

Gas-Liquid-Solid Volumetric Phase Distribution Estimation in a Cold Slurry Bubble Column System for Hydro-Conversion Processes

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Abstract Hydro-cracking slurry bubble column design, scale-up, and operation are strongly influenced by a fluid-dynamic parameter known as volumetric phase distribution. This parameter depends on the operating conditions (gas flow, liquid flow, pressure, and temperature) as well as on the gas, liquid, and solid physical properties. Experiments were carried out at ambient temperature and atmosphere pressure (cold conditions) in a 120 mm inner diameter plexiglas column (without any gas sparger) with air and CO₂, mineral oil, and coke with average particle sizes of 630 microns. The column was operated to up-flow continuous recirculation with superficial gas velocities ranging from 3 to 10 cm/s and a constant liquid-solid flow about 29 l/h. Experimental measurements were done by two methods: direct phase trapping and pressure drop. Measurement results indicate that the volumetric gas phase is highly affected by the superficial gas velocity. However, the superficial gas velocity effect on solid concentration is negligible. The experimental results were also compared with experimental data from other authors, obtaining a good agreement. A gas volumetric phase correlation was proposed.

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1 Introduction

Slurry bubble column reactors (SBCR) are used nowadays as part of hydro-cracking units, having a notably importance for heavy crude oil processing. Several advantages of their use include: high heat and mass transfer rates, high conversion levels by strong gas-liquid-solid interactions, and improvement of catalyst recovery processes (Chengtian et al. 2008). Also, these reactors reduce maintenance costs due to the absence of moving parts or without any internal and improved control of the phase residence time inside the equipment (Shah et al. 1982).

The design and scale-up of these equipments is strongly influenced by the fluid-dynamic parameters. Some of the parameters with a major impact are the gas-liquid-solid phase volumetric distribution, the pressure drop, the flow pattern, and the mixing regime. Several of these variables, validated in an operation range and under certain physical conditions, have been cleared through empirical and semi-empirical correlations, which predominate as the bases of slurry bubble column reactor design. However, there is not enough to get from a universal methodology to describe the whole behaviour of a slurry bubble column (Hikita et al. 1981; Akita and Yoshida 1973; Shah et al. 1982). Additionally, process variables, like gas and liquid superficial velocities and material physical properties, influence the fluid-dynamic performance, which is a particular characteristic in each kind of process.

This work deals with the study of the gas-liquid-solid phase distribution based on two physical properties (specific particle range size and gas density) and one operation condition. For hydro-conversion processes, it is relevant to know the gas-liquid-solid volumetric phase distribution since it affects the conversion rate, which can be considered as the most important variable in these units.

2 Methodology and Experimental Setup

2.1 Experimental System and Materials

A schematic diagram of the slurry bubble column (SBC) used in this work is presented in Fig. 1. The core of the system is a plexiglas column of 120 mm internal diameter (ID) with an entrance at the bottom and an exit at the top, without any internal parts. The experimental equipment has a slurry piston pump for the feedstock, a slurry centrifugal pump for recycle, a mechanical agitated tank for the feedstock, and two discharge tanks. The measurement variable system has a mass flow-meter transmitter (FT) for gas Endress + Hauser Promass 83A 4–20 mA output range 0–450 kg/h error $\pm 0.50\%$, a pressure transmitter (PT) ABB 2600T model 261T dual wire 4–20 mA output range 13.7–397 mbar accuracy $\pm 0.15\%$, a pressure differential transmitter (DPT) Rosemount model 115T Smart 4–20 mA output range 0–20 bar error $\pm 0.25\%$, and videographer recorder ABB model Screen Master 200 with four input channel.

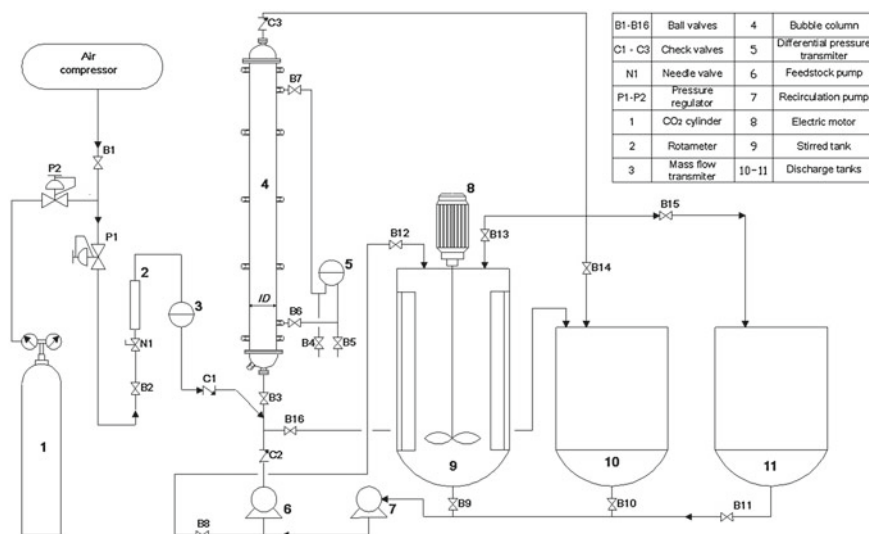


Fig. 1 Schematic diagram of slurry bubble column system

Table 1 Physical properties of fluid phases

Material	Properties		
	Viscosity (cSt)	Density (kg/m ³)	Superficial tension (dyn/cm)
Mineral oil (VASSA)	2.49	815	27.4
Air 25° C; 1 atm	0.018	1.185	
CO ₂ 25° C; 1 atm	0.015	1.808	

Commercial mineral oil VASSA LP-90 was used as the liquid phase because it has low toxicity and due to the fact that its physical properties (density and viscosity) at ambient temperature are similar to those of heavy oil, residual oil, or any other heavy hydrocarbon in operation conditions of hydro-cracking processes. Air and carbon dioxide were used at atmospheric pressure in the injection into the column. The air was supplied from the compression system from pilot plant services, which has about 6.8 bar, and carbon dioxide was supplied from gas cylinders, which have about 55.1 bar, connected to a pressure regulator to make pressure drop to 6.8 bar. The solid phase was ground petroleum coke. The particles sizes were measured by laser scattering of HORIBA device. The properties of fluids and solids are shown in Tables 1 and 2.

Table 2 Physical properties of solid phases

Material	Density (kg/m ³)	Particle size range (μm)	Mean size (μm)	Median size (μm)
Coke	1370	200–1100	629	617

2.2 Experimental Methodology

The unit was operating in recycling mode. In order to start the unit, a concentration of 1.5 wt % of solid with respect to the feedstock was mixed in the stirred tank. Then valves 2, 3, and 4 were opened (see Fig. 1), and after that pumps were started to begin the loading of the column.

The volumetric phase fraction was determined by phase trapping. This method consists in the rapid closing of the inlet and outlet valves of the column. This allows for an instantaneous and direct measure of the gas phase fraction inside the column. In operation, once the column reaches a steady value of pressure drop, the volume of gas phase is determined between the inlet and outlet valves of the column. This method has been used in vertical and horizontal pipes for oil production and transportation (Hagedorn and Brown 1965; Beggs and Brill 1973; Griffith et al. 1975) and also in the study of cold models in bubble column and slurry bubble column reactors (Tai-Ming et al. 1987; Wenge et al. 1995; Jena et al. 2008).

The experimental measurements were done when the stabilization time was reached, defined as the time when the differential pressure, between the fluidized zone, achieved an almost constant value. The estimated stabilization time was 12–16 h, approximately. Once the system has stabilized, the sampling procedure consists in closing the inlet valve just before the lower cone, and simultaneously opening the by-pass valve number 2. After that, it is necessary to keep the column for at least 12 h, until the phases have separated inside it. Finally, the volumes are measured directly through observation.

In addition, during the tests with air/oil/coke particles the gas density effect on the gas volumetric fraction was tested. After each test, the gas phase was switched to CO₂ during 30 min. This time was considered enough to get gas stabilization in this case.

3 Results and Analysis

The gas-liquid-solid phase distribution was determined in a slurry bubble column by means of cold modelling, following the experimental procedure described above. The temperature was kept at 25 ± 2 °C at atmospheric pressure. Figure 2 shows that the gas volume fraction increases with the gas flow. This agrees with the results reported by Akita and Yoshida (1973); Shah et al. (1982); Pino et al. (1990a,b, 1992); Hoefsloot and Krishna (1993); Wenge et al. (1995) and Jena et al. (2008).

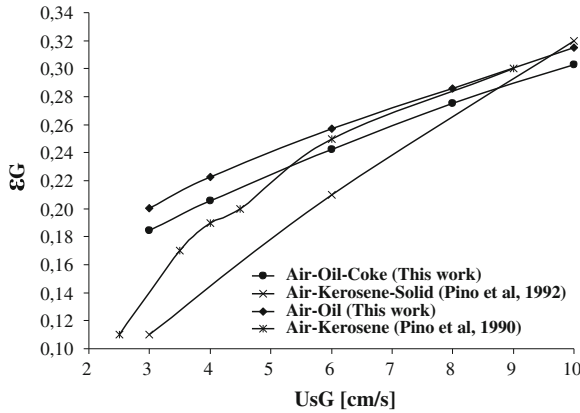


Fig. 2 Average gas volume fraction as a function of superficial gas velocity. The experimental results are compared with other experiments in the literature

The accumulated gas volume holds an average value between 0.17 and 0.30 v/v for all superficial gas velocities and solid phases tested.

Figure 2 also shows comparisons between two and three-phase studies reported by Pino et al. (1990a, 1992) and these experiments. The former authors used a bubble column of 100 mm internal diameter without gas sparger. The gas superficial velocity was varied in the range between 2 and 10 cm/s. In continuous mode operation, the average slurry superficial velocity was 0.5 cm/s. The solid concentration was 126 kg/m^3 with a particle range size of 3–180 microns and density of $4,530 \text{ kg/m}^3$. In the air-oil system mode, the gas volumetric phase is higher than in the air-kerosene system. This behaviour is due to the effect of the superficial liquid velocity, which is higher in the air-kerosene test. However, this effect is rather small in the two systems. On the other hand, the gas volumetric phase in the air-kerosene-solid system is lower than in the air-oil-coke testing. This performance is due to the fact that the solid concentration in the air-kerosene-solid system, is greater than in the air-oil-coke test, which increases the occurrence of bubble coalescence, thus inducing the formation of large bubbles with a high rise velocity and, consequently, a fast disengagement of the column, which then allows the gas volumetric phase to decrease (Kantarci et al. 2005; Behkish et al. 2007). Moreover, a lower average particle size facilitates particle movement throughout the bubble column, thus increasing the area of the solid-liquid contact. This also causes a decrease of the gas volumetric phase.

In Fig. 3, the effect of the gas density on the gas volumetric fraction is shown as a function of the gas velocity for the air/oil/coke and the CO_2 /oil/coke systems. The trend shows that the gas volume increases with the gas velocity a little more (between 1 and 2 %) for the CO_2 system than for the air system, suggesting a small effect of the gas density between these two gases at the operation conditions tested. However, in systems with a pressure higher than atmospheric, it was found that the gas volumetric fraction increased and the number of fine bubbles formed with residence

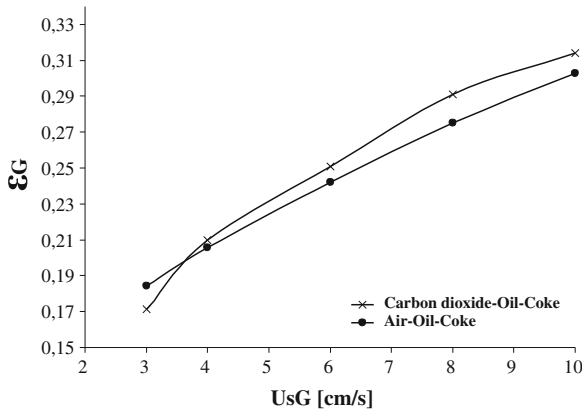


Fig. 3 Effect of the gas density on the gas volume fraction as a function of the superficial gas velocity

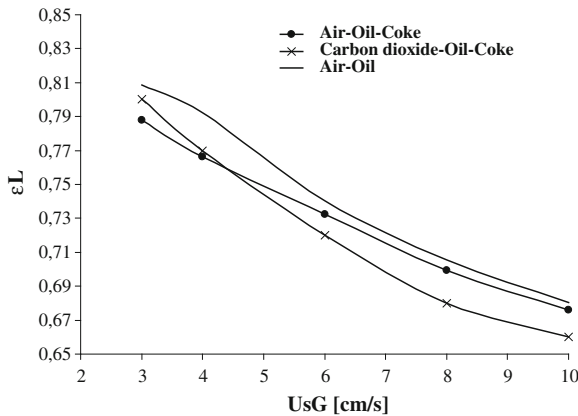


Fig. 4 Average liquid volume fraction as a function of the superficial gas velocity

time in the system was significantly higher. For columns with a pressure higher than atmospheric, the gas density can be affected in a higher proportion (Krishna et al. 1991). A large gas-phase density increases the kinetic energy and momentum of the bubbles, and this leads to an increase of the collision energy which then promotes bubble rupture (Inga and Morsi 1999). These parameters could increase appreciably the gas volumetric fraction.

According to the data plotted in Fig.4, the average volume fraction of liquid decreases from 0.79 to 0.68 v/v as the gas velocity increases from 3 to 10 cm/s. There is a difference in the average of the liquid volume between 1 and 2%, for the CO₂ system when compared with the air system, for gas superficial velocities of 6, 8, and 10 cm/s. This effect is, however, smaller at lower gas velocities of 3 and 4 cm/s.

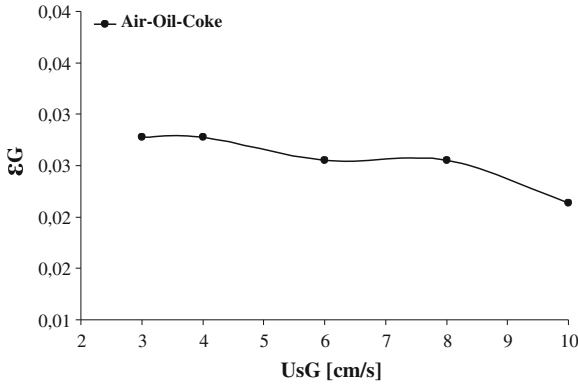


Fig. 5 Average solid volume fraction as a function of the superficial gas velocity

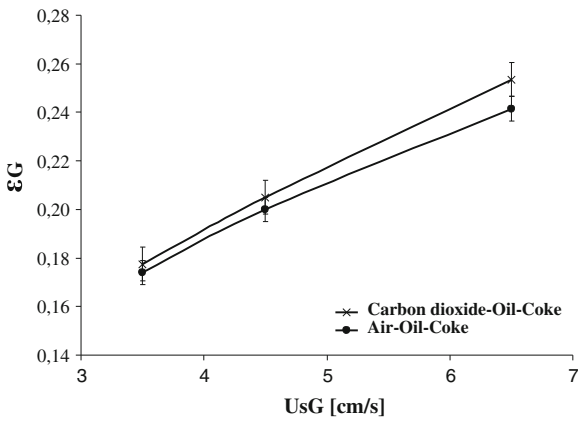


Fig. 6 Average gas volume fraction as a function of the superficial gas velocity with error bars

Looking at Fig. 5, we may see that the solid volume also decreases as the superficial gas velocity increases in a range between 0.03 and 0.02 v/v for carbon particles at the gas superficial velocity tested. The low accumulation of coke inside the column, for all gas superficial velocities tested here, indicates a high solid drag exerted by the liquid. Moreover, due to the low solid concentration inside the column, the influence on the average volume fractions of liquid is small. However, the solids might cause an antifoaming effect, decreasing the gas volume and increasing the liquid volume in the column (Guitian and Joseph 1997).

Figure 6 shows the error bars with three experimental points for the air/oil/coke and the CO₂/oil/coke tests. There can be seen a standard deviation of 0.5 % for the air/oil/coke test and of 0.7 % for the CO₂/oil/coke test. From the analysis of the experimental data obtained the following correlation was developed for the three-phase systems:

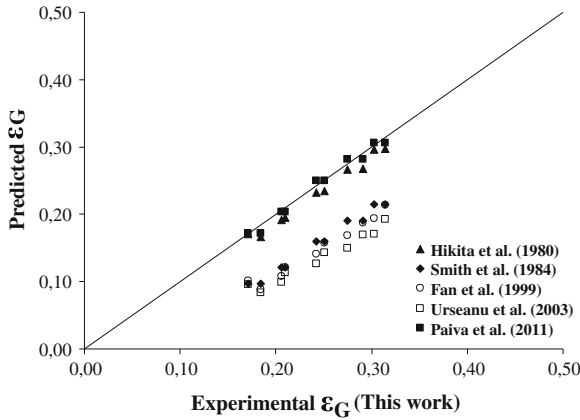


Fig. 7 Parity between the experimental (this work) and predicted average gas volume fractions

$$\varepsilon_G = 1.115 \times 10^{-1} \ln(U_s G) + \left(\frac{U_s G + \rho_s}{dp} \right)^{5.993 \times 10^{-3}} - 9.549 \times 10^{-1}.$$

This correlation takes into account particle properties, which appear to improve the correlation between the experimental and predicted gas volume fractions. It has an average error of 2 %, a standard deviation of 1 %, and a maximum error of 4 %, which is lower than the values obtained from other correlations in the literature as, for example, those reported by Hikita et al. (1980); Smith et al. (1984); Fan et al. (1999) and Urseanu et al. (2003) for different systems. Hikita et al. (1980) correlation had the lower average error (5 %), assuming for the gas volume fraction calculation that the slurry properties were given by the liquid properties, because their correlation applies only to two-phase systems (i.e., gas-liquid). As expected, from Fig. 7 we may see that the parity for the correlation developed here is higher than for the other correlations.

Finally, Fig. 8 shows a parity graph between the correlation obtained in this work and the experimental data of Pino et al. (1992). This latter correlation has an average error of 34.5 % in this case. This occurs because there are appreciable differences in the solid particle characteristics, the concentration of particles, and the superficial liquid velocity between both sets of data.

4 Conclusions

The gas, liquid, and solid phase distribution was estimated in a bubble column under cold conditions. It was found that the gas density does not affect significantly the volumetric fractions of gas and liquid. The low solid concentration obtained during the experiments denotes a high drag exerted by the liquid and a small effect of the

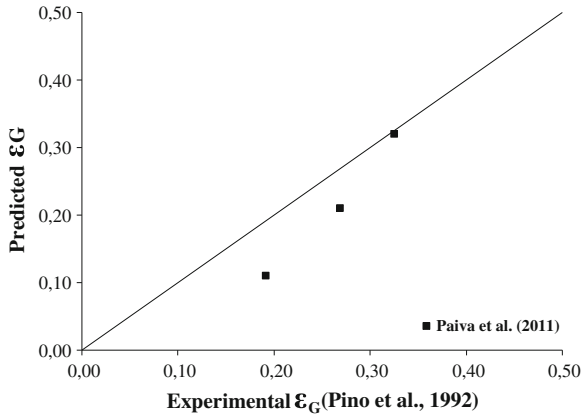


Fig. 8 Parity between experimental average gas volume fractions of Pino et al. (1992) and the predicted ones

superficial gas velocity on solid concentration. On the other hand, the comparison between two-phase and three-phase systems along with experimental data from the literature indicates that in systems with a tendency to foaming, the solids, even in small amounts, reduce the gas volume fraction. A new correlation for predicting the gas volume fraction in three-phase systems was developed, which has shown good agreement with experimental data that takes into account particle properties.

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