Application of X-Ray Diffraction In-line to Monitor Solution Mediated Phase Transformations between Polymorphs During Batch Crystallization Processes.

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Abstract

In-line power X-ray diffraction was applied using a flow-through cell and a chemometric method developed previously [Enhancing the signal-to-noise ratio of X-ray diffraction profiles by smoothed principal component analysis, Chen, Zeng Ping; Morris, Julian; Martin, Elaine; Hammond, Robert B.; Lai, Xiaojun; Ma, Caiyun; Purba, Elida; Roberts, Kevin J.; Bytheway, Richard, Analytical Chemistry (2005), 77(20), 6563-6570] for the quantitative analysis of a polymorphic phase transformation, within a slurry, during crystallization that converted the metastable alpha-form of L-Glutamic Acid (LGA) to the stable beta-form at a half litre scale-size.

The study revealed the detection limits to be 0.4 wt% for β -form of LGA in LGA-methanol slurries containing mixtures of both polymorphs of LGA. A method of fitting individual peaks was used to evaluate the rate of inter-conversion. The rate constants describing the growth and dissolution processes were evaluated and the activation energies were calculated through the application of the Arrhenius relation.