity, m/s   |
| $u_s$                | superficial velocity, m/s  |
| $u_t$                | terminal velocity of crystals, m/s   |
| $x$                  | vertical position in the fluidized bed, m  |
| $z$                  | expansion index  |
| <i>Greek letters</i> |  |
| $\varepsilon$        | local bed voidage, dimensionless   |
| $\mu$                | viscosity of the solution, kg/m s  |
| $\rho$               | density of the solution, kg/m <sup>3</sup>   |
| $\rho_w$             | density of the water, kg/m <sup>3</sup>  |
| $\rho_p$             | density of the crystal, kg/m <sup>3</sup>  |
| $\tau$               | crystal residence time in the bed, s   |

crystallizer with liquor recycling. The results of this study can provide valuable information for design and operation of an industrial Oslo-Krystal cooling crystallizer.

## 2. Model

The operation of the Oslo-Krystal cooling crystallizer may be described in Fig. 1(a) [1,16]. A small quantity of warm concentrated feed solution (0.5–2% of the liquor circulation rate) enters the crystallizer vessel at point A, located directly above the inlet to the circulation pipe B. The recycle stream drawn off at the top of the fluidized bed, together with the fresh feed solution, is circulated by pump C through the tubes of heat exchanger D. On cooling, the solution becomes supersaturated in metastable region. The supersaturated solution flows down pipe E and emerges from the outlet F, located near the bottom of the crystallizer vessel. The nucleation rate is expected to be small, because the solution is maintained within the metastable zone at all times, no solid crystals make contact with any moving parts (*i.e.* an agitator) and fluid shear is not expected to be very large. Any nuclei that do form are rapidly transported to the top of the bed and either removed or allowed to slowly grow and fall back through the bed as seed crystals. Seed crystals are introduced at the top of the bed at a controlled rate and settle downward slowly through upwardly-

flowing fluid. Thus the crystals segregate in the bed with small ones at the top and large ones at the bottom.

The solution velocity is chosen so that the crystals are uniformly suspended in the fluidized state. As the supersaturated solution flows upward through a fluidized bed crystallizer, the liquor contacting the bed relieves its supersaturation on the growing crystals and subsequently the supersaturation decreases along the upward direction. Crystals that have grown to the required size fall to the bottom of the vessel and are discharged from the outlet G, continuously or at regular intervals. Any excess fine crystals floating near the surface of the solution in the crystallizer vessel are removed in a small cyclone separator H, and the clear liquor is introduced back into the system through the circulation pipe. A mother liquor overflow pipe is located at point I.

To model the performance of an Oslo-Krystal cooling crystallizer, a schematic diagram is shown in Fig. 1(b). It is assumed that the seed crystals are added at the top of the fluidized bed and the recycle stream drawn off at the top of the fluidized bed, together with the fresh feed solution, flows upward through the fluidized bed crystallizer. The crystals then settle downward slowly through upward fluid and are continuously withdrawn from the bottom of the crystallizer as product. In the development of the model it is further assumed that: (1) No uncontrolled nucleation occurs and a constant input of  $N$  seed crystals per unit time is added at the top of the fluidized bed, which corresponds to the practical case where all excess nuclei are withdrawn and dissolved [11,17,18]. (2) The seeds are uniform-sized. (3) For simplicity, the liquid phase moves upward through the fluidized bed in plug flow, although some researchers have applied computational fluid dynamics to simulate the fluid velocity profile for a multiphase flow of monodispersed suspensions in a fluidized bed crystallizer [19–21]. (4) The crystals in the fluidized bed are perfectly classified. (5) The crystal growth rate depends only on the supersaturation and not on hydrodynamic conditions (*i.e.* the fluid flow rate). This assumption is good if the crystal growth rate is limited by the surface integration step. If crystal growth rate is limited by mass transport, then it is possible that the hydrodynamic conditions could have an effect on crystal growth rate, but that effect is likely to be small and is not considered in this work.

In the following, the variations of solute concentration, crystal size, and bed voidage in a continuous fluidized bed crystallizer with liquor recycling will be derived. A mass balance of the solutes over an incremental distance  $\Delta x$  along the fluidized bed at steady state can be described as

$$(1 + R)Q_0(C|_x - C|_{x+\Delta x}) - \frac{1}{2} \frac{(1-\varepsilon)\Delta x A}{k_v L^3} k_a L^2 G \rho_p = 0 \quad (1)$$

where  $\frac{(1-\varepsilon)\Delta x A}{k_v L^3}$  represents the number of crystals within the incremental distance  $\Delta x$  and then  $\frac{1}{2} \frac{(1-\varepsilon)\Delta x A}{k_v L^3} k_a L^2 G \rho_p$  denotes the rate of growth on the overall surface of all these suspended crystals. The factor 1/2 arises because the growth rate is commonly defined as the growth rate of equivalent diameter of crystals. Similarly, a mass balance of the crystals over an incremental distance  $\Delta x$  at steady state can be described as

$$N k_v (L^3|_{x+\Delta x} - L^3|_x) \rho_p + \frac{1}{2} \frac{(1-\varepsilon)\Delta x A}{k_v L^3} k_a L^2 G \rho_p = 0 \quad (2)$$

Dividing Eqs. (1) and (2) by  $\Delta x$  and taking limits, one obtains

$$\frac{dC}{dx} = - \frac{A k_a \rho_p}{2 k_v (1+R) Q} \frac{(1-\varepsilon)}{L} G \quad (3)$$

$$\frac{dL}{dx} = - \frac{A k_a}{6 k_v^2 N} \frac{(1-\varepsilon)}{L^3} G \quad (4)$$

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