



Liquid–solid wetting factor in trickle-bed reactors: its determination by a physical method

Filippo Pironti^a, Daniela Mizrahi^b, Andreína Acosta^b, Dosinda González-Mendizabal^{b,*}

^a*Inelectra S.A., Edif. Oficentro, San Luis, Caracas 1061, Venezuela*

^b*Departamento de Termodinámica y Fenómenos de Transferencia, Universidad Simón Bolívar, Sartenejas, Apartado Postal 89000, Caracas 1086-A, Venezuela*

Received 2 June 1997; accepted 30 November 1998

Abstract

The solid–liquid wetting factor, f , was experimentally determined in a fixed-bed three-phase reactor with downward concurrent gas and liquid flows (trickle-bed reactor). The method employed consisted of comparing the shear stress obtained when two phases (liquid and gas) circulated through the bed, with that obtained at liquid-full bed conditions, maintaining the intrinsic liquid and gas velocities constant. The shear stresses were related with pressure drop and liquid hold-up. In this way, a non-intrusive method can be applied to any chemical trickle-bed reactor by only measuring pressure drop and liquid hold-up. Experiments were carried out in a pilot unit, where the flow of both phases was modified, obtaining a direct dependency of f with the flow and hold-up of liquid and f values in agreement with those reported by other methods. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Wetting factor; Wetting efficiency; Trickle-bed reactor; Pressure drop; Liquid hold-up; Shear stress

1. Introduction

The wetting factor, f , or sometimes named as the contacting effectiveness, is defined as the fraction of the external area of the catalyst pellet effectively wetted by the liquid flowing down the bed. From the literature data, f , has been obtained either with a chemical method – comparison of reaction rates in a two-phase operation and in a reactor completely filled with the liquid (Satterfield, 1975; Morita and Smith, 1978; Specchia et al., 1978; Herskowitz et al., 1979; Mata and Smith, 1981; Herskowitz and Mosseri, 1983; Leung et al., 1987; Lakota and Levec, 1990; Llano et al., 1997) – or with a dynamic tracer technique (Colombo et al., 1976; Scharz et al., 1976; Sicardi et al., 1980; Mills and Dudukovic, 1981, 1982; Burghardt et al., 1990, Al-Dahhan and Dudukovic, 1995).

Colombo et al. (1976) measured intraparticle effective diffusivity for two-phase gas–liquid and liquid-full operation at the same liquid flow rate and they defined the

external surface-wetting fraction wetted by its ratio:

$$f = \frac{(D_{\text{eff}})_{2\text{-phase}}}{(D_{\text{eff}})_{\text{liquid-filled}}}. \quad (1)$$

Duduković (1977) and Baldi and Gianetto (1979), derived a revised formula in which the external contacting was related to the square root of the ratio of the effective diffusivities in two-phase and liquid-filled operation:

$$f = \sqrt{\frac{(D_{\text{eff}})_{2\text{-phase}}}{(D_{\text{eff}})_{\text{liquid-filled}}}}. \quad (2)$$

Schwartz et al. (1976) developed a method which involved feeding two tracers into the system: an adsorbing one (heptane) and a non-adsorbing one (benzene or naphthalene). Helium and hexane were used as the gas–liquid system. They defined the external catalyst contacting efficiency as the ratio of the apparent adsorption equilibrium constant, observed in two-phase flow, to the adsorption equilibrium constant for completely wetted particles as in a liquid-filled column:

$$f = \frac{(K_A)_{\text{apparent}}}{(K_A)_{\text{liquid-filled}}}. \quad (3)$$

*Corresponding author. Fax: 00582 9063743; e-mail: dosinda@usb.ve.

Morita and Smith (1978) estimated the fraction of particle surface wetted by liquid from the data for two catalyst activities and from the intrinsic rate constant established by the liquid-full data in a batch-recycle system. Herskowitz and Mosseri (1983) calculated wetting efficiency from intrinsic kinetic parameters measured in a stirred tank and with a correlation for the liquid–solid mass transfer.

Lakota and Levec (1990) and González-Mendizabal et al. (1998), defined the wetting efficiency as the ratio of the volumetric mass transfer coefficients in the two-phase flow to those in single-phase (liquid) flow at the same intrinsic velocity of the liquid:

$$f = \frac{(k_S a_{LS})_{2\text{-phase}}}{(k_S a)_{\text{liquid-filled}}} \quad (4)$$

It has been shown experimentally (in the hydrodesulfurization reaction in a pilot reactor; Bondi, 1971), in the hydrogenation of oils (Montagna et al., 1977), in the denitration of hydrogenated oils (van Klinken and van Dongen, 1980), in the dehydrogenation of crotonaldehyde over a palladium catalyst at ambient conditions (Sedricks and Kenney, 1973), and in the hydrogenation of benzene (Satterfield and Ozel, 1973), that the degree of wetting exerts a substantial influence on the overall rate of a heterogeneous catalytic reaction. Furthermore, the degree of wetting also depends upon many other variables. Various empirical equations, which determine the degree of wetting of the packing, as a function of the liquid and gas velocities, the physical and chemical properties of the liquid, and the size of the packing have been developed. However, as noted by Satterfield (1975), the applicability of these equations, as generalized in Schwartz et al. (1976), for calculations of the degree of wetting of catalyst particles is questionable, and requires the performance of experiments under conditions corresponding to such operations.

In this work, we propose a new method by which we compare the liquid–solid shear stress times the specific area in the two-phase flow to that in liquid-full bed at the same intrinsic liquid and gas velocities, i.e.

$$f = \frac{(\tau_{LS} a_{LS})_{2\text{-phase}}}{(\tau_{LS} a)_{\text{liquid-full bed}}} \quad (5)$$

The advantage of this method is that the external wetting can be determined directly from the pressure drop and liquid hold-up measurements without disturbing appreciably the chemical system.

2. Model

In trickle flow the liquid phase trickles over the packing while the gas phase, which is continuous, moves in the remaining void space. The driving force of the gas flow is

the pressure drop while the driving force of the liquid flow is mainly gravitational, dominated by the liquid–solid viscous shear stress. It has been found by Rode et al. (1995), that the overall mass transfer rate is proportional to one-third the power of the overall shear-rate characteristic for a laminar mass-transfer mechanism where liquid flow is dominated by viscous forces even at high interaction regimes. Considering that the analogy between mass and momentum transfer is in this case applicable, a knowledge of the average liquid and gas shear stresses enable us to establish simplified expressions of the average force balances for the liquid and gas phases; with the following assumptions: the phases are treated as continua; the flows are isothermal and steady with constant and uniform voidage and liquid hold-up values; acceleration effects are negligible; and there is no radial pressure gradient. By overall force balance over the liquid and gas phase we can relate each shear stress with pressure drop and liquid hold-up measured under two-phase and liquid- and gas-filled operation.

For liquid-full bed operation, we have for the liquid:

$$(\tau_{LS} a)_{\text{liquid-full bed}} = \frac{\varepsilon_B (\Delta P)_{\text{liquid-full bed}}}{L} + \varepsilon_B \rho_L g \quad (6)$$

For gas-filled operation, we have for the gas:

$$(\tau_{GS} a)_{\text{gas-filled}} = \frac{\varepsilon_B (\Delta P)_{\text{gas-filled}}}{L} + \varepsilon_B \rho_G g \quad (7)$$

For the two-phase operation, we have for the liquid the following force balance:

$$AL(\tau_{LS} a_{LS})_{2\text{-phase}} = AL(\tau_{LG} a_{LG}) + A\varepsilon_L (\Delta P)_{2\text{-phase}} + \varepsilon_L \rho_L g AL \quad (8)$$

And for the gas:

$$AL(\tau_{LG} a_{LG}) + AL(\tau_{GS} a_{GS})_{2\text{-phase}} = A(\varepsilon_B - \varepsilon_L) (\Delta P)_{2\text{-phase}} + (\varepsilon_B - \varepsilon_L) \rho_G g AL \quad (9)$$

To account for the wetting efficiency, a comparison was made between the shear stress for the two-phase and to that in single-phase (liquid-full bed) flow at the same intrinsic velocity of the liquid:

$$f = \frac{(\tau_{LS} a_{LS})_{2\text{-phase}}}{(\tau_{LS} a)_{\text{liquid-full bed}}} \quad \text{at the same } v_L^* \quad (10)$$

Using the dynamic liquid hold-up data, the intrinsic velocity v_L^* was calculated by

$$v_L^* = \frac{Q_L}{A\varepsilon_L d} \quad (11)$$

The new liquid Reynolds number is defined as

$$Re_L^* = Re_L \frac{\varepsilon_R}{\varepsilon_{Ld}} \quad (12)$$

It is postulated that the gas–solid shear stress for the two-phase flow is related to the single-phase flow (gas-filled) by the fraction of the packing not wetted by the liquid, $(1 - f)$, at the same intrinsic gas velocity,

$$1 - f = \frac{(\tau_{GS} a_{GS})_{2\text{-phase}}}{(\tau_{GS} a)_{\text{gas-filled}}} \quad \text{at the same intrinsic gas velocity.} \quad (13)$$

And the new gas Reynolds number is defined as

$$Re_G^* = Re_G \frac{\varepsilon_B}{(\varepsilon_B - \varepsilon_L)} \quad (14)$$

By eliminating the gas–liquid shear stress from Eqs. (8) and (9) and by taking into account Eqs. (10)–(14), an expression for the wetting fraction, f , is obtained:

$$f = \frac{g[\rho_L \varepsilon_L + \rho_G(\varepsilon_B - \varepsilon_L)] + \varepsilon_B((\Delta P_{2\text{-phase}})/L) - (\tau_{GS} a)_{\text{gas-filled}}}{(\tau_{LS} a)_{\text{liquid-full bed}} - (\tau_{GS} a)_{\text{gas-filled}}} \quad (15)$$

where $(\tau_{LS} a)_{\text{liquid-full bed}}$ and $(\tau_{GS} a)_{\text{gas-filled}}$ are given by Eqs. (6) and (7), respectively.

3. Experimental

The experimental apparatus, bench scale, used to measure pressure drops and hold-up for both single-phase and trickle-bed operation is shown in Fig. 1. The 2 m high column is made of a carbon steel tube in an internal diameter of 4 in, with four pressure taps of 1/4 in each one separated by 0.6 m. The column has a thick optically clear acrylic window to observe the behavior of the two-phase flow near the wall surface. The gas–liquid distributor is similar to the one used by Herskowitz and Mosseri (1983).

Distilled water was stored in a 200 l reservoir, and it was pumped through a rotameter up to the top of the column. The flow was regulated manually by needle valves. Compressed air, regulated to a constant pressure of 60 psig, was driven through gas rotameters and introduced to the top of the column at a given flow manually fixed by a needle valve. For all the experimental runs, the liquid and gas phases, were at ambient conditions (25°C and atmospheric pressure).

The column is packed with cylindrical extruded catalyst material. The characteristics of the column and the particle used are shown in Table 1. The column-to-particle diameter ratio is about 26, enough to avoid

channeling. As stated in the literature (Sylvester and Pitayagulsarn, 1975; Hirose et al., 1976; Satterfield et al., 1978; Wang et al., 1982; Losada and Pironti, 1988), the above ratio may vary between 5 and 47 with no significant effect.

On the top and at the bottom of the column there are two solenoids valves connected to a switch that suddenly and simultaneously interrupts the entry and the exit of fluids to the column allowing the measurement of external liquid hold-up. The exit valve of the column is opened, allowing the liquid to drain for a time not less than 30 min, after that the liquid collected is weighed. Then the dynamic liquid hold-up is calculated by

$$\varepsilon_{Ld} = \frac{W_L}{\rho_L V} \quad (16)$$

Pressure drops across the particle bed, were measured with pressure transducers connected to the top and the bottom of the reactor bed. Then the signal is recorded by a data acquisition system. For each gas and liquid flow rate, a minimum of 20 min was allowed for the pressure drop and liquid hold-up measurements to ensure that the data reflected the true steady state (Holub et al., 1993). The mass flux range adopted in this work varied from 1.76 to 14.48 kg m⁻² s⁻¹ for liquid flow and varied from 0.1 to 0.31 kg m⁻² s⁻¹ for gas flow. Under these operating conditions, visual observation through the acrylic window indicates the existence of continuous gas flow regime in the column. This was corroborated by stable and steady pressure drop measurements.

4. Results and discussion

To find the wetting factor, according to the model proposed in this work (Eq. (15)), single-phase pressure drops, liquid hold-up and pressure drops for two-phase runs, are needed.

To predict pressure drop when only one-phase experiments are carried out through the column, Ergun's equation can be used:

$$\frac{\Delta P}{L} = E_1 \frac{(1 - \varepsilon_B)^2 v \mu}{\varepsilon_B^3 d_p^2} + E_2 \frac{(1 - \varepsilon_B) v^2 \rho}{\varepsilon_B^3 d_p} \quad (17)$$

However, as Holub et al. (1993) recommends, the constants, E_1 and E_2 , must be determined for each particular packed material, since they depend on the tortuosity and rugosity of the bed particles. The single-phase flow Ergun constants were determined from pressure drop measurements using air over dry packing. The experimental results obtained are shown in Fig. 2, together with the Ergun's equation with constants: $E_1 = 150$ and $E_2 = 1.8$. These constants were obtained by regression (Raw $R^2 = 0.995$) from pressure drop experimental values and were reproduced with an error less than 5%.

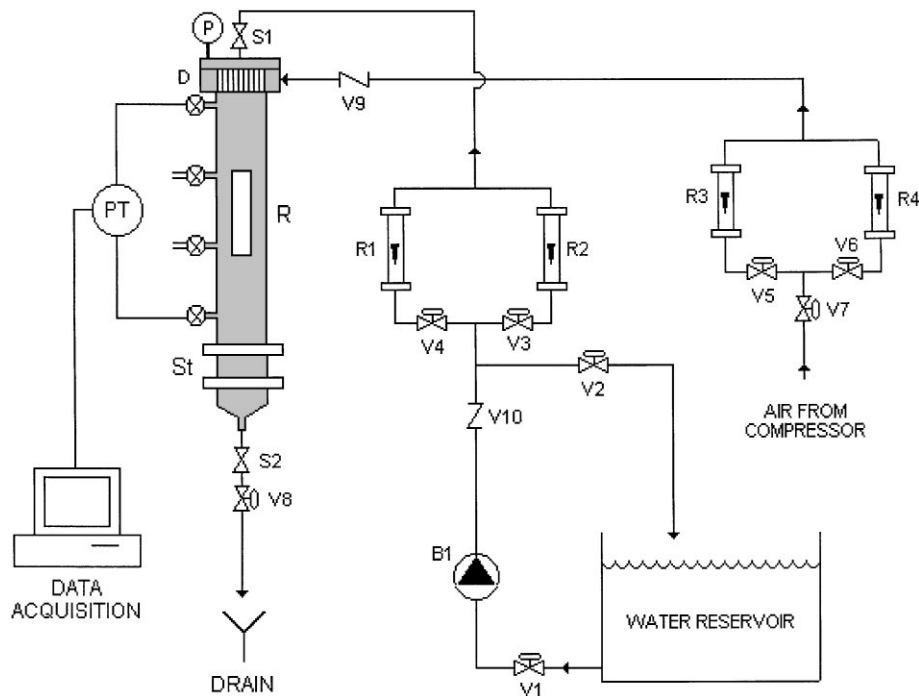


Fig. 1. Schematic diagram of the experimental set-up; (B1) pump; (D) distributor; (PT) pressure transducer; (R) reactor; (R1–R4) rotameters; (S1, S2) solenoid valves; (St) terminal section; (V1–V9) valves.

Table 1
Characteristics of solids, fluids and column

Column diameter	10.16 cm
Column volume (V)	16,863.21 cm ³
Void fraction of the bed (ϵ_b)	0.41 ± 0.01
Static holdup (ϵ_{LS})	0.036 ± 0.001
Mean equivalent diameter of the particles (d_e)	3.68 ± 0.01 mm

Values for the dynamic liquid hold-up in the gas continuous regime are shown in Fig. 3, as a function of liquid Reynolds numbers for different gas Reynolds numbers. The reproducibility of the experimental method, employed for the determination of the dynamic liquid hold-up, was good with a deviation less than 2%, which affects the wetting factors results, calculated by Eq. (15), by approximately 2%. A direct dependence of the liquid Reynolds number upon the dynamic liquid hold-up is observed. However, the gas Reynolds number seems to exert less influence on the dynamic liquid hold-up, as the gas Reynolds number increases the liquid hold-up decreases. The above results can be correlated to obtain the following expression:

$$\epsilon_{Ld} = 0.048 Re_L^{0.403} Re_G^{-0.077} \quad (\text{Raw } R^2 = 0.998), \quad (18)$$

valid for the range: $11 \leq Re_L \leq 90.4$ $9 \leq Re_G \leq 110$.

Eq. (18) predicts the experimental values with a standard deviation less than 3.7%.

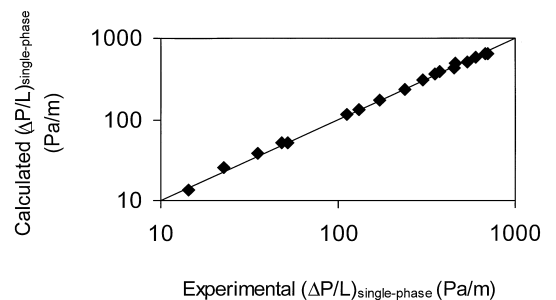


Fig. 2. Experimental single-phase pressure drop compared with values calculated from the Ergun equation.

Basically, all correlations reported in the literature for the calculation of the dynamic liquid hold-up, show a direct dependency upon liquid superficial velocity or liquid Reynolds number, as an exponent in accordance with the movement of the liquid through the bed (laminar film, turbulent films or spray) and the particle texture. Generally, the values of this exponent oscillate between 1/3 for laminar films and 0.5–0.6 for turbulent films (Wammes et al., 1990). However, contradictory results have been found, for instance, Goto and Smith (1975), reported an exponent of 1/3 for lower liquid Reynolds numbers, while Kohler and Richarz (1984) related the liquid dynamic hold-up, ϵ_{Ld} , with a 0.53 exponent for very low Re_L (0.1–5). In our case, the exponent is around 0.4, which is an indication that the flow regime is laminar, where the

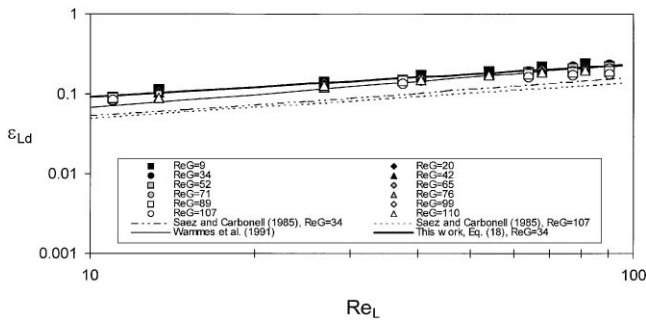


Fig. 3. Dynamic liquid hold-up vs. liquid Reynolds numbers, for different gas flows.

energy loss of the fluid is mainly due to viscous effects between the liquid and the packing (Wammes et al., 1991). Regarding the effect of the gas upon the liquid hold-up, Shah (1879) and Ramachandran and Chaudhari (1983) reported that at atmospheric pressure it was not appreciable. Their results are in agreement with those obtained in this work. However, at high pressure, the gas flow reduces the liquid hold-up considerably, due to the increase of the drag force in the liquid–gas interface.

Correlation from Saez and Carbonell (1985) – for two gas Reynolds numbers – Wammes et al. (1990) and Eq. (18) are also shown in Fig. 3, indicating that the experimental data of this work is in the range of values reported. However, there is a difference between the slope of the line through the experimental points (Eq. (18)) and that of the correlation of Wammes et al. (1990). A maximum deviation of 17.5 and 6.5% for all liquid Reynolds of this work and $Re_G = 9$ and $Re_G = 110$ respectively, was found, when experimental values of ϵ_{Ld} were compared with those predicted by the correlation of Wammes et al. (1990).

The pressure drop for two-phase operation, as a function of liquid Reynolds number for several gas Reynolds numbers, is shown in Fig. 4. It is clearly observed in this figure, that the pressure drop increases with both Reynolds numbers. The reproducibility of the experimental method, employed for the determination of the two-phase pressure drop, was good with a deviation less than 5%, which affects the wetting factors results, calculated by Eq. (15), by approximately 2%. Pressure drop values reported by Saez and Carbonell (1985) and Larkins et al. (1961), for two different gas Reynolds numbers, are also shown in Fig. 4, indicating that the experimental data of this work is in agreement with other results. However, for $Re_G = 107$ all measured data points are higher than predicted by the correlation of Larkins et al. (1961) and by Saez and Carbonell (1985). The deviation found between our experimental values and those reported by Larkins et al. (1961) was 16% for $Re_G = 34$ and 23% for $Re_G = 107$, for all Re_L covered in this work.

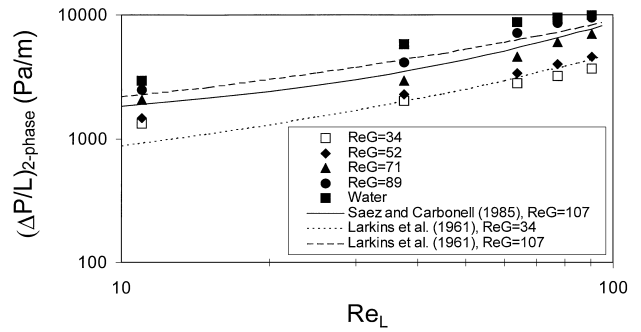


Fig. 4. Pressure drop for two-phase flow vs. liquid Reynolds numbers for different gas flows.

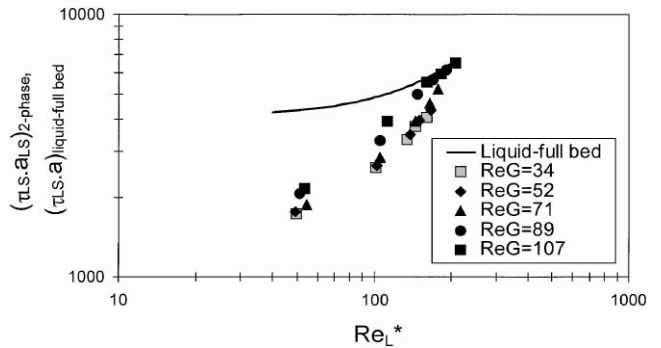


Fig. 5. Liquid–solid shear stresses as a function of modified Reynolds number for two-phase and single-phase (liquid-full bed) flow.

Shear stress for single-phase operations was calculated from Ergun equation with the constants determined by measured single-phase pressure drops and Eqs. (6) and (7). Liquid–solid shear stresses for two-phase operation together with the liquid-full bed results, are shown in Fig. 5 as a function of modified liquid Reynolds numbers ($Re_L^* = Re_L \epsilon_B / \epsilon_{Ld}$) for several gas Reynolds numbers. The same behavior predicted by Lakota and Levec (1990) and González-Mendizabal et al., (1998), is clearly observed from this figure. As the gas and liquid flow increases, the wetting factor should come closer to unity, since the shear stress for two-phase operation is approaching the liquid-full bed values, even more if the Reynolds number is based on the intrinsic liquid velocity over the particles.

In Fig. 6 values of gas–liquid shear stresses estimated from Eqs. (8) and (9), are reported as a function of operating conditions. As it is observed, most of the experimental points are on the 45° slope line, which is an indication of the concordance between results calculated by either equation, and there is no danger that by subtracting two numbers a large error could be generated.

The wetting factors were calculated by means of Eq. (15). Fig. 7 shows the results plotted against the Reynolds numbers for both phases and for all the experiments done in this work. The figure shows that the

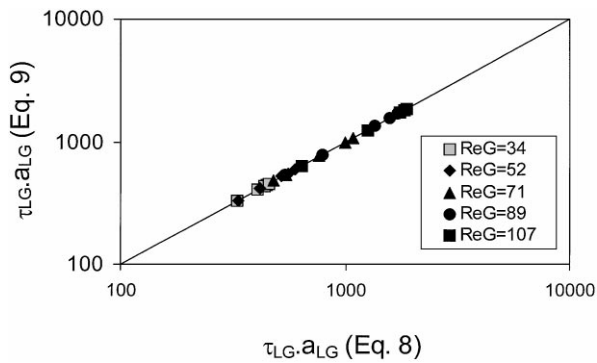


Fig. 6. Parity plot of gas-liquid shear stresses calculated by Eqs. (8) and (9).

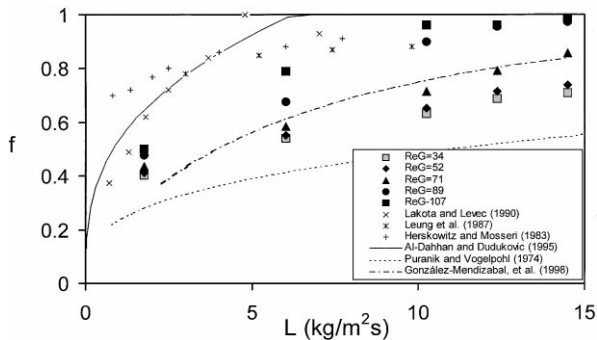


Fig. 7. Wetting factors as a function of superficial liquid mass velocity and gas Reynolds numbers for two-phase flow.

wetting efficiency increases with the liquid and gas Reynolds numbers. These results agree with those reported by numerous authors (Satterfield, 1975; Herskowitz and Mosseri, 1983; Leung et al., 1987; Lakota and Levec, 1990; Al-Dahhan and Dudukovic, 1995; Llano et al., 1997; González-Mendizabal et al., 1998), and disagree with Morita and Smith (1978), and Levec and Pavko (1978), who got constant f values for different liquid flow values.

Results of this work are compared in Fig. 7 with those reported by other researchers. This figure shows that the f values obtained here are within the wide spread found in literature for this parameter. This is basically because authors propose their own equation based on specific fluid phases and packing characteristics. Therefore, a small change in solid texture or shape difference greatly modifies the f value.

The method proposed in this work was checked by employing correlations reported in the literature by Wammes et al. (1990) for the dynamic liquid hold-up, and by Larkins et al. (1961) and Saez and Carbonell (1985) for two-phase pressure drop. In Fig. 8, a reasonable agreement is observed, in a parity plot, between the f values calculated by substituting these correlations in Eq. (15) and our experimental results.

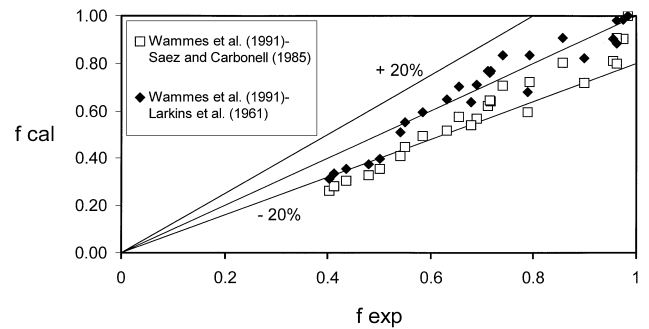


Fig. 8. Parity plot of wetting factors calculated from Eq. (15) and known correlations of ΔP and ε_{Ld} , and experimental values obtained in this work.

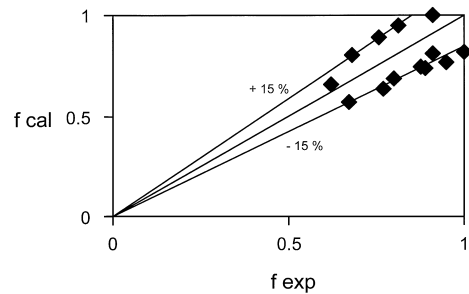


Fig. 9. Parity plot of wetting factors calculated from Eq. (15) and ΔP and ε_L data of Al-Dahhan and Dudukovic (1994), and experimental values obtained by the same authors (1995).

Furthermore, the model proposed in this work (Eq. (15)) was used to calculate wetting factors with experimental published data, $\Delta P/L$ and ε_L , reported by Al-Dahhan and Dudukovic (1994). These calculated values were compared with reported wetting factors by the same authors in 1995. Shear stresses for liquid- and gas-full bed were calculated from the Ergun equation with the constants reported by these authors for the packing they used. Results obtained by our model reproduced the data of Al-Dahhan and Dudukovic (1995) with a maximum deviation of 15%, as shown in Fig. 9, for low gas velocities (less than 8 cm/s) and different pressure. For high gas velocities our model predicts values of f higher than one, probably because it fails to take into account the gas-liquid turbulent stresses now present.

The f values obtained in this work corroborated the feasibility of using the method proposed here to determine solid-liquid wetting factors, with the advantage to be a non-intrusive method, requiring only the knowledge of Ergun's constant characteristics of the packing bed, the pressure drop and liquid hold-up under three-phase operation. These parameters can be taken either from experimental data or from published correlations applicable to the operating conditions.

5. Conclusions

A non-intrusive method for determination of solid–liquid wetting factors in the gas continuous regime was proposed for a three-phase operating column. The model is simple and required only the knowledge of two-phase pressure drop, liquid hold-up and Ergun's constant characteristics of the packing.

Solid–liquid wetting factors were determined with the model proposed in this work, obtaining a direct dependency of the wetting fraction with the liquid and gas flows. The experimental values were contrasted against those reported by the literature and it was found that they are within the ample band of values proposed by other authors for the same liquid and gas flow combinations.

Notation

a	effective interfacial area of particles per unit volume of bed, m^{-1}
A	column cross section, m^2
d_e	average equivalent particle diameter defined as six times the volume to surface ratio, m
d_p	particle diameter, m
D_{eff}	intraparticle effective diffusivity
E_1, E_2	Ergun constants for the single-phase flow on the packing (describes bed tortuosity and roughness)
f	wetting factor, dimensionless
k_{sa}	volumetric liquid–solid mass transfer coefficient without reaction, s^{-1}
K_A	adsorption equilibrium constant
L	superficial liquid mass velocity, $\text{kg m}^{-2} \text{s}^{-1}$
Q	volumetric flow, $\text{m}^3 \text{s}^{-1}$
Re	Reynolds number defined as $(Q/A\varepsilon_B)(\rho/\mu)$
Re_{G^*}	modified gas Reynolds number, $Re_{G^*} = Re_G \varepsilon_B / (\varepsilon_B - \varepsilon_L)$
Re_{L^*}	modified liquid Reynolds number, $Re_{L^*} = Re_L \varepsilon_B / \varepsilon_{Ld}$
v	superficial velocity, m s^{-1}
v_{L^*}	intrinsic liquid velocity, $v_{L^*} = Q_L / (A\varepsilon_{Ld})$
V	column volume, m^3
W_L	weight of the liquid, kg

Greek letters

$\Delta P/L$	pressure drop per unit of length, Pa m^{-1}
ε_B	packed-bed void per reactor volume (bed porosity), dimensionless
ε_L	total external liquid hold-up per reactor volume ($\varepsilon_L = \varepsilon_{Ld} + \varepsilon_{Ls}$), dimensionless
ε_{Ld}	dynamic liquid hold-up per reactor volume, dimensionless

ε_{Ls}	static liquid hold-up per reactor volume, dimensionless
μ	viscosity, $\text{kg m}^{-1} \text{s}^{-1}$
ρ	density, kg m^{-3}
τ	shear stress, Pa

Subscripts

G	referred to gas phase
GS	referred to gas–solid phases
L	referred to liquid phase
LG	referred to liquid–gas phases
LS	referred to liquid–solid phases

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