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

by K.M. Primrose*

Synopsis

Electrical tomography has developed rapidly over the last few years in its sophistication and practical use to enable three-dimensional information on the distribution of phases within flowing mixtures to be observed[1]. This paper reviews the current state of application from the industrial perspective, drawing on applications case studies based on scaling studies in the laboratory right through to installations on plant. In the laboratory, applications have looked at scale-up of mixing[2] processes involving gas-liquid and solid-liquid suspensions[3] with a particular interest in verifying computational fluid dynamics simulations or in checking scale-up design[4,5]. In the processing plant, the circumstances are usually bespoke and may range from simple detection of the degree of homogeneity of a flowing mixture through to the need to measurement a sophisticated batch process such as crystallization, sedimentation, filtering and drying. Examples drawn from the chemical and pharmaceutical industries will be presented and related to the benefits that can be gained through application of the method.

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 of sensors 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.

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 sulphate[11,12]

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)

* *Industrial Tomography Systems plc., Manchester, UK.*

Table I

Electrical resistance tomography conditions

Process requirement	Example of environment
Intrinsic safety	Solvents present in pressure filter[6]
High temperature	Parr reactor/autoclave for Fischer Tropsch reaction research; bespoke autoclave for study of nylon polymerization
High pressure	Parr reactor/autoclave for Fischer Tropsch reaction research; bespoke autoclave for study of nylon polymerization[7]
Physical resistance	Abrasive environment such as hydrocyclone; pumping of slurries for land reclamation[8]
Chemical resistance	Monitoring of precipitation of highly active radio-nucleotides in 6 molar nitric acid[9]
Large-scale sensor	Monitoring of dryness in pressure filtration (circular sensor 4 m diameter) and monitoring of cloud height in storage tanks (4 m height); monitoring of homogeneity in FCC riser (1 m diameter)[7]
Long cable lengths	50 m cable run from sensor to data acquisition system[9]
Conductivity	Low conductivity such as polar organic solvent. High conductivity such as monitoring of precipitation of highly active radio-nucleotides in 6 molar nitric acid[9]

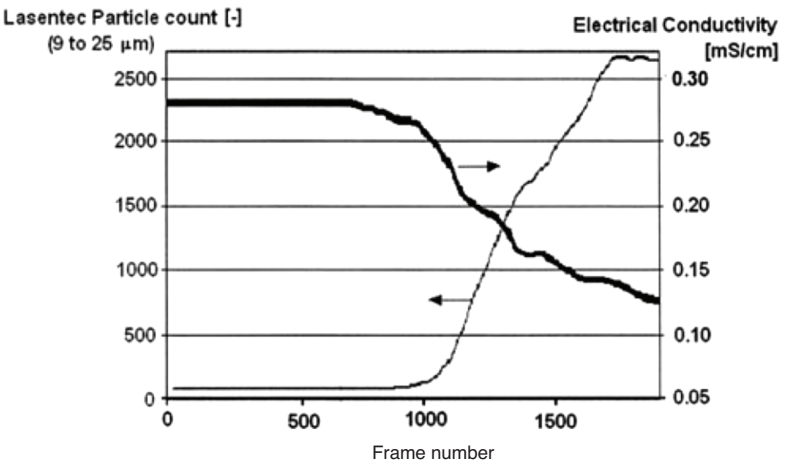

Figure 1—Chart plotting FBRM and ERT measurements during crystallization (Richard *et al*., 2005)[10]

– 0.1, 0.2, 0.4 mol/litre.

The reaction was scaled up, matching power per unit volume and addition time. In each case the vessel geometries were matched (vessel with 4 baffles). (Figure 2).

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

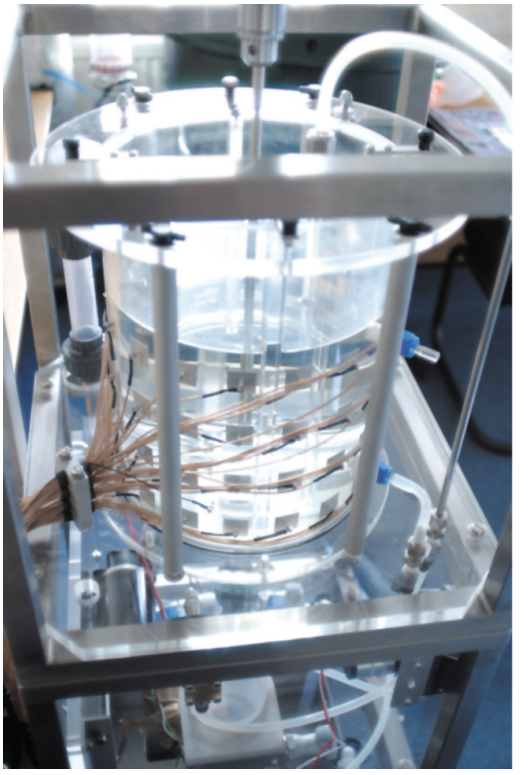

Figure 2—Seven-litre experimental ERT reactor

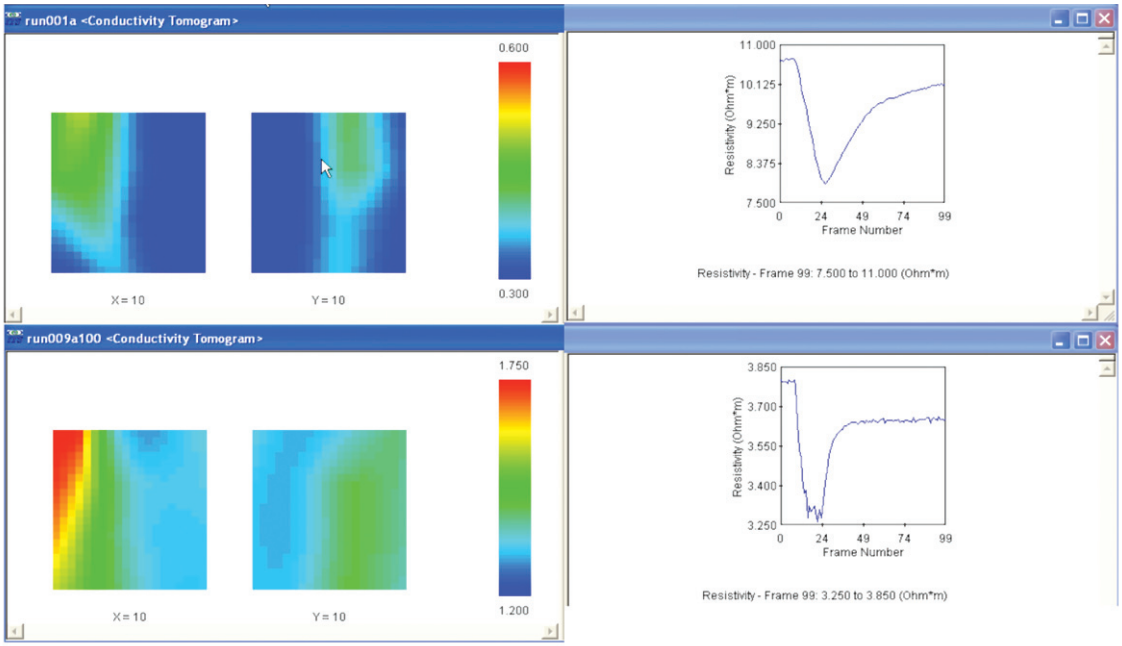

Figure 3—Slow (top) and rapid (lower) mixing conditions for small scale

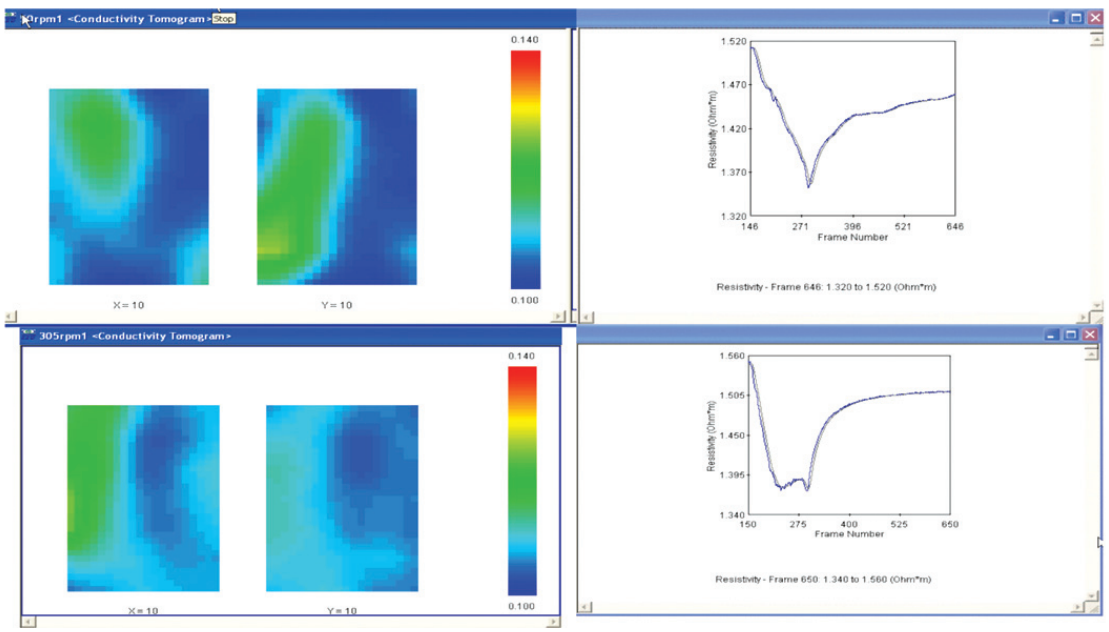

Figure 4—Slow (top) and rapid (lower) mixing conditions for large scale

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

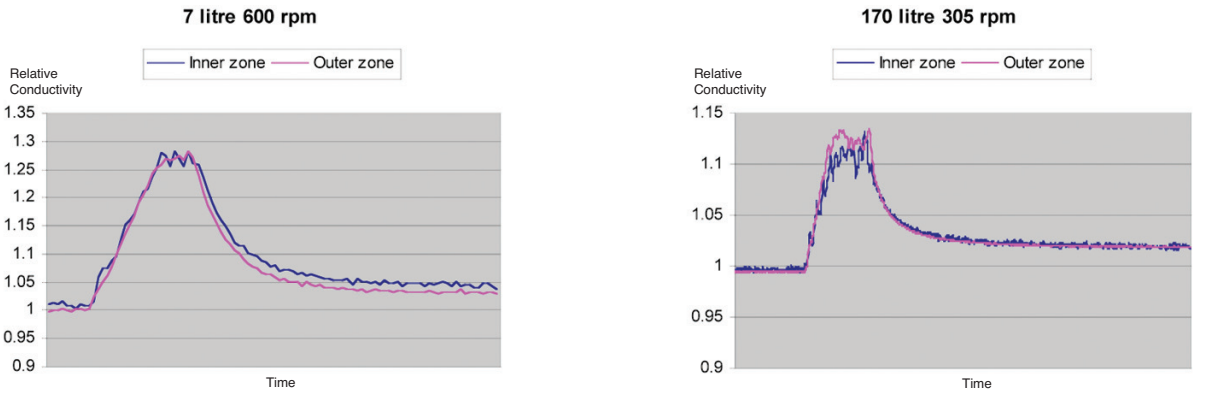

Figure 5—Relative conductivity for inner and outer region for rapid mixing conditions

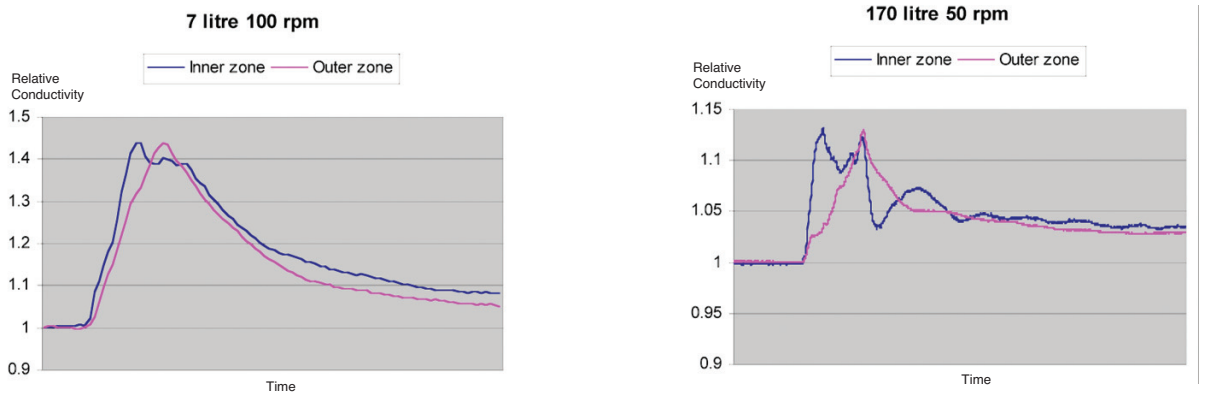

Figure 6—Relative conductivity for inner and outer regions for slow mixing conditions

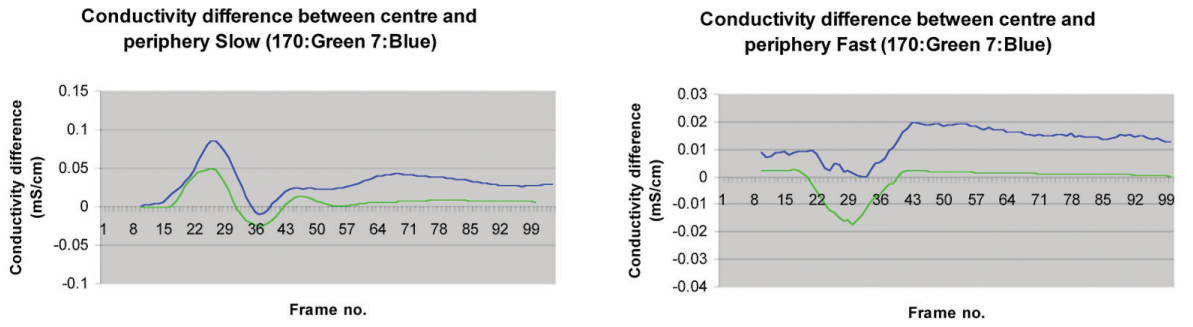

Figure 7—Conductivity difference (mS/cm) between centre and circumferential regions for rapid (left) and slow (right) mixing conditions

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 aa 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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