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NOTE

Liquid Holdup Measurement Techniques in Laboratory High Pressure Trickle Bed Reactors

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Three different techniques, which are tracer, drainage and weighing methods, are used to measure liquid holdup in a laboratory trickle-bed reactor operated under high pressure. The holdup measurements are compared to determine the applicability of these methods at high pressure operation. It was found that tracer and drainage techniques give comparable values for the total liquid holdup. Although several investigators recommended the weighing method over the others based on their experiments performed at atmospheric conditions, it was found that the weighing method failed to measure liquid holdup properly at high pressure operation.

On a eu recours à trois techniques différentes - traçage, drainage et pesage - pour mesurer la rétention liquide dans un réacteur à lit ruisselant de laboratoire fonctionnant à pression élevée. Les mesures de rétention ont été comparées afin de déterminer l'applicabilité de ces méthodes pour un fonctionnement à pression élevée. On a trouvé que les techniques de traçage et de drainage donnaient des valeurs comparables pour la rétention liquide totale. Bien que plusieurs chercheurs recommandent plutôt la méthode de pesage d'après leurs expériences réalisées à des conditions atmosphériques, on a trouvé que la méthode de pesage ne mesurait pas convenablement la rétention liquide à une pression élevée.

Keywords: measurement techniques, liquid holdup, high pressure trickle bed reactors.

Trickle bed reactors are fixed beds of solid catalyst particles contacted by cocurrent downflow of gas and liquid. They are used widely in industry and are usually operated at high pressure (up to 20 or 30 MPa). Liquid holdup is an important parameter for the design, scale-up and operation of trickle bed reactors. The total liquid holdup (ε_{Lt}) (the ratio of the amount of liquid in the bed at any time to the volume of the empty reactor) can be divided into two types: external and internal. Internal liquid holdup (ε_{Lint}) is the ratio of the volume of the liquid held by capillary forces in the pores of porous catalysts to the reactor volume (usually the catalyst pores are filled with liquid due to capillary forces, especially when the bed is flooded before operation), while the external liquid holdup (ε_{Lext}) is the ratio of the volume of the liquid outside the catalyst pores partially occupying the void volume of the bed to the reactor volume. The external liquid holdup can further be divided into dynamic (ε_{Ld}) and static (ε_{Ls}) liquid holdup. The dynamic liquid holdup is the free flowing fraction of the liquid which is usually measured as the ratio, to the reactor volume, of the volume of liquid that would drain from the reactor when the inlets to the reactor are shut off simultaneously and the liquid is allowed to drain until it stops draining freely. In such measurement however, the name so-called dynamic holdup is not accurate since part of the remaining external liquid which contributes to the dynamic holdup during the normal operation is not accounted for. The dynamic liquid holdzup is dependent upon the hydrodynamics of the flow. The static liquid holdup is the ratio of the volume of stagnant liquid and the liquid retained between and around the contact points of the catalyst particles after draining to the reactor volume. It is independent of the reactor pressure, gas flow rate and liquid flow rate. However, it depends on liquid physical properties, particle shape, size and wettability (Al-Dahhan, 1993; Wammes, et al., 1991; Charpentier, et al., 1968; Shulman, et al., 1955). It is called static holdup, although part of it would contribute to the dynamic holdup. However, liquid holdup sometimes is called liquid saturation, which is based on the void volume of the reactor. For example, the external liquid saturation (β_{Lext}) is defined as the ratio of the volume of liquid present outside the catalyst particles to the void volume of the reactor. The relationship between the various types of holdups can be described by the following equations:

$$\varepsilon_{Lt} = \varepsilon_{Lext} + \varepsilon_{Lint} = \varepsilon_{Ls} + \varepsilon_{Ld} + \varepsilon_{Lint} \quad \ldots\ldots\ldots\ldots \text{(1a)}$$

$$\varepsilon_{Lext} = \varepsilon\, \beta_{Lext} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(1b)}$$

Both dynamic and static liquid hold up have been widely studied in the literature and many correlations have been proposed for their prediction at atmospheric and high pressure operation (Bennett and Goodridge, 1970; Schubert, et al., 1986; Levec, et al., 1986; Saez, et al., 1991; Wammes, et al., 1991; Larachi, et al., 1991,a; Tukac and Hanika, 1992; Holub, et al., 1992, 1993; Mao, et al., 1993; Al-Dahhan, 1993; Al-Dahhan and Dudukovic, 1994; Tsamatsoulis and Papayannakos, 1994; Urrutia, et al., 1996, Al-Dahhan et al., 1997, 1998; Saroha and Nigam, 1996 and many others).

Different techniques have been used to measure liquid holdup in laboratory trickle bed reactors. They can be divided into integral, semi-integral and local measurements methods as follows:

i. Integral Measurement Methods: These methods provide information over the entire volume of the bed.
   - Draining Method. Liquid holdup is measured by draining the liquid when inlets and outlets to the reactor are shut off simultaneously. Both dynamic and static liquid holdup can be measured (Larkins et al., 1961; etc.).
   - Weighing Method. Liquid holdup is measured by weighing the reactor while liquid flows through it and either subtracting from this the weight of dry bed to obtain total holdup or by subtracting the weight of drained bed to obtain dynamic holdup (Crine and Marchot, 1981; Holub, 1990; etc.).

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• Tracer Method. Liquid holdup is measured by measuring the liquid residence time distribution and its mean to obtaing total holdup (El-Hisnawi, 1981; Mills and Dudukovic, 1981; etc.)

• Closed Loop Method. Liquid holdup is measured by circulating in a closed loop of a known amount of liquid through the packed bed. The total holdup is the difference between the loop volume and the volume of the liquid outside the bed (Charpentier et al., 1968)

• Electrical Conductivity Method. Liquid holdup is measured by measuring the apparent electrical conductivity in the presence of a conducting liquid between two electrode positioned at the top and the bottom of the bed or at two positions across the cross section of the bed (Prost and Le Goff, 1964; Achwal and Stepanek, 1975; Le Goff, 1967; Basic, 1992). This method can be used for various applications depending on how to configure and design the experiment. It was also used to measure the liquid texture in trickle bed reactors (Le Goff and Prost, 1966; Chartenpier et al., 1968). Prost (1967) and Blok and Drinkenburg (1982) used such a method to study the pulsing flow regime in trickle bed reactors and to monitor locally the fluctuations of liquid in this regime. The conductivity method can also be used for semi-integral and local holdup measurements (Basic, 1992; etc.).

ii. Semi-Integral Measurement Methods: These methods provide information over a section of the bed and/or for a line-integral over a line drawn through the system. More complete information can be obtained by applying the techniques at many different positions.

• Absorbance Methods. Liquid holdup is measured for example by exposing the bed to a beam of electromagnetic radiation (e.g. X-Ray and γ-Ray) and measuring the attenuation of the beam due to presence of the phases according to Lambert-Beer's law (Hewitt, 1978; Seo and Gidaspow, 1987; etc.). The advantage of these techniques is that they are non-invasive. Detailed discussion about various techniques of this kind and its applications in multiphase flow systems can be found in Chaouki et al. (1997).

• Electrical Conductivity Method. This method as mentioned earlier can be also used for a semi-integral measurement based on the design of the experiment.

iii. Local Measurements Methods: These methods provide a local measurements by inserting a sensor or a probe at a certain position in the bed. They are used based on different physical principles, such as optical signal by utilizing optical fiber probe, absorbance of electromagnatic radiation, electrical conduction, etc. (Hackett and Mann, 1985; Blok and Drinkengurg, 1982; Hewitt, 1978; etc.).

Three of these methods, which are the drainage, tracer and weighing methods, have been used extensively in laboratory trickle bed reactors and they have been performed at either atmospheric pressure (Crine and Marchot, 1981; Tukac and Hanika, 1992; Mao, et al., 1993; Ellman et al., 1990; Holub, et al., 1992, 1993; Urrutia, et al., 1996; etc.) or at elevated pressures (Wammes, et al., 1991; Wammes, 1990; Larachi, et al., 1991,a,b,c; Al-Dahhan, 1993; Al-Dahhan and Dudukovic, 1994; Tsamatsoulis and Papayannakos, 1994).

In this work, these three methods are discussed and the total holdups measured by each method are compared over a wide range of operating conditions, particularly examining their applicability at high pressure operation.

Descriptions of the holdup measurement techniques

TRACER TECHNIQUE

The tracer technique involves measuring the residence time distribution (RTD) in a reactor by analyzing the response curve of a tracer injection (El-Hisnawi, 1981; El-Hisnami et al., 1982; Mills and Dudukovic, 1981; Dudukovic, 1986; Larachi, et al., 1991,a,b,c; Tukac and Hanika, 1992; Mao, et al., 1993; Al-Dahhan, 1993; Tsamatsoulis and Papayannakos, 1994; Al-Dahhan and Dudukovic, 1995, 1996). It has been used often in multiphase systems to provide important information about the flow through these systems, in addition to liquid holdup (Hanratty and Dudukovic, 1990, 1992). Dudukovic (1986) gave a sumary of the techniques of tracer methods and provided examples of their use for packed beds. Typically, an impulse injection of tracer or a step increase or decrease in tracer concentration is used. The impulse injection can be used to find total external holdup (in beds of nonporous particles), the dynamic holdup and the static holdup. However, the step decrease in tracer concentration is considered as a better approach for finding static holdup (Schubert, et al., 1986). By plotting the total external holdup versus the volumetric liquid flow rates and extrapolating to zero liquid flow rate, static liquid holdup can be determined (Bennett and Goodridge, 1970). The concentration of tracer at the outlet is measured, as a function of time, by some signal which is proportional to tracer concentration, such as light absorption, current, voltage, reflection, etc. In order to interpret this signal in terms of the residence times of tracer, 1) the tracer must be nonvolatile and nonadsorbing, 2) it must enter and leave the system only by bulk flow, 3) the tracer injection must be proportional to flow, 4) the rate at which tracer leaves the system must be the integral of the product of the velocity times concentration integrated in a vectorial sense over the whole exit boundary, 5) the system must be at steady state except with respect to the tracer concentration, 6) the response curve must be proportional to the mass of tracer injected, 7) the tracer must behave almost identically to the carrier fluid, 8) there must be only one inlet and outlet, 9) there must only be a single flowing phase and single homogeneous phase in the system and 10) the tracer injection must not perturb the system (Dudukovic, 1996). Also, the liquid volume external to the reactor should be minimized compared to the reactor volume and it is very important to close the tracer mass balance (i.e. account for all of the tracer mass).

For an impulse injection of tracer, the total liquid holdup, ε_{Lt}, can be evaluated as follows (Al-Dahhan and Dudukovic, 1995, 1996; Dudukovic, 1986; Dudukovic and Felder, 1983):

$$\bar{t} = \int_0^\infty t\,E(t)dt = \frac{V_r \varepsilon_{Lt}}{Q} \qquad (2)$$

where:

$$E(t) = \frac{Q\,C(t)}{m_T} \qquad (3)$$

If the tracer concentration is linearly proportional to the output signal of the analytical equipment used, $E(t)$ then can be evaluated directly from the measured signal as:

$$E(t) = \frac{R(t)}{\int_0^\infty R(t)dt} \qquad (4)$$

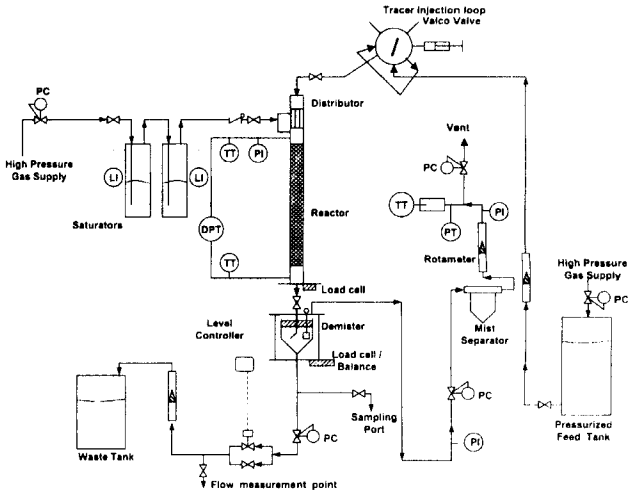
The advantage of the tracer method is that it can be performed without interrupting fluid flow and can easily be used in industrial reactors, but it lends itself to errors if not performed correctly, such as poor injection and collection methods.

Drainage technique

The drainage method is simple and widely used in laboratory scale experiments (Larkins et al., 1961; Goto and Smith, 1975; Crine and Marchot, 1981; Wammes, et al., 1991; Tukac and Hanika, 1992; Mao, et al., 1993; Al-Dahhan, 1993; Al-Dahhan and Dudukovic, 1994; Urrutia, et al., 1996). The drainage method consists of simultaneously shutting off inlet and outlet streams of the reactor when the reactor is at steady state. The liquid that then drains from the reactor when the outlet is opened is the draining holdup. Some researchers refer to this as the dynamic holdup (Urrutia, et al., 1996). It would not be the exact dynamic holdup because part of the liquid remaining in the bed after drainage would contribute to the dynamic holdup if there was still flow through the bed (Al-Dahhan, 1993). The drained bed can then be weighed and the weight of a dry bed is subtracted. This is the weight of the remaining holdup (static and internal holdups). It is generally assumed that the pores of catalysts are completely filled with liquid (due to capillary forces, particularly if the bed was flooded before operation) making the internal holdup and total pore volume equal. By subtracting the total catalyst pore volume from the remaining holdup, the static holdup can be determined. The sum of the static holdup, the draining holdup and the catalyst pore volume is the total liquid holdup. The advantage of this method is its simplicity, as far as laboratory scale experiments are concerned. The disadvantages are that it cannot be performed without disturbing the operation of the system, it can be inaccurate due to measuring small differences between heavy weights (i.e., the wetted and dry reactor) and weighing a reactor (wet or dry) or weighing the draining holdup is impractical to be performed on an industrial scale.

Weighing method

The weighing method, also known as the gravimetric method (Levec, et al., 1986), has been mainly used for laboratory scale trickle bed reactors under atmospheric pressure (Crine and Marchot, 1981; Levec, et al., 1986; Lakota and Levec, 1990; Holub, et al., 1993; Al-Dahhan, 1993). Based on the experimental work performed at atmospheric pressure, this method has been recommended over the methods mentioned previously (Holub, et al., 1993; Levec, et al., 1986; Sato, et al., 1973). In the general weighing method, the reactor is weighed during two phase flow operation, without disturbing the operation and the weight of the dry or drained reactor is subtracted to obtain the total holdup or the dynamic holdup, respectively. The principle of this method is to measure the body force, which is the gravity, acting on the liquid in the bed. The downward body force (or the gravity) is measured by mounting the entire bed on or suspending it from a load cell or a balance (Holub, 1990; Levec et al., 1986; Sato et al., 1973). Crine and Marchot (1981) measured liquid holdup under atmospheric pressure by using an inductive displacement sensor to measure the displacement of the reactor supported on springs working in the linear domain. The dynamic liquid holdup was found by subtracting from the weight of the reactor under operation, the weight of the reactor after interrupting the liquid feed and draining the reactor. The advantage of the weighing method is it can be performed while the system is in operation. Its disadvantage is the inaccuracy of measuring differences between heavy weights, it is difficult to use in industrial practice and it will be shown that the use of this method is questionable under high pressure operation.



DPT: Differential Pressure Transducer, LI: Level Indicator, PC: Pressure Regulator or Back Pressure Regulator, PI: Pressure Indicator, PSV: Pressure Safety Valve, PT: Pressure Transducer, TT: Thermocouple

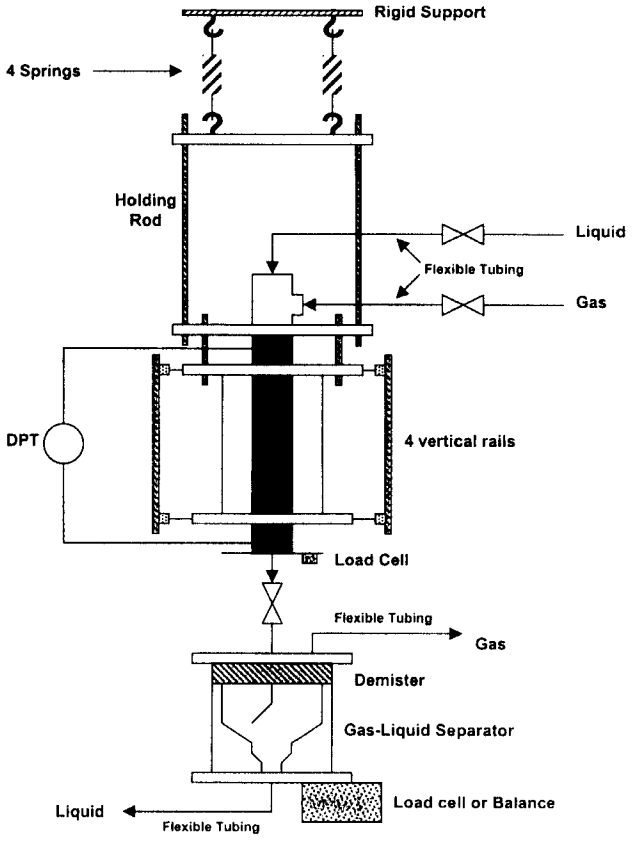


Figure 1 — a. Experimental set-up diagram.
b. Trickle bed reactor arrangement for the implementation of the weighing method.

Experimental setup

The experimental setup, shown in Figure 1a, was used to measure liquid holdup by the three methods discussed above (i.e. tracer, drainig and weighing methods). The set-up can

TABLE 1
The Range of Operating Conditions Studied

Pressure (MPa)	0.31 - 5
Superficial gas velocity (cm/s)	1 - 8.5
Liquid flow rate (cm^3/min)	22.4 - 93.2
L ($kg/m^2 \cdot s$)	0.65 - 4.1
Reactor diameter (cm)	2.2
Bed length (cm)	51.6
Spherical packing d_p (cm)	0.152
Void of its bed, ε_B	0.412
Cylindrical packing $(d_p)_{eq}$ (cm)	0.199
Size (cm)	0.157 × 0.43
Void of its bed, ε_B	0.355

be operated at low to high pressure (up to 7 MPa). The range of the operating conditions used in this study is summarized in Table 1. The liquid phase is distributed via 21 tubes of 1.6 mm inside diameter, while the gas phase is distributed through the space of 0.98 mm surrounding the tubes (21 holes of 3.55 diameter surrounding the tubes) and through 18 holes in between of 0.76 mm diameter (Al-Dahhan, 1993).

The facility was setup to execute the tracer method by using a tracer loop mounted in close proximity to the reactor inlet. The tracer technique used *n*-heptane as a tracer in hexane as a solvent. The tracer response was analyzed using either a differential refractometer (DR, Water 410) for low pressure operation or a gas chromatograph (GC, GOW MAC 69-550) for high pressure operation. A sampling stream was split from the liquid stream exiting from the gas/liquid separator, which was funnel shaped to minimize the liquid volume external to the reactor. This stream was either passed continuously to the DR or used to take samples for injection in the GC. It was found that the DR was unsuitable for use in two phase flow at high pressures due to bubbles generated in its low pressure sample cell; hence, samples were taken and injected manually into the GC.

The drainage method was executed with our system by simultaneously shutting off the liquid and gas inlet valves and the reactor outlet valve. The reactor and its dry bed were weighed a priori. The gas/liquid separator was emptied and the reactor outlet was opened to allow liquid to trickle out. The drained liquid was collected and weighed, to measure its volume. The reactor was then weighed to obtain the remaining holdup (static and internal holdups).

To implement the weighing method in our system, the reactor in use was mounted on four vertical rails system that allowed free vertical movement of the reactor setup with minimal friction as shown in Figure 1b. Flexible tubes were used to connect the reactor/separator to the gas and liquid inlet and outlet lines. The reactor would then rest on either a load cell or on an electronic balance to record the difference between a dry bed and the wetted bed. Because of the difficulty in trying to measure the small difference between two large weights due to liquid holdup, the reactor was counterbalanced by five springs. The springs were attached to a fixed plate that was part of the support structure above the reactor. The other ends were attached to a moving plate further attached to the top of the reactor. By this method, the total weight on the load cell or the balance could be adjusted and reduced.

Comparison of holdup measurements

Comparison between tracer and drainage techniques measurements at elevated pressure

The operating conditions used in this investigation are summarized in Table 1. Figure 2 shows a comparison

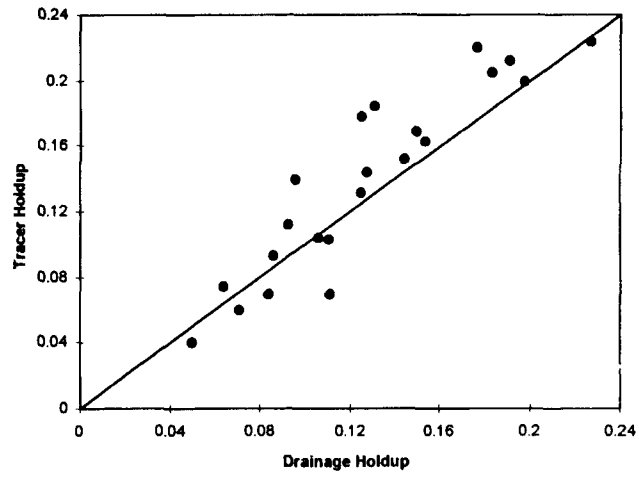


Figure 2 — Holdup values obtained by the tracer and drainage methods at elevated pressure in beds of cylindrical and spherical packing. The operating conditions are listed in Table 1.

between the total holdup measured by tracer method and that measured by drainage method at low to high pressure. It reveals acceptable agreement between these methods at elevated pressure. The comparison is similar to what Tukac and Hanika (1992) found at atmospheric pressure. Some of the liquid holdup obtained by tracer method is larger than that by the draining method. This would be due to a systematic experimental error associated in measuring liquid holdup by either method. It is suspected that the draining method may underestimate static holdup which is obtained by subtracting of weights similar in magnitude while the tracer method would overestimate slightly external holdup. This combined effect would lead to the slight bias of larger holdups measured by tracer method in comparison to the draining method. Such hypotheses for the causes of this discrepancy have not been proven (Al-Dahhan, 1993). However, Kushalkar and Pangarkar (1990) have reported that liquid saturation obtained by tracer technique depends on the form of the inlet curve (i.e. type of the tracer injection). They have shown at atmospheric conditions that the static liquid holdup determined by a tracer technique is always smaller than the value determined by drainage technique (Saroha and Nigam, 1996).

Weighing method measurements at elevated pressure

Although this method has been recommended based on atmospheric experiments (Holub, et al., 1993; Levec, et al., 1986; Sato, et al., 1973; Crine and Marchot, 1981), it fails to accurately measure the liquid holdup at high pressure operation. Crine and Marchot (1981) measured the liquid holdup using both the weighing method and the drainage method at atmospheric pressure and found good agreement between the two methods. In this work, detailed experiments (Table 1) were conducted for single-phase (gas) as well as two-phase (gas–liquid) systems over a range of pressures and only results from selective experiments will be presented here to show the inability of the weighing method to give reliable results under high pressure conditions.

For single-phase flow experiment, nitrogen was used as the gas phase flowing downward in a bed packed with dry, spherical particles. In order to maintain an elevated pressure within the reactor, back pressure regulators are required

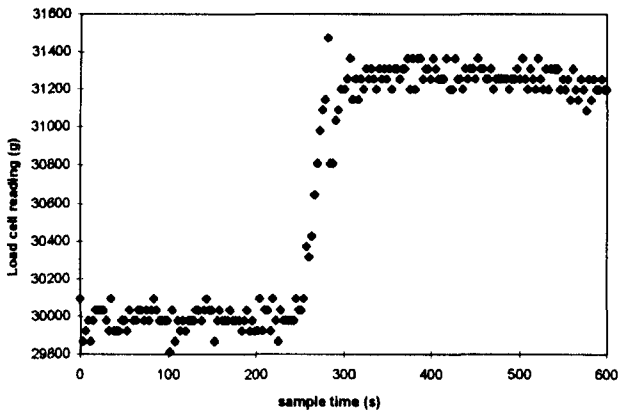


Figure 3 — Increase in the weight by introducing nitrogen gas-only into a dry bed of spherical particles. Pressure: 3.51 MPa (500 psig), Superficial gas velocity: 1 cm/s.

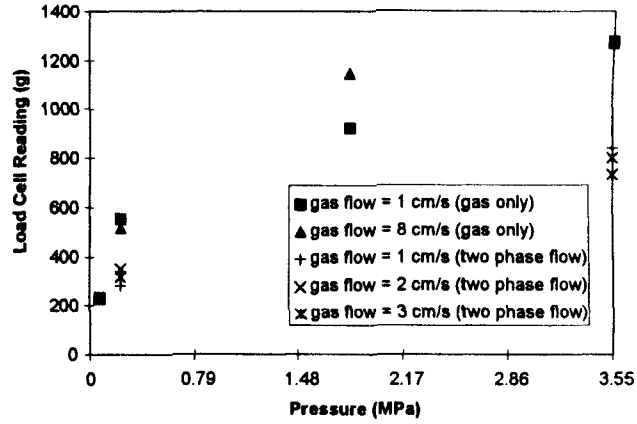


Figure 4 — Total holdups measured by the weighing method at various operating pressures and superficial gas velocities in a bed of spherical particles. Liquid flow rate: 30 cm^3/min.

downstream. In these experiments, the weight of the dry reactor was first recorded. The gas was then introduced at high pressure and the increased weight recorded by the load cell was significantly more than the added mass of the dense gas would induce, as shown in Figure 3. There was no liquid flow at this point. When the gas was introduced at higher pressures and at constant actual superficial velocity (still with no liquid flow), the weight recorded by the load cell was much larger, as shown in Figure 4. This phenomenon seems due to the impingement of the gas inlet jets on the bed in which the gas phase imparting a downward force in addition to the body force when it collides with the packing, a force that increases with gas density, causing the load cell or balance to read a larger value than the mass of the gas would induce (i.e. the body or the gravity force). It is not clear at this time whether by redesigning the distributor and the reactor/separator set-up, or by increasing the size of the inlet pipe and the holes of the distributor, such phenomena would be eliminated or minimized by dissipating the gas inlet jets. When the reactor is operated at atmospheric pressure by opening its bottom to the atmosphere, this phenomenon was not observed.

In two phase flow operation, the following procedure was employed: the bed was soaked in liquid overnight, then it was operated at high liquid and gas flow rates (the high interaction regime), the flow rates were then reduced to the point of interest. This procedure was necessary to avoid hysteresis (Levec et al., 1986; Kan and Greenfield, 1979; Holub, 1990).

Under steady-state operation, the weight recorded by either the load cell or the balance was considerably larger than the addition in weight from the liquid holdup and gas holdup. The typical weight of the static and/or dynamic liquid holdup found using the drainage method in our reactors operated at low to high pressure was in the range of 8.0 to 38.0 g, whereas, by the weighing method, the increased mass of two phase flow over the dry bed was approximately 280 g at the lowest pressures investigated. At higher pressures, the recorded weight is increased to as much as 800 g while the liquid holdup decreases (Al-Dahhan and Dudukovic, 1994). Obviously, this is much larger than any holdups found using the drainage method under similar conditions. However, the recorded weights in two phase flow operation, as shown in Figure 4, is lower than those recorded at gas-only flow rate operation. The difference between the weight recorded in two phase flow and gas-only flow is increased noticeably at high pressure operation. This would be due to that the presence of the liquid flow dissipated the gas jets and hence, reduces the force imparted by the flowing fluids on the system. The phenomena are complex and the causes of such observations are not clear at the moment. Hence, it is not easy to extract the body force that represents only the liquid phase weight from such measurements to obtain a reliable holdup at high pressure operation.

Therefore, weighing method failed to measure liquid holdup properly at high pressure operation for the experimental set-up used in this investigation compared to atmospheric operation and it is not recommended accordingly for high pressure laboratory trickle bed reactors.

Concluding remarks

The tracer technique and the drainage technique give comparable values for the total liquid holdup at high pressures, similar to what was found by Tukac and Hanika (1992) at atmospheric pressure. The tracer technique is a more practical technique on an industrial scale than either the drainage method or the weighing method, due to the tracer technique's capability of being performed without interrupting the flow and the fact that it does not involve weighing any large reactors or liquid volumes. Although Crine and Marchot (1981) found good agreement between weighing and draining methods at atmospheric pressure, the weighing method failed to measure properly liquid holdup at high pressure operation. Hence it is not recommended for use at elevated pressure. It overestimates significantly the liquid holdup due to the force imparted by the flowing fluids on the packing at elevated pressure (high gas density).

Nomenclature

C = tracer concentration at time t out of the reactor, (kg/m^3)
C_0 = tracer concentration at the inlet, (kg/m^3)
d_p = particle diameter, (cm)
E = residence time density function
m_T = mass of tracer injected, (kg)
Q = liquid flow rate through the reactor, (m^3/s)
R = signal response of analytical equipment (i.e. voltage, light absorption, etc.)
V_r = volume of reactor, (m^3)
t = time, (s)
$\bar{t}$ = mean residence time, (s)

Greek letters

ε_{Lt} = total liquid holdup (external and internal holdups)
ε_{Lext} = total external liquid holdup
ε_{Lint} = total internal liquid holdup
ε_{Ls} = static liquid holdup
ε_{Ld} = dynamic liquid holdup
ε_{B} = bed voidage
ε_{Lext} = total external liquid saturation

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