INVESTIGATION OF STIRRED TANK MIXING-PRECIPITATION PROCESSES

Lars Vicum [a], Marco Mazzotti [a], and Jerzy Bałdyga [b]

[a] Institute of Process Engineering, Sonneggstrasse 3, CH-8092 Zürich.
[b] Department of Chemical and Process Engineering, Warsaw University of Technology, PL-00-645 Warsaw, Poland.

Most of sparingly soluble crystals are produced through precipitation processes. The precipitation sub-processes (chemical reaction, nucleation, crystal growth) are usually very fast, so that mixing can strongly affect the course of precipitation and the resulting quality of the product (CSD, morphology). Identification and understanding the interaction mechanisms between hydrodynamics, mixing and precipitation enable formulation of adequate mixing-precipitation models. Once the models are formulated and validated experimentally, they can be used in industrial applications for prediction of effects of process parameters on the product quality and for scale-up.

In this work the effect of turbulent mixing on the course of a precipitation process in a stirred tank has been investigated experimentally and through modeling for the model substance barium sulfate. The main focus has been to identify new models for stirred tank mixing-precipitation processes. Two classes of models were developed. On one hand short-cut models characterizing the main mixing and precipitation mechanisms were realized. These models allow to study the effect of the key operating parameters, e.g. agitation rate, feed point position and reactant concentrations, on the mean particle size within a reasonable computational time.

On the other hand detailed CFD based models were developed. By implementing reaction and particle formation kinetics, population balance equations as well as a multi-scale-mixing model into the CFD code, a tool was developed that enables to describe the dynamics of a mixing-precipitation process in great detail. In this work it is presented for the first time how such a model is applied to a process in the complex geometry of a stirred tank.

Both tools have been applied to the precipitation of barium sulfate upon mixing of aqueous Na_2SO_4 and $BaCl_2$ solutions in a stirred vessel for conditions where particle formation is controlled by nucleation and growth exclusively. Particle formation kinetics were described based on a detailed $BaSO_4$ electrolyte model, accounting for the effect of various ions present in the solution and the association of ions to form ion-pairs. The short-cut models as well as the detailed CFD closure models characterize mixing phenomena from the macro- to the meso- and microscale employing suitable multi-scale models. The performance of both kind of mixing models was evaluated by applying them to the yield prediction of competitive chemical reactions, which were carried out experimentally in a stirred tank under various operating conditions.

Precipitation experiments were performed in a flat-bottom cylindrical vessel, equipped with four baffles and a Rushton type flat blade turbine. The evolution of the particle size distribution during the process was monitored using a FBRM device and offline particle characterization was performed using laser light diffraction measurements and a SEM image analysis technique. Results of the different models and experimental data are compared, thus highlighting the strength of the different models for particle size prediction.