

- The cooling rate was lowered from 1 to 0.75 °C min<sup>-1</sup> in an attempt to slow the generation of supersaturation and oiling out was noticeably reduced. A further reduction of the cooling rate to 0.5 °C min<sup>-1</sup> prevented oiling out from occurring all together.
- Seeding was employed at the cooling rate of 1 °C min<sup>-1</sup>; the addition of seeds in the undersaturated region worked better.

In summary, encrustation occurred in NiTech DN15 crystallisers when operated incorrectly which can be avoided entirely when appropriate operational and seeding strategies are applied.

### 3.5.5 PAT Implementation

In NiTech crystallisers, the saturated solution becomes supersaturated when flowing through the pre-established temperature zones. The increased specific heat transfer areas provide ample sites for PAT tools that can readily be placed anywhere along the length of the COBC. This is the unique advantage in comparison to industrial batch operation where it is impossible to implement any lab scale PAT tools in scale up vessels, preventing the science learnt in labs from realisation.

Stability of continuous crystallisation and steady states of process parameters in continuous crystallisation, such as solute concentration, crystal size and supersaturation, can be evaluated experimentally with the help of PAT probes. In crystallisation of palm oil melt, two FTIRs and two PVMs were placed at two strategic locations of a known distance along the length of the DN15 crystalliser; the analysis of solute concentrations from FTIRs and particle size from PVMs at two locations *at the same time* provides *spatial* characteristics, *e.g.* the rate of decrease in solute concentration, the rate of supersaturation depletion, the rate of crystal growth and the stability of operation, while the analysis of data at two time intervals at *a specific location* gives the *temporal* characteristics. This allows the determination of the degree of stability of crystal size and solute concentration, the stability of supersaturation *etc.*<sup>224,225</sup> Crystallisation kinetics (growth) was directly calculated from experimental data based on the changes of solute concentration from FTIR as a function of time and equally, experimental data can also be fed into a population balance model to determine crystallisation kinetics. Evolution of crystal particle size distributions (PSD) in a seeded cooling crystallisation of L-glutamic acid was investigated and showed that a constant PSD or steady state was obtained after the 2nd residence time in a seeded crystallisation, while PSD varied and steady state was not achievable in an unseeded case.<sup>147</sup>

FBRM and a UV-NIR spectrophotometer were employed to monitor particle counts and absorbance spectra of methylene blue dye in a COBC to determine residence time distribution of dispersed solid and liquid phase.<sup>46</sup> Other types of PAT tools have also been used in COBC, for example, immersion Raman, non-invasive Raman and THz Raman.