

Reaction Engineering

The conductivity of substrates in a reactive system frequently change during the course of a reaction. For example the driving off of water, during a condensation reaction or the precipitation of ions from solution during crystallisation. Tomography information can provide valuable insights to the location and progress of reactions.

Radial flow CFD validation

Achieving an even flow distribution in large packed bed reactors is critical to their performance. In many petrochemical processes there is significant energy incentive to minimise pressure drop. This has led to the development of a number of variants on the radial flow bed. Johnson Matthey Catalysts used computational fluid dynamics (CFD) to assess the flow distribution in a new radial flow reactor design.

To validate the CFD model, a model was constructed and fitted with 8 x 16-electrodes to allow full interrogation of the process volume by ERT. An ITS p2000 8-channel ERT system was used for data acquisition. A high conductivity tracer was added to the inlet feed to measure the flow pattern within the reactor.

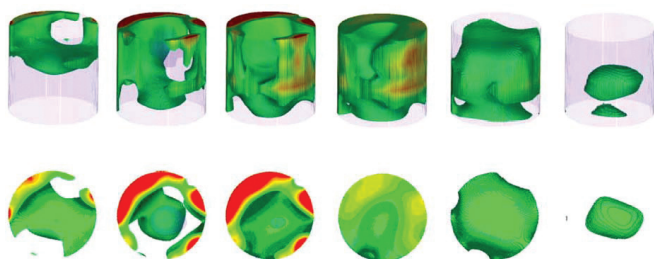


Figure 1: Time series of '3D solid-body' images following a high conductivity tracer as it flows through the reactor

Figure 1 shows 3D solid-body for several frames of data after the high conductivity tracer addition. The simultaneous movement of the tracer down through the bed and towards the central collector can be seen.

Further analysis was performed using pixel x pixel correlation to track the flow of the high conductivity tracer. Both a radial and axial were combined to provide a velocity magnitude and direction throughout the reactor to validate CFD results previously obtained.

The experimentally derived velocity map agreed qualitatively well with the CFD results with some uncertainties towards the bottom of the reactor where the results were less robust. Overall, there was confidence in the new design concept of a more efficient radial flow reactor.

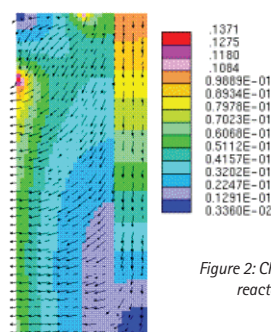


Figure 2: CFD simulated velocity map of model reactor at experimental conditions

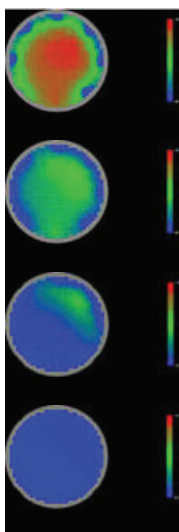


Figure 3: Tomogram showing different stages of nylon polymerisation, red regions correspond to high conductivity regions where water is present

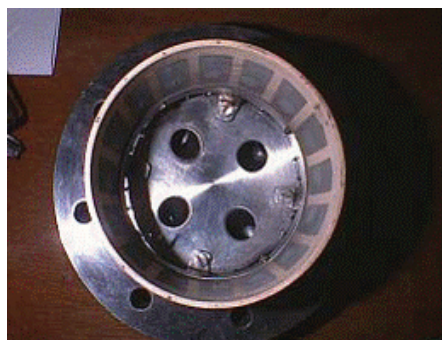


Figure 4: Ceramic-based tomography sensor used in an autoclave

Polymerisation

Many polymers are formed by reacting single molecules together at high pressure, often a catalyst. The molecules join together into larger molecules with polymeric properties. These properties, critical to processing, are often determined by the "length" of the polymer chains. The chain lengths are a consequence of the conditions in the reactors and the effectiveness of the mixing.

The challenge is to measure the polymer properties as they are formed and the conditions, particularly mixing, in the reactors. This is made difficult by the high pressure conditions involved in most polymerisation reactors.

Two sensor strategies have been successfully deployed for investigating such challenging reaction conditions:

- Ceramic sensors to withstand high temperatures and pressures to characterise the polymerisation of nylon. Nylon polymerisation is essentially a condensation reaction. Changes in conductivity show the reaction's progress. This can help improve product quality through improving consistency in the reactor.
- High temperature polymers have also been deployed to investigate reactions at moderately high temperatures and high temperatures.

Key Benefits

- Determination of concentration conditions within process volume
- Characterise homogeneity to improve process consistency
- Identification of end point of polymerisation

Case Study: Crystallisation

The Challenge

Crystallisation is a key manufacturing operation. While the target is always to grow the desired crystals within a narrow size distribution, the nature of the crystallisation process can make it difficult to achieve this level of control. One particular challenge is scaling up production to achieve the ideal particle size (which influences the final drug's performance in the patient) and the ideal crystalline form (which can drastically affect biological performance). Achieving perfect crystals involves perfect mixing and the control of concentration and temperature. It isn't easy.

The Solution

Electrical resistance tomography (ERT) offers the opportunity to monitor how crystallisation develops in different regions of a vessel and as a result provides a useful tool for process scale-up. ERT can monitor conductivity at in excess of 200 points in a vessel at rates of 20-40 times per second.

There is a substantial change in conductivity as ions move from solution to solid form. Figure 1 shows the tracking of crystallisation of paracetamol using ERT, compared to FBRM. It shows that ERT is able to pick up crystallisation much closer to nucleation (as it does not require the build up of particles).

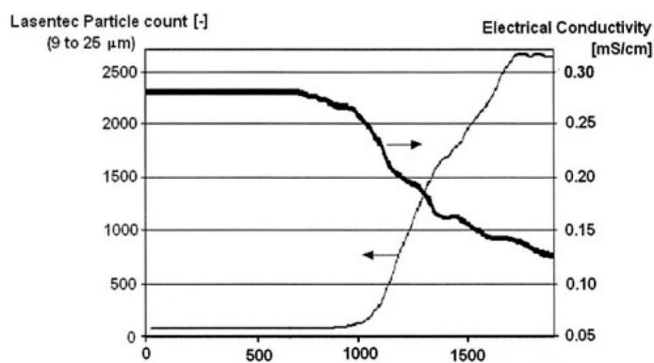


Figure 1: Chart plotting FBRM and ERT measurements during crystallisation (Ricard et al., 2005)

Experimental

The precipitation of barium sulphate (surface addition of barium chloride to sodium sulphate) was observed at:

- Two scales (7 and 170 litres)
- Three mixing speeds
- Three different concentrations (all equimolar)

The scale up from 7 to 170 litres was based on power per unit volume and addition time. Process tomography was able to confirm this successful application of these rules by monitoring reaction conditions.

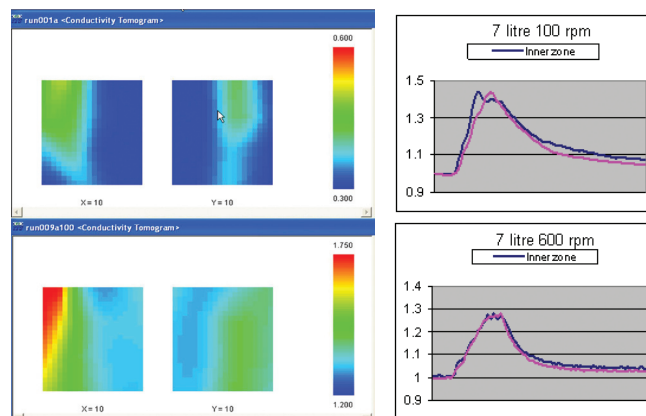


Figure 2: Slow (top) and rapid (lower) mixing conditions for small scale vessel

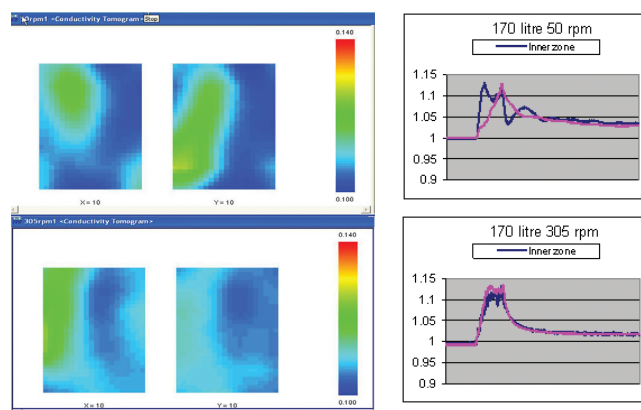


Figure 3: Slow (top) and rapid (lower) mixing conditions for large scale vessel

These conductivity images provide a snapshot of the highly conductive region where crystallisation occurs (rapid in centre, slow along baffle) and the conditions during the reaction (slow with variable conditions and extended time to complete, rapid with relatively constant conditions and reaching completion more rapidly).

Customer Benefits

Electrical Resistance Tomography provides an effective tool for characterising reaction conditions in crystallisation

Complement other on-line tools such as Lasentec its sensitivity at stages close to nucleation where many particle characteristics are determined

Improved understanding of the early stages of the crystallisation process

References

Ricard, F, Brechtelsbauer, C, Xu, XY, Lawrence, CJ (2005) Monitoring of multiphase pharmaceutical processes using electrical resistance tomography, Chemical Engineering Research and Design, 83, A7, pp 794-805

Bolton, GT, Bennett, M, Wang, M, Qiu, C, Wright, M, Stanley, SJ and Rhodes, D (2007) Development of an electrical tomographic system for operation in a remote, acidic and radioactive environment, Chemical Engineering Journal, Vol. 130, Issues 2-3, pp 165-169