Effect of partial wetting on liquid/solid mass transfer in trickle bed reactors

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Abstract

The wetting efficiency of liquid trickle flow over a fixed bed reactor has been measured for a wide range of parameters including operating conditions, bed structure and physico-chemistry of liquid/solid phases. This data bank has been used to develop a new correlation for averaged wetting efficiency based on five different non-dimensional numbers. Finally liquid/solid mass transfer has been determined in partial wetting conditions to analyse what are the respective effects of wetting and liquid/gas flow turbulence. These effects appear to be separated: wetting being acting on liquid/solid interfacial area while the liquid/solid mass transfer coefficient is mainly connected to flow turbulence through the interstitial liquid velocity. A correlation has been proposed for liquid/solid mass transfer coefficient at very low liquid flow rate.

Keywords: Multiphase reactors; Hydrodynamics; Trickle bed reactors; Trickle flow; Wetting efficiency; Mass transfer

1. Introduction

Trickle bed reactors (TBR) are randomly packed beds of catalyst particles in which liquid and gas phases flow concurrently downward and are widely used mostly in oil refinery and petrochemical industry. Among multiphase reactors, TBR have probably the most complex hydrodynamics as trickling flow exhibits the unique feature of solid partial wetting under some conditions, mainly at low liquid flow rate. In the development and industrial operation of these reactors, partial wetting is a major issue since it can result in a poor utilization of the catalyst and in hot spots formation. At industrial scale, higher residence times are required to increase sulphur removal or high compounds conversion resulting in lowering liquid velocity that may increase partial wetting. At pilot scale, reactors are usually operated at the same liquid hourly space velocities as the commercial reactors, leading to very low liquid velocities and partial wetting of the catalyst in downward flow configuration. As the volume of catalyst tested has to be decreased for practical and economical reasons, partial wetting becomes a major issue for the correct operation of small size pilot plant. Among its many possible effects on three phase catalytic reaction, this work investigates how it could modify liquid/solid mass transfer.

Several correlations have been proposed in the past for wetting efficiency inside TBR (Mills and Dudukovic, 1981; El-Hisnawi et al., 1982; Burghardt et al., 1990, 1995; Al-Dahhan and Dudukovic, 1995) but the models give very dispersed results over the same operating conditions range and it is very difficult to choose the more accurate one. Furthermore, these correlations express wetting efficiency mainly as a function of gas/liquid flow hydrodynamics but none of them include the effect of solid intrinsic wettability. The liquid/solid mass transfer in trickle bed reactors has been largely studied and numerous correlations have been proposed for liquid/solid mass transfer coefficient k_{LS} (Goto and Smith, 1975; Specchia et al., 1978; Satterfied et al., 1978; Rao and Drinkenburg, 1985; Lakota and Levec, 1990). The validity domain of these correlations remains limited since they are all issued from experimental data obtained with aqueous solution as liquid phase and air as gas phase. Almost no results have been reported with organic fluids. Furthermore, the liquid/solid mass transfer coefficient has always been measured without a separated measurement for wetting efficiency. The solid wetting efficiency is thus always intrinsically included in

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the expression of k_{LS}. This approach is not totally acceptable since the dependence on hydrodynamic and physico-chemistry parameters is not a priori the same for wetting efficiency and liquid/solid mass transfer. A new research effort was thus necessary to study liquid/solid mass transfer independently from wetting efficiency with organic liquids.

This paper summarizes a part of a collaborative work between IFP Lyon and LGC Toulouse devoted to several aspects of partial wetting in TBR.

In a first step, a parametric study has been performed to determine the averaged wetting efficiency as a function of liquid/solid interaction (wettability), liquid and gas flow rates, operating pressure, particle shape and size. A correlation based on selected non-dimensional numbers has been established to predict wetting efficiency as a function of operating parameters.

In a second step, liquid/solid mass transfer coefficient has been determined as a function of liquid and gas flow rates in partial wetting conditions. Its dependence to wetting efficiency has been studied. A new correlation has been proposed for k_{LS} as a function of a Reynolds number based on interstitial liquid velocity.

2. Parametric study of wetting efficiency

Two techniques have been implemented to quantify averaged wetting efficiency inside a catalytic trickle bed: residence time distribution (RTD) analysis and image analysis after dye adsorption on porous bed particles. The first technique based on tracer analysis involves a detailed model of tracer RTD including liquid axial dispersion, mass transfer at the catalyst boundary and non-symmetrical diffusion in the catalyst pores due to partial wetting of the catalyst boundary. This complex modelling is described by Julcour-Lebigue et al. (2007). With the second technique, a dye colorant injected stepwise at the column inlet colorizes the particle external surface in contact with the liquid film. Images of solid particles in beds cross-sections are processed to detect wetted surface and determine the wetting efficiency. This technique, which gives very detailed local information, even at the pellet scale, is fully described in Baussaron (2005) and Baussaron et al. (2007).

Many operating parameters have been investigated. The most important contribution of this work concerns liquid/solid interaction (wettability) characterized by the contact angle and varied by changing the liquid phase—water, ethanol and heptane. In addition, liquid and gas flow rates, operating pressure and gas density, particle shape and size have also been varied. The ranges for these parameters are listed in Table 1. Other features as effects of liquid inlet distribution and bed prewetting have been pointed out and are reported in Baussaron et al. (2007). Nevertheless all values of averaged wetting efficiency that are presented in this paper were obtained using an

Table 1
List of studied parameters and corresponding varying ranges

Parameters	Range
Liquid superficial velocity (m/s)	5×10^{-4} to 10^{-2}
Gas superficial velocity (m/s)	0–0.2
Pressure (MPa)	0.1–1
Gas density (kg/m^3)	1.2–33
Liquid/solid contact angle (°)	0–65
Particle shape	Spherical, cylindrical
Particle size (m)	2.5×10^{-3} to 5.5×10^{-3}

Table 2
Qualitative effect of studied parameters on averaged wetting efficiency

Studied parameters	Effect on wetting efficiency	
Better liquid flow distribution	↗↗	
Increase of liquid superficial velocity: V_{SL} ↗	↗↗↗	
Increase of gas superficial velocity: V_{SG} ↗	↗	
Pressure or gas density increase: P or ρ_G ↗	–	
Particle diameter decrease: d_p ↘	↗	
Particle shape	–	
Bed prewetting	↗↗↗	
Liquid/solid wettability ↗	$V_{SL} > 2 \cdot 10^{-3}$ m/s: –	$V_{SL} < 2 \cdot 10^{-3}$ m/s: ↗↗

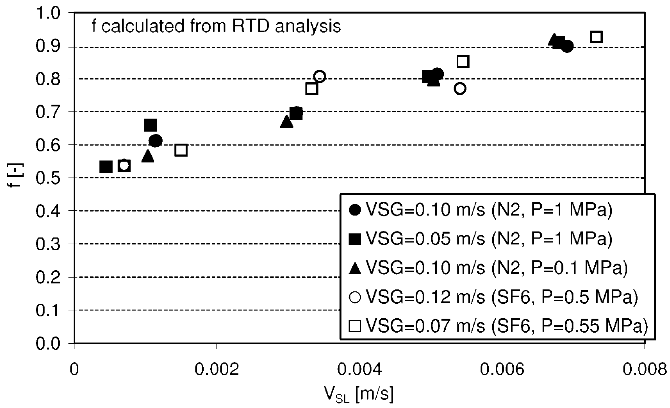

Fig. 1. Wetting efficiency variation as a function of liquid superficial velocity for different gas superficial velocities, operating pressures and gas molecular weights.

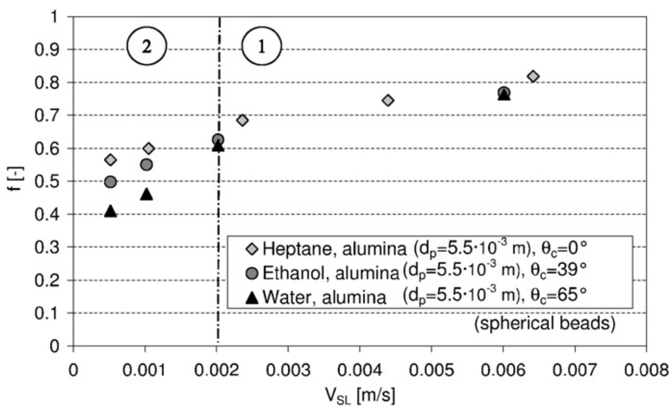

Fig. 2. Wetting efficiency variation as a function of liquid superficial velocity for liquid/solid systems with various contact angles between 0° and 65°.

Table 3
Correlations from literature used for wetting efficiency prediction

Authors	Correlation for wetting efficiency
El-Hisnawi et al. (1982)	$f = 1.617 Re_L^{0.146} Ga_L^{-0.071}$ with $Ga_L = \left(\frac{d_p^3 g \rho_L^2}{\mu_L^2}\right)$
Burghardt et al. (1990)	$f = 0.0381 (\rho_L V_{SL})^{0.222} (\rho_G V_{SG})^{-0.083} d_p^{-0.373}$
Burghardt et al. (1995)	$f = 3.38 Re_L^{0.222} Re_G^{-0.083} Ga_L^{-0.512}$ with $Ga'_L = d_p \left(\frac{g \rho_L^2}{\mu_L^2}\right)^{1/3}$

optimized inlet distributor with bed pre-wetting to avoid influence of liquid flow distribution on global wetting efficiency.

Table 2 summarizes the main trends deduced from parametric study. While several observed tendencies are expected and sound physical, two results are less obvious or have been weakly studied:

- The effects of gas density (varied with pressure or gas molecular weight) or gas superficial velocity on catalyst wetting are quite weak. As can be seen in Fig. 1, wetting efficiency measured by RTD analysis varies with liquid flow rate, but changes of gas density (from 1.2 to 33 kg/m^3) or gas velocity (from 0.05 to 0.12 m/s) have almost no influence on wetting. The gas inertia appears to have effect on liquid film thickness and thus liquid retention, but do not induce significant liquid film spreading. Sederman and Gladden (2001) have also observed a weak effect of gas flow on wetting efficiency when measuring catalyst wetting inside a TBR using MRI imaging technique.
- The effect of intrinsic solid wettability is only significant at very low liquid superficial velocity (lower than 2×10^{-3} m/s). The wetting efficiency is plotted in Fig. 2 as a function of liquid superficial velocity for three different liquid/solid systems with contact angle from 0° to 65°. The three curves are almost superposed for liquid superficial velocity higher than 2×10^{-3} m/s. Above this limit, the wetting of liquid film is mainly controlled by flow dynamics in restricted volume and no more by liquid/solid affinity.

The whole data bank of measured averaged wetting efficiencies has been compared to predicted values using existing correlations of El-Hisnawi et al. (1982), Burghardt et al. (1990) and Burghardt et al. (1995). These correlations are detailed in Table 3 and the comparison results are presented in Figs. 3–5 as parity diagrams. In Figs. 4 and 5, data obtained with no gas flow are not reported since Burghardt's correlations cannot handle trickle flow condition without any gas flow. Experimental data are overestimated by the correlation of El-Hisnawi et al. (1982) that predicts wetting efficiencies between 15% and 60% higher than measured ones. On the contrary the correlations developed by Burghardt et al. (1990, 1995) provide results that underestimate the measured wetting efficiency data. The deviation remains moderate for data acquired at low pressure (close to ambient pressure) but for data corresponding to higher pressure (1 MPa) or with denser gas (SF6) the deviation increases largely with predicted value 50% lower than measured data.

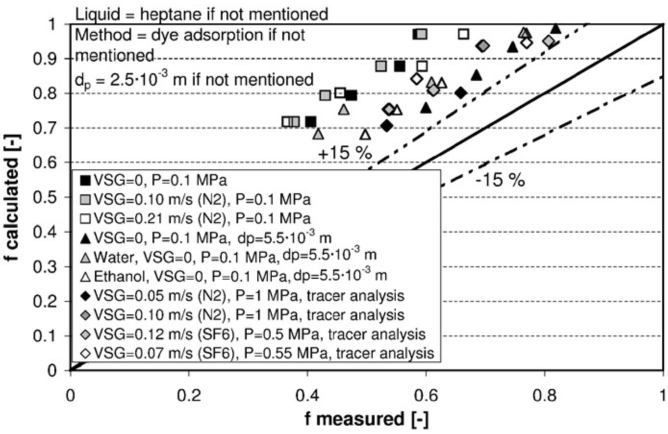

Fig. 3. Comparison of measured wetting efficiency data with predicted values with correlation from El-Hisnawi et al. (1982).

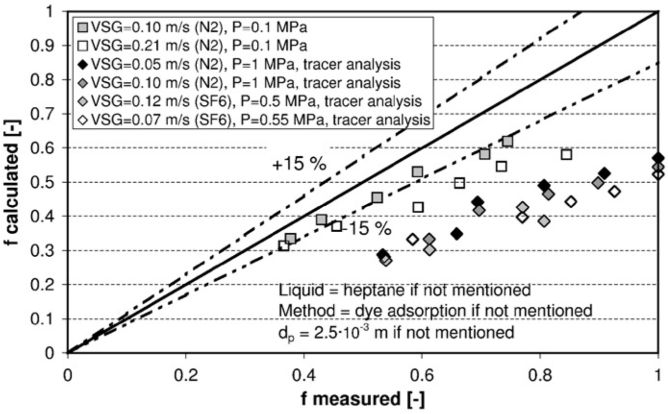

Fig. 4. Comparison of measured wetting efficiency data with predicted values with correlation from Burghardt et al. (1990).

Nevertheless, it can be noted that the dimensionless correlation developed by Burghardt et al. (1995) improves the prediction accuracy when compared to the dimensional one (Burghardt et al., 1990). These correlations are not accurate enough to predict solid wetting in a broad range of operating conditions, pressure or gas density effects being clearly overestimated.

The data bank of wetting efficiencies has been thus used to develop a new correlation. A dimensional analysis has shown that wetting efficiency can be expressed as a function of five independent non-dimensional numbers: Reynolds numbers for gas and liquid flows, Galileo number and two capillary numbers based on gas/liquid and liquid/solid surface tensions.

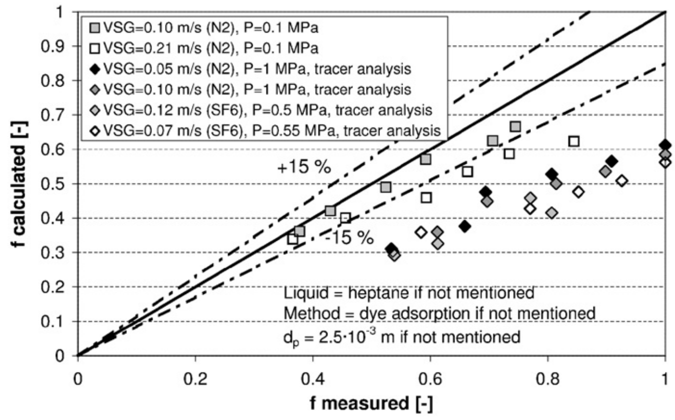

Fig. 5. Comparison of measured wetting efficiency data with predicted values with correlation from Burghardt et al. (1995).

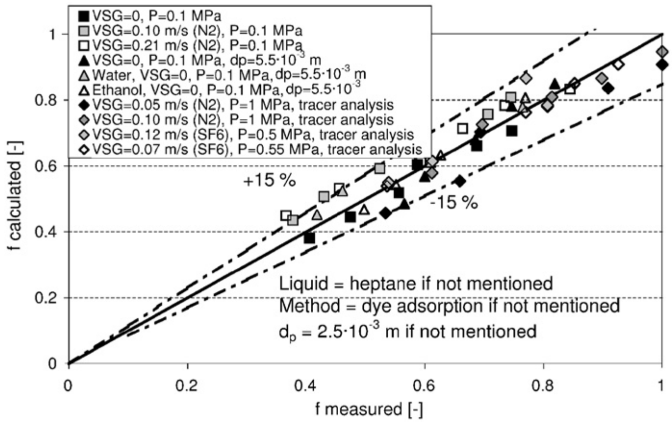

Fig. 6. Parity diagram for the wetting efficiency prediction with the new developed correlation.

$$f = 0.96\left(\frac{\rho_L V_{SL} d_p}{\mu_L}\right)^{0.22}\left(1+\frac{\rho_G V_{SG} d_p}{\mu_G}\right)^{0.046}\left(\frac{\varepsilon_B^3 \rho_L^2 d_p^3 g}{(1-\varepsilon_B)^3 \mu_L^2}\right)^{0.083} \times\left(\frac{\mu_L^2}{\gamma_{LS} d_p \rho_L}\right)^{0.052}\left(\frac{\mu_L^2}{\gamma_L d_p \rho_L}\right)^{0.12}. \qquad (1)$$

This correlation has been established within the following validation domain:

$$2 \leqslant \left(\frac{\rho_L V_{SL} d_p}{\mu_L}\right) \leqslant 60; \quad 0 \leqslant \left(\frac{\rho_G V_{SG} d_p}{\mu_G}\right) \leqslant 530;$$

$$9.2 \times 10^4 \leqslant \left(\frac{\varepsilon_B^3 \rho_L^2 d_p^3 g}{(1-\varepsilon_B)^3 \mu_L^2}\right) \leqslant 1.5 \times 10^6;$$

$$2.0 \times 10^{-6} \leqslant \left(\frac{\mu_L^2}{\gamma_{LS} d_p \rho_L}\right) \leqslant 1.6 \times 10^{-5};$$

$$2.0 \times 10^{-6} \leqslant \left(\frac{\mu_L^2}{\gamma_L d_p \rho_L}\right) \leqslant 1.5 \times 10^{-5}.$$

For non-spherical particle, the particle diameter d_p is expressed as: $d_p = 6V_p/A_p$ with V_p the solid particle volume and A_p the particle surface area. As expected the gas inertia term and liquid/solid capillary number appears with a low exponent since these physical phenomena have a weak effect on wetting efficiency. The correlation is still valid when operating without a gas flow.

Fig. 6 shows the parity diagram for wetting efficiency prediction with the developed correlation. The whole data set corresponding to a large range of operating conditions is correctly predicted using Eq. (1) with a maximum deviation of 13%.

The effect of particle diameter and bed void fraction variation in the validation domain is correctly taken into account by correlation (Eq. (1)) but the tendency of wetting efficiency correlation with particle diameter alone is not consistent with physical observed tendency. Due to very low number of tested values for particle diameter and bed porosity, the sensitivity of wetting efficiency to Galileo number is not still very accurate and will be optimized in the future by testing independently the effect of bed void fraction and particle diameter variation.

3. Liquid/solid mass transfer in partial wetting conditions

To determine the effect of partial wetting on liquid/solid mass transfer, the coefficient k_{LS} has been measured independently on the same experimental tool to propose a model in partial wetting condition. k_{LS} has been determined by analysis of solid dissolution in the liquid flow. The solid particles were spheres of β-naphthol that are slowly dissolved by trickling n-heptane flow. The β-naphthol has been chosen due to its very low solubility in hydrocarbon (1 kg/m^3 at 25 °C in n-heptane). The liquid/solid mass transfer coefficient k_{LS} is deduced from the liquid concentration of β-naphthol measured at the bed outlet, C, using a piston flow model:

$$\frac{C}{C^*} = 1 - \exp\left(-\frac{k_{LS} a_{LS} S Z}{Q_L}\right), \qquad (2)$$

where C^* is the β-naphthol concentration at equilibrium condition and a_{LS} is the effective liquid/solid interfacial area expressed by:

$$a_{LS} = f\,\frac{6\varepsilon_B}{d_p} \qquad (3)$$

with f the wetting efficiency measured by RTD analysis technique for the same operating conditions not to include additional error of wetting efficiency correlation in the study of wetting efficiency coupling with liquid/solid mass transfer.

The assumption of piston flow has been previously checked by implementing RTD measurement inside a bed of glass beads of same size within the same operating conditions. The experiments are performed in a column of 0.05 m in diameter and 1.2 m length. The active bed is inserted downstream 1 m of bed of porous alumina particles in order to have an established liquid flow at active bed entrance that is representative of trickle flow through a catalytic bed. Downstream the active bed of β-naphthol particles, a layer of glass beads is inserted in order to avoid β-naphthol adsorption on solid particles before liquid

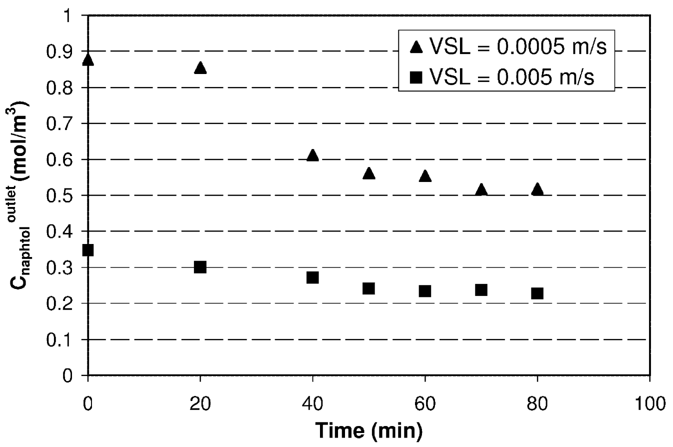

Fig. 7. Variation of β-naphthol concentration at bed outlet as a function of time.

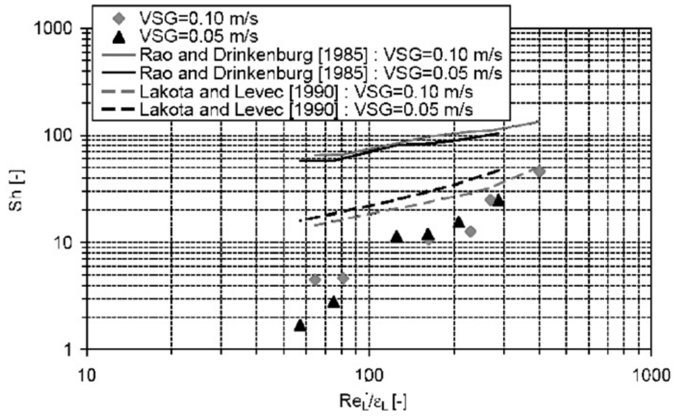

Fig. 8. Variation of liquid/solid Sherwood number as a function of a Reynolds number based on liquid interstitial velocity.

sampling. The optimal length for active bed of β-naphthol particles has been estimated in order to minimize error on k_{LS} estimation when using Eq. (2) and keep β-naphthol concentration at bed outlet lower than 70% of saturation value (C^*). Upstream liquid sampling point, the liquid flow is collected through a converging section in order to get the averaged concentration over the bed cross-section. β-naphthol concentration is measured using UV spectrometer for several samples collected successively. The time concentration profile shows that steady state conditions for liquid/solid mass transfer are reached after about 1 h (see Fig. 7). These steady state values are used to determine k_{LS}. The β-naphthol amount that may be stripped into gas phase by gas/liquid mass transfer is negligible since the maximum β-naphthol flux that can be caught by the gas flow (at equilibrium condition) is only 0.01% of the total flux.

k_{LS} has been measured as a function of liquid superficial velocity for two different gas flow rates. The effect of liquid flow rate appears to be very strong on k_{LS}, contrary to the weak effect of gas flow rate. The increase of gas flow rate mainly contributes to reduce the liquid retention and thus to increase the interstitial liquid velocity, that enhances liquid/solid mass transfer. The effect of gas flow rate on liquid/solid mass transfer coefficient can be integrated by modelling k_{LS} as a function of a Reynolds based on liquid interstitial velocity. Therefore, the local liquid/solid mass transfer coefficient and the corresponding Sherwood number can be fully predicted once knowing the liquid flow rate and dynamic liquid holdup ε_L inside the bed (see Fig. 8). In these experiments, the dynamic liquid holdup has been measured in the same operating condition using a quick closing valves system. As one can see in Fig. 8 the experimental data are largely underestimated by the correlation of Rao and Drinkenburg (1985). The correlation of Lakota and Levec (1990) predicts liquid/solid mass transfer with a better accuracy at high value of liquid interstitial velocity but the discrepancy increases for Reynolds number based on liquid interstitial velocity lower than 100. The effect of gas flow rate is furthermore overestimated by the correlation of Lakota and Levec (1990).

Despite the low number of available data, a first correlation has been proposed to interpret them and express the liquid/solid Sherwood number as a function of liquid Reynolds number based on liquid interstitial velocity:

$$Sh = \frac{k_{LS} d_P}{D_m} = 5.64 \times 10^{-4} \left(\frac{Re_L}{\varepsilon_L}\right)^{1.6} Sc_L^{1/3}. \quad (4)$$

Since particle diameter variation was very low during the test and was dependent on liquid velocity, it was not possible to really validate the sensitivity of k_{LS} to particle diameter alone. The validation of this correlation is thus restricted to a particle diameter between 2.5 and 3 mm and illustrates mainly the effect of liquid interstitial velocity on liquid/solid mass transfer.

It must be noticed that the exponent of Reynolds number is larger than those proposed by existing correlations for $f \cdot k_{LS}$ (Satterfied et al., 1978; Rao and Drinkenburg, 1985; Lakota and Levec, 1990). Nevertheless, in these previous correlations, partial wetting was not taken into account, the whole solid surface being referred as the transfer surface, and only water/air system was used. In the present experimental study performed with hydrocarbon fluid, the effect of liquid velocity on mass transfer conductance appears to be quite significant at very low liquid superficial velocity. This correlation gives an accurate prediction of k_{LS} except for $Re_L/\varepsilon_L < 100$ where gas inertia has a greater effect which overpasses the change of local liquid velocity. For $V_{SL} < 10^{-3}$ m/s the gas momentum may modify the structure of thin films (less zones with stagnant films) and thus enhance the liquid/solid mass transfer. Nevertheless with only two values of gas flow rate and two liquid velocities in this range it is not possible to properly account for gas inertia in the k_{LS} model for the lowest liquid superficial velocities ($V_{SL} < 10^{-3}$ m/s).

The following correlation can also be used to determine global liquid/solid mass transfer efficiency:

$$f \times Sh = 8.58 \times 10^{-4} \left(\frac{Re_L}{\varepsilon_L}\right)^{1.91} Sc_L^{1/3}. \quad (5)$$

4. Conclusions

A wide parametric study has been performed to determine the variation of the wetting efficiency as a function of operating conditions (pressure, flow rates), bed geometry (particle shape and size) and physico-chemistry (liquid/solid contact angle). This data bank has been used to develop a new correlation for wetting efficiency since deviation was too large with previous ones. Finally liquid/solid mass transfer has been evaluated in partial wetting conditions by solid dissolution in an organic liquid. The results show that wetting efficiency only impacts on liquid/solid interfacial area and does not interfere with the liquid/solid mass transfer conductance that mainly relies on a liquid interstitial Reynolds number.

Notation

a_{LS}	effective liquid/solid interfacial area, m^2/m^3 ($a_{LS} = f\frac{6\varepsilon_B}{d_p}$)
C	β-naphthol concentration measured at bed outlet, mol/m^3
C^*	β-naphthol concentration measured at equilibrium condition, mol/m^3
d_p	particle diameter, m
D_m	molecular diffusion coefficient of β-naphthol in heptane, m^2/s
f	wetting efficiency, [–]
Ga_L, Ga'_L	liquid Galileo numbers, [–] $Ga_L = \left(\frac{d_p^3 g \rho_L^2}{\mu_L^2}\right)$, $Ga'_L = d_p\left(\frac{g\rho_L^2}{\mu_L^2}\right)^{1/3}$
k_{LS}	liquid solid mass transfer coefficient, m/s
Q_L	liquid flow rate, m^3/s
Re_L	liquid Reynolds number based on superficial velocity, [–] ($Re_L = \frac{V_{SL} d_p \rho_L}{\mu_L}$)
S	bed cross-section area, m^2
Sc_L	liquid Schmidt number, [–] ($Sc_L = \frac{\mu_L}{\rho_L D_m}$)
Sh	liquid/solid Sherwood number, [–] ($Sh = \frac{k_{LS}}{d_p D_m}$)
V_{SG}	superficial gas velocity, m/s
V_{SL}	superficial liquid velocity, m/s
Z	β-naphthol particles bed length, m

Greek letters

γ_L	liquid/vapour surface tension, N/m
γ_{LS}	liquid/solid surface tension, N/m
ε_B	bed porosity, [–]
ε_L	liquid holdup, [–]
θ_c	contact angle, °
μ_G	dynamic viscosity of gas, Pa s
μ_L	dynamic viscosity of liquid, Pa s
ρ_G	gas density, kg/m^3
ρ_L	liquid density, kg/m^3

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