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Application of Synchrotron Radiation FTIR Microscopy to Mapping the Spatial Distribution of Supersaturation in Crystal/Solution Slurries

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Beamline(s): U2B

Introduction:

Study of the spatial and temporal distribution of crystal mass and solution concentration in crystallising systems is necessary to improve the understanding of nucleation and crystal growth phenomena, and to create reliable algorithms for their control. Variation in concentration and supersaturation impacts on the crystal size and thus measurements and control of supersaturation offer a route to control of Crystal Size Distribution (CSD) in batch reactors.

The feasibility of the synchrotron microscopy as a tool for studying concentration and supersaturation spatial distributions in crystal/solution slurries down to 10 μm scale has been demonstrated through an examination of crystals of monosodium glutamate slurried with saturated aqueous solution mother liquor. For the first time, this technique has been applied to monitor the distribution of crystal mass as well as the solute concentration in supersaturated solutions at the same time.

Methods and Materials:

A Nicolet Nic-Plan IR microscope using a collimated synchrotron radiation at the Beam Line U2B was used in this work. Two types of objects were studied: single crystals and crystal aggregates. Line and area maps of the intensity of the absorbance/ transmittance of IR light over a scanned area were obtained to determine concentration and supersaturation spatial distribution in the slurry. The spectral range of scanning was limited to 1400-1600 wavenumbers (characteristic absorbance range of the crystallized substance) in order to observe the concentration changes. An ATR-FTIR calibration model based on ratio of the characteristic peaks for the solute and solvent^{1,2} was used to qualitatively determine the solute and crystal mass concentration in the slurry.

Results:

The absorbance/ transmittance intensity and hence concentration of species close to the crystal face is non-linear, non-uniform and non-smooth. The crystal area is characterised by a concentration profile that shows non-homogeneity of the concentration distribution over that area. The points having maximal concentration doesn't belong to the area of crystals, but to their immediate surroundings. This might be due to refraction of light from other crystals or from rough crystal surfaces, noisy signal, background change etc. The areas with relatively higher concentration represent the position of the crystals, but the shape of the contours does not correspond to the fixed crystal surfaces. The concentration distribution is non-linear and non-uniform and it shows spots of lower concentration in the area close to crystals. However, a positive concentration gradient exists around crystal edges.

Conclusions:

The solute concentration in the slurry is non-uniformly distributed and within the studied mapping range we cannot confirm so far the expected concentration profiles in terms of the boundary layers theories. A new calibration model needs to be developed for transmission microscopic IR spectroscopy to account for the specific conditions of the spectroscopic measurements and for the presence of crystals as well as solution in the system. Faster mapping techniques are required for on-line monitoring of concentration and crystal mass distribution in crystal/solution slurries.

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References:

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