Radioisotope Applications in the Petrochemical Industry: An Overview

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ABSTRACT

1. INTRODUCTION

Radioactive isotopes, particularly in the form of radiotracers and radioactive sealed sources, have been used over a broad spectrum of process industries for equipment and process troubleshooting, monitoring, control, inspection, optimization, and many other purposes. Radioisotope based measurement techniques are best suited in multiphase flow systems of interest in industrial practice, particularly in petrochemical processes, since these systems are opaque and contain a large volume fraction of the dispersed phase. However, the applications of the radioisotope based techniques in these systems are not trivial, and hence, research and developments are required for proper use and reliable information. In multiphase flow systems, two or more phases (gas, liquid, solid) are involved and interact with each other in various forms. Multiphase flow systems have found extensive applications in a wide range of industrial processes, such as chemical, petrochemical, petroleum, gas processing, polymer and materials, food and feed, biochemical, pharmaceutical, environmental, bio-energy and alternative energy, biomass conversion, water and wastewater treatment, and mineral processes. In the petrochemical industry various types of multiphase flow systems are used, such as multiphase reactors, blenders/mixers, separators, etc. Multiphase reactors in particular are essential parts of petrochemical processes, as their selected types and performance dictate the number of separation/mixing/blending units needed in the processes and their load (before and after the reactors) and thus, affect profoundly the overall economics of the processes. Furthermore, the reactor is the key to environmentally friendly processing in preventing pollution at the source. In practice, over 99% of reactors are multiphase in character. Multiphase reactors in general are complex, as they can take various configurations and types based on the way the phases are contacted and interact. Various types of multiphase flow reactors have been employed in the petrochemical industry, such as bubble columns (gas-liquid system), slurry bubble columns (gas-liquid-fine solid system), packed beds (gas-solid, liquid-solid, gas-liquid-solid systems), fluidized beds (gas-solid, liquid-solid, gas-liquid-solid systems), mechanically mixed equipment (gas-solid, liquid-solid, gas-liquid, gas-liquid-solid systems), and many others. All these systems are opaque, as mentioned earlier, and hence only radioactive isotope based techniques can probe and visualize them, as light based techniques cannot be applied. Furthermore, quantification of the performance of these multiphase reactors and flow systems in general requires proper understanding and description of the hydrodynamics, complex interaction among the phases, phase distribution, velocity field, mixing, flow pattern, etc. Integrating such knowledge and information with transports, kinetics and events on the eddy and molecular scales is essential for properly sizing the reactor, reliable predictive modeling, design, scale up, and process optimization of these systems. This knowledge enables cleaner, safer, and more energy efficient petrochemical and industrial processes in general. In addition, the knowledge, information and findings obtained by these radioisotope based techniques will not only advance the fundamental understanding of these systems but will also provide bench mark data for evaluation and validation of multiphase fluid dynamics (CFD) codes and their closures, which in turn allows us to develop better, more reliable reactor or system scale based models, thus reducing pollution and improving productivity and selectivity for benefit of society and environment. Also, full dynamic model based process control and optimization depend on a reliable reactor scale model. Achieving all these is a challenging task due to the complex interaction among the phases and the opaque nature of these reactors and equipment. Therefore, it is important to develop and employ means to visualize and quantify the flow field in these systems. In this contribution, radioisotope based techniques are outlined, and their applications in petrochemical processes are overviewed and discussed with examples. More information and results about the use of radioactive isotope in the petrochemical industry and the needed future work, research and development will be presented in the plenary lecture.

2. RADIOISOTOPE BASED TECHNIQUES

A variety of optically based techniques have been developed for flow visualization and quantification in transparent systems.

However, photons of visible light fail to pass through opaque objects. Since, multiphase systems are opaque, high energy gamma ray photons are required, because they can penetrate these systems to provide information about phase distributions, flow, and mixing. Traditionally, gamma emitters were used in nuclear gauge densitometry to establish liquid levels or provide an estimate of a line average holdup of phases. Radioactive isotopes were used to 'trace' the phases and provide residence time distributions (RTDs). Only recently, full quantification of the density distribution via computer tomography and full Lagrangian description of the flow by particle tracking become possible, with the advent of high computational power and advanced data acquisition technology. Many radioisotopes can be used in industrial applications such as Scandium-46, Cesium-137, Cobalt-60 and 58, Selenium-75, Americium-24 1, Gold, Sodium-22, Manganese-54, Oxide on Manganese-56, Yttrium-88, Ziconimu-95, Niobium-95, Ruthenium-103, etc. Various radioisotope based techniques have been developed and used for research at the laboratory scale and pilot plant scales and for site applications in industrial processes. The following are some examples of these radioisotope based techniques:

1. radiotracer method for the measurement of the residence time distribution (RTD) and its utilization for trouble shooting and diagnostics. Various radioisotope types and amounts have been used for gas, liquid and solid phases. Also, radiotracer-based multiphase flow meters have been developed and implemented.

2. Sealed sources for gamma ray and x ray transmission measurements. Various techniques have been developed and used for industrial and research measurements, such as level monitoring and control using nuclear gauge densitometry, chord (line) average densities (phase holdups) using gamma ray densitometry, time and cross sectional averaged phases holdup distribution along the reactor or equipment height using computed tomography (single energy, dual or multiple energy), single photon emission tomography, positron emission tomography, and Compton scattering tomography. Different isotopes have been used and tested in these techniques.

3. Radioactive particle tracking (RPT) techniques using a single radioactive particle or multiple radioactive particles and either collimated or non-collimated detectors. These techniques have been used to measure in 3D the flow field, phase trajectory, velocity, RTD, turbulent parameters, stagnant zones, diffusivity, turbulent kinetic energy, and many flow related aspects of the tracked phase (either liquid or solid phase). Also, various types of isotopes have been used and tested.

4. Positron emission particle tracking (PEPT) techniques for measurements similar to those obtained by the radioactive particle tracking techniques outlined in (3) above.

For all these, techniques various mathematical models and reconstruction algorithms have been developed, evaluated, and implemented for image reconstructions. Furthermore, various mathematical methodologies, procedures and algorithms have been developed from post processing of the reconstructed image to estimate the desired parameters, such as phase holdups, phase velocities, phase turbulent parameters, etc. Implementing these techniques properly is not a trivial task. For example, implementing radioactive particle tracking techniques requires devices and specialized knowledge for particle preparation, calibration, performing the experiment, safe handling of the radioactive materials, and data reconstruction and processing. Chaouki et al. (1997) and IAEA reports (2007, 2006, 2005) outline and discuss some these radioisotope based techniques.

The desirable characteristics of the radioisotope based techniques in particular, and of any experimental diagnostic and measurement techniques in general, can be summarized as follows:

1. Good spatial and temporal resolution in both velocity and volume fraction (holdup measurements),

2. Capability to provide instantaneous (snapshot) measurements so that one could, in principle, be able to quantify the turbulent and dynamic flow structures,

3. Ability to probe opaque systems in which the dispersed phase volume fractions are high,

4. Statistically reproducible results obtainable in a finite time,

5. Amenability to automation, so as to minimize human involvement in the data collection process,

6. Portability of the technique and its applicability to larger units, such as pilot plant and industrial units,

7. Affordable cost,

8. Safety of the personnel involved in the experimentation

At present, unfortunately, no single experimental technique satisfies all these characteristics. However, research in the direction of the long term goal to achieve the above requirements is constantly in progress (Chaouki et al., 1997; IAEA, 2007; Varma et al. 2008).

In the past and present, classical methods for trouble shooting and for "blackbox" model development have been used. These techniques are: 1) tracer impulse response to find RTDs, dispersion extent and dispersion coefficient, holdup, or to match the response to a model, and 2) gamma ray densitometry to obtain line average holdups and, from a number of measurements, to determine radial profiles and assess potential problem areas within the tested equipment. These techniques are very valuable, and advances have been made in the technology used for their applications (for example, see www.tracerco.com). Due to the advances in technology and computational methods, various modern techniques have been developed and employed as outlined above. Among these, gamma ray computed tomography, and radioactive particle tracking techniques are discussed here. Examples of their applications on key multiphase flow systems employed in petrochemical processing are briefly demonstrated. As mentioned earlier, detailed information and more examples will be presented in the plenary lecture.

3. GAMMA RAY COMPUTED TOMOGRAPHY (CT)

Gamma ray CT is a non-invasive technique to measure time and cross-sectional averaged gas holdup distribution at any desired column height. The principle of CT is based on Beer – Lambert's law. The gamma ray beam is attenuated based on the medium it passes through. From the measured attenuation of the beams of radiation (projections) through the two phase mixture, the distribution of the phases in the cross-section can be calculated. Different configurations and generations of gamma ray CT have been used. The fan-beam (third generation CT) based CT consists of an array of NaI detectors (2 inch in diameter) arranged in an arc on one side of the object and a gamma ray source with suitable strength on the other side. Cesium-137 and Cobalt-60 with various activities have been used as encapsulated sources for CT units. Figure 1 shows one of our Chemical Reaction Engineering Laboratory (CREL) CT units (Kumar, 1994; Rados, 2003). The detectors and the source are mounted on an automatically moved rotating plate by a stepper motor, which makes it possible to scan 360 degrees around the object. The source is placed in a source collimated device which provides a fan beam of 40 degrees in the horizontal plane. The beam is further collimated using a 20x20x10 cm lead brick with a central slit, so that the emerging beam has a thickness of 6.5 mm at a distance of 28 cm from the source. The detectors are collimated using a collimator made of lead 6.35 cm deep and 7.62 cm high so that the detectors are completely shielded by the collimator. In front of each detector there is a rectangular hole in the collimator of 5x10 mm or 2x10 mm for sampling the beam. For a given view (each angular movement of both the source and detectors), the detectors and collimators are moved automatically by another independent stepper motor to collect additional chordal transmittance measurements (projections) for each detector. Hence, depending on the number of positions of the fan beam and the number of detectors thousands of projections can be obtained for different sizes of columns or objects to be scanned. The spatial resolution of the CT is within 2-5 mm, and the density resolution of about 0.04 g/cm3. In general the spatial resolution of CT is affected by the total number of projections and the number of detectors used, and the dimensions of the source and collimated detectors. On the other hand, the temporal resolution is affected by the number of projections per unit time (the duration must be smaller than the system time scale), the minimum data acquisition time or counts integration time (which is a function of the data acquisition hardware), the source strength, the area of the collimated detectors and the time required to make a complete scan. These factors determine the number of counts received by the detectors and the quality of their statistics. The CT scan is usually performed for about 2 or more hours, depending on the type of experiment, to obtain time and cross sectional averaged holdup distributions. Among a number of image reconstruction algorithms reported in the literature, such as convolution or filter back projection, algebraic reconstruction, and estimation-maximization (EM) algorithms, EM based on maximum likelihood principles was found to be the best (Kumar 1994). Recently, the alternating minimization (AM) algorithm was found to outperform the EM technique (Buhsarapu et al., 2005; Varma, et al., 2008).

It is noteworthy that the CT unit can also be used as a gamma ray densitometry technique by utilizing the middle detector, which receives the attenuated gamma ray line passing through the column diameter. Recently an industrial based scanner has been developed and implemented on a 2 ft industrial pilot plant unit as shown in Figure 2 (Al-Dahhan et al., 2007).

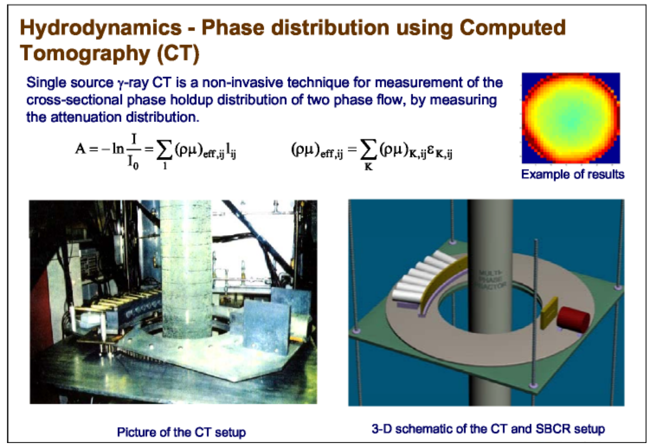

Figure 1: One of the Chemical Reaction Engineering Laboratory's (CREL) CT Techniques.

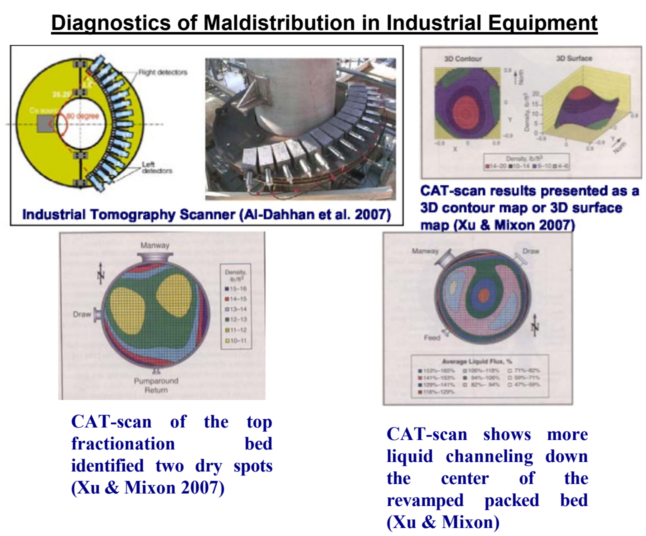

Figure 2: Industrial Tomography Scanner (ITS) and Its Application in A Pilot Plant and Commercial Scales Units

4. RADIOACTIVE PARTICLE TRACKING (RPT)

The RPT technique is based on following the motion of a single radioactive particle in the 3D domain of the whole system. The particle is made of irradiated Scandium-46, Gold-…, Cobalt-60 or another isotope of a gamma ray emitter. A tracer particle that is identical in size, density, and shape to the solids is used to track the solids' motion in gas-solid, gas-liquid-solid, and liquid-solid systems. To track the motion of liquid, a small (1 mm or below) tracer particle that is neutrally buoyant is used in a gas-liquid system. In the first case a coated particle is used, while in the second case, a polypropylene or polyethylene bead of 0.8 – 2 mm diameter is used. A hole is machined to house the irradiated element and the empty gap of the hole is filled with an amount of glue sufficient to obtain the same density as the tracked liquid phase. A Metallic paint or coating ensures that bubbles do not adhere to the particle. The activity of the irradiated particle ranges from 150 to 500 µCi. The technique was introduced to monitor the motion of solids in fluidized beds by Lin et al. (1985), and it was adapted for the measurement of liquid velocity and its flow field in a bubble column by Devanathan (1990). Since then, the technique has advanced and the spatial resolution has improved. The instantaneous particle position is identified by simultaneously monitoring the radiation intensities received by a set (16-32) of 2 inch NaI detectors arranged strategically around the column as shown in Figure 3. The radiation intensity recorded at each detector decreases exponentially with increasing distance between the particle and the detector. In order to reconstruct the position of the particle during the tracking experiment, a calibration experiment using a suitable calibration device and methodology needs to be performed at the same conditions as the radioactive particle tracking (RPT) experiment. This can beachieved by placing the radioactive particle at various known locations and monitoring the radiation received by each detector. Using the information acquired, calibration curves are established that relate the intensity received at a detector to the distance between the particle and the detector. Once the distance of the particle from the set of detectors is estimated, various reconstruction algorithms are used to estimate the position of the particle at a given sampling instant in time. These algorithms include a weighted regression scheme (Devenathan, 1990; Degaleesan, 1997), a modified weighted regression scheme (Rados, 2003; Luo, 2005, Vesvikar, 2006), cross correlation (Bhusarapu, 2005), and Monte Carlo simulation (Larachi et al, 1994; Gupta, 2002; Ong, 2003; Bhusarapu, 2005). A wavelet based filtering algorithm (Degaleesan, 1997) is employed to remove the noise in position estimations created by the statistical nature of gamma radiation.

Once the calibration step is completed, the tracer particle is let to freely move in the system while data is collected by the detectors. The operating conditions are controlled and kept constant during whole the period of the experiment. The sampling frequency is adjusted to ensure good accuracy. For example, for bubble columns, stirred tanks and circulating fluidized beds, frequencies of 50 Hz, 100-200 HZ, and 200Hz have been found suitable. Using the calibration data and algorithm, a sequence of instantaneous position data is obtained that yields the Lagrangian position of the particle at successive sampling instants (i.e., Lagrangian trajectory) (Figure 4).

Recently, the resolution of the technique has been improved to within 1 mm (Luo, 2005). The performance of RPT is assessed by two indicators: resolution (R), which is related to the uncertainty in tracer position reconstruction, and sensitivity (S), which is related to the fractional change in counts with a change in the tracer position. For better RPT performance, R needs to be minimized and S needs to be maximized. Many challenges must be met to achieve the desired R and S, as many factors can affect these indicators. These factors include tracer particle source strength, photon energy, detector (crystal) size, positioning of detectors, and number of detectors used (e.g., staggered configuration, number of detectors per plane, medium attenuation and its holdups fluctuation, sampling frequency, dynamic bias, reconstruction algorithm, and grids size (Larachi et al., 1994; Roy et al., 2002). Particle trajectories, at constant superficial gas velocities, are monitored and mapped over a long period of time. Differentiation of the data yields instantaneous particle velocities. The column is divided into fictitious small compartments and the estimated instantaneous velocity is assigned to the middle compartment. Ensemble averaging at each location (compartment) provides the time averaged quantities and the spatial flow field for the whole column. The RPT experiment will continue until the time averaged velocity in each compartment reaches a plateau where adequate statistics have been achieved. The instantaneous and time averaged velocities can then be used to determine various turbulent parameters (Reynolds stresses, turbulent kinetic energy, turbulent eddy diffusivities, etc.) Figure 4 shows the steps of data processing for RPT experiments. An additional wealth of information can be obtained from RPT data, such as overall and local residence time distributions, and stagnant zones and sizes. RPT is the only non-invasive technique that maps in 3D the flow field in the whole reactor and provides particle Lagrangian velocities throughout the column. It is also the only technique that can accomplish these measurements with no limitation on gas velocity, operating conditions of pressure and temperature, opacity, and system designs and configurations.

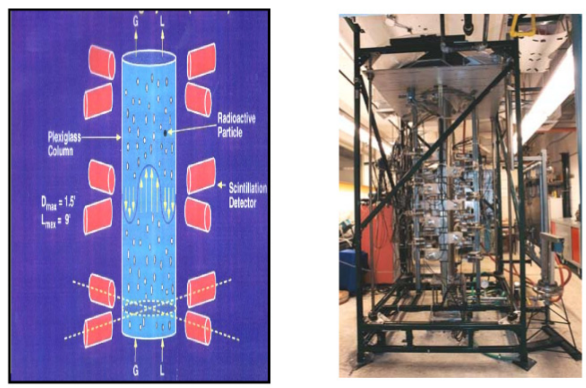

Figure 3: One of the CREL's Radioactive Particle Tracking (RPT) Techniques

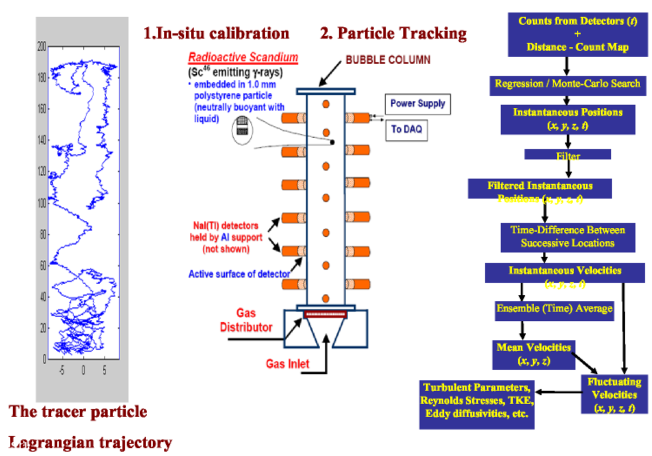

Figure 4: Lagrangian Trajectory and Data Processing Steps of RPT Technique

5. APPLICATIONS OF RADIOISOTOPE BASED TECHNIQUES IN THE PETROCHEMICAL INDUSTRY

Various types of multiphase flow systems are employed in the petrochemical industry, such as multiphase reactors, blenders/mixers, and separators. Radioisotope based techniques have been applied to these systems for diagnostics, monitoring, process and advanced model development. Examples of bubble and slurry bubble columns and trickle bed reactors are briefly outlined here. Additional examples and results of these techniques used for other types of multiphase flow systems related to petrochemical processes will be summarized in the lecture, such as distillation columns, structured beds, fluidized beds, circulating beds, mechanical mixing equipment, etc.

6. BUBBLE AND SLURRY BUBBLE COLUMN REACTORS

Bubble and slurry bubble columns are used extensively in a variety of petrochemical processes for hydrogenation, oxidation, hydro-formulation, and chlorination. Due to their excellent heat transfer characteristics, they have recently been identified as reactors of choice for syngas (a mixture of carbon monoxide, carbon dioxide and hydrogen) conversion, such as liquid phase methanol synthesis and Fischer-Tropsch (FT) synthesis Dudukovic et al. (2002). In addition, they are used in a variety of bioprocesses and in wastewater treatment. Figure 5 schematically shows bubble and slurry bubble columns. They are cylindrical columns where the gas is sparged at the bottom, resulting in buoyancy driven flow which creates strong liquid recirculation. When fine catalyst particles are used (typically 60 microns or less), the reactor is called a slurry bubble column. As long as the liquid superficial velocity is an order of magnitude smaller than the gas superficial velocity, it is the gas velocity that dictates the fluid dynamics of the whole system. Hence, whether the liquid is processed batch-wise or flows either co-currently or counter-currently will not affect the hydrodynamics of the system. Although in some applications bubbly flow (2-3 cm/s of superficial gas velocity) is practiced, the current industrial interest is the churn-turbulent flow regime, where superficial gas velocity ranges 10 cm/s up to 60 cm/s.

Recently, due to the use of advanced radioisotope based techniques, the fundamental understanding of the hydrodynamics of these multiphase reactors has been noticeably advanced. This improves the reactor scale modeling, computational fluid dynamics (CFD) codes and closures, and scale-up and design methodology. Figure 6 shows an example of the gas-holdup distribution and profiles using gamma ray computed tomography (CT). Also, using CT, the flow regime transition between bubbly and churn-turbulent flow regimes has been identified, as shown in Figure 7 It is clear that in these systems and at a high range of superficial gas velocities, gas holdup is higher in the central region of the column than near its wall region, causing pressure or buoyancy imbalance between these two regions. This causes a liquid or slurry circulation between the central region of the column, where the flow is upward, and the wall region, where the flow is downward. Such a flow structure has been confirmed by RPT results, as shown for example in Figures 8 and 9. Both CT and RPT results have been used to develop reactor scale mechanistic models, to evaluate and validate CFD codes and closures, and to develop a new methodology for scale-up of these reactors. The knowledge obtained by CT and RPT has been integrated with the results obtained by using the radioisotope based tracer technique to measure the RTD of the gas, liquid, and solid phases in industrial pilot plant slurry bubble columns for methanol synthesis. The proper characterization of its hydrodynamics is outlined below.

6.1 Integrating Classical and Modern Radioisotopes Based Techniques

Figure 10 shows an example of how radioisotope based techniques (namely as mentioned above, a radioactive tracer for gas, liquid, and solid (RTD), gamma ray computed tomography (CT), and radioactive particle tracking (RPT)) have used to advance the modeling and hydrodynamics understanding of slurry bubble columns for methanol synthesis from syngas (CREL submitted DOE reports, 2002). The mechanistic models developed based on the results of CT and RPT have been used along with validated CFD results to predict properly the RTD responses measured using radioisotopes (irradiated Argon-41, is used for the gas phase, irradiated powder oxide of Mn-56 (Mn2O3) is used for the liquid phase and irradiated catalyst particles doped with an oxide of Mn-56 are used for the catalysts). The system was an 18-inch pilot plant slurry bubble column (Laporte, Texas) operating under industrial operation for methanol synthesis.

6.2 Trickle Bed Reactors

Trickle bed reactors are the most frequently used fixed bed reactors, with two phase flow in industry in general and in the petrochemical industry in particular. In these reactors, gas and liquid phases flow co-currently downward over a bed of catalyst particles randomly packed. This mode of operation allows for various flow regimes depending on the chemistry of the process and based on the flow rates of phases, bed characteristics, and physical properties. Also, such a mode of operation is the most flexible with respect to varying throughput demands, as compared to other modes of operation (i.e., co-current upward and counter-current flow of gas and liquid phases). Trickle bed reactors have been used extensively in various applications, such as residuum and vacuum residuum desulfurization, hydrodesulfurization, catalytic dewaxing, sweetening, hydrocracking, hydrodenitrofication, isocracking, wax synthesis from syngas, Fischer-Tropsch synthesis, production of lubricants, hydrogenation and selective hydrogenation, oxidation, hydration, and many others (Dudukovic, et al., 2002).

Radioisotopes have been used in both classical tracer techniques (i.e., RTD measurement) and modern gamma ray computed tomography. On-site radioisotope tracer techniques can be used to characterize the extent of dispersion of both gas and liquid phases and to identify qualitatively the existence of stagnant zones or internal circulation. Gamma ray computed tomography (CT) can be used to investigate the time averaged cross sectional phases distribution along the reactor height, quantify the effect of design and operating parameters, study the impact of distributor design on liquid and gas distribution, obtain bench mark data for evaluating and validation of CFD, characterize the bed viodage distribution and the effect of the method of packing, etc.

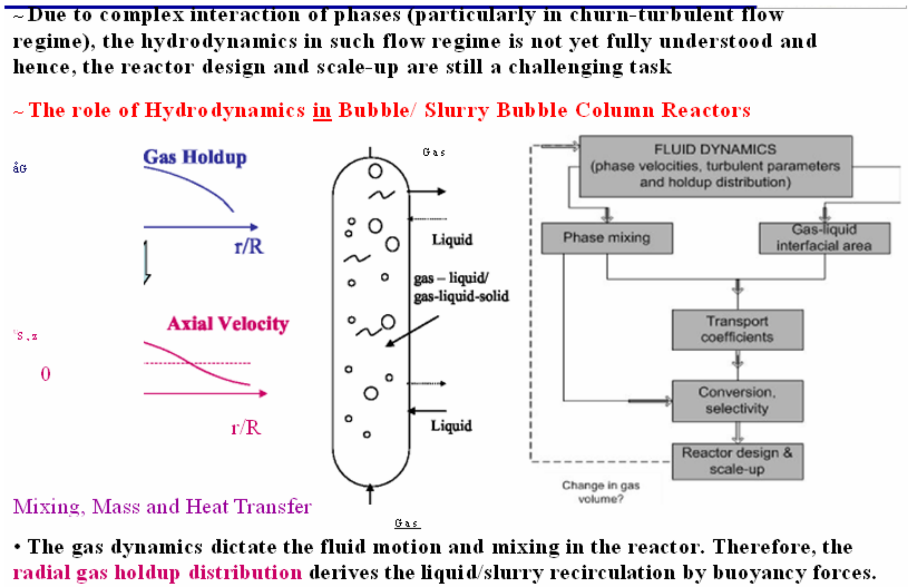

Figure 5: A Schematic Diagram of Bubble and Slurry Bubble Columns

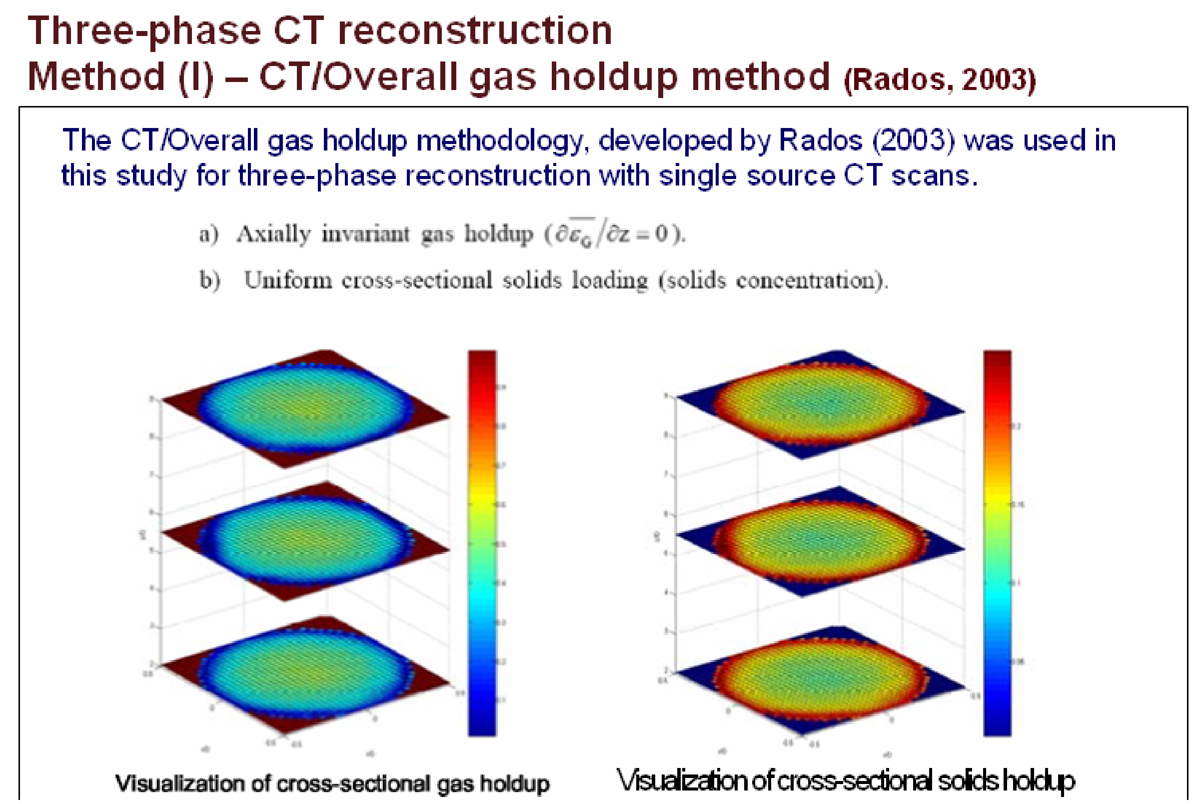

Figure 6: Example of Results of Gas and Solids Hold-up Cross Sectional Distributions along the Slurry Bubble Column Height Using CT Technique

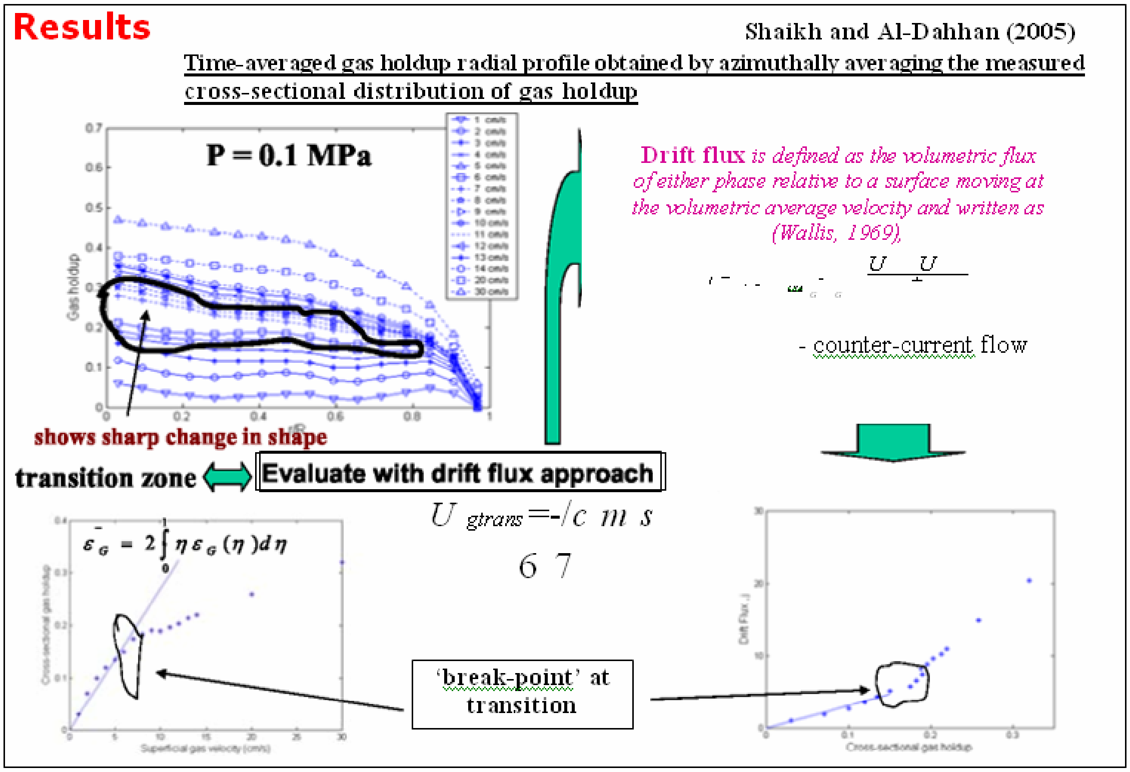

Figure 7: Identification of Flow Regime Transition in Bubble Column using CT technique

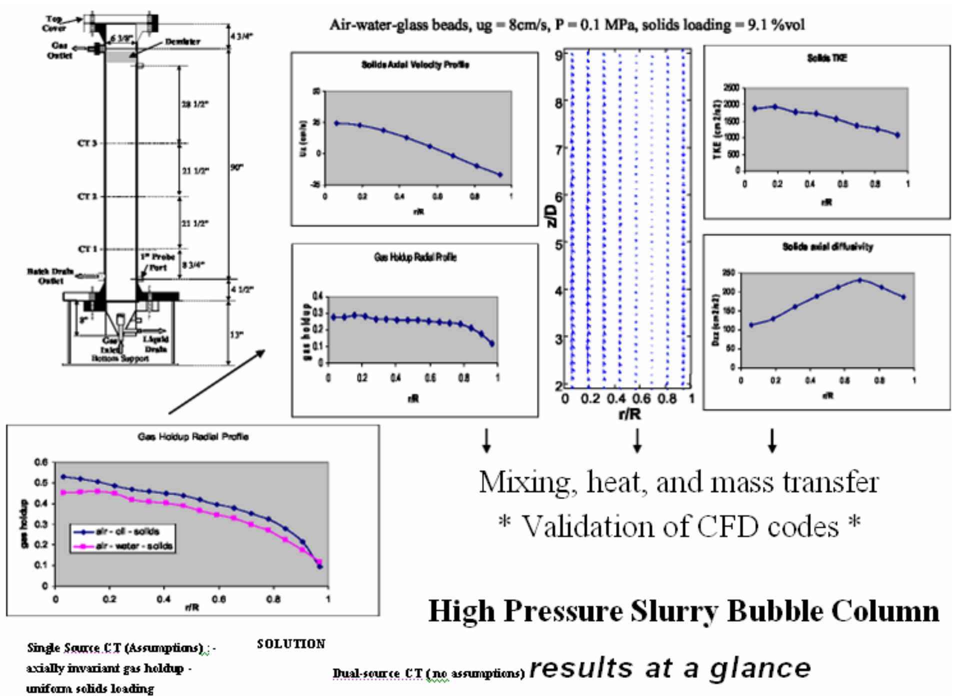

Figure 8: Flow Structure of Slurry Bubble Column obtained by RPT and CT Techniques

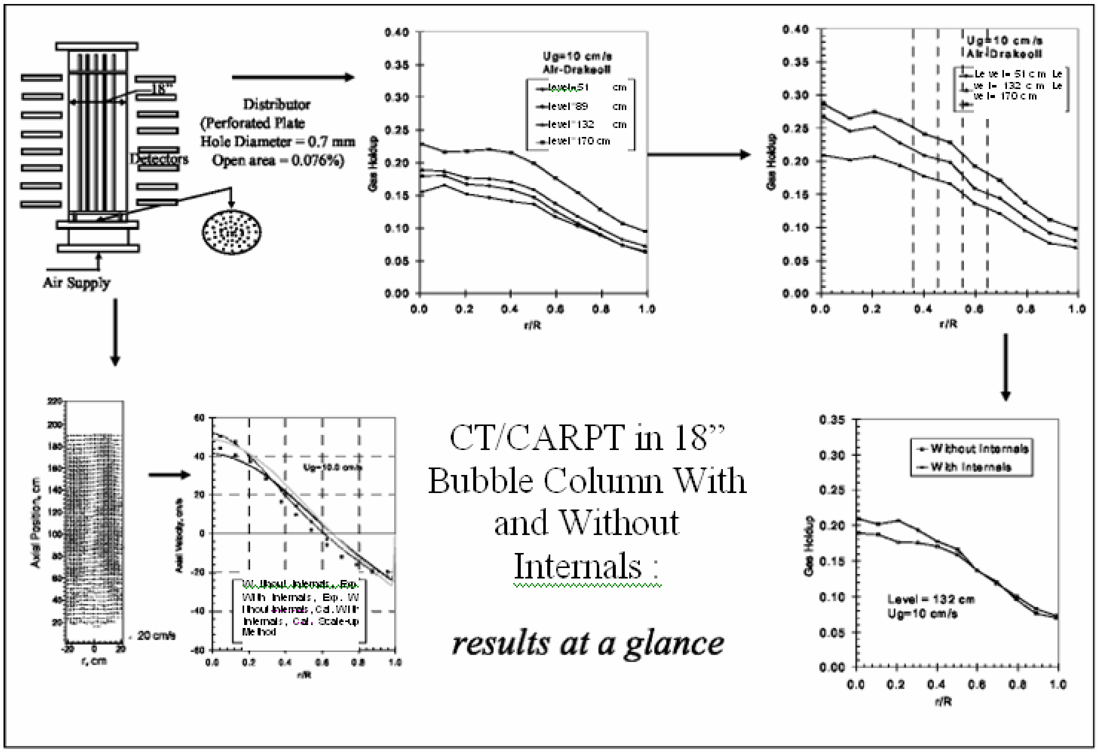

Figure 9: Flow Structure of Bubble column with and without Heat Exchanging Internals Obtained by RPT and CT Techniques.

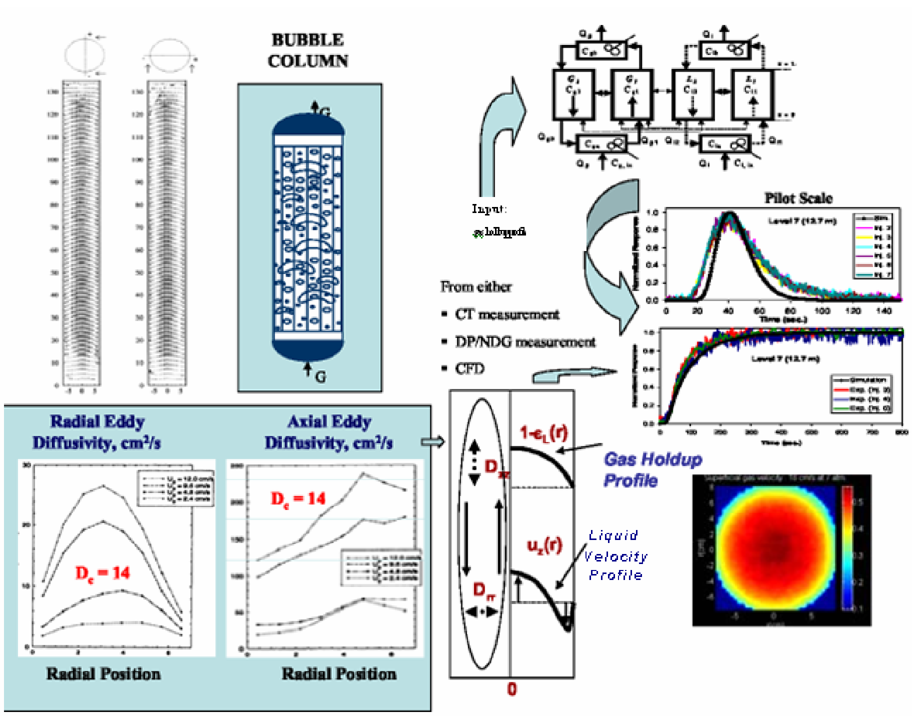

Figure 10: Integrating the Results of Radioactive tracer RTD, Densitometry, CT and RPT Techniques to Advance the Design of Bubble and Slurry Bubble Column for Methanol Synthesis from Syngas (Laporte, Texas, Alternative Fuels Development Unit).

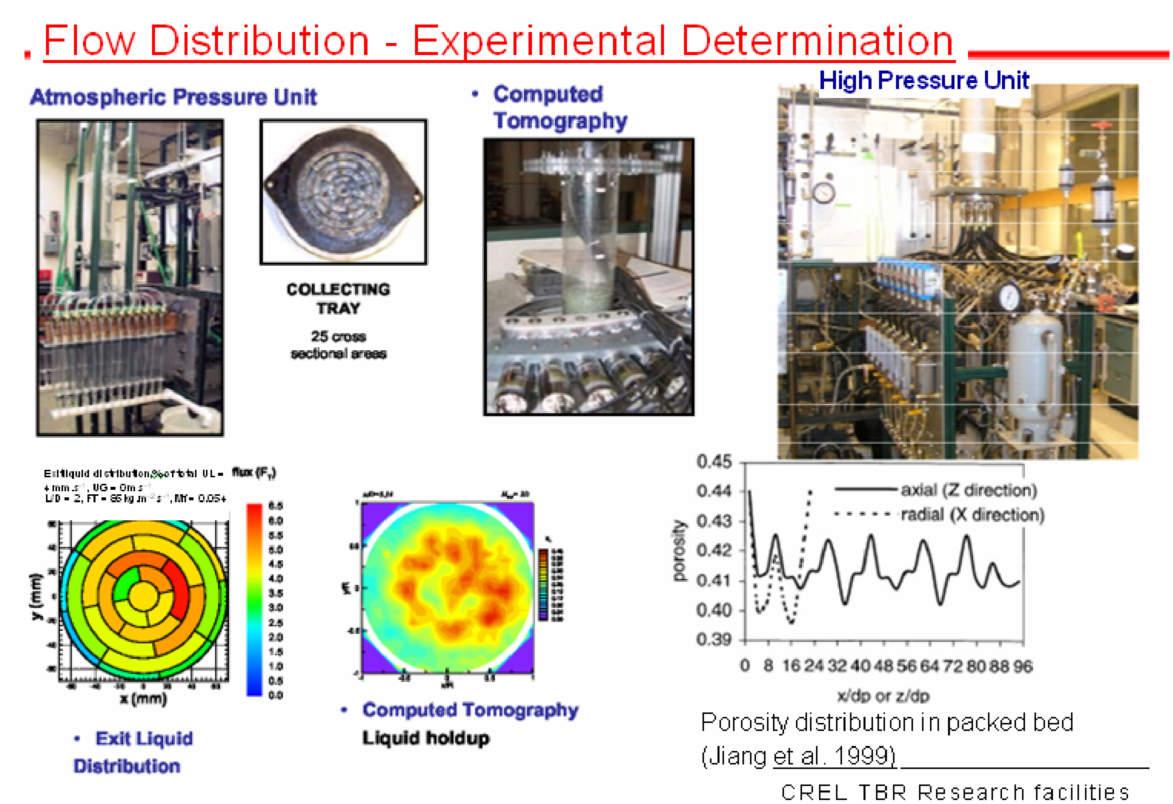

Figure 11: Example Of CT Technique Results in a Trickle-Bed Reactor and their Comparison with the Liquid Flux Collected at the Bottom.

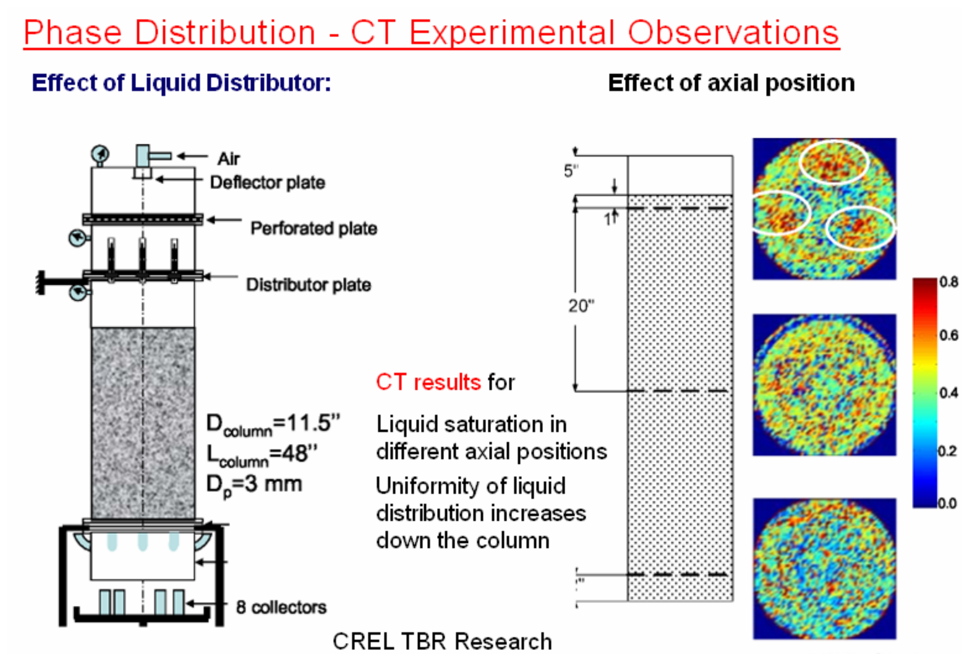

Figure 12: CT Technique application to Study the Distributor Design in a Pilot Plant Scale Trickle-Bed Reactor

Figure 11 shows a sample of results of CT in a trickle bed reactor, while Figure 12 shows the application of CT to study the impact of distributor design on the flow distribution of gas and liquid over the catalyst bed.

7. REMARKS

It is evident that radioisotopes play a major role in advancing the design, scale-up, and performance of various petrochemical processes by furthering the understanding and modeling of their key multiphase flow systems. The radioactive isotope based techniques in general are invaluable tools for laboratory and pilot plant research and for on-site industrial application for diagnostics, monitoring, advanced mechanistic modeling development for performance prediction and process optimization, and for obtaining bench mark data for CFD evaluation and validation. Further investigation and development are needed to advance these techniques, to develop new ones, and to improve their measurements and applications on industrial scale processes.

8. ACKNOWLEDGEMENTS

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