OBJECTIVES OF THE PROJECT

To design, install, test and operate a 0.108 m internal diameter slurry bubble column for heat transfer investigations at ambient conditions employing heat transfer probes of different diameters and a seven-tube bundle. To conduct measurement of heat transfer coefficients for slurries involving solids of different sizes (0-100 μm) and concentrations (0-30 weight percent) in water and Therminol-66, for different air and nitrogen velocities (0-0.30 m/s) and slurry velocities (0-0.01 m/s).

To design, install, test and operate a 0.305 m internal diameter Pyrex glass slurry bubble column and conduct heat transfer measurements up to 523K. To employ single-probe, five-, seven-, and thirty-seven tube bundles for heat transfer investigations. To measure heat transfer coefficients for slurries of water and Therminol-66 involving different size powders (0-100 μm) and concentrations (0-30 weight percent) for different superficial air and nitrogen velocities (0-0.25 m/s).

To conduct related hydrodynamic measurements with a view to characterize the two- and three-phase systems for conditions under which the above mentioned heat transfer measurements will be performed. To accomplish this detailed gas holdup (total and local), limited measurements of bubble size, and visual observations of bubble formation, bubble coalescence and liquid circulation patterns will be undertaken.

To compare these hydrodynamic and heat transfer data with the available data wherever possible, and with correlations and model expressions with a view to provide an assessment of their adequacy for heat transfer coefficient and gas holdup. To develop new correlations, as necessary, for these two properties based on the broad data base generated in this endeavor, to assist scaleup efforts of indirect coal liquefaction industrial plants.

Based on this comprehensive experimental and related theoretical effort, to suggest future research work so that reliable heat exchanger configurations can be designed which will not unfavorably impair the mass transfer, gas conversion and active catalyst life in the bubble column and at the same time will have efficient heat removal characteristics.

ABSTRACT

Two bubble columns (0.108 and 0.305 m internal diameter) were set up and experiments were conducted to determine gas holdup and heat transfer coefficients. These columns were equipped with either single heat transfer probes of different diameters, or bundles of five-, seven- or thirty-seven tubes. The experiments were conducted for two-and three-phase systems; employing for gas phase: air and nitrogen, liquid phase: water and Therminol-66, and solid phase: red iron oxide (1.02, 1.70 and 2.38 μm), glass beads (50.0, 90.0, 119.0 and 143.3 μm), silica sand (65 μm), and magnetite (28.0, 35.7, 46.0, 58.0, 69.0, 90.5, 115.5 and 137.5 μm). The column temperature was varied between 298-523 K, gas velocity between 0 - 40 cm/s, and solids concentration between 0 - 50 weight percent.

The holdup and heat transfer data as a function of operating and system parameters were employed to assess the available correlations and semitheoretical models, and to develop new correlations. Information concerning the design and scale-up of larger units is presented. Specific research work that need to be undertaken to understand the phenomena of heat transfer and gas holdup is outlined so that efficient gas conversion and catalyst usage may be accomplished in slurry bubble columns. The new results obtained in this Department of Energy sponsored research project are published in twenty-eight technical research papers.

1. EXECUTIVE SUMMARY

To accomplish the major objectives of this research program and to understand the heat transfer phenomenon in slurry bubble columns, two experimental facilities have been designed, fabricated, installed, tested and operated using several different systems for a range of operating conditions with the results presented in this final report of the DOE sponsored project.

One such facility consists of a 0.108 m internal diameter vertical bubble column with its associated gas supply, temperature and pressure distribution measuring systems and can be operated in both semi-batch and continuous mode at ambient conditions. Its detailed conceptual design was presented by Saxena and Shrivastav [1], and the final form of test unit by Saxena [2]. The conceptual design of a second and larger 0.305 m internal diameter vertical bubble column with its associated supply and measuring units was reported by Saxena and Vadivel [3] , and that of final test unit by Saxena, Vadivel and Saxena [4]. The data obtained on these two units for different systems and operating variables are described briefly in the following, first for the small column and then for the larger column.

The smaller unit was operated in the semi-batch mode at ambient conditions for the air-water system in different flow regimes (discrete bubbling, bubble coalescing or churn turbulent, and coalesced bubble or slugging) as the air velocity was gradually increased up to 0.38 m/s. The axial local and total air holdup were measured for increasing and decreasing air velocities and the hysteresis effect was observed. The effect of slumped liquid column height was established. The influence on gas holdup of liquid velocity (up to 1.2 cm/s) was also studied. The validity of available correlations was evaluated. Heat transfer coefficient was measured for a 19 mm cylindrical axial heat transfer probe after establishing the axial and radial temperature profiles and its transient behavior. These results were employed to judge the adequacy of five theoretical models and six data sets available in the literature. All these investigations related to gas

*References are listed at the end of this executive summary.

holdup and heat transfer coefficient studies were published in four technical papers [2, 5 - 7].

Using high speed cine-photography and fiber optic probe techniques, we made some exploratory investigations to establish the dependence of bubble diameter on gas velocity and radial position These results published in an article by Saxena, Rao and Saxena [8] revealed that bubble diameter for the air-water system is independent of air velocity in the range 3.6 to 9.2 cm/s. The bubble diameter is smaller at the column wall than at distances farther away from it and closer to the column axis.

Gas holdup was measured in air-water system with single probes of three different diameters, Saxena and Patel [9], a five-tube bundle [10], and a seven-tube bundle [11]. These investigations revealed that baffled columns have larger holdup than for unbaffled columns under otherwise identical conditions. This is consistent with our findings [8] that the bubble size is smaller near the walls in a bubble column than in those portions of the column which are free of immersed surfaces. The influence of slumped liquid column height was found negligible in the range 1.1 to 1.7 m.

Gas holdup was also measured involving a more viscous fluid (119 cP at 293 K) Therminol-66 and the nitrogen holdup was found much smaller than for the less viscous air-water system, Saxena, Rao and Thimmarapuram [12]. The nitrogen holdup was found to increase for a baffled column in comparison to a single tube for otherwise identical conditions, Saxena, Rao and Kagzi [13]. This result is consistent with our above stated findings for the Air - Water system. It was also noted that there is no appreciable hysteresis and slumped liquid height effect on gas holdup. The commonly used correlations were found to predict values which are two- to four-fold larger than the experimental values both for three single tubes of different diameters and for the seven-tube bundle except for the correlation of Smith et al. which reproduces the data well at low velocities but the deviations increase up to 40% at the highest gas velocity, 0.26 m/s.

Experiments were conducted with air-water-red iron oxide (1.02 and 2.38 μm) system and a 19 mm single probe, Saxena, Vadivel and Saxena [14], for air velocity range up to 0.36 m/s and slurry concentrations up to 30 wt%. The gas holdup decreased with slurry concentration and increased with air velocity. The particle diameter and hysteresis effects were negligibly small. Experiments were also conducted under somewhat similar conditions with three single tubes of different diameters and a seven-tube bundle for the nitrogen-Therminol-red iron oxide system, Saxena, Rao and Kagzi [13]. In all cases foaming was absent, gas holdup increased with increase in gas velocity, effect of slurry concentration was negligible, and single tube data were slightly greater than the corresponding data for the seven-tube bundle. Theoretical models over estimated the holdup data by about 200 to 300 % except for Smith et al. correlation which reproduced the data satisfactorily. A modified approach based on the drift-flux theory successfully correlated the data.

Saxena, Verma, Vadivel and Saxena [15] examined the air-water-glass bead system for the 19 mm single tube and for slurries of three different particle sizes and concentrations ranging up to 30 %. Hysteresis effect was present for the slurry concentration till about 10 wt% but was absent at higher concentrations. The holdup decreased with increase of solids proportion in the slurry. Experiments of similar scope have also been conducted with three single-tube internals, Saxena and Patel [9], and with a seven-tube bundle Saxena and Patel [16]. Tube diameter effect on holdup was found negligible but for the seven-tube bundle the holdup was greater than the single tube data in conformity with the corresponding two-phase system.

Extensive experiments were conducted for the air-water-magnetite system involving magnetite powders of six different sizes (35.7, 58.0, 69.0, 90.5, 115.5 and 137.5 μm), slurry concentrations up to 30 weight percent, and air velocities up to 0.33 m/s, Saxena, Rao and Saxena [17, 18]. These measurements revealed that the air holdup increased with air velocity, had almost no dependence on slurry concentration, and is feebly dependent on particle size; having a constant value for particles < 100 μm and somewhat lower values for particles > 100 μm. Important conclusions were drawn regarding the appropriateness of different theoretical correlations. Experiments of similar nature were also performed with a seven-tube bundle, Saxena, Rao and Patel [11]. It was found that the presence of solids decreased the holdup, and the decrease was more with the increase of particle size in the slurry. The air holdup values with bundle were usually larger than for a single tube.

Gas holdup for the nitrogen-Therminol-magnetite (27.7, 36.6 and 46 μm) system was investigated with single probes of three different diameters (19.0, 31.8 and 50.8 mm) and a seven-tube bundle, Saxena, Rao and Yousuf [19]. There was no foaming and the hysteresis effect was absent. These results suggested negligible influence of tube diameter, slurry concentration and particle size. The models failed to reproduce the data, overpredicting the experimental values up to 300 percent. The drift-flux theory, treating the rise velocity of a single bubble in an infinite medium as an adjustable parameter, has proven successful in correlating the data.

Now a brief review of the heat transfer measurements conducted in the small bubble column will be presented. Heat transfer coefficients were measured for the air-water system using a 19 mm axial probe with the heated elements located in three different regions of the bubble column and for the air velocity range up to 0.37 m/s, Saxena [2], and Saxena and Rao, [7]. The heat transfer rates were the same along the entire column length and were dependent on gas velocity, increasing rapidly in the beginning and became almost constant for velocities greater than 0.1 m/s. It was found that literature heat transfer coefficient data from six different sources were sufficiently different amongst themselves and also from the present data. Reasons for these discrepancies were given. The data were also compared with six available models and their inadequacies were discussed. Data were also taken for the five-tube bundle, Saxena, Vadivel and Verma [5], and it was found that heat transfer coefficients were appreciably greater (20-30 %) for the tubebundle than for the single tube. Similar results were also found for the seven-tube bundle in contrast to the single tube results, the heat transfer rates were greater in the middle and upper regions of the column as compared to the lower region. There was no radial dependence of heat transfer coefficients in the middle and upper column regions.

Experimental results for the nitrogen-Therminol system for the three single probes and a seven-tube bundle were obtained similar to air-water system. Values for the latter system were about an order of magnitude larger than for the former. Also in contrast to the air-water system, nitrogen-Therminol system data for the seven-tube bundle were appreciably smaller than for the single tube data over the entire velocity range, up to 0.26 m/s. Data were not reliably predicted by the models.

Heat transfer coefficients were measured for the air-water-red iron oxide (1.02 and 2.38 μm) system for the 19 mm probe and slurry concentrations ranging up to 30 wt%. For both the powders, it was found that the heat transfer coefficient decreased with increase in slurry concentration. In this range the influence of particle diameter was negligible. The available theoretical expressions could not predict the experimental data, Saxena, Rao and Saxena [20]. Saxena and Patel [21] measured heat transfer coefficients for this system and a seven-tube bundle under similar conditions. The heat transfer coefficients decreased only slightly with increase in slurry concentration, but increased appreciably in comparison to the single-tube values, and the particle diameter effect was negligible. Values for the nitrogen-Therminol-red iron oxide system were found to be an order of magnitude smaller than for the air-water-red iron oxide. For the three single tubes of different diameters and a seven-tube bundle, heat transfer coefficient was found to increase with increase in solids concentration (up to 50 wt%) and gas velocity. The values for the tube bundle were smaller than for the single tubes. The models were found inadequate to represent the data on the whole and an empirical approach successfully correlated the data.

Saxena, Verma, Vadivel and Saxena [15] measured the heat transfer coefficients between a 19mm probe and a dispersion of air-water-glass bead system involving slurries of three powders (50.0, 117.6 and 143.3 mm) and for different concentrations. These data could not be reproduced by the commonly used theoretical models and were correlated by a semitheoretical model, Saxena, Saxena and Rao [22]. Saxena and Patel [16] measured heat transfer coefficients for the same system and conditions but for a seven-tube bundle. It was found that the values were almost independent of particle diameter and slurry concentration, and were almost identical with the single tube values except for the smallest particle, 50μm.

Heat transfer coefficients for the air-water-magnetite system were measured for the 19 mm probe and slurries of six different size powders in the concentration range up to 30%. Heat transfer coefficients were independent of the slurry concentration and were only slightly greater than the corresponding two-phase values. Particle size of the powder had a small involved dependence. Available correlations were examined but none could correlate the data adequately over the entire range, Saxena, Rao and Saxena [18]. Experiments conducted with a seven-tube bundle indicated very little dependence on particle size, slurry concentration, and were somewhat greater than the single tube results. Nitrogen-Therminol-magnetite system heat transfer rates were an order of magnitude smaller than for the corresponding air-water-magnetite results. Heat transfer coefficients increased with nitrogen velocity (up to 0.2 m/s), and with slurry concentration (up to 50 wt%), and were almost independent of mean particle diameter (27-46 μm). The single tube (19 mm) values were about 30% greater than the corresponding seven-tube bundle results. Models other than that of Deckwer et al. underpredicted the heat transfer data. A correlation was proposed along the same lines as for the other two- and three-phase systems.

The gas holdup and heat transfer data taken on the larger 0.305 m diameter bubble column, will be discussed now, first the holdup and then the heat transfer data. Air holdup was measured for the air-water system, Saxena, Vadivel and Saxena [4], for air velocities up to 0.38 m/s and for different slumped heights. The air holdup increased with increase in air velocity and decreased with increase in slumped height. Hughmark's correlation approximately reproduced the data. Saxena, Rao and Saxena [23] measured the holdup for this system at four temperatures in the range 297-343 K. Gas holdup showed no hysteresis effect at temperatures of 311K and above, holdup decreased with increase in temperature first rapidly, and exhibited little variation in the range 323-343 K. The data were compared with seven correlations which were found inadequate to represent the temperature dependence. Saxena, Rao and Saxena [24] measured holdup for a single tube, five- and seven-tube bundles at temperatures in the range of 297-343 K. At 297 K, the single-tube data were smaller than five- and seven-tube data while the latter two were almost identical. At higher temperatures the five- and seven-tube bundle data were almost identical. Saxena, Thimmapuram and Rao [25] have measured holdup for the thirty-seven tube bundle and air-water-glass bead system in the temperature range, 298-353 K and for air velocities ranging up to 0.18 m/s. At all temperatures, the holdup was significantly greater than the corresponding values for the single-, five- and seven-tube bundles. This clearly demonstrates the importance of internals in splitting bubbles in baffled bubble columns and thereby influencing the holdup in bubble columns. The available correlations could not reproduce the data while the drift-flux theory approach was most successful in correlating the data.

For the thirty-seven tube bundle arrangement, the gas holdup was measured by Saxena, Rao and Khan [26] for the nitrogen-Therminol system at temperatures 296-523K for the nitrogen velocity values up to 0.14 m/s. At a given temperature, the nitrogen holdup increased with nitrogen velocity; while it decreased with increase in temperature first up to about 309 K and increased with increase in temperature up to 428 K, and thereafter remained constant. At temperatures above 423 K, appreciable foaming and hysteresis effects were observed while for temperatures below 423 K these were negligibly small.

For the three-phase air-water-glass bead system, gas holdup measurements were taken [23] for the seven-tube bundle in the temperature and air velocity ranges of 297-343 K and 0-0.28 m/s. Powders of mean sizes 50.0, 90.0 and 143.3 μm were used in making slurries of concentrations up to 20 wt%. Gas holdup increased with increasing gas velocities, was almost independent of temperature and slurry concentration for the two smaller size powders. For the largest size powder, the temperature effect became less pronounced as the temperature was increased for slurries of 10 and 20 wt% while it was significant for 5 wt%. None of the correlations could reproduce the data.

The gas holdup data for the air-water-sand (65 μm) system were taken by Saxena, Rao and Saxena [24] at three temperatures (297, 323 and 343 K) and two slurry concentrations (5 and 10 wt %) in the gas velocity range up to 0.26 m/s. Most of the holdup data were found to be independent of slurry concentration, temperature, but increased with increase in air velocity. The data were not reproduced by the available correlations and were synthesized by the modified version of the drift-flux theory.

Saxena, Rao and Saxena [27] measured the holdup for the air-water-magnetite (50.0 and 90.0 μm) system at temperatures of 297, 323, and 353 K for slurry concentrations of 3, 5 and 10 wt%. The holdup values increased with increase in air velocity, decreased with increase in temperature, and were almost independent of particle size and slurry concentration. These could not be represented by most of available correlations but the modified drift-flux theory approach did successfully represent the data. Saxena, Rao and Khan [26] measured data for nitrogen-Therminol-magnetite system covering the ranges of gas velocity up to 0.14 m/s, temperatures 296-523 K, and solids concentrations(for mean particle diameter of about 36 μm) up to 40 wt%. This study revealed that holdup increased with nitrogen velocity, and also with temperature up to about 428 K, but remained constant thereafter. It decreased with slurry concentration at 298 K, remained constant with temperature up to about 378 K, and decreased thereafter up to 523 K.

The heat transfer data for the two-phase systems [4, 23-27] (air-water, and nitrogen-Therminol) were dependent on system properties and the values for the latter system were almost an order of magnitude smaller than the former. The data for the two systems revealed that heat transfer coefficient increased rapidly in the beginning and finally attained a constant value as the gas velocity was increased. For both the systems, heat transfer coefficient increased with temperature primarily because of the decrease in the viscosity. The available models could not correlate the data and the disagreement was more pronounced at higher temperatures. The air-water system with a single 19 mm probe revealed that heat transfer rates are larger in the upper section of the column as compared to the lower section. The air-water system data were successfully synthesized on the basis of an empirical approach. The thirty-seven tube bundle data agreed well with the seven-tube data for the air-water. For Therminol-nitrogen system with thirty-seven tubes, heat transfer coefficients have been measured for different radial and axial positions.

The heat transfer data for the air-water-glass bead system [23] revealed that heat transfer coefficient increased with increase in air velocity and acquired a constant value beyond 0.1 m/s, was independent of slurry concentration (0-20 wt%), and particle diameter (50-143 μm), and increased with increase in temperature. The data could not be correlated with models and the disagreement increased with increase in temperature. The air-water-silica sand system data [24] suggested that heat transfer coefficient were not dependent upon solids concentration (up to 10 wt%), but increased with increasing temperature and gas velocity. Models again failed to reproduce the data while our empirical approach accomplished this goal. Air-water-magnetite system [27] investigated for a seven-tube bundle demonstrated that heat transfer coefficients were not dependent on solids concentration (up to 10 wt%) and particle size (50 and 90 μm). The values, however, increased with temperature and gas velocity. Models failed to correlate the data but the empirical approach did accomplish this goal. For the nitrogen-Therminol-magnetite system [26], the heat transfer coefficients increased with increase in nitrogen velocity (up to 0.14 m/s) and temperature (up to 523 K), but increased only moderately with slurry concentration up to 40 wt%. The semi-empirical approach successfully correlated the heat transfer coefficient data. Interesting conclusions concerning the scaleup of bubble columns are outlined by Saxena [28] from the data obtained of these two properties holdup and heat transfer coefficient on the two columns (0.108 and 0.305 m internal diameter) operating either with and without baffles (internals) simulating the heat exchanger tube bundles.

The major conclusions of this experimental research program are briefly summarized in the following. The heat transfer work done with two liquids and relatively high viscosity values has revealed that heat transfer coefficient increases with gas velocity but approaches to a constant value in the fully developed churn-turbulent flow regime. For viscous fluids the heat transfer coefficient decreases with increase in viscosity and this decrease is more than an order of magnitude. The heat transfer coefficient increases rapidly with temperature under otherwise identical operating and system conditions. With internals present in the column, heat transfer coefficient increases and this increase is related with the changes that internals bring about in the column, particularly in relation to liquid circulation and mixing patterns. This observation is corroborated with limited direct measurements of bubble diameters and their size distribution using the photographic and fiber optic probe techniques. More work in this direction will be useful in understanding the mechanism of heat transfer on immersed surfaces in a bubble column. To further assist in this direction an experimental technique has been developed to measure the temperature-history of an element of immersed surface and the same is employed to explain several observed phenomena in slurry bubble columns. There is a small variation of heat transfer coefficient in axial and radial directions in a bubble column and it is related with the design of gas sparger. However, a distinct increase in heat transfer coefficient is observed in the larger diameter column (0.305m) as compared to a laboratory scale model (0.108 m) and this is due to the better liquid mixing which is achieved in larger size columns.

Several interesting observations are possible on the data generated for three-phase systems involving solids of different physical properties in varying concentrations. In general, the three-phase systems have somewhat similar qualitative variations, as for the two-phase systems, with changes in physical operating parameters. Quantitatively, the differences are appreciable and these must be included in any careful design work. The heat transfer coefficient increases monotonically with increase in gas velocity and it approaches a constant value at sufficiently high gas velocities. The heat transfer coefficient increases with increase in temperature, solids concentration, and increase in column diameter. The size of the particles in the powder has different influence depending upon their size. The smaller particles influence heat transfer more through their influence on the viscosity of the suspension while larger particles (greater than 100 μm) enhance the heat transfer process by direct participation. Most of our data are generated in the semi-batch mode of operation (liquid flow velocity is zero) as our limited work conducted for both two- and three-phase systems in the continuous mode revealed that heat transfer coefficient values are negligibly influenced as long as the liquid or slurry velocity is about one cm per second or smaller.

The existing correlations and model based semi-theoretical expressions fail to predict the heat transfer process both qualitatively as well as quantitatively. Only an empirical approach developed on the basis of our experimental data has successfully correlated the data. However, more research work is needed to justify it and develop suitable formulations of the empirical constants in terms of the system properties and operating parameters. The data base generated in this experimental program has provided a firm basis to undertake such a research program.

The gas-phase holdup is also measured in these investigations and is regarded as one of the basis to characterize the systems for which we have reported the heat transfer data. It is found that the holdup increases with increase in gas velocity and hysteresis effect is encountered in the data. Hence majority of the data are taken for decreasing gas velocity mode. Liquid phase velocity, in the range up to 1 cm/s, influences the gas-phase holdup negligibly. The bubble dynamics is important and influences the holdup in different ways depending upon the bubble column internal configurations and rheology of the suspension. Thus, the axial phase variation, presence of solids in the column, configuration of internals, concentration of solids and their size distribution, physical properties of the liquid, column temperature, all influence the gas-phase holdup. These variations have been adequately explained on this concept and have been synthesized on a modified version of the drift-flux theory. More work needs to be done to realistically substantiate the parameters of this model derived on the basis of experimental data. However, this approach is considered significant in view of the fact that all available models and correlations fail both qualitatively and quantitatively to represent and reproduce the experimental findings of the current effort.

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