UNIVERSITY OF BIRMINGHAM

University of Birmingham Research at Birmingham

Measuring gas hold-up in gas-liquid/gas-solid-liquid stirred tanks with an electrical resistance tomography linear probe

Forte, Giuseppe; Alberini, Federico; Simmons, Mark J. H.; Stitt, E. Hugh

DOI:

10.1002/aic.16586

License:

Other (please specify with Rights Statement)

Document Version Peer reviewed version

Citation for published version (Harvard):

Forte, G, Alberini, F, Simmons, MJH & Stitt, EH 2019, 'Measuring gas hold-up in gas-liquid/gas-solid-liquid stirred tanks with an electrical resistance tomography linear probe', *AIChE Journal*, vol. 65, no. 6, e16586. https://doi.org/10.1002/aic.16586

Link to publication on Research at Birmingham portal

Publisher Rights Statement:

Checked for eligibility: 19/03/2019

This is the peer reviewed version of the following article: Forte, G, Alberini, F, Simmons, MJH, Stitt, EH. Measuring gas holdup in gas—liquid/gas—solid—liquid stirred tanks with an electrical resistance tomography linear probe. AlChE J. 2019;e16586., which has been published in final form at: https://doi.org/10.1002/aic.16586. This article may be used for non-commercial purposes in accordance with Wiley Terms and Conditions for Use of Self-Archived Versions.

General rights

Unless a licence is specified above, all rights (including copyright and moral rights) in this document are retained by the authors and/or the copyright holders. The express permission of the copyright holder must be obtained for any use of this material other than for purposes permitted by law.

- Users may freely distribute the URL that is used to identify this publication.
- Users may download and/or print one copy of the publication from the University of Birmingham research portal for the purpose of private study or non-commercial research.
- User may use extracts from the document in line with the concept of 'fair dealing' under the Copyright, Designs and Patents Act 1988 (?)
- Users may not further distribute the material nor use it for the purposes of commercial gain.

Where a licence is displayed above, please note the terms and conditions of the licence govern your use of this document.

When citing, please reference the published version.

Take down policy

While the University of Birmingham exercises care and attention in making items available there are rare occasions when an item has been uploaded in error or has been deemed to be commercially or otherwise sensitive.

If you believe that this is the case for this document, please contact UBIRA@lists.bham.ac.uk providing details and we will remove access to the work immediately and investigate.

Download date: 14. Jun. 2020

Measuring Gas Hold-up in Gas-Liquid/Gas-Solid-

Liquid Stirred Tanks with an Electrical Resistance

Tomography Linear Probe

G. Forte^{1, 2*}, F. Alberini², M.J.H. Simmons² & E.H. Stitt¹

¹School of Chemical Engineering, University of Birmingham, Edgbaston Campus, B152TT, Birmingham, UK.

² Johnson Matthey Technology Centre, TS23 4LB, Billingham, UK

ABSTRACT

An ERT linear probe was used to measure gas hold-up in a two-phase (gas-liquid) and three phase (gas-solid-liquid) stirred tank system equipped with a Rushton turbine. The ERT linear probe was chosen rather than the more commonly used ring cage geometry to achieve higher resolution in the axial direction as well as its potential for use on manufacturing plant. Gas phase distribution was measured as a function of flow regime by varying both impeller speed and gas flow rate. Global and local gas hold-up values were calculated using ERT data by applying Maxwell's equation for conduction through heterogeneous media. The results were compared with correlations, hard-field tomography data and CFD simulations available in the literature, showing good agreement. This study thus demonstrates the capability of ERT using a linear probe to offer, besides qualitative tomographic images, reliable quantitative data regarding phase distribution in gas-liquid systems.

Topical Heading Transport Phenomena and Fluid Mechanics

Key words Gas-liquid, Gas-solid-liquid, Electrical Resistance Tomography, gas hold-

up distribution, in situ measurement

Introduction

Gas-liquid stirred tanks are widely used in biochemical and chemical manufacture, driven by the process requirements for bulk mixing and interphase contact. As a consequence, substantial work has been conducted in the past few decades aimed towards the understanding of two-phase turbulent mixing in these units^{1, 2}. Studies on overall gas hold-up and gas dispersion have been carried out both experimentally and using computational simulations³.

A significant challenge remains in the application of diagnostic methods and sensors which can provide local information on mixing performance, flow regime and phase distribution at process scale, either for troubleshooting or as a means of controlling process or product attributes during production. The most common methods are limited to either transparent media or global observations. For example, bulk gas volume fraction in a stirred tank is commonly obtained by determination of the increase in liquid height given by the presence of the gas with respect to the non-gassed condition either visually⁴⁻⁶ or using level probes⁷. Applied techniques for local parameter investigations (velocity fields, local bubble size and interphase contact) and for overall gas content remain the tools of the industrial or academic researcher on the grounds of practicality or cost, e.g. Computer Automated Particle tracking (CARPT)^{8,9}, ultrasonics¹⁰⁻¹³, Particle Image Velocimetry (PIV)^{14,15} Positron Emission Particle Tracking (PEPT)¹⁶ and Laser Doppler Anemometry (LDA)¹⁷.

Tomographic methods, however, have some considerable potential. Whilst X-ray¹⁸ and γ-ray¹⁹ tomography and Magnetic Resonance Imaging²⁰ have been used successfully to obtain local values in opaque media, the need for high energy radiation, and thus significant health and safety precautions, often represent a practical limit for these techniques on plant. An alternative is the use of electrical tomography^{12, 21-23}, though image reconstruction is more complex and error prone since the methods are soft field rather than hard field²⁴ and the applicability of the method is dependent upon the conductivity of the phases present. Electrical Resistance Tomography (ERT), which is applicable for a conductive continuous phase, has been applied for mixing studies for single phase systems for a little under twenty years^{23,25}, at first using a cylindrical 8 plane electrode cage each containing 16 electrodes. Subsequently, this technique has been employed on a qualitative and quantitative basis for gas-liquid systems in bubble columns²⁶, stirred tank reactors^{21, 22, 27}, for immiscible liquid-liquid (L-L) systems²⁸, and to assess homogeneity in solid-liquid suspensions^{29,30}. Data obtained from multiplane sensors are commonly linearly interpolated to reconstruct volumetric conductivity and therefore phase distribution. Some attempts have been done in resolving three phase systems using ERT in combination with other techniques. For example, circular array ERT was used in combination with a fibre optic probe to investigate gas-liquid-solid circulating fluidized bed by Razzak³¹.

Although circular array ERT gives useful information³², the issues related to plant installations and re-engineering of existing reactors make this sensor design essentially suitable only for pilot and laboratory scale studies. Thus, alternative sensor designs which are sufficiently robust and are easy to engineer within the process environment have been sought; such a design is a linear array, or probe. The first works reporting the use of the linear arrangement of electrodes for ERT were by Richardson³³ and Bolton³⁴ for monitoring solids distribution in a conductive liquid

phase. Despite employing the same measurement strategy of the circular design, the linear configuration allows acquisition of data with higher resolution in the axial direction, therefore giving information of axial homogeneity and distribution, useful in multiphase applications. It is reasonable to state that in industrial application (e.g. for evaluating solids deposition or local gas hold-up), for rotationally symmetrical tanks, axial mixing efficiency, rather than the radial distribution, is the most important parameter in terms of quality control. Although potentially applicable on larger scale and for in situ monitoring, this configuration has received little attention in the literature after Bolton's³⁴ work. Ricard et al.³⁰, for example, applied a linear sensor in combination with CFD modelling to investigate L-L dispersion and solids suspension in stirred tanks.

This paper describes the application of ERT using a linear probe for quantitative measurement of gas and liquid phase distribution and overall hold-up in a stirred tank reactor equipped with a Rushton turbine operating in the turbulent flow regime. The capability of the linear probe to discriminate the gas and liquid phases, as a function of gas-liquid flow pattern is assessed. These data are analysed using Maxwell's equation, already successfully used for investigating bioreactors^{35, 36} for similar gas fraction using an ERT cage sensor, to determine global and local gas hold-up.

For validation purposes, the two-phase results were compared with results for similar systems from the literature using hard-field techniques and computational methods. Ford et al. 18 used X-ray computed tomography (CT) to measure gas dispersion in a 0.21 m diameter stirred tank agitated with a Rushton turbine (Fr = 0.09-1.1, Fl = 0.03-0.08, with gas flow rate between 0.86 and 1.7 vvm). Experiments were carried out at three different regimes (flooding, loading and complete dispersion) and both gas hold-up maps and axial distribution plots were produced,

deriving local time-averaged gas hold-up in the axial direction at different velocities. The CT measurement domain covered one third of the tank height (with the impeller at the bottom of the field of view), focusing on the impeller zone where the gas hold-up was observed to be at its highest value in the loading and complete recirculation regimes. At the flooding condition, inefficient gas dispersion was observed with a flatter vertical profile characterising the axial gas volume fraction: the power input is insufficient to retain gas that flows through the impeller to the surface.

Khopkar et al.⁸, instead, have combined γ -ray CT measurements with CARPT and computational fluid dynamic studies (CFD) on a stirred tank with diameter of 0.20 m also equipped with a Rushton turbine. This study was carried out at low impeller speed (Fr = 0.0755) and at a volumetric gas flow rate of 0.4-0.8 vvm (Fl = 0.042-0.084), therefore with the system working in the flooding regime. The CT measurements showed higher hold-up in proximity of vessel wall with CFD capturing an inward movement of the gas in the rise through the tank, generating a secondary loop in the top half of the vessel. Both techniques captured the low level of sparging achieved in the flooding regime and the obtained axial gas hold-up profile does not exceed 5% v/v.

As an extension to the two-phase system study, the present work also considers the capability of the method to discriminate the presence of an additional dispersed solid phase as a means of providing a first step towards the translation of this technology to the operation and control of three-phase reactors.

Theory

Gas – liquid flow regimes and hold-up

Gas hold-up is one of the most important parameters for scale-up, performance evaluation and for model development, directly influencing the G/L interfacial area. Amongst others, Smith³⁷ identified three key parameters in such applications: the scale of vessel, the impeller shape and dimension and the volumetric gas flow rate. The main flow patterns, or regimes characterising gas-sparged stirred tanks (generally dependent upon tank geometry, gas flow rate, Q_g , and impeller speed, N) are defined as flooded, loaded, completely dispersed, and gas recirculation³⁸. In flooded condition ($0 < N < N_f$), minimum dispersion is achieved, and a plume of gas rises centrally close to the impeller. At higher impeller speeds when $N_f < N < N_{cd}$ the system is in the loaded regime: the gas is more radially distributed but does not occupy the entire volume of the tank due to the buoyant forces exceeding the radial drag force. In the completely dispersed regime ($N_{cd} < N < N_r$) the gas is well dispersed through the whole reactor at low power. Beyond N_r , the full gas recirculation regime is observed. The two dimensionless numbers used to characterise the operating regime are the Froude number ($Fr = N^2 D / g$) and the Flow number³⁹ ($Fl = Q_g / (ND^3)$) and based upon these parameters a characteristic regime chart⁴⁰ can be drawn to identify the operating regime.

Many empirical correlations have been published to estimate gas hold-up. Based on the parameters described above and the Reynolds number $\left(Re = \frac{ND^2\rho}{\mu}\right)$, Smith⁴¹ proposed a correlation for tanks with T > 0.44 m:

$$\varepsilon_g = 0.85 (\text{Re} \cdot Fr \cdot Fl)^{0.35} \left(\frac{D}{T}\right)^{1.25}$$
 (1)

Another common approach to predict gas fraction in agitated vessel refers to the power dissipated by the impeller:

$$\varepsilon_{\rm g} \propto \left(\frac{{\rm P_g}}{{
m V}}\right)^{\rm A} {\upsilon_{\rm s}}^{\rm B}$$
 (2)

The parameters A and B are system dependent but generally are within the range of 0.2-0.7 with a higher value for A in the case of non-coalescing systems compared with coalescing systems⁶.

For three-phase mixing, many studies have been carried out with focus on solids suspension and effect of gas presence on the Zwietering "just suspended" impeller speed⁴², N_{js} , defined as:

$$N_{is} = S v^{0.1} d_n^{0.2} (g \Delta \rho / \rho)^{0.45} X^{0.13} D^{-0.85}$$
(3)

It is reported that the presence of gas, unsurprisingly, affects suspending power of the impeller⁴³ and that in three-phase systems the solid suspension mechanism is mainly determined by the gas-liquid interaction hydrodynamics at the impeller blade⁴⁴. For this reason, it is usually advised that the $N_{js,g}$ in presence of gas is higher than N_{js} in the bi-phasic system⁴⁵. Studies conducted by different researchers have shown various different effects of solids presence on the gas holdup depending on relative density and solid concentration⁴⁶; Chapman⁴⁷ reports no significant influence on gas distribution in presence of solids for X lower than 20%.

ERT for gas hold-up

ERT has been applied to reconstruct the gas volume fraction over the height of the tank and to calculate overall hold-up. Maxwell's model given in Equation (4), was used to compute gas volume fraction \mathcal{E}_g within the tank⁴⁸:

$$\varepsilon_{g} = \frac{\left[1 - \left(\frac{\sigma_{m}}{\sigma_{c}}\right)\right]}{\left[1 + 0.5 \cdot \left(\frac{\sigma_{m}}{\sigma_{c}}\right)\right]} \tag{4}$$

Where σ_c is the conductivity of the continuous phase and σ_m the conductivity of the biphasic mixture. An alternative approach was proposed by Montante and Paglianti⁴⁹ and applied extensively by Jamshed⁵⁰, which used a dimensionless conductivity ε ($\varepsilon = 100(1-\sigma_m/\sigma_c)$) proportional to the gas hold-up to describe sparging performance of different impellers, however the authors indicate this parameter as proportional to the gas hold-up and not as an absolute value. Furthermore, in the investigated range of gas hold-up (approximately 1-15%), the simplification used by Montante [49] can be considered proportional (with a factor of approximately 0.7) to the Maxwell's equation. For this reason, Maxwell's equation is used in this work. Limitations to the application of this method could be encountered when approaching very high or very low (next to monophasic) hold-up.

Materials and Methods

Stirred tank configuration

The investigated system for two-phase and three-phase experiments was an agitated cylindrical Perspex tank equipped with a ring sparger at the bottom of the vessel. The tank, with diameter T, was equipped with four baffles with width B equal to T/10. The impeller used in this study was a stainless steel six blade Rushton Disc Turbine (RDT6) with diameter 0.056 m (D/T=2/5), additional information on system design is given in Table 1. The impeller was earthed to the tomographic acquisition system to reduce electro-magnetic disturbance on the imposed electric field. In order to cover all the electrodes within the vertical linear probe, the liquid level H was set to 0.21 m (H/T=3/2).

Nickel nitrate hexahydrate (99,99% Sigma Aldrich®) solution in demineralised water, having a conductivity of 2.7 mS cm⁻¹, was used as the liquid phase. Air was sparged into the tank using a metal ring sparger located at the bottom of the vessel concentric with the impeller shaft. The diameter of the sparger was 0.04 m; designed smaller than the impeller diameter in order to allow the rising bubbles to be caught in the radial flow induced by the impeller⁴⁰. The ring was equipped with 8 orifices of diameter $0.5 \cdot 10^{-4}$ m and connected to a compressed air source. The air flow rate was set within a range of $8.33 \times 10^{-5} - 16.7 \times 10^{-5}$ m³ s⁻¹ using an inline rotameter, corresponding to 1.5 and 3 vvm respectively. In Figure 1, the operating conditions are reported in a Fr-Fl chart redrawn from Middleton et al.⁵¹. The boundaries between the flow regimes are obtained by the equations reported in 38,52 .

Flow number was varied between 0.024 and 0.32, whilst the value of Fr was kept between 0.016 and 2.5. The Reynolds number was thus between 15,000 and 60,000 ensuring the system was maintained in the turbulent regime.

For the three-phase mixing experiment, stainless steel particles were used as dispersed phase $(p_s = 8000 \text{ kg m}^{-3} \text{ and particle size between } 0.177 \times 10^{-3} \text{ and } 0.420 \times 10^{-3} \text{ m})$ at concentrations of 3, 4 and 5 % w/w. Conductive particles have been chosen for having an opposite effect on conductivity compared to the gas. The "just suspended" impeller speed was calculated using the Zwietering correlation (3). The achievement of such state was optically verified with particles being present on the bottom of the tank for not longer than 2 seconds. Biphasic measurements (solid-liquid) were taken at different impeller speeds $(1/3 N_{js}, 2/3 N_{js} \text{ and } N_{js})$ for comparison with the three-phase measurements. The effect of gas on suspending potential of the system was taken into account correcting the N_{js} speed as suggested by Nienow⁴⁵, resulting in a insignificant increase in the investigated conditions (3 rpm at the most).

Measurement Configuration

The ERT apparatus used in the experiments is comprised of a linear probe and a four-channel p2+ Data Acquisition System (DAS) supplied by Industrial Tomography System (ITS Ltd., Manchester, UK [https://www.itoms.com/]). The linear probe was equipped with 18 electrodes arranged in a 140 mm long strip as shown in Figure 2(a, b). The stainless-steel electrodes are uniformly spaced 4 mm apart and have a width of 8 mm and a length of 4 mm. The bottom and the top electrodes do not actively take part in the measurement, instead they are earthed and act as guard electrodes. The polyethyl ether ketone (PEEK) support forming the body of the probe, isolates the coaxial cables that connect each electrode to the DAS, from the aqueous system.

The linear probe was placed behind one of the baffles (in the direction of impeller rotation), to minimise its influence on the flow field, as shown in Figure 2(c). The electrode array was placed so that it faces the interior of the vessel across a chord to avoid the noise caused by the stirrer. The latter was grounded to the DAS to avoid any potential difference between the mixing equipment and the tomographic apparatus. The portion of fluid covered by the ERT extends approximately 70 mm from the surface of the probe and the obtained output is a tomogram 10×20 pixels in size. The field of view of the probe extends from the baffle to the impeller zone, however the penetration is a function of the gas hold-up and will decrease dramatically in case of very high gas hold-up. In this work, where hold-up does not exceed 20%, the penetration is considered constant throughout the investigated cases, and equal to the minimum suggested by the manufacturer.

The excitation scheme is analogous to the traditional cage geometry: each pair of adjacent electrodes are used to input a current through the fluid, while the potential difference is measured for each pair of remaining neighbour electrodes along the sensor. The acquired data are processed using the ITS p2+ software that runs a Linear Back Projection (LBP) algorithm in order to reconstruct the conductivity distribution across the field of view of the probe. The chosen algorithm guarantees a fast response and has been previously applied in similar studies for gas liquid^{49,54} and solid-liquid⁵⁵. The intensity of the injected current was 30 mA for all the experiments and the frequency was kept at 9600 Hz and 60 frames were taken at each condition.

The probe was specifically selected to evaluate axial conductivity in the stirred tank and to guarantee a certain flexibility in installation. Unlike the circular cage, its geometry causes the covered portion of space to not having the same electric field density: the periphery of the probe (top and bottom) has a lower density of measurement than the central region. The effect of this

is that the accuracy of the projected image is not uniform, with the top and bottom corners away from the probe reported as having poor reliability⁵⁶. Previous investigations⁵⁷ suggest the periphery of the tomogram is characterised by low reliability due to the linear geometry of the sensor, therefore in the conducted data processing, the tomograms are reduced to a 8×16 pixel grid. Specifically, the top and bottom two rows and the last two columns of pixel are removed from the analysis.

The tomograms are commonly displayed using conductivity scale, showing zones with higher or lower conductivity. To facilitate reading of gas volume fraction, the tomograms were converted into local gas hold-up tomograms, by applying Maxwell's equation (4) to each pixel, taking a reference values obtained from monophasic runs. Figure 3, in which tomograms are represented using a smoothing graphical tool, gives an example of a tomogram converted from conductivity to gas hold-up map.

Results and Discussion

Two phase Gas-Liquid Mixing

Global gas hold-up

To evaluate the impact of gas presence on the average conductivity across the tank, a monophasic test was run with gas feed valve shut and the impeller speed was varied between 0 and 1300 rpm. In Figure 4, a flat trend is visible up to 1000 rpm, after which the average conductivity moves towards lower values because some air is entrained at the surface. In the same figure, conductivity values with standard deviation error bars are shown for the bi-phasic system at the two different gassing rates used.

The higher the feed rate, the lower the detected average conductivity, indicating an increasing gas hold-up. The same trend is observed when the impeller speed is increased. The obtained values for conductivity are converted into gas hold-up data applying Maxwell's Equation (4). In Equation (4), the used value for conductivity for mixture and continuous phases are taken at corresponding impeller speeds. Figure 5, plots interpreted global gas phase fraction against the product of the Froude number, Fr, the gas flow number, Fl, and impeller Reynolds number, Re, according to Equation (1).

In Figure 5, as proposed by Smith [41], the calculated values can be approximated by a straight line. The calculated gradient for this experimental dataset is 1.8, different from the value of 0.85 derived by Smith. Nonetheless, the work reported by Smith was on a larger system (T > 0.41), and it can be reasonably argued that the size of tank may affect the proportionality constant as well as the different properties of the used aqueous solution. More importantly, it should be noted that the holdup data derived from ERT measurements correspond well to Smith's linear hypothesis, leading to the conclusion that the observed trend well represents the evolution of gas retained in the liquid bulk.

Local gas hold-up

The linear probe geometry allows the use of ERT to visualise in real time (measurement frequency up to 50 s⁻¹) the presence of gas in the tank and to assess quantitatively the homogeneity of distribution within the tank. Figure 6 shows a comparison between tomograms and images taken by a camera for the different flow regimes obtained for the lower gas feed rate (1.5 vvm).

The effect of increasing the impeller speed, as expected, appears to cause the total hold-up to shift towards higher values. In particular, the impeller zone seems to be most influenced. Similar effects can be seen in Figure 7, reporting pictures and gas hold-up tomograms for the different regimes but with a gas feed rate of 3 vvm.

A direct visual comparison can be drawn between the two figures. As would be expected, increasing the air flow rate causes an increase of gas hold-up, although in both cases the distribution has a similar profile, with the gas volume fraction decreasing towards the top of the tomogram (and thus the tank).

In order to better visualize the variation of the gas fraction over the height of the vessel, an axial gas hold-up plot can be obtained, shown in Figure 8, by averaging the pixel values along each horizontal row in the tomogram.

Figure 8 emphasizes observations made above, that similar trends are obtained for 1.5 and 3 vvm, with the latter having significant higher gas retention. The profile is relatively flat at low impeller speeds (in flooding condition), with the impeller having little effect on gas dispersion: the gas flows along the shaft, with a slightly higher hold-up towards the top of the tank. At loading, the profile gains more inflection, with hold-up increasing in the impeller region. It can be observed that the higher the value of impeller speed, the higher is the gas concentration around the impeller. This parabolic profile in the impeller area is due to the gas capture caused by the impeller motion⁵⁸. The increase in impeller speed has relatively less effect on dispersion towards the top of the tank and even at complete dispersion regime gas hold-up does not exceed 5% and 8% at the top of the tank, for the lower and higher gas rates respectively.

Comparison with literature

Axial gas hold-up profiles extracted from Ford's X-ray¹⁸ and Khopkar's⁸ γ -ray tomography and CFD are compared with data obtained using ERT at similar regimes, in Figure 9. Given the difference in set up of the different studies as well as hard rather than soft field measurement methods, this comparison has the objective of validating the capability of ERT to detect meaningful gas hold-up profiles and at the same time, to analyse its limitation in accurately representing axial variations in gas distribution.

CT and CFD data are compared with ERT at flooding condition (Figure 9(a)). As expected at this regime, the axial profile is rather flat, with air flowing axially through the impeller and along the shaft; sometimes also referred to descriptively as "bubble column mode". Data from the three studies agree with the theoretical expectation and describe similar profiles. Gas hold-up does not exceed a value of 4% and its maximum can be located approximately at half way up the tank, with little or no axial variance.

Increasing the impeller speed, moving to the loading condition, the gas now locates mainly around the impeller where is "thrown" radially by the impeller. Visually, bubble size decreases and the gas covers a higher portion of the tank, as was shown in the corresponding tomograms reported in Figure 6 (b, c) and 7 (b, c). This condition is picked up in the axial plots (Figure 9(b)) where both data from the literature and ERT show an overall increase in hold-up compared to the flooding condition, together with an increase in axial variance. Indeed, the impeller region is characterised by a significantly higher hold-up compared to the rest of the tank. It is possible to notice how the profile obtained by CT measurements quickly decreases tending to a constant value as exiting the impeller region; differently ERT shows a smoother trend with no sudden

changes in the axial profile. Nonetheless, all studies are in agreement, indicating a significantly higher gas hold up in the impeller region.

A similar trend is observed for the complete dispersion regime. In this regime the optical images (Figure 6 (d, e) and 7 (d, e)) show decreased bubble size with particularly high concentration in the impeller zone. Although the ERT technique does not allow observation of the recirculation loops in the tank, it is able to capture the high gas fraction around the impeller region coming from sparging in the first place and the radial motion given by the impeller discharge and recirculation. The high-hold up is also well depicted in Figure 9(c) in which ERT and CT gas hold-up profiles are compared. Both techniques indicate a similar trend to loading condition with a significant overall shift toward higher values of gas fraction. Although the comparison shows an overall condition of higher aeration achieved in this work (due to higher vvm gas rate), a good agreement in trends is observed. A smoother profile from the ERT data is again observed. This apparent smoothing is most probably related to the lower resolution of ERT and in particular to the inherent smoothing caused by the linear reconstruction applied to derive the conductivity tomograms. Nevertheless, in this comparison, ERT is not presented as a direct replacement for hard field CT in terms of accuracy for research studies, but rather as a lower cost alternative that is additionally deployable into manufacturing vessels.

Gas-Liquid-Solid System

The addition of stainless steel particles to the system has an influence on the electric field generated by the ERT electrodes and therefore it affects the measured conductivity. Given the conductive nature of the used solids, the measured average conductivity increases from the monophasic condition as more solids are suspended from the bottom of the tank. This increase is

however very small relative to the impact of the presence of gas. Indeed, the relative increase in conductivity at N_{js} for 5 w/w % in the solid-liquid case is approximately 0.4% while the addition of gas causes a decrease in the average conductivity between 8-20% from the base conductivity of the continuous phase. This difference can be seen in Figure 10 where axial profile in conductivity are shown for the Solid-Liquid run at different impeller speeds and, with a different conductivity (x axis) scale, the same runs are plotted in comparison with Gas-Solid-Liquid run at the same impeller speeds.

Figure 10 (a) shows how increasing the impeller speed causes a global increase of conductivity, given by the higher number of conductive particles suspended. At 1/3 Njs a few particles are suspended and they are concentrated in the bottom part of the tank; moving towards Njs it is possible to see how the profile becomes flatter and globally higher than the profiles at lower speed. However, reaching the N_{js} condition does not guarantee a homogenous dispersion of the particles across the tank; this condition is picked up by the ERT profile that still shows higher values in the impeller region than in any other height in the tank. The obtained profiles are consistent to what observed in Carletti's work⁵⁹ in which glass beads were suspended in aqueous solution. Although the different electrical properties of the solids used in this case causes an opposite effect on conductivity, similar trends are observed as the impeller speed is increased.

Figure 10(b) reports conductivity profiles for the same conditions (viz. with solids), but now with a gassing rate of 3 vvm. The scale in changes of conductivity are markedly larger with the gas. From this, it can reasonably be argued that, despite the presence of solids, the ERT system can still measure substantially the gas hold-up in the tank, with only minor influence from the dispersion of the particles.

In Figure 11, the trend of global gas hold-up for the three-phase case is compared with data for the Gas-Liquid only system. The data show similar values of gas hold-up for the three-phase case at the higher impeller speeds; the right-hand side of the plot. For the condition of low agitation however, the presence of the solids at the bottom seems to inhibit the retention of gas as already observed by Chapman⁴⁷. However, increasing the power provided by the impeller, the presence of solids seem to have little effect on the gas hold-up, as previously reported by Warmoeskerken⁶⁰.

Conclusions

In this work, an ERT linear probe was used to visualize and measure gas distribution in a two-phase and three-phase stirred tank at different regimes. ERT was able to successfully measure global gas hold-up in every observable regime in agreement with predictions from the literature. Local gas distributions were also measured and agreed with measurements on comparable systems conducted using hard-field techniques. Suspension of conductive solids was measured using ERT and it was assessed that they have little or no effect on the measurement of gas hold-up, therefore it can be concluded that the technique is suitable for measuring gas volume fraction also in presence of particles.

Differently from the traditional circular cage where the focus is on radial concentration, the chosen geometry of the probe allows to observe the axial profile of gas fraction within the tank in a quantitative fashion. The linear probe has the portability, ease of retrofitting and flexibility characteristics that make it a desirable tool for real-time measurements in an industrial environment. Overall, ERT has proven to offer, beside qualitative visual information on gas

location, quantitative measurements of gas hold-up in a small scale stirred tank with high potential to be scaled for inline monitoring of multiphase operations at industrial scale.

Acknowledgements

Giuseppe Forte is an EngD student at the University of Birmingham funded jointly by the Engineering & Physical Sciences Research Council (EPSRC) Centre for Doctoral Training in Formulation Engineering (EP/L015153/1) and Johnson Matthey.

Nomenclature

D Impeller diameter, m

D_p Diameter of suspended particles, m

Fl Flow number

Fr Froude number

g Standard acceleration due to gravity, m s⁻²

N Impeller rotational speed, rps

N_{cd} Complete dispersion critical impeller speed, rps

N_f Flooding critical impeller speed, rps

N_{is} 'Just suspended' impeller speed, rps

N_r Recirculation critical impeller speed, rps

P_g Gassed power, W

 $Q_g \qquad \qquad \text{Volumetric gas flow rate, m}^3 \text{ s}^{\text{-}1}$ $S \qquad \qquad \text{Geometrical constant in Zwietering correlation}$ $V \qquad \qquad \text{Volume of the tank, m}^3$ $v_s \qquad \qquad \text{Superficial velocity of the gas, m s}^{\text{-}1}$

X Percentage mass ratio of solids to liquid in suspension

Greek letters

Gas volume fraction ϵ_{g} Dimensionless conductivity[49] 3 Dynamic viscosity of liquid, kg m⁻¹ s⁻¹ μ Kinematic viscosity of liquid, m² s⁻¹ ν Density of liquid, kg m⁻³ ρ Density of solid particles, kg m⁻³ ρ_s Difference between solid and liquid density, kg m⁻³ Δρ Electrical conductivity, mS cm⁻¹ σ

List of Figure captions

- Figure 1. Flow map for the Rushton turbine with operated condition at different gas flow rate redrawn from Middleton et al.⁵¹

- Figure 2. Picture (a) and schematic (b) of the ERT linear probe and its position in the tank⁵³ (c)
- Figure 3. ERT tomograms representing conductivity map (a) and gas holdup (b)
- Figure 4. Average conductivity for monophasic and biphasic system at different impeller speeds.
- Figure 5. Global gas holdup values at 1.5 and 3 vvm
- Figure 6. Comparison of pictures and conductivity tomograms for flooding (a), loading (b, c) and completely dispersed (d, e) for 1.5 vvm
- Figure 7. Comparison of pictures and conductivity tomograms for, flooding (a), loading (b, c) and complete dispersion (d, e) for 3 vvm
- Figure 8. Axial gas hold-up at Flooding (Fr=0.016, Fl=0.32), Loading (Fr=0.11, Fl=0.14 for circles and Fr=0.24, Fl=0.080 for triangles), and Recirculation regimes (Fr=0.98, Fl=0.040 for asterisks, and Fr=1.5 Fl=0.032 for crosses), for 1.5 vvm (a); axial gas hold-up at Flooding (Fr=0.14, Fl= 0.21) Loading (Fr= 0.24 Fl=0.16 for circles, and Fr=0.55 Fl=0.11 for triangles) and Recirculation regimes (Fr=1.5, Fl= 0.063 for asterisks, and Fr=2.5 Fl=0.049 for crosses) for 3 vvm (b). The dashed lines delimit the impeller zone.
- Figure 9. Comparison of axial gas hold-up between ERT experimental data and data from the literature at different regimes: flooding (a), loading(b) and complete dispersion (c), with the dashed lines delimiting the impeller zone
- Figure 10. Axial conductivity plot for 5% w/w solids content in the case of Solid-Liquid mixing (a) and in the case of Solid-Liquid mixing compared with Gas-Solid-Liquid mixing with gas feed flow of 3 vvm (b)

- Figure 11. Global gas hold-up trend for two-phase and three-phase systems

List of Table captions

- Table 1 - Geometric properties of stirred tank

References

- 1. Ranade V, Deshpande VR. Gas-liquid flow in stirred reactors: Trailing vortices and gas accumulation behind impeller blades. Chem. Eng. Sci., 1999;54(12–14): 2305–2315,.
- 2. Smith, JM, Warmoeskerken, MMCG. The dispersion of gases in liquids with turbines. 5th European Conference on Mixing, Wurtsburg, West Germany, 1985:115–126.
- 3. Wang H, Jia X, Wang X, Zhou Z, Wen J, Zhang J. CFD modeling of hydrodynamic characteristics of a gas-liquid two-phase stirred tank, Appl. Math. Model., 2014;38(1): 63–92.
- 4. Rushton J H, Bmbinet JJ. Holdup and flooding in air liquid mixing. Can. J. Chem. Eng., 1968; 46(1): 16–21.
- 5. Saravanan K, Joshi JB. Fractional gas hold-up in gas inducing type of mechanically agitated contactors. Can. J. Chem. Eng. 1996; 74(1): 16–30.
- 6. Yawalkar AA, Pangarkar VG, Beenackers AACM. Gas hold up in stirred tank reactors. Can. J. Chem. Eng. 2002; 80:158–166.
- 7. Gao Z, Smith JM, Müller-Steinhagen H. Gas Dispersion in Sparged and Boiling Reactors. Chem. Eng. Res. Des., 2001;79(8):973–978.
- 8. Khopkar AR, Ranade VV. CFD simulation of gas–liquid stirred vessel: CARPT/CT measurements and CFD simulations. AIChE J. 2005; 52(5):1654–1672.
- 9. Chen JW, Kemoun A, Al-Dahhan MH, Dudukovic MP, Lee DJ, Fan LS. Comparative hydrodynamics study in a bubble column using computer-automated radioactive particle tracking (CARPT)/computed tomography (CT) and particle image velocimetry (PIV). Chem. Eng. Sci., 1999;54(13–14):2199–2207.

- 10. Cents AHG, Brilmant DWF., Versteeg GF. Ultrasonic investigation of hydrodynamics and mass transfer in a gas-liquid(-liquid) stirred vessel. Int. J. Chem. React. Eng. 2005;3(1).
- 11. Stamatiou IK, Muller FL. Determination of mass transfer resistances of fast reactions in three-phase mechanically agitated slurry reactors. AIChE J. 2017;63(1):273–282.
- 12. Chaouki J, Larachi, F, Dudukovic MP. Non-Invasive Monitoring of Multiphase Flows (first edition). Elsevier Science, 1997.
- 13. Utomo MB, Warsito W, Sakai T, Uchida S. Analysis of distributions of gas and TiO2 particles in slurry bubble column using ultrasonic computed tomography. Proc. 5th Int. Conf. Gas-Liq. Gas-Liq. Solid React. Eng., 2001;56(21):6073–6079.
- 14. Laakkonen M, Honkanen M, Saarenrinne P, Aittamaa J. Local bubble size distributions, gas-liquid interfacial areas and gas holdups in a stirred vessel with particle image velocimetry, Chem. Eng. J., 2005;109(1–3):37–47.
- 15. Chung KHK, Simmons MJH, Barigou M. Local gas and liquid phase velocity measurement in a miniature stirred vessel using PIV combined with a new image processing algorithm. Exp. Therm. Fluid Sci., 2009;33(4):743–753.
- 16. Fishwick RP, Winterbottom JM, Parker DJ, Fan XF, Stitt EH, Hydrodynamic measurements of upand down-pumping pitched-blade turbines in gassed, agitated vessels, using positron emission particle tracking. Ind. Eng. Chem. Res. 2005;44(16): 6371–6380.
- 17. Dudukovic MP, Opaque multiphase reactors: Experimentation, modeling and troubleshooting. Oil Gas Sci. Technol.-Rev. Ifp Energ. Nouv., 2000;55(2):135–158.
- 18. Ford JJ, Heindel TJ, Jensen TC, Drake JB. X-ray computed tomography of a gas-sparged stirred-tank reactor. Chem. Eng. Sci. 2008;63(8):2075–2085.
- 19. Veera UP, Patwardhan AW, and Joshi JB. Measurement of Gas Hold-up Profiles in Stirred Tank Reactors by Gamma Ray Attenuation Technique. Chem. Eng. Res. Des. 2001;79(6):684–688.

- [20] A. J. Sederman and L. F. Gladden, 'Magnetic resonance imaging as a quantitative probe of gas-liquid distribution and wetting efficiency in trickle-bed reactors', Chem. Eng. Sci., vol. 56, no. 8, pp. 2615– 2628, Apr. 2001.
- [21] M. V. Sardeshpande, S. Gupta, and V. V. Ranade, 'Electrical Resistance Tomography for Gas Holdup in a Gas-Liquid Stirred Tank Reactor', Chem. Eng. Sci., vol. 170, pp. 476–490, Apr. 2017.
- [22] M. Wang, Dorward, A., Vlaev, D., and R. Mann, 'Measurements of gas-liquid mixing in a stirred vessel using electrical resistance tomography (ERT)', Chem. Eng. J., vol. 77, no. 1–2, pp. 93–98, 2000.
- [23] P. J. Holden, M. Wang, R. Mann, F. J. Dickin, and R. B. Edwards, 'Imaging stirred-vessel macromixing using electrical resistance tomography', AIChE J., vol. 44, no. 4, pp. 780–790, 1998.
- [24] M. Wang, Industrial Tomography Science and Applications, 1st ed. Woodhead Publishing Series in Electronic and Optical Materials, 2015.
- [25] R. Mann, F. J. Dickin, M. Wang, Dyakowski, T., R. A. Williams, and Edwards, R. B., 'Application of Electrical Resistance Tomography to Interrogate Mixing Processes at Plant Scale', Chem. Eng. Sci., vol. 52, pp. 2087–97, 1997.
- [26] H. Jin, S. Yang, M. Wang, and R. A. Williams, 'Measurement of Gas Holdup Profiles in A Gas Liquid Cocurrent Bubble Column Using Electrical Resistance Tomography', AIP Conf. Proc., vol. 914, no. 1, pp. 869–876, Jun. 2007.
- [27] Abdullah, B., 'Evaluation of gas-liquid mass transfer in gas induced stirred tank reactor using electrical resistance tomography', J. Chem. Technol. Amp Biotechnol., vol. 92, no. 8, pp. 2123–2133, 2017.
- [28] J. Kourunen, R. Kayhko, J. Matula, J. Kayhko, M. Vauhkonen, and L. M. Heikkinen, 'Imaging of mixing of two miscible liquids using electrical impedance tomography and linear impedance sensor', Flow Meas. Instrum., vol. 19, no. 6, pp. 391–396, Dec. 2008.

- [29] S. T. L. Harrison, R. Stevenson, and J. J. Cilliers, 'Assessing solids concentration homogeneity in Rushton-agitated slurry reactors using electrical resistance tomography (ERT)', Chem. Eng. Sci., vol. 71, pp. 392–399, Mar. 2012.
- [30] F. Ricard, C. Brechtelsbauer, X. Y. Xu, and C. J. Lawrence, 'Monitoring of multiphase pharmaceutical processes using electrical resistance tomography', Chem. Eng. Res. Des., vol. 83, no. A7, pp. 794–805, Jul. 2005.
- [31] S. A. Razzak, S. Barghi, J.-X. Zhu, and Y. Mi, 'Phase holdup measurement in a gas-liquid-solid circulating fluidized bed (GLSCFB) riser using electrical resistance tomography and optical fibre probe', Chem. Eng. J., vol. 147, no. 2–3, pp. 210–218, Apr. 2009.
- [32] C. Carletti, G. Montante, C. De Blasio, and A. Paglianti, 'Liquid mixing dynamics in slurry stirred tanks based on electrical resistance tomography', Chem. Eng. Sci., vol. 152, pp. 478–487, Oct. 2016.
- [33] Richardson, D. J. and Holdich, R. G., 'A Novel and Low Cost Probe for Monitoring Dispersed Solids Concentrations in Liquids', Chem. Eng. Res. Des., vol. 83, pp. 569–574, 2001.
- [34] Bolton, G. T., Qiu, C. H., and M. Wang, 'A Novel Electrical Tomography Sensor for Monitoring the Phase Distribution in Industrial Reactors', in 7th UK Conference on Mixing, Bradford, 2002.
- [35] M. Hamood ur Rehman, F. Ein Mozaffari, and Y. Dahman, 'Dynamic and local gas holdup studies in external loop recirculating airlift reactor with two rolls of fiberglass packing using electrical resistance tomography', J. Chem. Technol. Biotechnol., vol. 88, no. 5, pp. 887–896, 2013.
- [36] Khalili, F., Jafari Nasr, M. R., Kazemzadeh, A., and Ein-Mozaffari, F., 'Analysis of gas holdup and bubble behavior in a biopolymer solution inside a bioreactor using tomography and dynamic gas disengagement techniques', J. Chem. Technol. Biotechnol., vol. 93, no. 2, pp. 340–349, 2018.
- [37] Smith, J. M., 'Dispersion of Gases in Liquids: the hydrodynamics of gas dispersion in low viscosity liquids', in Ulbrecht, J.J. and Patterson, G.K., eds, New York, 1985, pp. 115–126.
- [38] Nienow, A.W., Warmoeskerken, M.M.C.G., Smith, J. M., and Konno, M., 'On the flooding/loading transition and the complete dispersal condition in aerated vessels agitated by a Rushton-turbine', in 5th European Conference on Mixing, Wurtsburg, West Germany, 1985, pp. 143–154.

- [39] M. M. C. G. Warmoeskerken and J. M. Smith, 'Flooding of disc turbines in gas-liquid dispersions: A new description of the phenomenon', Chem. Eng. Sci., vol. 40, no. 11, pp. 2063–2071, Jan. 1985.
- [40] Middleton, J. C. and Smith, J. M., 'Gas-Liquid Mixing in Turbulent Systems', in Handbook of Industrial Mixing: Science and Practice, John Wiley and Sons, 2003, pp. 585–638.
- [41] Smith, J. M., 'Simple performance correlations for agitated vessels', in 7th European Congress on Mixing, Brugge, 1991, pp. 233–241.
- [42] T. Zwietering, 'Suspending of Solid Particles in Liquid by Agitators', Chem. Eng. Sci., vol. 8, no. 3–4, pp. 244–253, 1958.
- [43] G. R. Kasat and A. B. Pandit, 'Review on mixing characteristics in solid-liquid and solid-liquid-gas reactor vessels', Can. J. Chem. Eng., vol. 83, no. 4, pp. 618–643, Aug. 2005.
- [44] V. Rewatkar and J. Joshi, 'Critical Impeller Speed for Solid Suspension in Mechanically Agitated three phase Reactors .2. Mathematical-Model', Ind. Eng. Chem. Res., vol. 30, no. 8, pp. 1784–1791, Aug. 1991.
- [45] A. W. Nienow and W. Bujalski, 'Recent Studies on Agitated Three-Phase (Gas-Solid-Liquid) Systems in the Turbulent Regime', Chem. Eng. Res. Des., vol. 80, no. 8, pp. 832–838, Nov. 2002.
- [46] Y. Y. Bao, Z. G. Hao, Z. M. Gao, L. T. Shi, J. M. Smith, and R. B. Thorpe, 'Gas dispersion and solid suspension in a three-phase stirred tank with multiple impellers', Chem. Eng. Commun., vol. 193, no. 7, pp. 801–825, Jul. 2006.
- [47] Chapman, C.M., A. W. Nienow, M. Cooke, and Middleton, J. C., 'Particle-gas-liquid mixing in stirred vessels. Part IV: Mass transfer and final conclusions', Chem Eng Res Des, vol. 61, no. 3, pp. 182–185, 1983b.
- [48] Maxwell, James Clerk, A Treatise on Electricity and Magnetism, 2nd ed., vol. 1. Clarendon Press, 1881.
- [49] G. Montante and A. Paglianti, 'Gas hold-up distribution and mixing time in gas-liquid stirred tanks', Chem. Eng. J., vol. 279, pp. 648–658, Nov. 2015.

- 50. Jamshed A, Cooke M, Ren Z, Rodgers TL. Gas-Liquid mixing in dual agitated vessels in the heterogeneous regime. Chem. Eng. Reas. Des. 2018;33:55-69.[51] J. C. Middleton, 'Gas-Liquid Dispersion and Mixing', in Mixing in the Process Industries, 2nd ed., Butterworth Heinemann, 1997, pp. 322–363.
- [52]B. W. Lee and M. P. Dudukovic, 'Determination of flow regime and gas holdup in gas-liquid stirred tanks', Chem. Eng. Sci., vol. 109, pp. 264–275, Apr. 2014.
- [53] Clark, P.J., 'Towards in-situ characterisation of formulated products', University of Birmingham, 2016.
- [54] M. S. Takriff, A. Ahmad, M. I. Rosli, and S. Jantan, 'ERT visualization of gas dispersion performance of aerofoil and radial impellers in an agitated vessel', J. Teknol. Sci. Eng., vol. 64, no. 5, pp. 75–78, 2013.
- [55] S. Hosseini, D. Patel, F. Ein-Mozaffari, and M. Mehrvar, 'Study of solid-liquid mixing in agitated tanks through electrical resistance tomography', Chem. Eng. Sci., vol. 65, no. 4, pp. 1374–1384, Feb. 2010.
- [56] Bolton, G. T., 'A Review of Linear Electrical Tomography Probes for Monitoring the Behaviour of Multiphase Mixing Processes', presented at the 12th European Conference on Mixing, Bologna, 2006.
- [57] G. Forte, P.J. Clark, O. Wale, J. F. Hall, N. Zumaeta, M. J. H. Simmons, E. H. Stitt, 'Extending the application of electrical resistance tomography using linear probes in fluid mixing diagnosis', in 8th World Congress on Industrial Process Tomography, Iguassu Falls, Brazil, 2016.
- [58] K. Van't Riet and J.M. Smith, 'The trailing vortex system produced by rushton turbine agitators', Chem Eng Sci, vol. 30, no. 91, pp. 1093–1105, 1975.
- [59] C. Carletti, G. Montante, T. Westerlund, and A. Paglianti, 'Analysis of solid concentration distribution in dense solid–liquid stirred tanks by electrical resistance tomography', Chem. Eng. Sci., vol. 119, pp. 53–64, Nov. 2014.
- [60] M. M. C. G. Warmoeskerken, van Houwelingen, M. C., Drijlink, J. J., and Smith, J. M., 'The role of cavity formation in stirred gas-liquid-solid reactors', Chem Eng Res Des, vol. 62, pp. 197–200, 1984.