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Review

Measurement, modelling, and closed-loop control of crystal shape distribution: Literature review and future perspectives

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ABSTRACT

Crystal morphology is known to be of great importance to the end-use properties of crystal products, and to affect down-stream processing such as filtration and drying. However, it has been previously regarded as too challenging to achieve automatic closed-loop control. Previous work has focused on controlling the crystal size distribution, where the size of a crystal is often defined as the diameter of a sphere that has the same volume as the crystal. This paper reviews the new advances in morphological population balance models for modelling and simulating the crystal shape distribution (CShD), measuring and estimating crystal facet growth kinetics, and two- and three-dimensional imaging for on-line characterisation of the crystal morphology and CShD. A framework is presented that integrates the various components to achieve the ultimate objective of model-based closed-loop control of the CShD. The knowledge gaps and challenges that require further research are also identified.

Contents

Introduction 2
Crystal shape and shape distribution: definitions and early modelling and measurement research 3
Structure (polymorph) and shape (morphology) of a crystal 3
CShD for a population of crystals 3
Relevant work on particle shape characteristics for a population of particles 4
On-line measurement of crystal shape based on 2D and 3D imaging 4
3D imaging systems 5
Image analysis and 3D shape reconstruction 5
Morphological population balance modelling 6
Multidimensional and morphological population balance models 6
Efficient solution of PB equations 8
MPBMs 9
Measurement and estimation of crystal faceted growth kinetics 10
Facet growth rates of single crystals 11
Growth rate estimation of crystals grown in reactors 11
MPBM-based model identification to estimate crystal facet growth rates and kinetics 12
Crystal morphology prediction for growth rate estimation 13
Optimisation and control of crystal shape and size distributions 13
Manipulated variables for crystal shape optimisation and control 14
Final remarks 15
Acknowledgments 16
References 16

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Introduction

For particulate products obtained by crystallisation, the crystal morphology is an important property because it not only directly affects the downstream processing of the particles, but also could affect the end-use properties of the final product. However, model-based closed-loop optimisation and control of the crystal shape for a population of crystals in a crystalliser has long been considered to be too challenging, mainly because of the limitations of available measurement techniques and modelling capabilities. Some people even question if there is such a concept as morphology for a population of crystals: while it is known how to define the morphology for a crystal, it is not as clear how to define the morphology for a population of crystals. In recent years, there has been important progress in the areas of on-line measurement of crystal morphology and process modelling of the dynamic evolution for a population of crystals during crystallisation. For on-line real-time measurement of the crystal morphology, the most impressive developments have been in two-dimensional (2D) and three-dimensional (3D) imaging (Borchert et al., 2014; Ferreira, Faria, Rocha, & Teixeira, 2011; Kuvadia & Doherty, 2011; Kwon, Nayhouse, Christofides, & Orkoulas, 2013; Larsen & Rawlings, 2009; Ma, Liu, & Wang, 2016; Ochsenbein, Schorsch, Vetter, Mazzotti, & Moran, 2014; Oullion, Puel, Fevotte, Righini, & Carvin, 2007b; Qu, Louhi-Kultanen, & Kallas, 2006; Simon, Abbou-Oucherif, Nagy, & Hungerbuhler, 2010; Singh et al., 2012; Su, Hao, Barrett, & Glennon, 2010; Wang, Roberts, & Ma, 2008; Zhang, Ma, Liu, & Wang, 2015; Zhou, Srinivasan, & Lakshminarayanan, 2009). For modelling the dynamic evolution of the crystal shape for a population of crystals in a crystalliser (i.e., the crystal shape distribution (CShD)), development has occurred because of the newly proposed multidimensional and morphological population balance models (e.g., Alatalo, Hatakka, Louhi-Kultanen, Kohonen, & Reinikainen, 2010; Borchert, Nere, Ramkrishna, Voigt, & Sundmacher, 2009; Briesen, 2009; Dandekar, Kuvadia, & Doherty, 2013; Hounslow, Lewis, Sanders, & Bondy, 2005; Kwon, Nayhouse, Christofides, & Orkoulas, 2014; Lindenberg, Krattli, Cornel, Mazzotti, & Brozio, 2009; Ma, Wang, & Roberts, 2007; Majumder, Kariwala, Ansumali, & Rajendran, 2010; Nagy & Braatz, 2012; Oullion, Puel, Fevotte, Righini, & Carvin, 2007a; Ramkrishna & Mahoney, 2002; Sato et al., 2008) and morphological population balance (e.g. Ma & Wang, 2008, 2012; Ma, Wang, & Roberts, 2008; Ramkrishna & Singh, 2014; Wang et al., 2008). In this article, CShD is used as the abbreviation for crystal shape distribution to differentiate it from crystal size distribution (CSD) because the latter is widely used in the crystallisation community to represent the crystal size distribution, where the size of a crystal is often defined as the diameter of a sphere with the same volume as the crystal. The new developments have led to proof of concept in model-based closed-loop automatic control of the CShD in crystallisation processes.

This review aims to not only provide a summary and critique of the relevant literature about the measurement and modelling of crystal shape, but also to present a framework that integrates the various components to achieve the ultimate objective of model-based closed-loop control of the CShD, and identify the knowledge gaps that require attention in future research. As a result, rather than beginning the review by discussing the details of the individual elements, an integral framework is first presented that gives a picture of how the individual topical elements are linked. The integral framework is schematically shown in Fig. 1. The main components include:

- On-line real-time measurement of the crystal shape and shape distribution using on-line 2D and 3D imaging instrument and image analysis techniques.
- Measurement of crystal faceted growth rates and growth kinetics using 2D and 3D images in crystal growth cells and stirred tanks, as well as estimation based on model identification methods.
- Modelling and simulation of the dynamic evolution of the CShD using morphological population balance models (MPBMs).
- Feedback and cascade control to track the optimum operating conditions, as well as on-line optimum control.

Measured or estimated facet growth kinetics, that is, the facet growth rate as a function of variables such as supersaturation, solvent, impurities, and crystal size, are needed by MPBMs (Wang et al., 2008). MPBMs can be used in multiobjective optimisation to obtain the optimum operating conditions, such as an optimum supersaturation curve, that leads to the desired CShD, yield, and other objectives. The control system configuration (e.g., simple feedback or cascade control) can then be designed to track the optimum process conditions, such as the optimum supersaturation conditions. However, such a control strategy cannot avoid batch-to-batch variation because of uncertainties, and on-line measurement of the CShD is therefore required. Through real-time image segmentation analysis and shape reconstruction, the real-time size/shape distributions of crystals can be estimated. For example, in cooling crystallisation tracking the optimum supersaturation curve, the real-time CShD information can be used to reoptimise the remaining optimum supersaturation curve.

Here, it needs to be pointed out that the presented framework might not be the only or best framework for integrating the topical elements to achieve closed-loop control of the CShD, and new and more innovative control frameworks are also open for future research (e.g., could CShD be directly used as the set point in a control configuration?). However, it will help the readers to understand how the individual components can be linked together in a framework for closed-loop control of the CShD. It also needs to point out that the framework does not consider other factors. For example, for scale-up in modelling large crystallisers, computational fluid dynamics (CFD) should be included and integrated with MPBMs to account for the mixing conditions. Furthermore, the framework was constructed with a batch or semibatch stirred tank crystalliser in mind. For continuous or other types of crystallisers, amendments need to be made.

In the remainder of the paper, the developments in relevant areas will be reviewed. The topics will cover:

(1) Description of crystal shape and definition of the CShD (see section "Crystal shape and shape distribution: definitions and early modelling and measurement research").
(2) On-line measurement and characterisation of the crystal shape and CShD using 2D and 3D imaging and image analysis techniques (see section "On-line measurement of crystal shape based on 2D and 3D imaging").
(3) MPBMs for modelling the dynamic evolution of the CShD subject to variations in the operational conditions (see section "Morphological population balance modelling").
(4) Direct measurement as well as model-based estimation of faceted crystal growth rates and faceted growth kinetics (see section "Measurement and estimation of crystal faceted growth kinetics").
(5) Model-based optimisation and closed-loop control of the CShD (see section "Optimisation and control of crystal shape and size distributions").

Knowledge gaps and suggestions for future research will be identified in the individual sections as well as in the Final Remarks section.