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Mass transfer measurements in a non-isothermal bubble column using the uncatalyzed oxidation of sulphite to sulphate

Peter Therning [a], Anders Rasmuson [a,b,*]

[a] School of Engineering, University College of Borås, S-501 90 Borås, Sweden
[b] Department of Chemical and Biological Engineering, Chalmers University of Technology, S-412 96 Gothenburg, Sweden

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Abstract

The volumetric mass transfer coefficient has been determined for a system comprising air and sodium sulphite (0.8 M) in a semibatch bubble column with a diameter of 0.2 m. The superficial gas velocity was varied in the range 0.03–0.2 m/s. The measurements were made at non-isothermal conditions between 19 and 36 °C, and it is shown that if the mass transfer is not chemically enhanced, the absorption rate of oxygen in a sodium sulphite solution will be constant and independent of the temperature. Based on this, it is possible to determine the volumetric mass transfer coefficient without any knowledge of the kinetics of the oxidation of sulphite to sulphate. Furthermore, it is found that the parameter $k_l a/\varepsilon_G$ in the heterogeneous regime for the system is about 0.5, which is in agreement with previous work of an air–water system. It is also shown that the mass transfer coefficient of small bubbles generated by a membrane or by a porous gas sparger is smaller compared to the mass transfer coefficient of larger bubbles formed in a gas sparger with 2 mm holes.

Keywords: Bubble columns; Volumetric mass transfer coefficients; Measurements

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1. Introduction

In bubble columns, the mass transfer from the gas phase to the liquid phase is a subject of great interest and is often the rate-limiting step due to the low solubility of the gas in the liquid, especially in bioprocesses. Therefore, the volumetric mass transfer coefficient $k_l a$ is a key parameter when designing bubble columns, and it is necessary to have knowledge of suitable methods for measuring the volumetric mass transfer coefficient. A survey of various methods is given by Gogate and Pandit [1]. It is interesting to note that Kulkarni et al. [2] and Kulkarni and Joshi [3] recently performed mass transfer measurements in a bubble column using one of the chemical methods, the oxidation of sulphite to sulphate. The authors compared these results with those estimated from instantaneous velocity–time data obtained using Laser Doppler Anemometry and found good agreement between the methods. The well-known sulphite method is discussed in more detail by Linek and Vacek [4]. Usually cobalt is used as a catalyst and by adjusting the cobalt concentration it is possible to keep the bulk concentration of oxygen close to zero while the reaction occurs at the interface between the liquid bulk and the liquid film. Wilkinson et al. [5] investigated the uncatalyzed sodium sulphite oxidation and replaced cobalt with sulphuric acid. They observed that the reaction rate increased if the initial sulphuric acid concentration increased, and that the reaction was of first-order with respect to oxygen. A pseudo first-order rate constant was expressed as,

$$k = k' C_{\mathrm{H_2SO_4}}^{n} \tag{1}$$

where $C_{\mathrm{H_2SO_4}}$ is the initial sulphuric acid concentration and n and k' are the constants depending on the initial concentration of sulphuric acid. The authors maintain that the constant reaction rate is advantageous in mass transfer measurements. Therefore, if low reaction rates are required, they recommended this reaction as a model reaction in mass transfer measurements as an alternative to the cobalt-catalyzed sulphite oxidation. However, only few authors have used this method to study mass transfer phenomena [6,7].

There is a large scatter in the published data of mass transfer measurements in bubble columns where oxidation of sulphite to sulphate has been used [2,3,6,8,9]. According to Linek and

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[*] Corresponding author. Tel.: +46 31 772 29 40; fax: +46 31 81 46 20.
E-mail address: rasmuson@chemeng.chalmers.se (A. Rasmuson).

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Nomenclature

a	specific interfacial area (m^{-1})
C	concentration (mol/dm^3)
D	diameter of the bubble column (m)
D_L	molecular diffusion coefficient (m^2/s)
g	gravitational acceleration (m^2/s)
h	Henry's law constant (dimensionless)
H	height of the gas–liquid dispersion (m)
H_c	clear liquid height (m)
Ha	Hatta number (dimensionless)
k	pseudo first-order rate constant (s^{-1})
k'	rate constant
k_l	liquid side mass transfer coefficient (m/s)
k_la	volumetric mass transfer coefficient (s^{-1})
k_M	constant in Eq. (8) (dimensionless)
K_L	the over all mass transfer coefficient (s^{-1})
M_B	molecular weight of solvent B (g/mol)
N	molar flux ($mol/m^2\,s$)
T	temperature (K)
U_G	superficial gas velocity (m/s)
V_A	molar volume of solute A at its normal boiling temperature (cm^3/mol)
z	axial coordinate (m)

Greek symbols

α	solubility of oxygen in sulphite (mol/dm^3 atm)
ε_G	gas holdup
Φ	association factor for solvent B
μ	dynamic viscosity (Pa s (in Eq; cP))
ρ	density (kg/m^3)
σ	surface tension (N/m)

Subscripts and superscripts

b	bulk of liquid
g	gas
i	interface
inlet	gas inlet at bottom of the bubble column
l	liquid
n	constant in Eq. (1)
outlet	gas outlet at the top of the bubble column

Vacek [4], the reaction shows a large scatter in the kinetic data due to the extreme sensitivity to metal ions. Due to the strong influence of impurities it is therefore not recommended to use kinetic data from the literature and kinetic investigations for the specific application being studied are therefore necessary. The same batch of sodium sulphite, catalyst and the same water as in the real mass transfer measurements should be used. However, it has to be noted that contamination products from the material in the bubble column or from the compressed air system are not considered in this procedure. Further, the exothermic reaction requires that the bubble column is equipped with some kind of control system to keep a constant temperature.

The objective with this work is to further evaluate the possibility of using the uncatalyzed sulphite oxidation to measure mass transfer coefficients in bubble columns and to apply the sulphite method to a semi batch bubble column without any form of temperature control. The purpose is also to investigate if the scatter in the results presented in the literature is a result of contamination or differences in the bubble column design.

2. Materials and methods

2.1. Materials

The experiments were performed in two bubble columns of glass, one with the diameter 0.2 m (BC1) and one smaller scale column, diameter 0.05 m (BC2). The first column (BC1) was equipped with a bottom section of stainless steel, and before entering the column the air, taken from an industrial compressed air system, passed both an oil mist collector and a humidifier. The gas sparger was made of stainless steel, free hole area 0.7%, hole diameter 2.0 mm. Deionized water was used and the dispersion height was 2 m. The initial concentration of the sulphite solution was 0.8 mol/dm^3 and the sulphuric acid concentration ranged from 0.06 to 0.035 mol/dm^3. Different grades of sodium sulphite were used, purum and pro analysis. The sampling point was located at half the dispersion height. The other bubble column (BC2) was equipped either with a 5 μm porous metal sparger or a plastic membrane, Filtroplast 40. The spargers in BC2 formed considerably smaller bubbles resulting in a higher gas holdup compared to BC1.The dispersion height in BC2 was 1 m. The bubble columns were not temperature controlled and, as a result, the temperature increased during each trial. The temperature increase depended on the measurement times, and was typically between 10 and 20 °C in BC1 and 3–5 °C in BC2.

2.2. Method

2.2.1. Gas holdup

The average gas holdup in BC1 was determined from the dispersion height and the measured height of the clear liquid after stopping the gas flow. In BC2, on the other hand, a known liquid volume was added to the bubble column. The gas holdup was calculated from

$$\varepsilon_G = \frac{H - H_c}{H} \tag{2}$$

2.2.2. The volumetric mass transfer coefficient

Chemical absorption techniques usually require knowledge of the kinetics of the reaction. As mentioned above, if the sulphite method is used this information has to be obtained from laboratory scale experiments. The reason for this is the large data scatter in the literature due to the extreme sensitivity to trace metals. As a result of the strong impact from impurities, an oxygen absorption test was performed in a flat interface stirred vessel. The test indicated a higher oxygen absorption rate in liquid samples taken from the bubble column compared to pure samples prepared in the laboratory. All samples contained the same quality of water, the same batches of sodium sulphite and sulphuric acid. It was therefore uncertain if the liquid in the bubble column was free of contamination products. Consequently, the normal procedure by using kinetic data from a "clean" test in a small laboratory reactor was not appropriate. Instead another approach was adopted.

If the system is entirely mass transfer controlled the oxygen absorption rate can be described by

$$N_{O_2} = k_l a (C^{i}_{O_2} - C^{b}_{O_2})\,(\text{mol oxygen/m}^3, \text{s}) \tag{3}$$

and

$$N_{O_2} = -0.5\,\frac{dC_{SO_3{}^{2-}}}{dt} \tag{4}$$

In line with Wilkinson et al. [6], the gas flow is considered as plug flow and the liquid phase is assumed to be perfectly mixed. The oxygen balance between the gas and the liquid at an arbitrary height, z, is then given by

$$U_G\,\frac{dC_{O_{2,g}}}{dz} = -k_l a (C^{i}_{O_{2,z}} - C^{b}_{O_{2,z}}) \tag{5}$$

Since the oxygen conversion was low during the experiments ($\leq$5%), U_G was assumed to be constant. In the case of a low bulk concentration of oxygen and if $C_{O_2,g} = hC^{i}_{O_{2,l}}$, Eq. (6) is derived by integrating Eq. (5) from $z = 0$ to H, where H is the height of the gas–liquid dispersion

$$k_l a = \frac{hU_G}{H}\ln\frac{C^{\text{inlet}}_{O_2,g}}{C^{\text{outlet}}_{O_2,g}} \tag{6}$$

The superficial gas velocity, U_G was calculated at the column midpoint pressure of the dispersion, and the distribution coefficient h was estimated by a correlation of oxygen solubility in sodium sulphate, Linek and Vacek [4], as shown by Eq. (7)

$$\alpha = 5.909 \times 10^{-6}\exp\left(\frac{1602.1}{T} - \frac{0.9407 \cdot C_{Na_2SO_4}}{1 + 0.1933 \cdot C_{Na_2SO_4}}\right) \quad (\text{mol/dm}^3, \text{atm}) \tag{7}$$

By knowing the amount of oxygen consumed by the reaction with sulphite, $C^{\text{outlet}}_{O_2,g}$ was calculated from an integral oxygen material balance over the entire bubble column. The oxygen consumed was followed indirectly by a standard iodine back titration method. A typical example of an experimental run is given in Fig. 1.

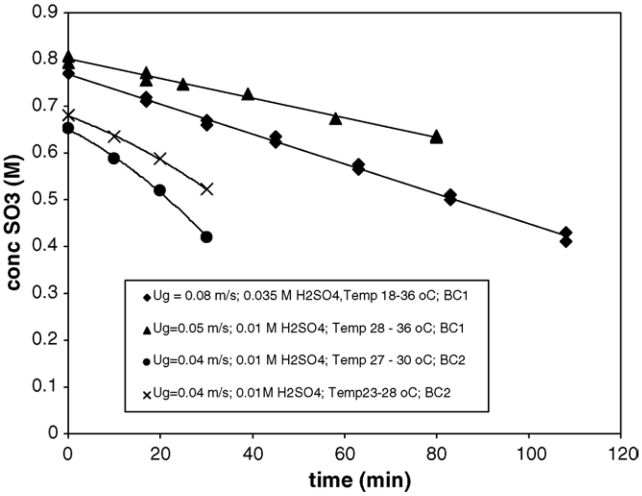

Fig. 1. The concentration of sulphite vs. time in the BC1 and BC2.

Despite the rise in temperature due to the exothermic reaction, the concentration of sulphite decreased linearly with the reaction time in BC1, whereas a small deviation from the linear relationship can be observed in BC2. The constant absorption rate of oxygen in BC1 may be explained by the fact that the reduced solubility is compensated by an equivalent increase in $k_l a$. If the temperature is increased 10 °C, the reduction of oxygen solubility is, according to Eq. (7), about 16%. The volumetric mass transfer coefficient, $k_l a$ is estimated to increase by approximately 16% in accordance with the correlation by Akita [10] which is valid for water–electrolyte solutions in bubble columns, as shown in Eq. (7),

$$k_l a = \left(\frac{k_M D_L}{D}\right)\left(\frac{\mu_l}{\rho_l D_L}\right)^{0.5}\left(\frac{gD^2\rho_l}{\sigma}\right)^{4/7}\left(\frac{gD^3\rho_l^2}{\mu_l^2}\right)^{2/7}\varepsilon^{1.18} \tag{8}$$

where k_M is 2.6 for a 0.8 mol/dm^3 sodium sulphite solution [6].

The temperature dependence of the diffusivity of oxygen in the sodium sulphite solution was estimated using the correlation by Wilke and Chang, as shown in Eq. (8), which is given by Reid et al. [11];

$$D_L = \frac{7.4 \cdot 10^{-8} \cdot (\Phi \cdot M_B)^{1/2} T}{\mu_B V_A^{0.6}} = \text{const} \cdot \frac{T}{\mu_B} \tag{9}$$

The temperature dependences of the surface tension, density and viscosity for aqueous solutions of sodium sulphite in the temperature interval from 25 to 35 °C were taken from Vazquez et al. [12] and the results were extrapolated to 0.8 M.

Since the absorption rate of oxygen is independent of the temperature, the non-isothermal conditions do not allow for evaluation of the data. The linear consumption of sulphite is also an indication of a mass transfer controlled transport of oxygen, but it is also characteristic of a low content of oxygen in the liquid bulk. According to Wilkinson et al. [5] and van Ede et al. [7], the uncatalyzed reaction is of first-order with respect to oxygen, which gives

$$C^{b}_{O_2} = \frac{-0.5\,\frac{dC^{b}_{SO_3{}^{2-}}}{dt}}{k} \tag{10}$$

No information is available in the literature about the temperature dependence of the rate constant, but the well-known rule of thumb makes it reasonable to assume that k increases by a factor 2 for a temperature rise of 10 °C. In this case the bulk concentration of oxygen $C^{b}_{O_2}$ reduces by 50%. Obviously, an increase in temperature decreases the bulk concentration of oxygen more rapidly than the concentration at the interface. This implies that the difference $C^{i}_{O_2} - C^{b}_{O_2}$ can only be considered to be constant during the experimental run if the bulk concentration of oxygen is negligible. This assumption is strengthened by Wilkinson et al. [6] who claim that the oxygen concentration in the liquid bulk were usually close to zero when using the uncatalyzed reaction in a bubble column for mass transfer measurements. Thus, Eq. (5) seems to be valid with respect to the assumption of low concentration of oxygen in the liquid bulk. It remains to show that the mass transfer is not enhanced by the reaction.

By changing the value of the rate constant it is possible to determine whether a heterogeneous gas–liquid reaction occurs in the liquid concentration boundary layer [6]. If the volumetric mass transfer coefficient is not influenced by a change in the rate constant, the absorption of oxygen is mass transfer controlled and the reaction occurs in the bulk. In this work two parameters were used to vary the rate constant of the reaction, namely the temperature and the sulphuric acid concentration. In the flat interface stirred vessel, mentioned earlier in this section, a strong influence of the temperature on the absorption rate of oxygen was observed, confirming the validity of using the temperature to vary the rate constant. However, it must be emphasized that the suitability of using the temperature is correlated to the constant consumption rate of sulphite, independent of the temperature. Experiments were therefore carried out at two different initial temperatures, 19 and 28 °C, and the concentration of sulphuric acid was varied between 0.006 and 0.0035 M.

The results from the different experiments in BC1 are shown in Table 1.

2.2.3. *The influence of temperature*

In Fig. 2 experimental data evaluated according to Eq. (5) are plotted against the initial temperature. The values of $k_{\mathrm{l}}a$ were converted to conditions at 20 °C. The results are obtained from the two bubble columns, BC1 and BC2. The gas spargers of the smaller bubble column create extremely small bubbles and thus a high gas holdup was obtained; $\varepsilon_G = 0.31$ by Filtroplast 10 and 0.37 by the porous metal sparger. The gas holdup in the larger bubble column can be studied in Fig. 3. It is evident that the volumetric mass transfer coefficient in the smaller bubble column is extremely sensitive to the temperature, while no such influence can be seen in BC1. The strong temperature dependence in BC2 can only be explained by the fact that the mass transport is enhanced by the chemical reaction and therefore these $k_{\mathrm{l}}a$-values must be considered to be affected by the reaction. This conclusion is also supported by the fact that BC2 shows a weak deviation from the linear decrease of sulphite versus time in Fig. 1. The consumption rate of sulphite seems to increase when the temperature is increased.

2.2.4. *The influence of sulphuric acid*

Fig. 4 shows the influence of the sulphuric acid concentration on the volumetric mass transfer coefficient at different superficial gas velocities. The measurements were made in BC1, the bubble column with the diameter of 0.2 m. According to Wilkin-

Table 1
Results of $k_{\mathrm{l}}a$ measurements in BC1

Sample no	U_G (m/s)	H_2SO_4 (M)	$k_{\mathrm{l}}a$ (s^{-1})	Quality of the sulphite
1	0.05	0.006	0.109	pa
2	0.05	?	0.086	pa
3	0.05	0.01	0.091	pa
4	0.05	0.036	0.128	pa
5	0.19	0.036	0.18	pa
6	0.21	0.006	0.171	pa
7	0.20	0.01	0.18	pa
8	0.20	0.025	0.20	pa
9	0.20	0.035	0.184	pa
10	0.20	0.035	0.166	purum
11	0.20	0.035	0.193	purum
12	0.032	0.01	0.061	purum
13	0.05	0.01	0.091	purum
14	0.08	0.01	0.119	purum
15	0.08	0.01	0.115	purum
16	0.08	0.025	0.116	purum
17	0.125	0.01	0.133	purum
18	0.16	0.01	0.16	purum
19	0.22	0.025	0.162	purum

Sample 1–11: initial temperature 18 °C; sample 12–19: initial temperature 28 °C.

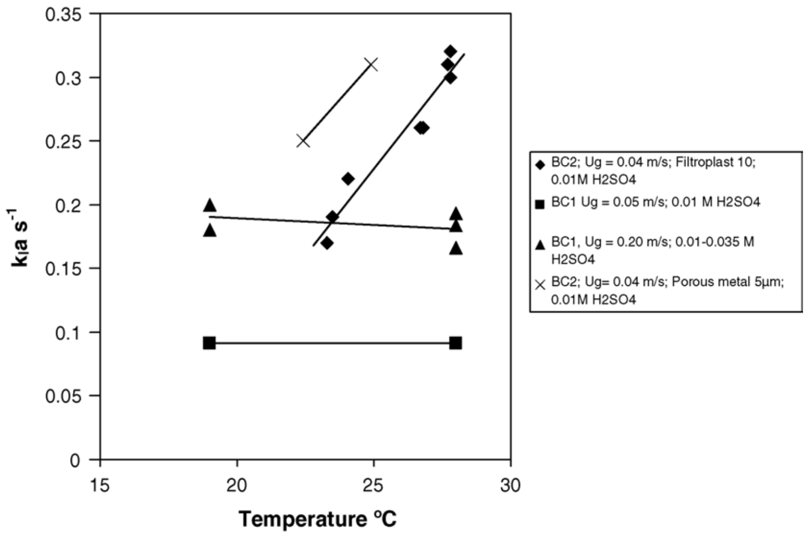

Fig. 2. The influence of the initial temperature on $k_{\mathrm{l}}a$ calculated from Eq. (6) (BC2—the $k_{\mathrm{l}}a$ values are related to the liquid volume).

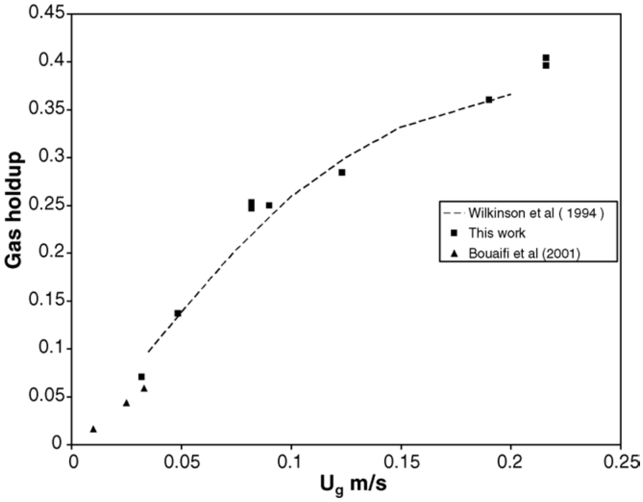

Fig. 3. The gas holdup as a function of the superficial gas velocity.

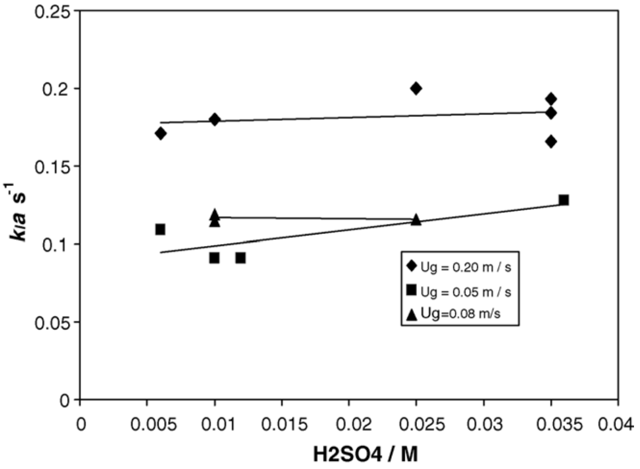

Fig. 4. The volumetric mass transfer coefficient vs. the concentration of sulphuric acid.

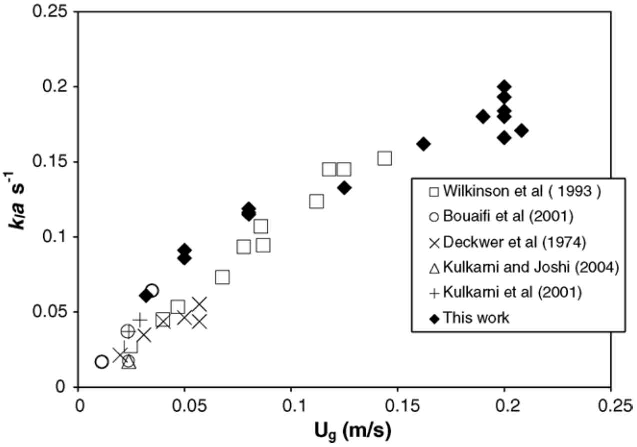

Fig. 5. The volumetric mass transfer coefficient plotted against U_{G} at atmospheric pressure.

son et al. [5] and Van Ede et al. [7], a rise in the concentration of sulphuric acid from 0.005 to 0.0035 M increases the reaction rate constant (Eq. (1)) by factors of 2.4 and 3.4, respectively. Virtually no influence of the sulphuric acid on $k_{\mathrm{l}}a$ can be observed at $U_{\mathrm{G}} = 0.08$ and 0.20 m/s, whereas a slight influence is observed at the lower velocity, $U_{\mathrm{G}} = 0.05$ m/s. Therefore, the experiments discussed above imply that the reaction proceeds in the liquid bulk with one exception, i.e., when the superficial gas velocity is 0.05 m/s. However, considering the lower sulphuric acid concentration of 0.01 M, no effect of the temperature is observed for BC1 in Fig. 2. Thus, the absorption at this lower sulphuric acid concentration is assumed not to be chemically enhanced.

3. Results

The calculated values of $k_{\mathrm{l}}a$ (20 °C) are plotted against the superficial gas velocity in Fig. 5 and against the gas holdup in Fig. 6. The values in this work are compared with results from

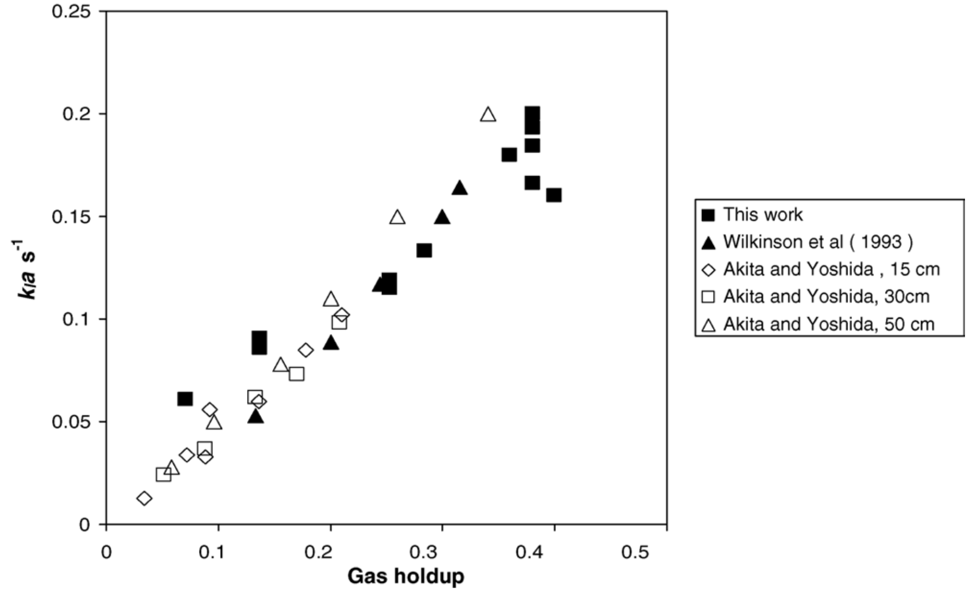

Fig. 6. The volumetric mass transfer coefficient as a function of the gas holdup at atmospheric pressure.

Table 2
Experimental conditions in this work and previous investigations

	This work	Wilkinson et al. [6]	Akita and Yoshida [8]	Bouaifi et al. [9]	Kulkarni et al. [2]	Kulkarni and Joshi [3]	Deckwer et al. [13]
Column diameter (m)	0.20	0.158	0.15–0.50	0.20	0.1/0.15	0.1/0.15	0.2
Gas distributor							
Hole diameter (mm)	2.0	10	5.0	2.0	0.8	0.8	1.0
Number of holes	69	19	1		54/122	54/122	56
Column height (m)	2	2	2–3	2	0.8/1.02	0.8/1.02	7.2
Concentration of sodium sulphite (M)	0.8	0.8	0.15	Low	0.2	0.2–0.8	–
Temperature (°C)	17–33	20.0	20	20	–	–	16

other studies. The trend is clear: an increase in the superficial gas velocity increases the volumetric mass transfer coefficient. At lower gas velocities and in the homogeneous region, the results obtained in this work deviate from the results by Kulkarni and Joshi [3], Wilkinson et al. [6], Akita and Yoshida [8], Deckwer et al. [13], but are comparable to the data obtained by Wilkinson and Akita and Yoshida in the heterogeneous region. The results at lower gas velocities are, however, the same as the results by Kulkarni et al. [2] and Bouaifi et al. [9]. The results from Kulkarni et al. [2] are estimated from data given in their article. The sulphite concentrations differ between these studies, as well as the design of the bubble columns, see Table 2. All authors except Deckwer et al. used sodium sulphite, whereas Deckwer measured the oxygen concentration in a sodium sulphate solution with a polarographic electrode.

In Fig. 7 the ratio $k_l a/\varepsilon_G$ is plotted against the gas velocity. A comparison is also made with Wilkinson et al. [6] and Vandu and Krishna [14]. The result of Vandu and Krishna was obtained in a bubble column with water as liquid and the authors used a sparger with a hole diameter of 0.5 mm. At higher gas velocities, in the heterogeneous region, the value of $k_l a/\varepsilon_G$ in this work is constant and approximately 0.5. This is in line with several authors [14–16].

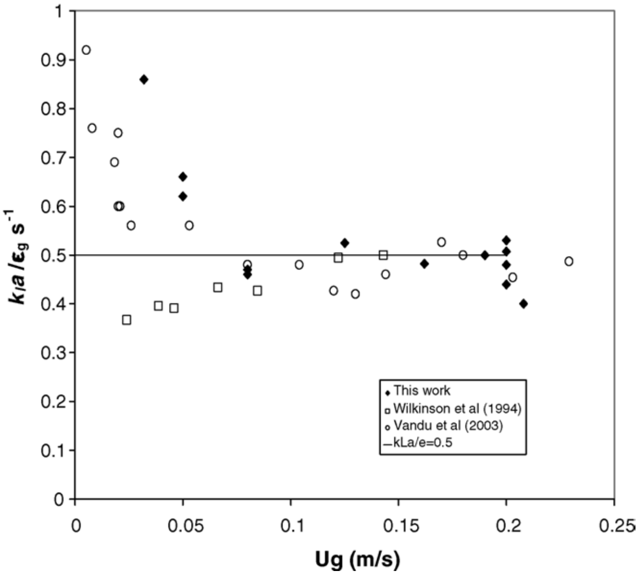

Fig. 7. The ratio $k_l a/\varepsilon_G$ vs. the superficial gas velocity at atmospheric pressure.

4. Discussion

First, it is relevant to discuss why the mass transfer in BC2 is enhanced by the reaction, while the mass transfer in the larger bubble column does not seem to be affected. A criterion that the chemical reaction proceeds in the liquid bulk, in the slow reaction regime, is that the Hatta number is less than 0.2 [17]. For a pseudo first-order reaction the Hatta number, Ha, is defined as:

$$Ha = \frac{\sqrt{D_L k}}{k_l} \tag{11}$$

From this point of view the difference between BC1 and BC2 can only be explained by a higher value of the liquid side mass transfer coefficient, k_l in BC1, where larger bubbles are formed. For a single gas bubble in tap water, Motarjemi and Jameson [18] observed a maximum value of the overall mass transfer coefficient K_L for oxygen at an optimal bubble size of 2 mm. In addition to the diffusivity, k_l for a single bubble also depends on the flow pattern around the bubble. The flow pattern is determined by the diameter and the geometry of the bubble and the rise velocity. However, in a swarm of bubbles, the repeated break-up and coalescence of bubbles increases the mass transfer. Also, the higher bubble terminal velocity created by the larger bubbles increases the turbulence and the mass transfer of the smaller bubbles. A higher value of k_l in BC1, where larger bubbles are formed, is therefore probable. This statement is supported by Zahradnik et al. [19]. At identical gas holdups, the authors observed higher values of $k_l a_l$ in a bubble column using a gas sparger with larger holes compared to a bubble column equipped with a porous sparger.

The higher value of $k_l a$ at low gas velocities studied here as compared to the results of Wilkinson, Akita and Deckwer may either be a result of differences in the design of the bubble columns or an undesirable impact of the chemical reaction. For the studies given in Table 2 important design parameters that may influence the mass transfer are given, such as the hole diameter of the sparger, the column diameter and the column height. The hole diameters of the gas spargers in the work by Akita and Yoshida and Wilkinson are considerably larger compared to the spargers used in the other studies. This may explain the higher value of the volumetric mass transfer coefficient obtained in this work and by Bouaifi et al. [9] and Kulkarni et al. [2]. It is worth noting that the $k_l a$ values reported by Kulkarni and Joshi

[3] and Deckwer et al. [13] are lower in spite of gas spargers with a small hole diameter. In the case of Deckwer, the height of the bubble column, 7 m, may be one possible explanation. The bubble column was also operated in a co-current mode with a liquid flow in the range of 0.4–6 m^3/h. Additional differences are the liquid, which was a sodium sulphate solution, and the measurement technique. However, according to Wilkinson et al. [6], it is generally accepted that the sparger design and the bubble column design does not influence the gas holdup provided the column diameter is larger than 0.15 m, the H/D is greater than 5 and the sparger hole diameter is larger than 1–2 mm. This is due to the coalescing of the gas bubbles. In this work the measured gas holdup is comparable with the result achieved by Wilkinson et al. [6], as shown in Fig. 3, and it is therefore reasonable to believe that the interfacial area in these two studies may be similar. From this point of view it is possible that the volumetric mass transfer coefficient achieved in this work at lower gas velocities and by Kulkarni et al. [2] and Bouaifi et al. [9] is affected by the chemical reaction. If this scenario is correct, the change from homogeneous flow to the heterogeneous flow regime also implies an increase in k_l. This may be a result of continuous coalescing and splitting up of bubbles, which increases the mass transfer.

5. Conclusions

1. The sulphite method, using sulphuric acid as a catalyst, has been used to determine the volumetric mass transfer coefficient in a bubble column with a diameter of 200 mm. The experiments were performed at non-isothermal conditions and it was found that the absorption rate of oxygen was independent of the increase in temperature if the absorption was not enhanced by the reaction. The method demonstrated in this work does not require any knowledge of the rate constant or the order of reaction.
2. In the homogeneous flow regime, the mass transfer coefficient k_l is smaller for fine bubbles generated by a porous gas sparger compared to larger bubbles generated by a gas sparger with the hole diameter of 2 mm.
3. When using the sulphite method it is recommended to test if the equipment or the compressed air generates impurities that can affect the absorption rate of oxygen. In the literature, measurements in bubble columns by the sulphite method show a large scatter in volumetric mass transfer data.
4. The ratio $k_l a/\varepsilon_G$ is constant and is of the same order (0.5) for a non-coalescing sodium sulphate solution as for a stronger coalescing system, such as air–water in the heterogeneous regime.

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