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Seeding strategies and residence time characteristics of continuous preferential crystallization

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ABSTRACT

This contribution investigates the effects of different seeding strategies and residence time characteristics on the dynamics of a Mixed Suspension Mixed Product Removal (MSMPR) crystallizer equipped with a fines dissolution unit. For the first time continuous preferential enantioselective crystallization is investigated. The fines dissolution is included as recycle streams around the MSMPR crystallizer. Moreover, primary heterogeneous and secondary nucleation mechanisms along with size-dependent growth rates are taken into account. A semi-discrete high resolution finite volume scheme (FVS) is employed for discretizing the derivatives with respect to the length coordinate. The resulting ordinary differential equations (ODEs) are solved by a Runge–Kutta method of order four. Several numerical case studies are carried out. The results support process design and optimization.

1. Introduction

An attractive process for gaining pure enantiomers from racemic mixtures is the so-called preferential crystallization (PC), which has been realized up to now in a discontinuous operation mode, see for example Jacques et al. (1981), Collet (1999), Alvarez-Rodrigo et al. (2004), Elsner et al. (2005), Coquerel (2007), Czapla et al. (2009), and Elsner et al. (2011). For elucidating the principle of a continuous enantioselective process one might consider a suspension crystallizer revealing MSMPR characteristics, i.e. a perfectly mixed tank (concerning both phases), which is continuously fed with a solution possessing a racemic composition of two enantiomers, see Fig. 1. Solid particles and liquid phase are continuously withdrawn. By a continuous supply of homochiral seed crystals of the preferred target enantiomer the preferential crystallization (PC) of only this enantiomer is initialized, i.e. growth of the seed crystals and possibly secondary nucleation of crystals of the seeded enantiomer, provided the crystallization takes place within the metastable zone where spontaneous, uncontrolled primary nucleation is kinetically inhibited. During a starting-up period, which strongly depends on the properties of the system as well as on the process parameters, the concentration of the target enantiomer is decreasing until a steady state is reached where the composition is determined by the mean residence time. Due to different kinetic mechanisms and their inherent different time constants, a different depletion of the supersaturation for each enantiomer can be realized by an appropriate choice of the process conditions. As long as a critical mean residence time, where primary nucleation may appear, is not exceeded, the concentration of the undesired counter enantiomer remains constant during the whole time. This fact reveals a benefit of this continuous process in comparison to the batch one. An optimal selection of the process conditions allows a constant production of the goal enantiomer at a high purity level. The mathematical modeling of PC as well as the optimization of the essential operating conditions requires high numerical precision (Qamar et al., 2008).

The population balance based modeling is regarded as a well suited and commonly accepted modeling approach for describing crystallization processes. The theory of population balances began in 1960s when Hulburt and Katz (1964) introduced it in the field of chemical engineering. A comprehensive overview on population balance modeling, nucleation and growth kinetics terms and methods of solution can be found in the book by LeVeque (2002), Mersmann (2001), Nývlt et al. (1985), and Ramkrishna (2000).

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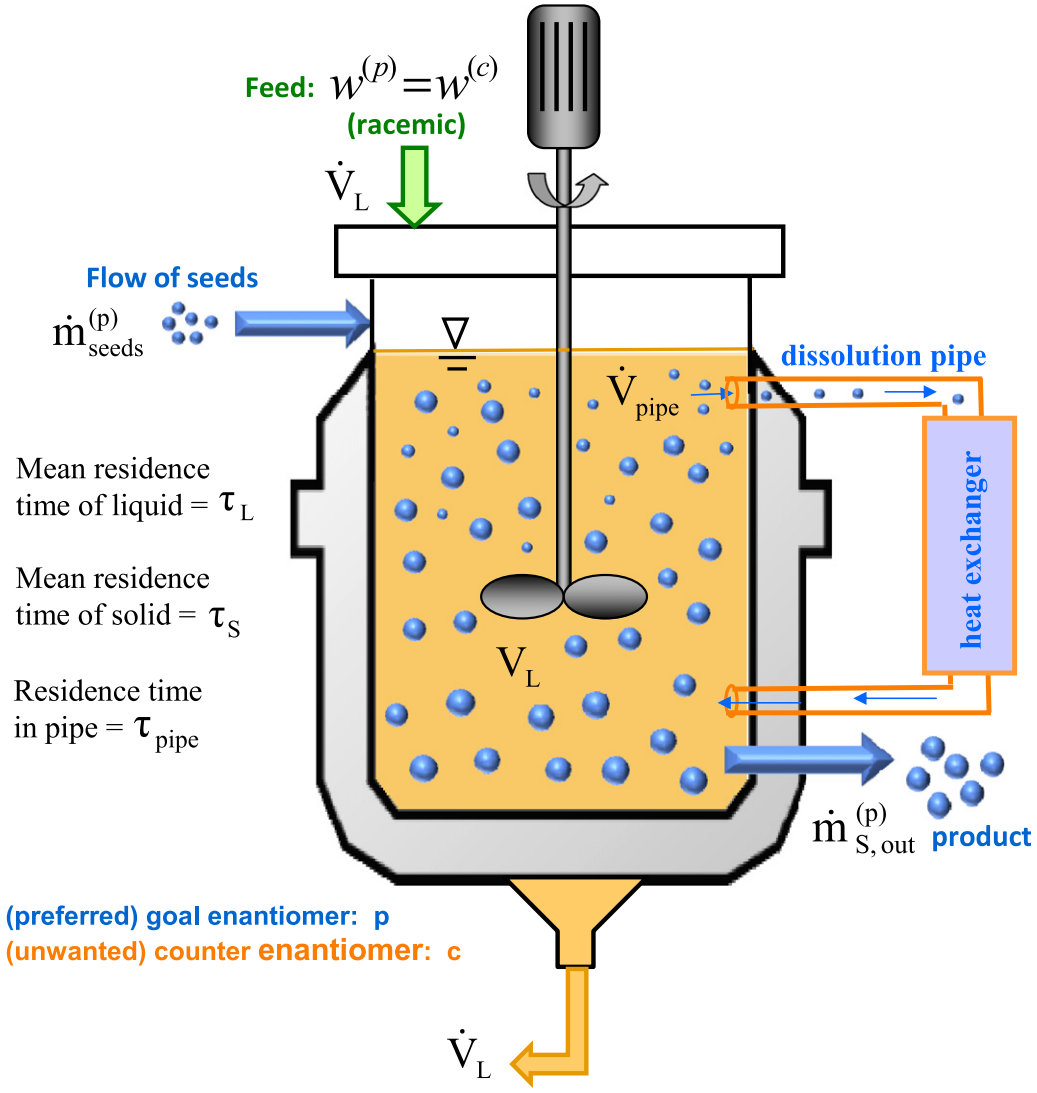

Fig. 1. Illustration of the principle of continuous enantioselective crystallization.

During the last decades many efficient methods were developed for solving population balance models (PBMs) such as the method of characteristics introduced in various fields by Rhee et al. (1986) and adopted for PBMs by Lim et al. (2002), Qamar et al. (2009), and Ramkrishna (2000), the method of weighted residual or orthogonal collocation by Rawlings et al. (1992), the Monte Carlo simulation by Smith and Matsoukas (1998) and Tandon and Rosner (1999), the fixed and moving pivot techniques by Kumar and Ramkrishna (1996a,b), and the high resolution finite volume schemes by Gunawan et al. (2004) and Qamar et al. (2006).

In this work a dynamic mathematical model is derived for simulating a continuously operated ideally mixed MSMPR crystallizer applied for continuous preferential enantioselective crystallization. The developed model includes the phenomena of primary heterogeneous nucleation, secondary nucleation, and size-dependent growth. A recycle pipe is attached to the crystallizer for the fines dissolution and it is assumed that fines are completely dissolved at the other end of the pipe. The effects of different seeding and operating strategies are investigated in simulations. The model and its parameters are based on experimental data obtained from batchwise crystallization. The semi-discrete flux-limiting finite volume scheme of Koren (1993) is implemented to solve the given model equations. The numerical results demonstrate the high order accuracy, efficiency and potential of the proposed numerical method for solving models describing continuous preferential crystallization of enantiomers and the potential of the process. The same performance of finite volume schemes was also observed by Gunawan et al. (2004), Hermanto et al. (2008), and Qamar et al. (2006) for solving polymorphic and batch crystallization models. Moreover, different contributions are available in the literature on continuous seeding and seed dissolution strategies in the batch crystallization process, see for example the articles of Aamir et al. (2010), Chung et al. (1999), and Woo et al. (2011) and reference therein.

The paper is organized as follows: in Section 2, a dynamic mathematical model based on population balances will be developed for a continuous MSMPR preferential crystallizer. In Section 3, the numerical method for solving the governing equations will be presented. In Section 4, some numerical case studies will be carried out. Finally, conclusions will be drawn in Section 5.

2. Continuous crystallization model

The model of the continuous crystallization process considers a two-phase dispersed system. The first phase is a continuous liquid phase containing the dissolved enantiomers and the solvent. The conditions in this phase determine the growth and