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Volumetric mass transfer coefficient in a slurry bubble column operating in the heterogeneous flow regime

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Abstract

We report the results of an experimental study of the gas holdup, ε_G, and volumetric mass transfer coefficient, k_La, in a bubble column slurry reactor of 0.1 m diameter operated at ambient temperature and pressure conditions. The superficial gas velocity U was varied in the range 0–0.4 m/s, spanning both the homogeneous and churn-turbulent flow regimes. Air was used as the gas phase. The liquid phase used was C_9–C_{11} paraffin oil containing varying volume fractions ($\varepsilon_S = 0$, 0.05, 0.10, 0.15, 0.20 and 0.25) of porous catalyst (alumina catalyst support, $10\% < 10\,\mu m$; $50\% < 16\,\mu m$; $90\% < 39\,\mu m$). With increasing slurry concentrations, ε_G is significantly reduced due to enhanced bubble coalescence. For superficial gas velocities $U > 0.10\,m/s$ the volumetric mass transfer coefficient per unit volume, k_La/ε_G, was found to be practically independent of U and has values in the range 0.36–0.55 s^{-1}. From estimations of the sizes of the bubble size from literature correlation it is found that the "large" bubble mass transfer coefficient is about one order of magnitude higher than predicted by literature correlations.

Keywords: Bubble columns; Slurries; Mass transfer coefficient; Heterogeneous flow regime

1. Introduction

Bubble columns are widely used in industry for carrying out a variety of chemical reactions such as hydrogenations, chlorinations, and oxidations (Deckwer, 1992). There is currently a great deal of academic and industrial interest in the Fischer–Tropsch (FT) synthesis in the context of the conversion of remote natural gas to liquid transportation fuels. It is now widely accepted that the bubble column slurry reactor is the best choice of reactor type for large scale plants with capacities of the order of 40,000 bbl/day of liquid hydrocarbon product (Davis, 2002; Eisenberg et al., 1999; Espinoza et al., 1999; Krishna and Sie, 2000; Sie and Krishna, 1999). The superficial gas velocity U in the FT bubble column reactor is in the range of 0.1–0.4 m/s depending the catalyst activity and the catalyst concentration in the slurry phase (Maretto and Krishna, 1999). For high reactor productivities, the highest slurry concentrations consistent with catalyst handleability should be used. In practice the volume fraction of catalyst in the slurry phase, ε_S, is of the order of 0.15–0.3 (Maretto and Krishna, 1999, 2001). At these high slurry concentrations the gas dispersion consists predominantly of fast-rising "large" bubbles (Krishna et al., 1997). The economic success of the FT process largely depends on the ability to achieve deep syngas conversions, say exceeding 95%. Reliable design of the reactor to achieve such high conversion levels, requires reasonably accurate information on gas hold-up ε_G and the volumetric mass transfer coefficient k_La. While there have been several experimental studies on mass transfer in slurry bubble columns (for literature surveys see Inga and Morsi, 1999, Behkish et al., 2002), most of the published work is restricted to values of $U < 0.2\,m/s$ with low slurry concentrations.

The major objective of this paper is to generate data on mass transfer in bubble columns operating under conditions

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relevant to the FT process, with superficial gas velocities ranging up to 0.4 m/s. We choose a liquid that mimics the properties (viscosity, surface tension, density) of the liquid phase in the commercial reactor under reaction conditions. The influence of slurry concentration is studied in steps ranging to 25 vol%. The generated data can be expected to be directly usable in scaling up the FT reactor.

2. Experimental

Gas holdup and volumetric mass transfer experiments were carried out in a 0.1 m-diameter polyacrylate bubble column. The experimental set-up is shown in Fig. 1. The column had a 1 mm-thick brass plate gas distributor with perforated holes of 0.5 mm diameter arranged on a triangular pitch of 7 mm. This resulted in a total of 199 holes on the distributor plate. The rate of air flow into the column was controlled by the use of one of four pre-calibrated *Sho-Rate Brooks* rotameters aligned in parallel. Nitrogen flow (employed for stripping out dissolved oxygen) was regulated by the use of a manually operated control valve connected to the bottom of the column. A venting system attached to the top of column safely lead and disposed of evaporating liquid.

Air was used as the gas phase in all experimental runs with a predominantly C_9–C_{11} *n*-paraffin mixture cut as the liquid phase. Sasol PURALOX ScCa 5/170, an alumina-based catalyst particle carrier was employed as the solid phase. The properties of the catalyst carrier are similar to those envisaged for use in the commercial FT reactor. Properties of the paraffin mixture and alumina particles utilised are given in Tables 1 and 2. The slurry concentration, ε_S was varied in the range 0, 0.05, 0.1, 0.15, 0.2 and 0.25. Slurry concentration is defined throughout this paper as the volume fraction of solids in gas-free slurry. The pore volume of the catalyst particles, which is liquid filled during experiments, was counted as being part of the solid phase. At the start of each experimental investigation, the clear liquid (or slurry) height, H_0 was set at 1.34 m (with no solids in the system) and 1.36 m (for slurry experiments). During experiments, the liquid phase was regularly replenished due to the loss of some liquid as a result of evaporation.

Gas holdup was determined by visual measurements. In determining the gas holdup in each experiment, the gas flow rate was adjusted using one rotameter at a time. Sufficient time was given for steady state to be reached in the column after which the increase in dispersion height was recorded. The total gas holdup, ε_G is defined as:

$$\varepsilon_G = \frac{H - H_0}{H}, \tag{1}$$

where H_0 is the ungassed column height and H is the column dispersion height due to the presence of gas bubbles.

The volumetric mass transfer coefficient $k_L a$ was determined by means of a dynamic oxygen absorption technique (Letzel et al., 1999). An oxygen electrode (*Yellow Springs Incorporated* Model 5331) inserted at a point that corresponded to the ungassed bed height was used to measure the change in dissolved oxygen concentration. Readings given by the electrode were fed to a PC via an ammeter and an analogue-to digital converter card (see Fig. 1). The change in dissolved oxygen concentration was reflected as a change in electrical current displayed on the ammeter. The oxygen electrode was made sensitive to the presence of dissolved oxygen by the application of a 0.13 g/ml KCl solution between its tip and an outer membrane, made of Teflon. Before the start of each experimental run, the membrane surrounding the tip of the electrode was changed and the time constant of the oxygen sensor determined. The sensor constant value corresponds to an inherent delay in readings obtained as a result of the fact that the oxygen sensor has a finite response time. In determining the sensor constant, two continuously stirred glass beakers were used, the first containing the liquid paraffin oil, and the other, the liquid paraffin and alumina particles in the same ratio as was present in the bubble column for a given experiment. Nitrogen was continuously bubbled into the first beaker and air into the other, such that the liquid phases in both beakers became completely saturated with dissolved gas over time. The sensor was first placed in the nitrogen-saturated liquid and after registering a negligible oxygen concentration, was instantaneously transferred to the oxygen-saturated liquid.

Assuming perfect mixing in the liquid phase, the oxygen concentration value indicated by the sensor, C_{sensor} is given by:

$$\frac{\mathrm{d}C_{\text{sensor}}}{\mathrm{d}t} = k_{\text{sensor}}(C_L^* - C_{\text{sensor}}), \tag{2}$$

Table 1
Properties of paraffin oil at 298 K

	Paraffin oil
Density	726 kg/m^3
Viscosity	0.85 mPa s
Surface tension	23.2 mN/m
Diffusivity of oxygen	3.69×10^{-9} m^2/s
Composition	$\leqslant C_8$: 3.3%; C_9 : 36.3%; C_{10} : 34.5%; C_{11} : 23.8%; $> C_{12}$: 1.9%

Table 2
Properties of catalyst carrier

Al_2O_3 content	98%
Skeletal density	3900 kg/m^3
Specific surface area	192 m^2/g
Particle porosity	70%
Particle size distribution	10% < 10 μm; 50% < 16 μm; 90% < 39 μm

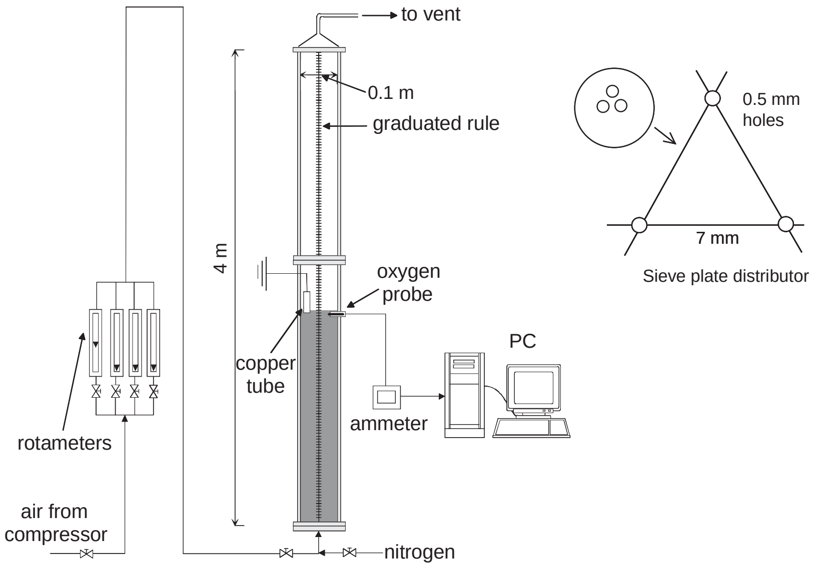

Fig. 1. Experimental set-up of the 0.1 m diameter slurry bubble column. Further details are available on our website (Vandu et al., 2004a,b).

where k_{sensor} is the sensor time constant. Integrating Eq. (2) gives:

$$\frac{C_{\text{sensor}}}{C_L^*} = 1 - e^{-k_{\text{sensor}} t}. \tag{3}$$

Fig. 2a shows a typical sensor response as well as the fit obtained with Eq. (3). The data presented in Fig. 2a is for the C_9–C_{11} *n*-paraffin oil slurry with $\varepsilon_S = 0.15$ in the oxygen-saturated beaker. The resulting k_{sensor} value in this case is $0.84\,\text{s}^{-1}$. Fig. 2b shows that there is no significant dependence of k_{sensor} on slurry concentration for the C_9–C_{11} *n*-paraffin liquid with k_{sensor} varying between 0.77 and $1.01\,\text{s}^{-1}$. The sensor constant k_{sensor} was determined thrice for each slurry concentration and the average value used in estimating $k_L a$.

For the determination of $k_L a$ in the bubble column, dissolved oxygen was stripped from the liquid phase to a negligible concentration by the use of nitrogen sparged through the gas distributor. After the stripping operation, a step input of air was introduced into the column, with the uptake of oxygen into the liquid phase continuously monitored by the oxygen sensor. Sufficient time was given in each experimental run for the oxygen saturation concentration in the liquid, C_L^* to be reached. With the assumptions of a perfectly mixed liquid and negligible oxygen depletion from the gas bubbles, the rate of oxygen uptake is described by:

$$\frac{\mathrm{d}C_L}{\mathrm{d}t} = k_L a_L (C_L^* - C_L), \tag{4}$$

where $k_L a_L$ is the volumetric mass transfer coefficient per unit volume of *liquid* in the bubble column. We verified that the assumption of well-mixed liquid phase was indeed a good one for our experimental conditions due to the high degree of liquid circulations in the churn-turbulent regime. The sensor delay equation resulting from the finite response time of the oxygen sensor is given by:

$$\frac{\mathrm{d}C_{\text{sensor}}}{\mathrm{d}t} = k_{\text{sensor}}(C_L - C_{\text{sensor}}), \tag{5}$$

Analytical solution of Eq. (4), along with the sensor delay equation (5) yields:

$$\frac{C_{\text{sensor}}}{C_L^*} = 1 - \frac{1}{k_{\text{sensor}} - k_L a_L} \times [k_{\text{sensor}} \mathrm{e}^{-k_L a_L t} - k_L a_L e^{-k_{\text{sensor}} t}]. \tag{6}$$

The volumetric mass transfer coefficient per unit volume of *dispersion* (gas + liquid + solid), $k_L a$ is obtained from:

$$k_L a = k_L a_L (1 - \varepsilon_G)(1 - f_S \varepsilon_S), \tag{7}$$

where f_S is the volume fraction of the solid particles due to the solid skeleton structure (i.e., not considering the solid pore volume contribution). Gas–liquid mass transfer can occur in the pores of the solid particles, which become filled with liquid during experiments, making it necessary to account for this contribution. Typical oxygen absorption dynamics for both liquids employed at three different values of U are shown in Fig. 2c for the paraffin oil slurry with $\varepsilon_S = 0.25$. It is important to note that even at the highest gas velocities employed, based on all experiments carried out, the oxygen absorption dynamics were at least three times slower than the sensor dynamics. This in effect meant that suitably accurate estimations of $k_L a$ could be obtained. In any event the sensor dynamics was routinely accounted for by use of Eq. (6).

Alumina particles are poor conductors of electricity but good supporters of electrostatic field i.e. they are excellent dielectric materials. One consequence of this property of

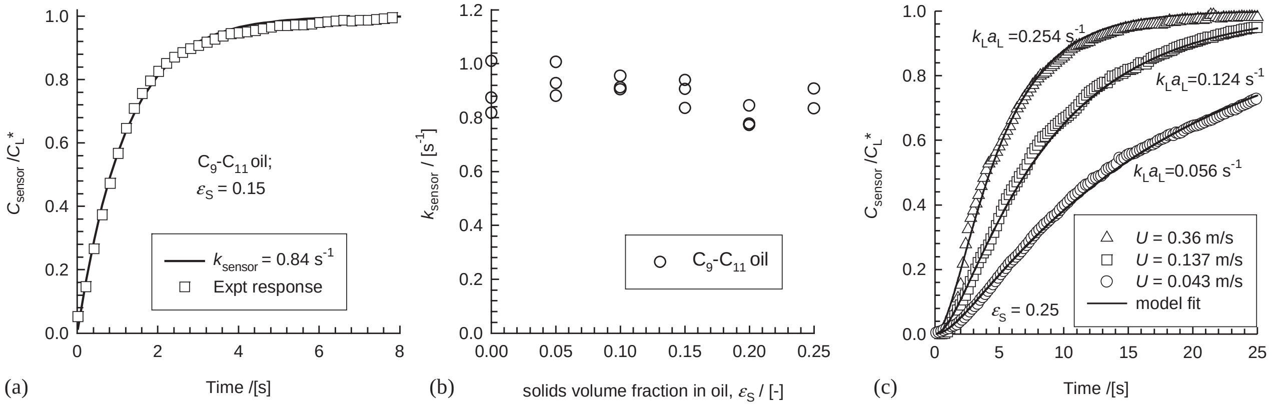

Fig. 2. (a) Typical sensor response obtained for $\varepsilon_S = 0.15$ in the C_9–C_{11} *n*-paraffin oil slurry with a resulting k_{sensor} value of $0.84\,\text{s}^{-1}$. (b) Effect of slurry concentration, ε_S on sensor time constant, k_{sensor}. (c) Oxygen absorption dynamics for C_9–C_{11} *n*-paraffin slurry with $\varepsilon_S = 0.25$.

alumina particles is that they can become fairly good conductors of electricity in the presence of large electrostatic fields. This in turn has an enormously negative effect on $k_L a$ measurements because the alumina particles were continuously deposited on the oxygen sensor, severely affecting its ability to detect for the presence of dissolved oxygen. This problem was fully overcome by inserting a 50 ml hollow copper tube in the column (see Fig. 1). The tube was properly earthed using a thin copper wire and provided a very large conducting surface compared to the oxygen sensor. In this way, during experiments, alumina particles were deposited on the surface of the copper tube but not at all on the oxygen sensor. During gas holdup experiments, the additional increase in dispersion volume resulting from the presence of the hollow copper tube was corrected for.

Further details of the experimental set-up and measurement techniques, including photographs are available on our website (Vandu et al., 2004a,b).

3. Results and discussion

The open symbols in Figs. 3a–f represent the gas holdup data obtained with various slurry concentrations ($\varepsilon_S = 0$, 0.05, 0.10, 0.15, 0.20 and 0.25) for a range of superficial gas velocities. Addition of catalyst particles tends to reduce the gas holdup, ε_G to a significant extent, consistent with our earlier work (Krishna et al., 1997). The reduction in gas holdup with increasing ε_S is due to the decrease in the small bubble population, resulting from an increase in slurry viscosity. Foaming was observed in the C_9–C_{11} paraffin oil at superficial gas velocities in the range $0.2 < U < 0.15$ when ε_S=0, but was fully suppressed when 0.05 volume fraction of catalyst particles was added. In Fig. 3a, for $\varepsilon_S = 0$, we note a small maximum in the gas holdup at U=0.07 m/s. Visual observations using fast video imaging techniques (Vandu et al., 2004a,b,c) showed that for the paraffin oil at $U = 0.07\,\text{m/s}$ we have formation of the first "large" bubble and therefore this point can be taken to be the regime transition gas velocity U_{trans}. With increasing slurry concentration the "window" of operation of the column in the homogeneous regime becomes progressively narrower. Estimates of the transition gas velocity U_{trans} and the corresponding gas holdup at the regime transition point, $\varepsilon_{\text{trans}}$ are summarized in Table 3. The gas holdup below the regime transition point consists predominantly of "small" bubbles. The rise velocity of these "small" bubbles V_{sb} can be determined from the slope of the holdup curve below U_{trans}; the values of V_{sb} thus obtained are summarized in Table 3.

The gas holdup for homogeneous regime of operation is given by

$$\varepsilon = \frac{U}{V_{Sb}}; \quad U < U_{\text{trans}}, \tag{8}$$

whereas in the heterogeneous flow regime, assuming two bubble classes, "small" and "large", we have the following gas holdup model (Krishna et al., 1999)

$$\varepsilon = \varepsilon_{Lb} + \varepsilon_{Sb} = \frac{(U - U_{\text{trans}})}{V_{Lb}} + \varepsilon_{\text{trans}}\left[1 - \frac{(U - U_{\text{trans}})}{V_{Lb}}\right]; \quad U > U_{\text{trans}}, \tag{9}$$

where V_{Lb} and ε_{Lb} represents the rise velocity and holdup, respectively, of the "large" bubbles. For estimation of the rise velocity of the large bubbles we use the extended Davies–Taylor relationship:

$$V_{Lb} = 0.71\sqrt{g d_{Lb}}(SF)(AF), \tag{10}$$

suggested by Krishna et al. (1999), wherein the scale correction factor SF:

$$\begin{aligned} &SF = 1, \quad \text{for } d_{Lb}/D_T < 0.125;\\ &SF = 1.13\exp(-d_{Lb} - D_T) \quad \text{for } 0.125 > d_b/D_T < 0.6;\\ &SF = 0.496\sqrt{D_T/d_{Lb}} \quad \text{for } d_b/D_T > 0.6. \end{aligned} \tag{11}$$

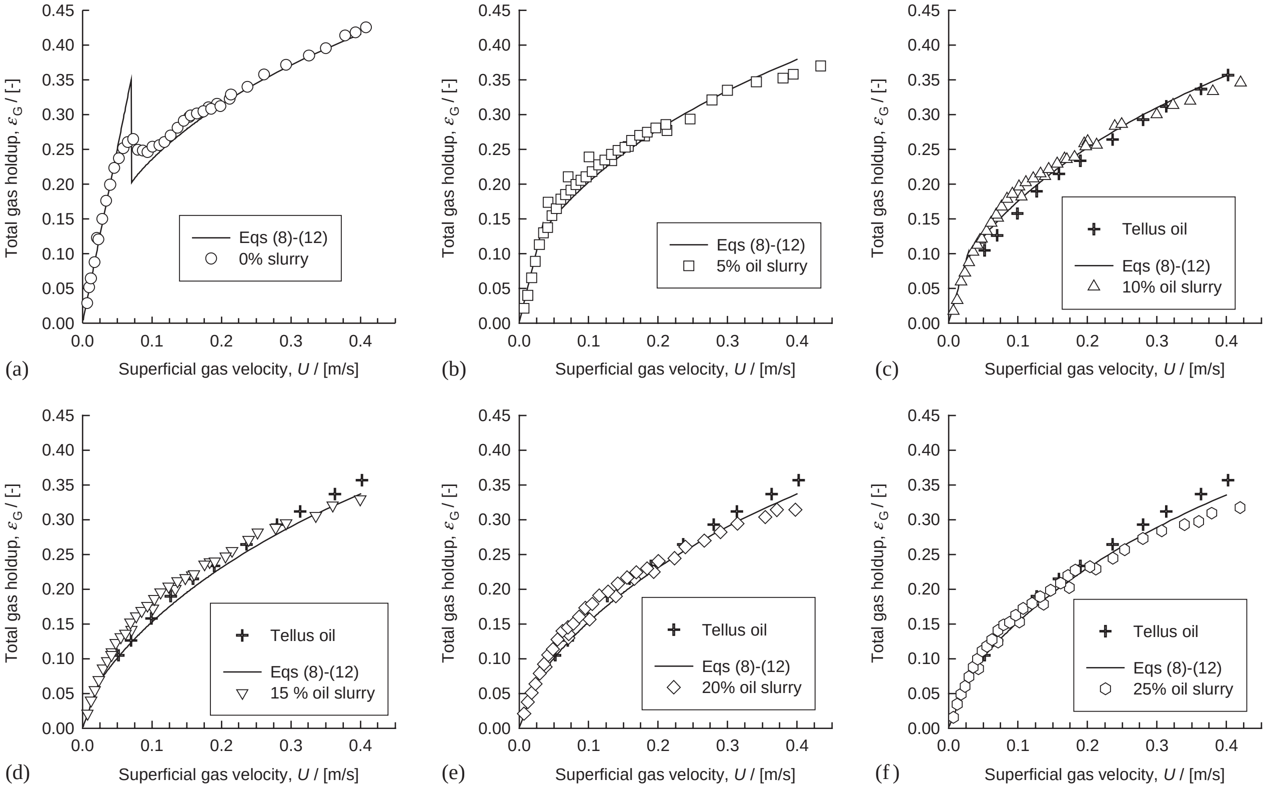

Fig. 3. Influence of superficial gas velocity U on gas holdup, ε_G for slurry concentrations $\varepsilon_S = 0$, 0.05, 0.10, 0.15, 0.20 and 0.25, with C_9–C_{11} paraffin oil as the liquid phase. Included are the gas holdup data for paraffin oil slurries reported by Vandu et al. (2004c) in a rectangular bubble column and for Tellus oil (denoted by cross-hairs) in a 0.1 m diameter column (Vandu and Krishna, 2004a).

Table 3
Data on transition velocity, transition gas holdup and small bubble rise velocity

Slurry concentration	U_{trans} (m/s)	$\varepsilon_{\text{trans}}$ (−)	V_{sb} (m/s)
$\varepsilon_S = 0.0$	0.07	0.2	0.2
$\varepsilon_S = 0.05$	0.035	0.13	0.24
$\varepsilon_S = 0.10$	0.03	0.095	0.28
$\varepsilon_S = 0.15$	0.025	0.08	0.32
$\varepsilon_S = 0.20$	0.025	0.065	0.36
$\varepsilon_S = 0.25$	0.02	0.06	0.4

These data were obtained from Vandu et al. (2004c).

and the acceleration factor AF

$$AF = 2.25 + 4.09(U - U_{\text{trans}}), \tag{12}$$

developed on the basis of measurements for Tellus oil ($\mu_L = 75\,\text{mPa/s}$). This acceleration is due to wake interactions and this factor increases as the distance between the large bubbles decreases. In our previous work (Krishna et al., 2000) we had demonstrated the equivalence in the hydrodynamics of bubble columns operating with Tellus oil and concentrated paraffin oil slurries. Indeed, the gas holdup data for Tellus oil (denoted by cross-hairs in Fig. 3) obtained in the same 0.1 m diameter column agrees very well with the data for paraffin oil slurries with catalyst concentrations in excess of 10%.

The continuous lines in Figs. 3a–f represent the calculations of the gas holdup model following Eqs (8)–(12). The agreement with the experimental data on gas holdup for paraffin oil slurries is very good indeed and provides justification of the two-bubble class approach to describe gas holdup.

The values of the volumetric mass transfer coefficient k_La for the C_9–C_{11} paraffin oil slurries are shown in Fig. 4a. Interestingly, we observe that k_La shows virtually no dependence on ε_S. In other published studies on the influence of slurry concentrations on k_La, a variety of trends have been discerned. In a 0.14 m diameter column for air–water system with glass beads as the solid phase, it was shown that k_La *decreases* with an increase in solid concentration (Shah et al., 1982). With hexane and iron oxides catalyst as well as a C_{10}–C_{16} oil and glass beads as the liquid and solid phases, it was shown that k_La generally decreases with an increase in slurry concentration in a 0.316 m diameter bubble column (Behkish et al., 2002). Furthermore, these investigators observed that in the hexane-iron oxides catalyst system, *increased* with slurry concentration in going from $\varepsilon_S = 0$ to 0.038 but then decreased with a further increase in catalyst concentration above this value. Thus, while a decrease in k_La with increasing ε_S had previously been

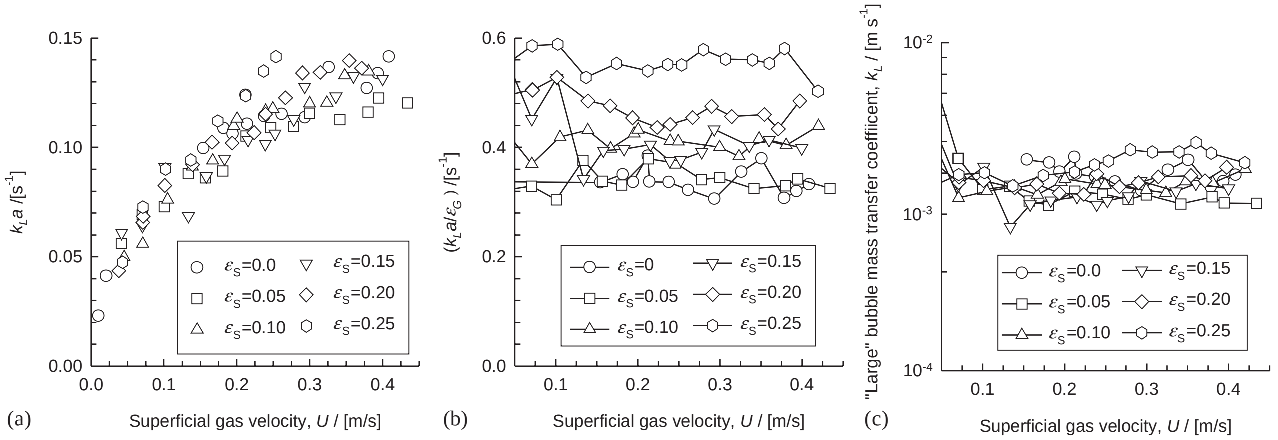

Fig. 4. Influence of superficial gas velocity U on (a) volumetric mass transfer coefficient, $k_L a$, (b) $k_L a/\varepsilon_G$ and (c) k_L for slurry concentrations $\varepsilon_S = 0$, 0.05, 0.10, 0.15, 0.20 and 0.25, with C_9–C_{11} paraffin oil as the liquid phase.

observed, though at much lower gas velocities than were used in this study, the same is not the case for an increase in $k_L a$ with increasing ε_S up to a value of 0.25. Our own earlier work for paraffin oil slurries with porous *silica* as the catalyst, we had observed a *decrease* in $k_L a$ with increasing ε_S (Vandu and Krishna, 2004a). Based on these observations it must be concluded that the *nature* of the solid particles *and* of the liquid phase are important determinants on the variation of $k_L a$ with ε_S. It is also clear that the correlation proposed by Behkish et al. (2002) will not be successful in describing our $k_L a$ data because this correlation anticipates a *decrease* with increasing ε_S.

Further insight is obtained when we consider the variation of the volumetric mass transfer coefficient per unit volume of bubbles, $k_L a/\varepsilon_G$ with U; see Fig. 4b. For superficial gas velocities $U > 0.10\,\mathrm{m/s}$ the value of $k_L a/\varepsilon_G$ is found to be practically independent of U and has values in the range 0.36–$0.55\,\mathrm{s}^{-1}$. For $U > 0.10\,\mathrm{m/s}$ we expect that the mass transfer to be dictated by the fast-rising large bubbles (De Swart et al., 1996). Recent work on video imaging in a rectangular bubble column indicated that the correlation of Krishna et al. (1999):

$$d_{Lb} = 0.069(U - U_{\mathrm{trans}})^{0.376}, \tag{13}$$

provides a good estimate of the "large" bubble size. From the estimate of the specific interfacial area of large bubbles, $a = 6\varepsilon_G/d_{Lb}$ we can back calculate the mass transfer coefficient k_L. The k_L values for large bubbles are practically independent of U and have values in the range of 0.002–0.003; see Fig. 4c. These k_L values are about one order of magnitude higher than that estimated from literature correlations (Akita and Yoshida, 1973, 1974). This high mass transfer coefficient can be attributed to the frequent breakup and coalescence phenomena (De Swart et al., 1996).

Our earlier work (Vandu and Krishna, 2004a,b) had shown that ε_G and $k_L a/\varepsilon_G$ are virtually scale independent for the churn-turbulent regime of operation and therefore the results presented in the present study are usable for scale up.

4. Conclusions

Gas holdup and mass transfer data have been generated in bubble columns operating with concentrated slurries at high superficial gas velocities. The following major conclusions can be drawn from this work.

1. The gas holdup is significantly reduced when the concentration of catalyst is increased. This reduction in gas holdup is primarily due to the reduction in the holdup of the small bubble population.
2. The variation in the gas holdup is well represented by the model of Krishna et al. (1999), summarized in Eqs. (8)–(12). For this estimation it is important to have reliable information on the transition gas velocity U_{trans} and the corresponding gas holdup at the regime transition point, $\varepsilon_{\mathrm{trans}}$.
3. For the paraffin oil slurry with alumina catalyst the volumetric mass transfer coefficient $k_L a$ is practically independent of the slurry concentration. This finding is not in agreement with the experiments reported in the literature, that show either an *increase* (Shah et al., 1982) or *decrease* (Behkish et al., 2002; Vandu and Krishna, 2004b) with increasing slurry concentration. It appears that the nature of the solid phase *and* the liquid is a crucial factor in determining the influence of increased catalyst concentrations. Further work is required to investigate the underlying physics.
4. Estimates of the mass transfer coefficient k_L for large bubbles show these to be about one order of magnitude larger than those predicted on the basis of literature correlations (Akita and Yoshida, 1973, 1974). This is due to frequent coalescence and breakup phenomena, as explained by De Swart et al. (1996).

Notation

a_L	gas–liquid interfacial area per unit liquid volume, m^{-1}
a	gas–liquid interfacial area per unit dispersion volume, m^{-1}
C_L	oxygen concentration in the liquid phase, arbitrary units
C_L^*	saturation concentration of oxygen in liquid, arbitrary units
C_{sensor}	liquid phase oxygen concentration given by sensor, arbitrary units
AF	acceleration factor, dimensionless
d_{Lb}	diameter of large bubble, m
f_S	volume fraction of catalyst that is (non-porous) skeleton , dimensionless
H	dispersion height in the column, m
H_0	height of ungassed column, m
k_L	liquid-side mass transfer coefficient, m/s
k_{sensor}	sensor time constant, s^{-1}
SF	scale correction factor, dimensionless
t	time, s
U	superficial gas velocity, m/s
V_{Lb}	rise velocity of large bubbles, m/s
V_{Sb}	rise velocity of small bubbles, m/s

Greek letters

ε_G	total gas holdup, dimensionless
ε_S	volume fraction of catalyst in the slurry, dimensionless

Subscripts

G	referring to gas phase
L	referring to liquid
Lb	referring to large bubble
trans	referring to the regime transition point
S	referring to porous solid (catalyst)
Sb	referring to small bubble

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