

Experimental measurements of volumetric mass transfer coefficient by the dynamic pressure-step method in internal loop airlift reactors of different scale

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Abstract

The volumetric mass transfer coefficient ($k_L a$) of oxygen was measured in internal loop airlift reactors of different scale using the dynamic pressure-step (DPM) method. The $k_L a$ values obtained by the DPM appear as the most reliable as they were found to be independent of the oxygen concentration in the inlet gas no matter the reactor scale. The effect of reactor scale on $k_L a$ values was studied in three airlift reactors of different working volume (12, 40 and 195 dm³) but with similar geometric configuration. The ratio between the riser and the downcomer cross-sectional areas and the slighthness of the column were taken as similarity criteria. As the liquid phase deionized water was used at a temperature of 27.5 °C. Both the gas hold-up (ε_G) and the mass transfer coefficient ($k_L a$) increased with increasing superficial gas velocity. The experimental dependencies of the $k_L a$ and ε_G values versus the superficial gas velocity were very similar, what indicated a strong dependency of the $k_L a$ values on gas hold-up.

The variation of the mass transfer coefficient with the gas hold-up was described using an appropriate correlation taken from the literature. The parameters of this correlation were almost the same for all working volumes of the reactor. Thus, it was found that even in larger reactors only the gas hold-up is the main key parameter influencing the $k_L a$ values.

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Keywords: Scale-up; Internal loop airlift reactor; Volumetric mass transfer coefficient; Dynamic pressure-step method

1. Introduction

Oxygen mass transfer is one of the most important design parameters of gas–liquid (–solid) reactors employed for chemical and biochemical applications. Any shortage of oxygen significantly affects the process performance. An ideal reactor should have a maximal transfer rate, with efficient mixing at a minimum energy input [1–3]. Bioprocesses are usually carried out in aqueous media, where the solubility of oxygen is low. However, the rate of oxygen utilization by microorganisms or enzymes is high. Thus, reliable estimation of oxygen mass transfer is important, particularly in the case of optimalization of

reactor design and in scale-up processes. The volumetric mass transfer coefficient ($k_L a$) is the rate of gas transfer across the gas–liquid interface per unit liquid volume and per unit driving force. Because of difficulties in calculating local values, the mass transfer coefficient is averaged over the whole volume of the reactor.

Generally, depending upon the principle used, the methods for determining mass transfer coefficients may be divided into two groups: chemical and physical methods. Due to the complex nature of oxygen mass transfer phenomena in biosystems and characteristics of the methods employed, most studies were performed just using the model media and consequently translated for describing real biosystems. The best should be the application of methods directly in the biosystem. Thus, “correct” values of $k_L a$ might be obtained without any damage of the biological environment.

In this work the dynamic pressure-step method (DPM) has been utilized. Due to the fact that this method does not use any

Abbreviations: ALR, airlift reactor; DPM, dynamic pressure-step method; ILALR, internal loop airlift reactor

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Nomenclature

A	cross-sectional area (m^2)
C_1	parameter in Eqs. (4) and (12) (m s^{-1}) ^{-2/3}
C_2	parameter in Eq. (12) (W m^{-3}) ^{-0.8}
D	diameter (m)
g	gravitation acceleration (m s^{-2})
H	height (m)
K	parameter in Eq. (1) ($\text{m}^{-0.4} \text{s}^{-0.6}$)
K_1	parameter in Eqs. (5) and (9) (s^{-1})
K_2	parameter in Eqs. (6) and (10) (m s^{-1}) ^{-(1.07-0.67n)} s^{-1}
K_3	parameter in Eq. (11) (W m^{-3}) ^{-0.8} s^{-1}
$k_{L,a}$	volumetric mass transfer coefficient (s^{-1})
m	exponent in Eqs. (4), (5) and (6)
n	exponent in Eqs. (1), (5) and (6)
P_G	power input (W)
R^2	correlation coefficient
t_c	circulation time (s)
U	superficial gas velocity (m s^{-1})
V	volume (m^3)

Greek letters

ε	gas hold-up
ρ	density kg (m^{-3})

Subscripts

B	distance from the bottom of ALR to the beginning of the riser
C	column
D	downcomer
G	gas
L	liquid
O	oxygen
R	riser
S	separator

chemicals and bioprocess inhibitors, it might be successfully applicable in bioprocesses [4].

The DPM had originally been developed for mechanically agitated tanks. The method was then applied for measurements of $k_{L,a}$ values in mechanically agitated bioreactors of different scale using coalescent and non-coalescent media, and pure oxygen or air as a gas supply [4–6]. In this type of bioreactors also the reliability of the DPM was confirmed.

To our knowledge, the DPM was tested in a pneumatically agitated bioreactor for the first time in the works of Blažej et al. [7–9]. These papers reported the measurements of $k_{L,a}$ values in a 40 dm³ internal loop airlift reactor (ILALR) using coalescent and non-coalescent media. Moreover, $k_{L,a}$ values obtained by the DPM in the 40 dm³ ILALR were compared with other methods (gassing-out and sulfite oxidation methods). Generally, the previous studies have shown the capability of implementing the DPM for evaluating $k_{L,a}$ values in a pneumatically agitated column.

Basing on the results of the works of Blažej et al. [7–9], the present deals with $k_{L,a}$ measurements using the DPM in three ILALRs of different scale (12, 40 and 195 dm³) and a coalescent medium. The total gas hold-up was measured as well. The measured values were fitted with the semi-empirical correlations originally derived by Bello et al. [10]. Finally, a comparison of the fitted parameters involved in the correlation describing the measured values was done.

2. Theory

2.1. The dynamic pressure-step method

The basic principle of the DPM lies in the employment of a small change of the total pressure (about ± 15 kPa) in a reactor. Thus, a simultaneous change in oxygen concentration in all gas bubbles in the dispersion is caused regardless of the flow pattern and the concentration change in the dispersion under the model assumption of ideal mixing of the gas phase [11]. A detailed description of the DPM and the equations used for evaluating $k_{L,a}$ values can be found in the original works of Linek et al. [11–13], and in our papers [7,9]. The oxygen concentration in the liquid starts to change as a consequence of a step change in pressure, owing to the change in the driving force for oxygen mass transfer. Evaluation of the experimental $k_{L,a}$ value was performed by fitting the experimental data (both the pressure and the liquid oxygen concentration) with the model [7]. Following assumptions for the model were made: constant gas hold-up, constant temperature, constant $k_{L,a}$ value, constant flow rate of the input gas and constant water vapour pressure in the gas phase as well as in the well-mixed gas and liquid phases referred to the pressure change.

2.2. Relating volumetric mass transfer values ($k_{L,a}$) to the gas hold-up (ε_G) and the superficial gas velocity (U_{GR})

The overall mass transfer coefficient is dependent on the gas hold-up and the superficial gas velocity as follows [10]:

$$k_{L,a} = K \left(1 + \frac{A_D}{A_R} \right)^{-0.4} U_{GR}^{0.4} \varepsilon_{GC}^{1-n} \quad (1)$$

The following relationship (2) between the gas hold-ups and the superficial gas velocity in the riser (U_{GR}) was proposed [10]:

$$\varepsilon_{GR} - \varepsilon_{GD} \propto U_{GR}^{2/3} \quad (2)$$

When a linear dependency between ε_{GR} and ε_{GD} is taken into account [10,14], relationship (2) can be rewritten into the following form:

$$\varepsilon_{GC} \propto U_{GR}^{2/3} \quad (3)$$

According to Bello et al. [10], ε_{GR} has been experimentally reported to decrease as the geometric parameter A_D/A_R increases at a constant value of U_{GR} . The same can be said about ε_{GC} values. Hence, a more appropriate form of relationship (3)

would be:

$$\varepsilon_{GC} = C_1 \left(1 + \frac{A_D}{A_R}\right)^{-m} U_{GR}^{2/3} \quad (4)$$

Using Eq. (4), Eq. (1) can be rewritten explicitly either as a function of ε_{GC} , Eq. (5), or as a function of U_{GR} , Eq. (6):

$$k_L a = K_1 \left(1 + \frac{A_D}{A_R}\right)^{-0.4+0.4m} \varepsilon_{GC}^{1.6-n} \quad (5)$$

$$k_L a = K_2 \left(1 + \frac{A_D}{A_R}\right)^{-0.4-m(1-n)} U_{GR}^{1.07-0.67n} \quad (6)$$

where the values of the parameters K_1 , K_2 , n and m have to be estimated experimentally.

In the original paper of Bello et al. [10] the semi-empirical relationship between $k_L a$ and the gas hold-up were related only to the gas hold-up in the riser. It is known that in the ILALRs the gas hold-up in the downcomer cannot be negligible [14,15]. It was reported that the ratio of the downcomer over the riser gas hold-up approaches values up to 0.9 depending on the working conditions [14]. Heijnen et al. [16] defined three circulation regimes in the ILALR. In Regime I, which is characteristic by low values of U_{GR} , the gas bubbles are present only in the riser (of course also in the bottom section and in the separator) and the downcomer gas hold-up is approximately equal to zero. In Regime II the gas bubbles penetrate into the downcomer. As

the liquid circulation velocity is increasing the head of the gas bubbles moves downward in the downcomer. When the head of the gas bubbles reaches the bottom of the downcomer, the gas bubbles start to circulate (Regime III). Hence, in this work the $k_L a$ values were related to the overall gas hold-up, Eq. (5).

2.3. Power input

In pneumatically agitated reactors, the power supply originates from two sources with respect to the liquid: isothermal expansion of the gas when it moves upward in the reactor and kinetic energy of the gas injected into the reactor. The kinetic energy contribution to the power input never exceeds 1.5% of the total power [17]. Consequently, in ALRs the specific power input can be written as a function of the superficial gas velocity:

$$\frac{P_G}{V_L} = \rho_{Lg} \left(1 + \frac{A_D}{A_R}\right)^{-1} U_{GR} \quad (7)$$

2.4. Experimental

The experimental equipment used in the investigation is schematically illustrated in Fig. 1. The measurements were carried out in three internal loop airlift reactors (IALRs) of different scale. The reactors were made of glass and the bottom was made of stainless steel. The total working volumes of the ILALRs were 12, 40 and 195 dm³. The three reactors were of

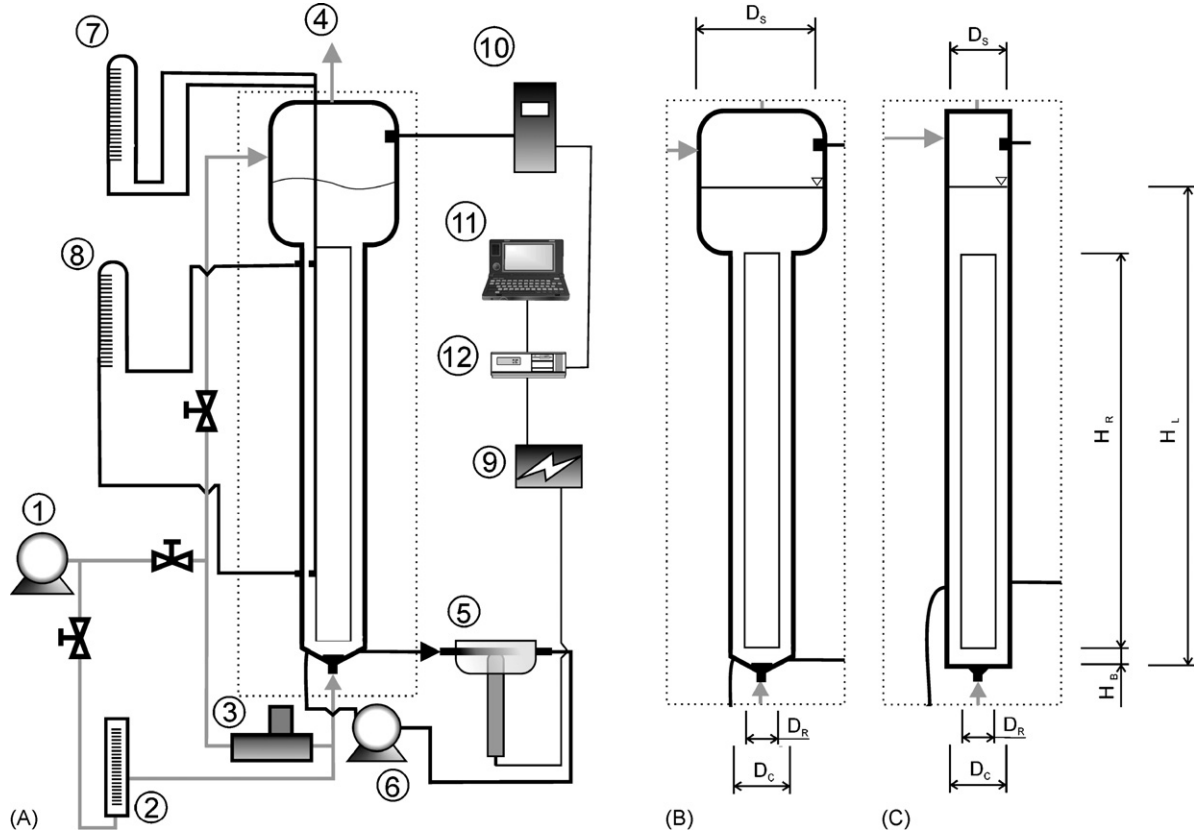


Fig. 1. (A) Experimental set-up: (1) air compressor, (2) rotameter, (3) inlet mass flow controller, (4) gas outlet, (5) oxygen probe placed in the bypass-cell, (6) peristaltic pump, (7 and 8) inverted U-tube manometers, (9) amplifier, (10) manometer, (11) PC, (12) A/D converter; (B) shape of the 12 and 40 dm³ ILALRs with an enlarged separator; (C) shape of the 195 dm³ ILALR with a simple separator.

Table 1
Geometrical details of the reactors used

Working volume (dm ³)	D_C (m)	H_L (m)	H_R (m)	D_R (m)	D_S (m)	H_B (m)	A_D/A_R	H_L/D_C
12	0.108	1.34	1.145	0.070	0.157	0.030	1.23	11
40	0.157	1.93	1.710	0.106	0.294	0.046	0.95	12
195	0.294	3.03	2.700	0.200	0.294	0.061	1.01	10

Table 2
Types of gas spargers used

Working volume (dm ³)	Material	Number of holes	Hole diameter (mm)
12	Teflon	25	0.5
40	Teflon	50	0.5
195	Stainless steel	90	1.0

similar geometry to avoid its influence on the hydrodynamic parameters. As similarity criteria were chosen:

- Column slowness (ratio of the column height to the column diameter).
- Ratio of the downcomer to the riser cross-sectional area (A_D/A_R).

Two smaller ILALRs (12 and 40 dm³) had an enlarged degassing zone and the 195 dm³ ILALR had a simple separator of the same diameter as that of the column, as can be seen in Fig. 1. Geometrical details of the ILALRs used are listed in Table 1. In all ILALRs only the internal tube was sparged, whereby the gas sparger was located at the bottom in form of a perforated plate made of teflon or stainless steel. The details of the gas spargers are given in Table 2.

In the reactors with the working volume of 12 and 40 dm³ the gas input was controlled by a rotameter. If a higher gas input was required (40 and 195 dm³ reactors), a mass flow controller (BROOKS-5853E) was employed. All experiments were carried out at a temperature of 27.5 °C, under atmospheric pressure. Air and deionized water were used as gas and liquid media.

The pressure-step increase was attained by means of an input gas bypass that was connected with the top section of the ILALR. Absolute pressure at the head of the column was measured by a digital manometer DB-1 (AIRFLOW, Lufttechnik GmbH). The concentration of dissolved oxygen in the liquid has been monitored using a polarographic oxygen probe (ASSET, Prague, Czech Republic) covered by a polypropylene (PP) membrane (thickness 8 μm) placed at the bottom of the reactor in the bypass-cell facing upwards. Both the oxygen probe and the absolute pressure signals were measured using A/D converters and recorded on a PC. A more detailed description of the experimental arrangement can be found elsewhere [7,9,14].

3. Results and discussion

Considering a reactor with circulation as one unit, the following criterium has to be fulfilled for $k_L a$ measurements [18]:

$$k_L a t_c \leq 2 \quad (8)$$

where t_c is the circulation time.

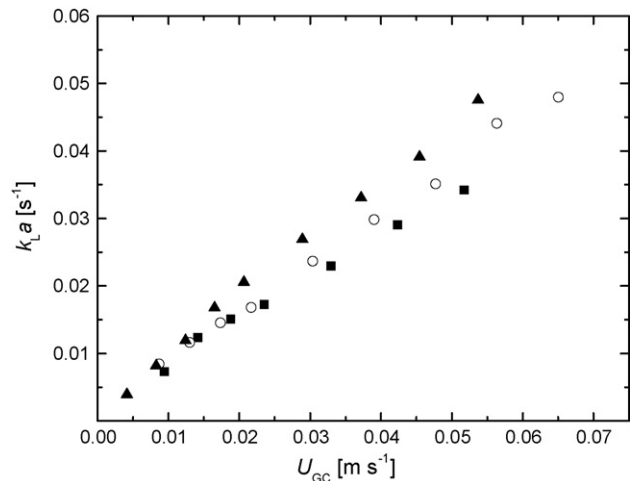


Fig. 2. Experimental dependencies of $k_L a$ values on U_{GC} (■) 12 dm³ ILALR, (○) 40 dm³ ILALR; (▲) 195 dm³ ILALR.

According to the values of circulation velocity in our reactors described previously [14] and the rate of oxygen transfer observed in this study, the above mentioned criterium is sufficiently fulfilled for all scales of our ILALRs and all experimental conditions used. In our case, the highest value of the term on the left hand side in Eq. (8) was 0.8. Therefore, all the ILALRs used were considered as one unit. Hence, the obtained volumetric mass transfer coefficients were related to the whole reactor volume.

The experimental dependency of $k_L a$ on U_{GC} is plotted in Fig. 2. A positive influence of the gas flow rate on $k_L a$ is evident

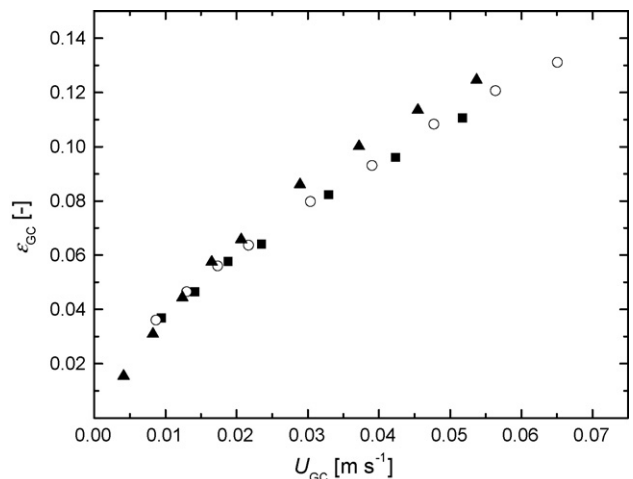


Fig. 3. Experimental dependencies of ε_{GC} values on U_{GC} (■) 12 dm³ ILALR, (○) 40 dm³ ILALR; (▲) 195 dm³ ILALR.

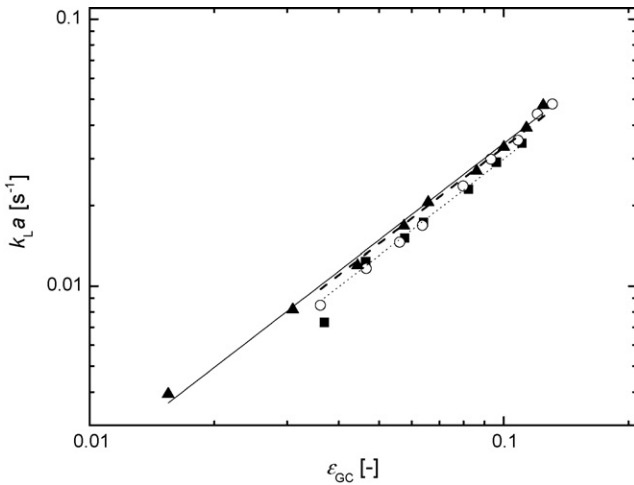


Fig. 4. Comparison of experimental data of $k_{L}a$ vs. ε_{GC} (symbols: (■) 12 dm³ ILALR, (○) 40 dm³ ILALR; (▲) 195 dm³ ILALR) with predicted values by Eq. (9) using the fitted parameter of K_1 presented in Table 3 (dotted line 12 dm³ ILALR, dashed line 40 dm³ ILALR and solid line 195 dm³ ILALR).

from the almost linear dependency of $k_{L}a$ on U_{GC} . Fig. 3 shows a plot of the experimental ε_{GC} values against U_{GC} .

Fig. 4 reveals a dependency of $k_{L}a$ on ε_{GC} . It can be noted that this dependency of $k_{L}a$ values on ε_{GC} is in fact independent on the ratio of cross-sectional areas (A_D/A_R). This observation is in a good agreement with the work of Bello et al. [10].

Hence, the exponent $(-0.4 + 0.4m)$ on the term $(1 + (A_D/A_R))$ in Eq. (5) can be put to zero; then m has the value of 1. According to Bello et al. [10] or Calderbank and Moo-Young [19], the parameter of n in Eq. (5) is put to 0.4. Hence, Eq. (5) becomes:

$$k_{L}a = K_1 \varepsilon_{GC}^{1.2} \quad (9)$$

The parameter of K_1 in Eq. (9) was determined by fitting the experimental dependencies of $k_{L}a$ values versus ε_{GC} shown in Fig. 4 in form of full lines. The estimated values of the K_1 parameter are 0.47, 0.52 and 0.54 s⁻¹ for respective volumes of ILALRs 12, 40 and 195 dm³ at respective correlation coefficients (R^2) of 0.99, 0.98 and 0.99. When the original value of the K_1 parameter (0.47 s⁻¹) proposed by Bello et al. [10] is applied for all experimental values of $k_{L}a$ in all ILALRs, correlation (9) describes the $k_{L}a$ values at a correlation coefficient of 0.977. As can be seen and deduced from the correlation coefficient, Eq. (9) describes the experimental $k_{L}a$ values sufficiently well.

Using the respective values of m and n of to 1 and 0.4, Eq. (6) becomes:

$$k_{L}a = K_2 \left(1 + \frac{A_D}{A_R}\right)^{-1} U_{GR}^{0.8} \quad (10)$$

and as a function of (P_G/V_L) using Eq. (7):

$$k_{L}a = K_3 \left(1 + \frac{A_D}{A_R}\right)^{-0.2} \left(\frac{P_G}{V_L}\right)^{0.8} \quad (11)$$

The experimental results (symbols) together with the fitting of experimental values by Eqs. (10) and (11) (lines) are plotted in Figs. 5 and 6. The variations of the experimental $k_{L}a$ values with U_{GR} and P_G/V_L are linear. Parameters of Eqs. (10) and

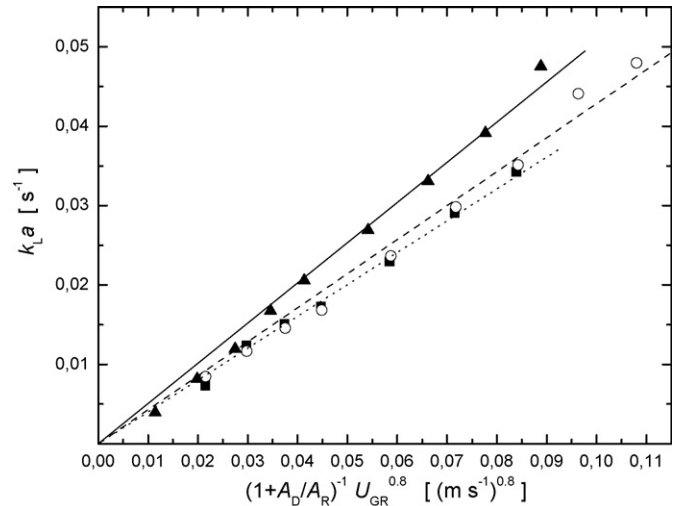


Fig. 5. Comparison of experimental data of $k_{L}a$ vs. $(1 + A_D/A_R)^{-1} U_{GR}^{0.8}$ (symbols: (■) 12 dm³ ILALR, (○) 40 dm³ ILALR; (▲) 195 dm³ ILALR) with predicted values by Eq. (9) using the fitted parameter of K_2 presented in Table 3 (dotted line 12 dm³ ILALR, dashed line 40 dm³ ILALR and solid line 195 dm³ ILALR).

(11) obtained by fitting the experimental values are presented in Table 3 together with the corresponding correlation coefficients.

Mass transfer from the gas phase to the liquid phase is straightly connected with the gas hold-up. Keeping the values of parameter m at the value of 1, Eq. (4) can be rewritten:

$$\varepsilon_{GC} = C_1 \left(1 + \frac{A_D}{A_R}\right)^{-1} U_{GR}^{2/3} \quad (12)$$

or as a function of (P_G/V_L) using Eq. (7):

$$\varepsilon_{GC} = C_2 \left(1 + \frac{A_D}{A_R}\right)^{-1/3} \left(\frac{P_G}{V_L}\right)^{2/3} \quad (13)$$

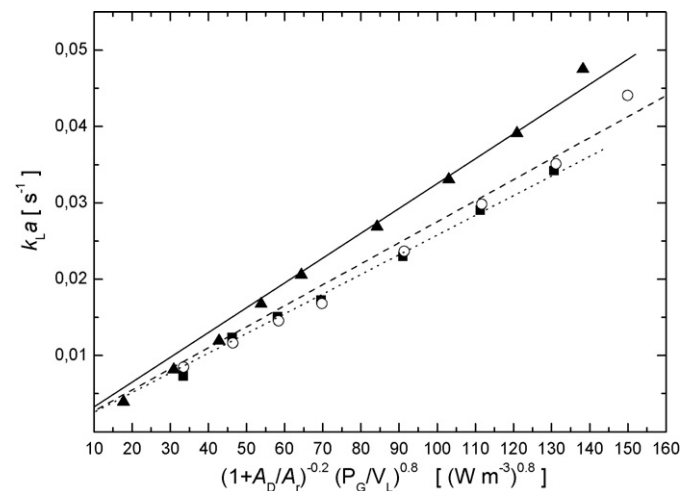


Fig. 6. Comparison of experimental data of $k_{L}a$ vs. $(1 + (A_D/A_R))^{-0.2} (P_G/V_L)^{0.8}$ (symbols: (■) 12 dm³ ILALR, (○) 40 dm³ ILALR; (▲) 195 dm³ ILALR) with predicted values by Eq. (9) using the fitted parameter of K_1 presented in Table 3 (dotted line 12 dm³ ILALR, dashed line 40 dm³ ILALR and solid line 195 dm³ ILALR).

Table 3
Parameters of correlations (9)–(13) used in this work determined from fitting the experimental values

Working volume (dm ³)	Eq. (9)		Eq. (10)		Eq. (11)		Eq. (12)		Eq. (13)	
	K_1 (s ⁻¹)	R^2	K_2 (m ^{-0.8} s ^{-0.2})	R^2	K_3 (W m ⁻³) ^{-0.8} s ⁻¹	R^2	C_1 (m s ⁻¹) ^{-2/3}	R^2	C_2 (W m ⁻³) ^{-0.8}	R^2
12	0.473	0.992	0.401	0.998	2.59×10^{-4}	0.998	0.999	1.00	2.18×10^{-3}	1.00
40	0.524	0.982	0.428	0.997	2.75×10^{-4}	0.997	0.946	1.00	2.07×10^{-3}	1.00
195	0.541	0.991	0.506	0.999	3.25×10^{-4}	0.999	1.060	0.999	2.32×10^{-3}	0.999

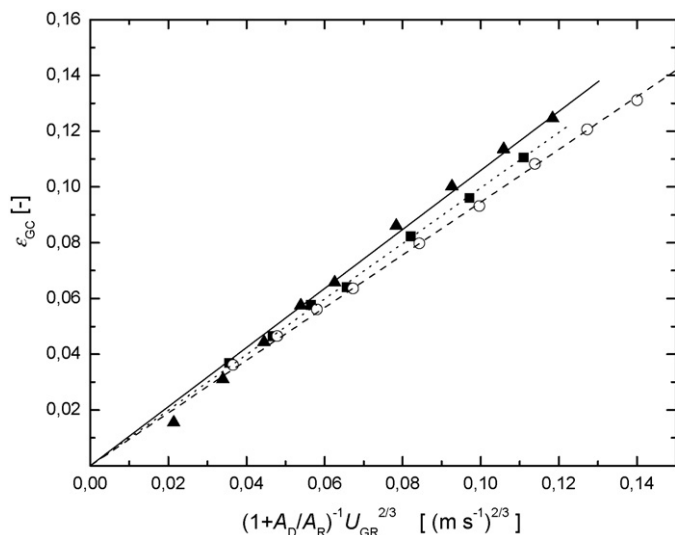


Fig. 7. Comparison of experimental data of ε_{GC} vs. $(1 + A_D/A_R)^{-1} U_{GR}^{2/3}$ (symbols: (■) 12 dm³ ILALR, (○) 40 dm³ ILALR; (▲) 195 dm³ ILALR) with predicted values by Eq. (9) using the fitted parameter of K_1 presented in Table 3 (dotted line 12 dm³ ILALR, dashed line 40 dm³ ILALR and solid line 195 dm³ ILALR).

The experimental results (symbols) together with the fitting of Eqs. (12) and (13) (lines) are plotted in Figs. 7 and 8. The total gas hold-ups were determined from the volume of the liquid free of gas and the volume of the gas–liquid dispersion. Due to the

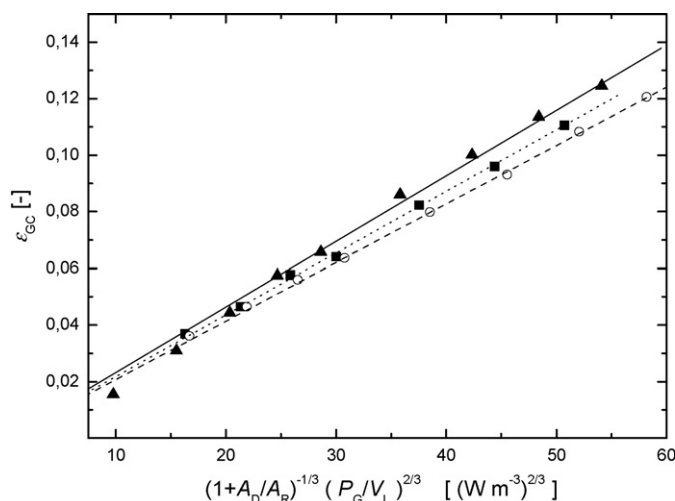


Fig. 8. Comparison of experimental data of ε_{GC} vs. $(1 + (A_D/A_R))^{-1/3} (P_G/V_L)^{2/3}$ (symbols: (■) 12 dm³ ILALR, (○) 40 dm³ ILALR; (▲) 195 dm³ ILALR) with predicted values by Eq. (9) using the fitted parameter of K_1 presented in Table 3 (dotted line 12 dm³ ILALR, dashed line 40 dm³ ILALR and solid line 195 dm³ ILALR).

unsteady height level of the dispersion, the experimental ε_{GC} values were determined with an error of up to 5%. Despite this fact, the experimental ε_{GC} values have shown linear dependencies on U_{GC} and P_G/V_L . Parameters of Eqs. (12) and (13) determined from the fitting of experimental values are presented in Table 3 together with the corresponding correlation coefficients.

4. Conclusions

Our previous work [7,8] showed the reliability of the dynamic pressure-step method for $k_L a$ measurements in pneumatically agitated reactors. Based on this knowledge, $k_L a$ values in three geometrically similar internal loop airlift reactors (ILAR) of different scale were determined. Besides these measurements, the experimental values of $k_L a$ and ε_{GC} were fitted with the semi-empirical correlations proposed by Bello et al. [10]. Parameters of the correlations used in this study are summarized in Table 3. All the correlations with empirical parameters described the experimental values well with respect to the correlation coefficients equal to 1 or almost equal to 1 (Table 3).

This work successfully verifies Bello's semi-empirical correlations for the prediction of $k_L a$ values as a function of the ε_{GC} values, according to Eq. (9). When using the original value of the parameter K_1 in Eq. (9) proposed by Bello et al. [10], all the experimental $k_L a$ values in all the ILALRs used can be described using correlation (9) with a sufficient correlation coefficient, given in Table 3. This relationship is independent on the geometric details of the reactor.

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