Applications of electrical tomography to improve the performance of crystallization, precipitation and mixing processes

by K.M. Primrose*

Introduction

In industrial processes, the application of process tomography technology requires the development of robust sensors—usually at larger scales than lab based experiments, data capture which is able to accommodate noisy process conditions and the manipulation of data to allow for meaningful interpretation of process conditions by plant operatives.

The process envelope for electrical resistance tomography encompasses the following conditions as indicated in Table I.

In each of the above cases, particular care has had to be taken with the choice of materials for the electrodes, the supporting substrate, shielding of signals along long cables, and use of ancillary equipment (such as zener diode barriers in the case of installation in a hazardous atmosphere).

Scale-up of crystallization

The control of crystallization, to the extent of specifying particle size distribution and crystal polymorph is both very challenging and important to many industries. For example, in the pharmaceutical sector the wrong polymorph can lead to serious side effects and the wrong particle size can reduce bioavailability of an active ingredient.

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
  – 100, 300, 600 rpm for 7 litre vessel
  – 49, 149, 305
➤ Three different concentrations (all equimolar)

Electrical resistance tomography (ERT) is able to monitor conductivity at in excess of 300 points in a circular measurement plane vessel at rates of 20–40 times per second per plane. It is possible to measure up to 8 planes in series, leading to 2 500 measurement points in the volume of a reactor.

There is a substantial change in conductivity as ions move from solution to solid form. This makes ERT a useful tool for tracking regions of a vessel of contrasting conductivities.

Figure 1 shows the tracking of crystallization of paracetemol using ERT, compared to FBRM. It shows ERT is able to pick up crystallization much closer to nucleation (as it does not require the build-up of particles). This is important as it is at the point of nucleation where a crystal’s polymorph is determined.

It is well known that as scale moves from millilitres to litres, mixing and flow processes can allow different regions of a vessel to have different concentrations and conditions. This means that scaling-up processes can lead to different product characteristics.

ERT offers the opportunity to monitor how crystallization develops in different regions of a vessel and as a result provides a useful tool for process scale-up. This work builds on previous studies of the precipitation of barium sulphate11,12

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Table I

<table>
<thead>
<tr>
<th>Process requirement</th>
<th>Example of environment</th>
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<tbody>
<tr>
<td>Intrinsic safety</td>
<td>Solvents present in pressure filter</td>
</tr>
<tr>
<td>High temperature</td>
<td>Parr reactor/autoclave for Fischer Tropsch reaction research; bespoke autoclave for</td>
</tr>
<tr>
<td></td>
<td>study of nylon polymerization</td>
</tr>
<tr>
<td>High pressure</td>
<td>Abrasive environment such as hydrocyclone; pumping of slurries for land reclamation</td>
</tr>
<tr>
<td>Physical resistance</td>
<td>Monitoring of precipitation of highly active radio-nucleotides in 6 molar nitric acid</td>
</tr>
<tr>
<td>Chemical resistance</td>
<td>Monitoring of dryness in pressure filtration (circular sensor 4 m diameter) and</td>
</tr>
<tr>
<td></td>
<td>monitoring of cloud height in storage tanks (4 m height); monitoring of homogeneity</td>
</tr>
<tr>
<td></td>
<td>in FCC riser (1 m diameter)</td>
</tr>
<tr>
<td>Large-scale sensor</td>
<td>50 m cable run from sensor to data acquisition system</td>
</tr>
<tr>
<td>Long cable lengths</td>
<td>Low conductivity such as polar organic solvent. High conductivity such as monitoring</td>
</tr>
<tr>
<td>Conductivity</td>
<td>of precipitation of highly active radio-nucleotides in 6 molar nitric acid</td>
</tr>
</tbody>
</table>

Results

Process tomography conditions were observed using ITS P2000 instrument, which takes data from up to 8 measurement planes (each with 16 electrodes in a circular array). In addition, particle size and micrographs were taken of the precipitate. The tomography data were used to determine:

➤ Reaction progress through averaging all electrical measurements
➤ Homogeneity of reactant conditions through analysis of standard deviation of vessel cross-sectional conductivity maps
➤ Reaction conditions through analysis of tomograms of vessel conditions.

Figures 3 and 4 show results from the high and low mixing conditions at small and large scale respectively.

The images on the left show two orthogonal ‘conductivity’ slices through the mid point of the vessel during the feed addition and the graph on the right shows the mean resistivity plotted from the entire process volume plotted over time. It can be seen from the conductivity slices that the key
difference between the reaction conditions is whether the high conductivity regimes are running along the baffles (rapid mixing) or at the centre of the vessel (slower mixing).

These conductivity images provide a snapshot of the highly conductive region where crystallization 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).

**Analysis**

As the high conductivity region is observed at the centre of the vessel in slow mixing conditions and at the baffle in rapid conditions, the average measurements from these zones were compared to characterize these two conditions.

Figures 5 and 6 show these average measurements for the fast and slow experiments respectively.

It can be seen that the scale-up criteria have effectively...
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reproduced similar reaction conditions. These are borne out by the particle size data taken at the two different scales.

Figure 7 shows the conductivity variation between the two regions for the slow and fast mixing experiments. It is clear that the conductivity difference between the regions is much less for the fast mixing case when compared to the slow case.

Conclusions

Electrical resistance tomography has been implemented in a range of industrial processes. It is one of the few technologies that can resolve differences within a process volume and can be used as a sensor in its own right or to complement information from other measurement devices.

It provides an effective tool for characterizing reaction conditions in crystallization.

Key benefits of ERT are the ability to detect conditions throughout a vessel and its sensitivity at stages close to nucleation where many particle characteristics are determined. In addition, the technique can be applied at different scales through both circular and probe based arrays.

Acknowledgments

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References