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Interfacing of EIT into an Industrial Pressure Filter
A Practical Example

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Abstract - *Pressure filtration is a generic manufacturing stage carried out across the process industry however unit efficiency and product quality are diminished by the lack of qualitative, real-time, monitoring techniques. This paper opens by outlining the control issues associated with pressure filtration and introduces electrical impedance tomography (EIT) as a potential solution. To verify the feasibility of this approach an initial investigation was carried out on a commercial 1.5m^3 pressure filter. A description is given of the engineering challenges which were met and the areas which were compromised, in lieu of future development. Specific reference is made to operation within metal walled vessels, hazardous area implications, novel sensor array and interconnection design to facilitate retrofitting, measurement within an electrically noisy environment, materials of construction, multi-modal operation and the characteristic requirements on speed, spatial resolution and signal sensitivity dictated by the batch manufacturing industry. Selected pertinent images are presented and their ramifications for control are highlighted. As pressure filtration is a subset of the much larger field of batch processing, the paper concludes with an overview of the anticipated lateral applications which may be addressed with an industrially compliant instrument*

Keywords - Process Tomography, Pressure Filtration, Electrical Impedance

1. INTRODUCTION

In late 1996 the UK government's Office of Science and Trade (OST) sanctioned support for the three year Foresight Process Tomography partnership. As one of the end-user companies within the consortium, Zeneca Ltd was charged with identifying suitable demonstrator applications and providing support to the academic members to develop the tomographic techniques from the laboratory environment towards applications of benefit to industry. In selecting the initial demonstration platform, the following criteria were employed;

- The measurement must be unavailable through existing technology
- It must be of commercial benefit, i.e. improves product quality, debottlenecks a process, assists in reducing environmental impact or facilitates safe plant operation
- It should be generically applicable across the process industry
- There must be real examples within the company
- There has to be a realistic chance of success within the duration of the project

An initial review created a series of applications of which two were selected for progression;

- Pressure filtration control
- Batch reactor monitoring

The pressure filtration control was seen as a subset of batch reactor monitoring, the latter encompassing such issues as multi-phase level control, in-vessel interface detection, indication of crystal nucleation and inferential analysis of reaction progress. The rationale was to address pressure filtration first as the engineering issues, and hence learning, associated with interfacing into such a unit would be similar to that for other plant items. In addition the application satisfies all the above criteria whilst being a relatively straightforward problem for two-dimensional qualitative process imaging.

2. THE REQUIREMENT FOR PROCESS TOMOGRAPHY WITHIN PRESSURE FILTRATION

The purpose of pressure filtration is to efficiently separate a solid fraction from a liquid phase. The solid may be generated within the filter, for example through crystallisation, or transferred as a slurry from an up-stream process.

With reference to figure 1, the liquid/solid combination is typically mixed within the filter by means of an agitator, this can both rotate and move axially within the vessel. Once a homogeneous slurry has been achieved the agitator is raised out from the liquor and the unit sealed and pressurised. The bottom run off valve (BRO) is then opened so that the liquid phase and small particulates (mother liquor) may pass through the filter mesh (cloth) and on for further processing or disposal. The point at which the mother liquor level drops so that the underlying solid may be observed is refereed to as detection of ‘dry land’. The solid phase is further dried and retained on top of the cloth to form a ‘filter cake’. The cloth tends to be formed from a fine metal or plastic mesh, which is reinforced by being stretched over a thick, often metallic, plate with a coarse series of holes drilled through it.

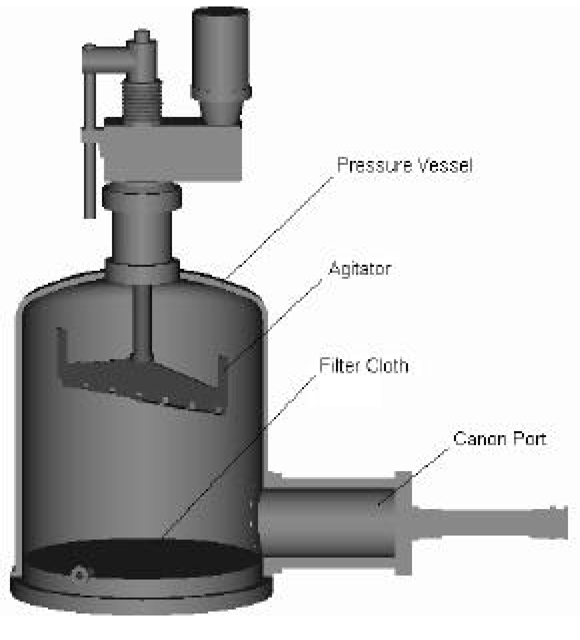

Figure 1: Typical Pressure Filter Layout

Pressure filtration alone may not remove all the mother liquor from the cake so further processing can be carried out via displacement of the original solvent with a ‘wash liquor’, which is evenly distributed over the cake through a spray bar in the top of the vessel, or re-dissolving the cake in a second solvent and filtering again. These clean-up stages may iterated several times for one batch.

On creation of the final cake, a large orifice is opened in the vessel wall (canon port) whilst the agitator carries out its second duty of removing the solid from the filter. This is achieved by slowly lowering the rotating blades into the surface of the cake so that it swept out of the unit through the canon port. To facilitate efficient removal of the solid the agitator blades are designed to operate in close tolerance with the filter walls.

Process Tomography may benefit this system both in terms of assisting process development and as a true on-line measurement. At present there are no process instruments available to give the following information;

- Real-time indication of end point of filtration
- Non-invasive imaging of imperfections in the filter cake
- Monitoring of solvent displacement of mother liquor

The lack of this information has direct impact on unit throughput as filtration times tend to be extended to accommodate the worst case conditions. In addition, non-homogeneous filtration will not be identified until the subsequent processing stage, or later, resulting in lengthy reworking of the batch. To compound this, any wash solvent usage may also be in excess as the efficiency of displacement is unknown, resulting in excess solvent costs and possible environmental impact when attempting to dispose of this unused material.

As pressure filtration is a common processing stage across the chemical sector, many attempts have been made in the past to automate measurement of the above parameters. For example through flow monitoring from the BRO valve or non-contact level detection within the vessel. Such approaches suffer from an inability to identify poor filtration when an inhomogeneous cake is formed or give an indication of the level of liquor remaining within the cake body. Similarly, manual methods of measuring filtration progress are limited to observation through the vessel sight-glass as sampling the cake would cause unacceptable imperfections to be created whilst being physically difficult to carry out within a pressurised unit.

Process tomography may be applied to resolve these issues by generating a two dimensional real-time image of the internals of the cake as it forms and is then washed. These images need not be quantitative as it is the change in solvent composition, and the mapping of this across the cake, which is of greatest importance. This information may be used in closed loop control of a production unit to optimise throughput and solvent usage and could create a development tool for visualising the effects of altering the process variables [1,2], e.g. programmed pressurisation of the vessel.

3. THE APPLICATION OF EIT TO PRESSURE FILTRATION

By the nature of filtration, the chemical composition of the bulk will change from one which is rich in solvent to one which is lean. Depending on the product nature this may be an organic or aqueous solvent being drawn off from an organic or in-organic solid. Given these matrix conditions, and the need to make measurements within a pressurised opaque vessel at relatively low-cost, a combination of electrical resistance (ERT) and capacitance tomography (ECT) was selected as the most appropriate sensing technique.

Though considerable experience has been gained within academia as to the application of ERT and ECT [3] this has been focused at such areas as multi-phase flow within pipelines and mixing within vessels, where the speed of response is at a premium. The industrial pressure filtration applications raise a new series of issues but also the flexibility to make some compromises on the earlier specification;

3.1 Electrical Sensitivity

To be of practical value the tomographic system must be capable of measuring across the wide range of materials that may be subjected to pressure filtration. This will span from purely organic matrices having low electrical conductivity through to fully dissociated ionic salts with low resistivity. However, in almost every case the addition or removal of a liquid from the solid cake will be associated with a perturbation in dialectric constant or resistance within the bulk. The resulting change in gain and phase angle may be subtle and frequency dependant , therefore, to optimise detection ideally a multi-modal instrument (ECT & ERT) would be required capable of operating at selected frequencies.

3.2 Vessel Aspect Ratio

Traditionally ERT measurements has been favoured over ECT when imaging within large diameter process units as the point electrodes required can be readily incorporated within an array mounted around the vessel wall. For mixing studies this does not create a problem as the matrix may be chosen to be conductive, e.g. brine water combinations. ECT dictates that the electrodes should be large with respect to the path length so as to increase the capacitance of the bulk to a level where it can be discriminated from the stray capacitances and electrical noise. As highlighted above, for pressure filtration the ECT mode may be required to operate in parallel with ERT, this dictates that a compromise in electrode size be found and a highly sensitive impedance measurement is employed.

3.3 Speed of Response

Applying tomography to pressure filtration, or most batch reactions, removes the necessity for the fast response times normally associated with process imaging. Filtration may occur over tens of minutes or several hours hence an image once every 30 seconds is adequate. This slow response has wide ranging implications for the instrument design, for example, signal averaging techniques may be employed to improve the electrical sensitivity and high precision but relatively slow commercial four-wire impedance spectrometers may be utilised to give the impedance information.

3.4 Sensor Array Design

If an electrode array is to be retrofitted within an existing filter a series of engineering challenges arise. The array must not protrude into the vessel as it will interfere with the action of the agitator sweeping out the dried cake, similarly any cabling must also be flush against the unit's wall. In addition the integrity of the vessel's pressure rating cannot be compromised by drilling into the wall either for fastening the electrodes or allowing cable entry. Finally the array must be at least as robust as the other components elsewhere within the filter. Some of these constraints may be relaxed if the electrodes were to be incorporated within a new custom filter, but such an approach would severely limit the uptake of the technology.

Because of the need to measure the capacitance element of the impedance signal, stray capacitance within the interconnecting cables must be minimised. Due to the size of a typical pressure filter this cable length may be several meters long. Therefore careful design of the transmission line shielding is required.

3.5 Materials of Construction

In terms of corrosion and erosion resistance the materials of construction for the wetted electrode array should be identical to that of the remainder of the vessel. However, the electrochemical half cell created on using some of the more common metals would be unacceptable both in terms of corruption of the measured signal and contamination of the chemical matrix through corrosion of the electrodes. A compromise must therefore be sought through selection of passive metals and investigation of the limiting voltages that may be applied for a particular chemical system, for example through cyclic voltammetry [4].

3.6 Metal Walled Vessel

The majority of industrial pressure filters, and reactors, contain metallic components either lined or exposed directly to the process. This creates a preferential path for any electrical signal to bypass the bulk, however, provided a discernible signal is transmitted through the process matrix then EIT may still be utilised.

3.7 Spatial Resolution

For the most part the spatial resolution of the system need not be high provided the electrical sensitivity within a 'pixel' is of a discernible level with respect to the loss of mother liquor during filtration. A correctly filtered cake should be approximately homogeneous, the tomographic system should be capable of detecting the occurrence of any imperfections during filtration however imaging the exact size and nature of these is less significant as the information would be used to prompt further off-line inspection of the filter components and methodology.

3.8 Hazardous Area Operation

To be of wide applicability across the process industry, any tomographic system must be compatible with the requirements for electrical certification in flammable atmospheres [5]. By the nature of the processes operated in the chemical sector there will often be flammable solvents operated within oxygen rich environments, pressure filtration is no exception to this. This will often dictate that the area inside the filter is designated, in Europe as, zone 0 and as both resistance and capacitance modes of impedance measurement will be required for the successful operation of the instrument this further forces the electrode array to be located within this environment. Under European law [6] the only method of protection which is adequate for this duty would be intrinsic safety (IS). Fortunately, the power associated with EIT measurements falls within that of IS and the nature of the electrode array should bring it under the jurisdiction of 'simple apparatus' allowing proprietary current clamping technology to be located within the instrument drive electronics. The latter may be located adjacent to the vessel and protected by a techniques such as a flame-proof enclosure, as this area will often be either zone 1 or 2.

5. PRACTICAL DEMONSTRATION ON A 1.5m^3 PRESSURE FILTER

5.1 Overview of Experimental Method

To gain confidence in the suitability of EIT when applied to an industrial scale vessel, it was decided to directly transfer the ERT system from the UMIST laboratory to a 1.5m^3 pressure filter at the Zeneca Huddersfield Engineering Labs (figure 2) under contrived conditions, i.e.;

- Over saturated brine used to create the filter cake giving a highly electrically conductive matrix
- Plastic lined vessel employed to reduce signal losses to the vessel walls
- Equipment operated under artificial conditions to overcome hazardous area certification

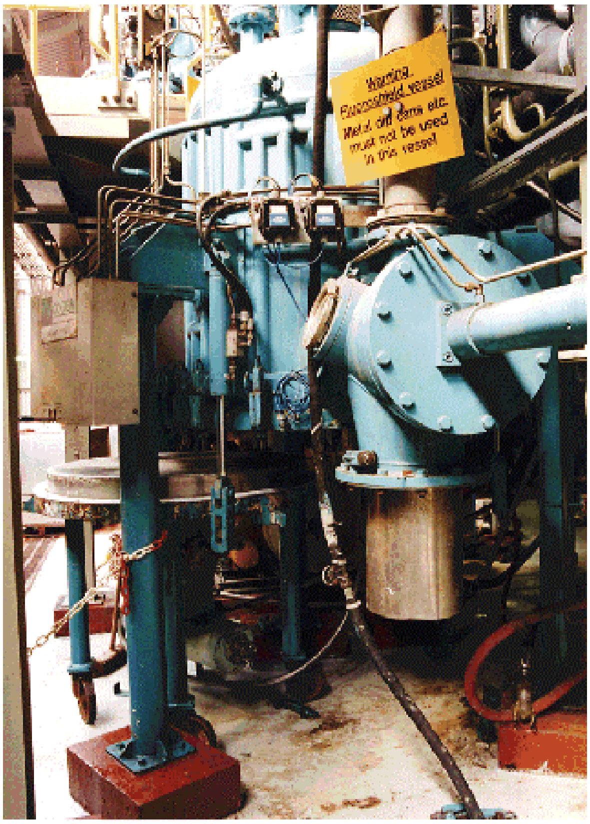

Figure 2: Demonstrator Filter Used in Study

As this was to be a temporary installation within an existing production unit, one of the principal concerns was to create an electrode array which could be mounted in the vessel without disturbing the pressure integrity or operation of the unit. The latter being particularly taxing as the array could not protrude further than a few millimetres into the vessel due to the action of agitator. To accomplish this a method of fabricating the array was devised by transferring techniques from the printed circuit board (PCB) industry. By incorporating the electrodes within a

3400mm long by 60mm wide flexible printed circuit board, a very thin array (<1mm) was created consisting of 16 gold plates (100mm x 40mm) in a single plane along with all the associated cabling which was terminated at a single multicore connector. This device was then temporarily mounted, with adhesive, to create a single plane of electrodes 100mm above the level of the filter cloth (figure 3) with a gap in the electrode ring at the point where the canon port enters the vessel. Fortuitously, the demonstrator vessel had a branch in the wall just above the filter cloth providing a route for cable entry and exit.

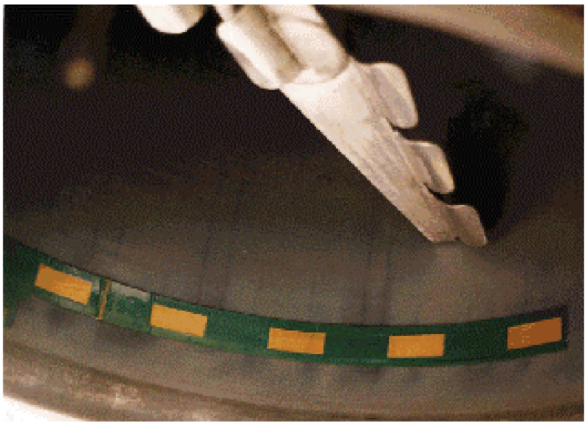

Figure 3: Internal View of Filter Showing Electrode PCB

The experimental programme consisted of creating an oversaturated brine slurry such that on filtration a cake of approximately 300mm depth would be created. This was filtered under a pressure of 1 barg nitrogen for approximately 2 hours. After drying the cake was then rewetted with brine at saturation concentration prior to re-filtration. This process was iterated several times whilst being monitored with the ERT system.

5.2 Results

One of the major problems with validating an experimental programme within a pressure filter is in attempting to relate the features seen on the tomographic images with that in the cake, as to open the vessel will disturb the system being monitored. A method of resolving this issue was devised by taking the dry filtered cake and re-wetting it with saturated brine through a single entry point, rather than the spray bar, at the top of the vessel. In this way a crater was created in the cake which could be clearly identified through the sight glass in the top of the vessel and on the tomographic images.

With reference to the following images, figures 4 to 5 show a dry filter cake over an 80 minute period after it has been re-wetted with brine. The lighter features around the periphery indicate the highly conductive brine soaking into the dryer, less conductive, cake.

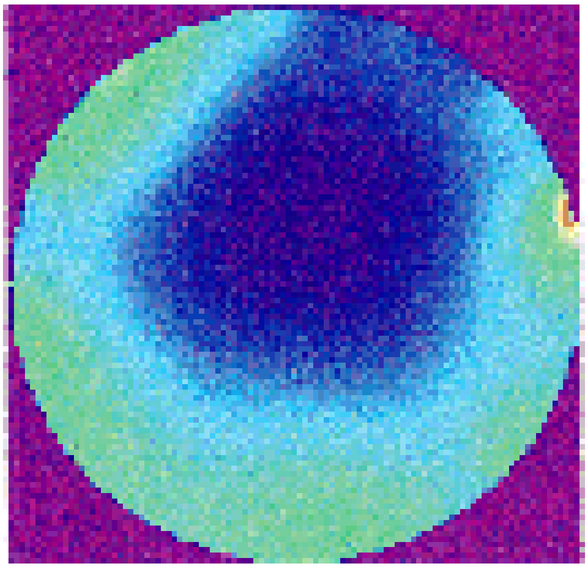

Figure 4: Filter Cake Tomographic Image at T=6 min

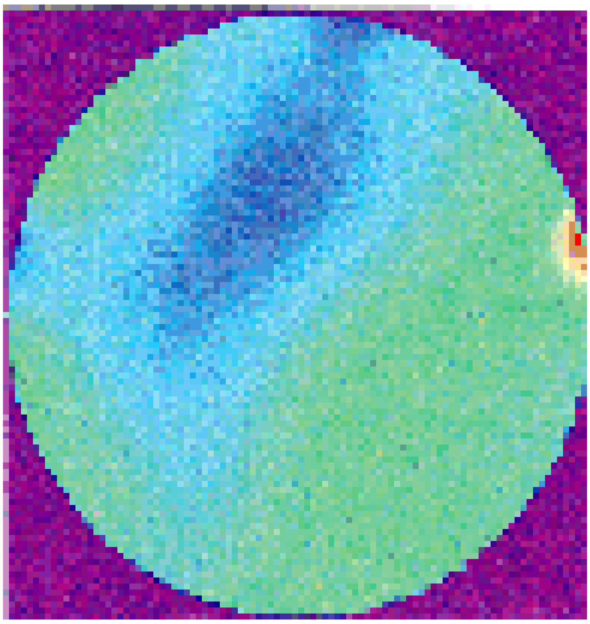

Figure 5: Filter Cake Tomographic Image at T=86 min

Figures 6 and 7 cover the next 4 minute period where the free brine has found a path of lower flow resistance through the cake at the point where the crater was formed. This area acts as a route for most of the remaining liquid to leave the solid forming the bright, conductive, spot in photo 8 surrounded by the dryer, less conductive, cake.

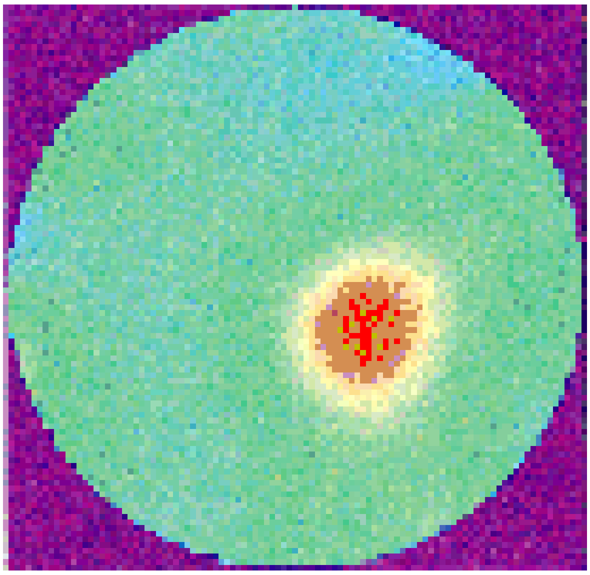

Figure 6: Filter Cake Tomographic Image at T=88 min

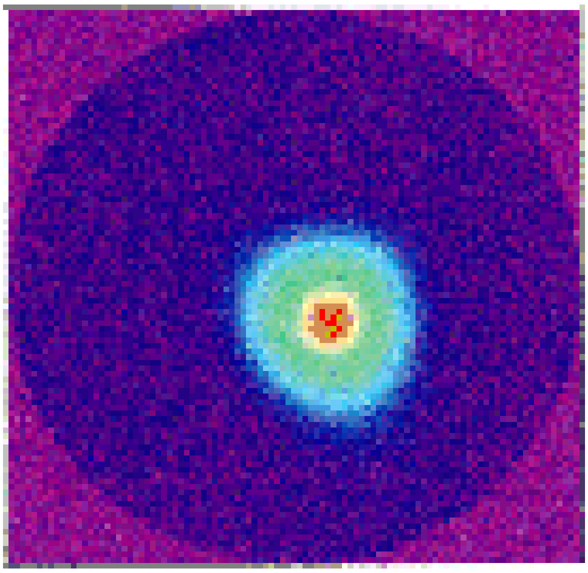

Figure 7: Filter Cake Tomographic Image at T=90 min

5.3 Review of Results

These results show that qualitative images can be generated throughout the filtration process on an industrial scale vessel and it is possible to identify inhomogeneities within the cake using a 16 electrode array mounted in a single plane around the periphery of the vessel. This study provides evidence that vessel based EIT is possible within an electrically noisy environment but raises many challenges with respect to understanding and over-coming the compromises accepted during these experiments.

6. FUTURE DEVELOPMENT FOCUS

6.1 Review of Instrument Specification

The experimental programme described above indicate that for batch processing the emphasis for the tomographic system is not so much on speed of response but on electrical sensitivity. This opens up the opportunity to apply a commercial precision impedance analysers as the measurement element and then to interface this through a custom switch matrix to the electrode array. In this way, a very precise and sensitive measurement of impedance may be achieved using readily available instrumentation, this may be several orders of magnitude above that previously available from dedicated ERT or ECT instruments.

6.2 Electrochemical Analysis

Imaging organic or aqueous slurries throughout the filtration process dictates that a multimodal approach be applied so as to sensitively monitor the bulk which may be changing from a predominantly resistive to a capacitive matrix or vice versa. To achieve this sensitively without corrupting the process stream an electrochemical model of the system is highly beneficial. This allows the optimum operating frequency, or frequencies, to be determined and the limiting drive potentials to be defined for a particular electrode material and chemical matrix combination. An understanding of the latter allows the sensitivity of the measurement system to be optimised and prevent corruption of the process stream, by release or take up of ions at the electrode interface.

6.3 Laboratory Simulation of Filtration

Given the difficulty in manipulating the cake formation within a closed pressure filter, a semi-tech scale open ambient pressure filter offers the opportunity to simulate the process in a controlled manner and readily modify the electrode array configuration.

7. CONCLUSIONS

This paper demonstrates that two-dimensional electrical impedance tomography may be applied on a large scale vessel, under limited conditions, employing a novel printed circuit based electrode array. The benefits and concerns with moving this technology into the industrial environment have been highlighted and a new instrument approach has been introduced to overcome these constraints. Once these engineering issues have been addressed for pressure filtration it is anticipated that transfer of this technology into other batch processing applications should be relatively straightforward. Some potential future imaging areas include; onset of crystallisation detection, distillation column control, multiphase level monitoring, bulk property analysis and batch reaction progress.

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