



Gas absorption in bubble column using a non-Newtonian liquid phase

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ABSTRACT

Gas–liquid mass transfer has been investigated in a bubble column using non-Newtonian media and pure carbon dioxide as absorbent and gas phase, respectively. The volumetric mass transfer coefficient and gas–liquid interfacial area has been measured under different operational conditions for the gas–liquid system analysed in this work. The non-Newtonian media used were in all the cases aqueous solutions of κ -carrageenan; the characterization of this polymer in relation to the medium molecular weight and rheological behaviour has been carried out as a previous step. The influence of rheological properties, polymer concentration and gas flow-rate upon the mass transfer rate has been studied. The results shown in the present work allowed us to understand the carbon dioxide transfer to a non-Newtonian liquid phase, as well as to evaluate the effect caused by the liquid phase characteristics.

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1. Introduction

Industrial operations where one or more components of a gas phase are absorbed into a liquid phase are common in chemical process industries. In many cases, this contact is useful to achieve the desired reactions between components of both two phases. Bubble reactors (columns and stirred vessels) have a wide range of applications in chemical, biochemical and pharmaceutical industries [1] due to the effective contact of a gas with a liquid phase they provide to carry out chemical or biochemical reactions. The mass transfer of a gas compound into a liquid is often the rate-limiting step. Most of the liquid phases employed in these processes are non-Newtonian fluids (in food industries, for example). Several studies [2,3] have analysed the effect caused by the presence of different polymers on the absorption process.

It is known that the volumetric mass transfer coefficient, $k_L a$, is one of the most important parameters that govern the performances of gas–liquid reactors. Therefore, extensive efforts have been carried out to estimate the volumetric mass transfer coefficient, although most of them are based on empirical analyses. The values of the volumetric mass transfer coefficient ($k_L a$) have been obtained for various impeller speeds, gas flow-rates and for different solutes concentrations. These values have been correlated with the operating variables, the geometric parameters of the system

and the broths' physical properties, using rigorous techniques in order to obtain a set of reliable and accurate data [4].

The behaviour of these non-Newtonian liquids is studied on the basis of the apparent viscosity variations. Some studies proved the important effect upon the mass transfer caused by the apparent viscosity of the liquid phase [5,6]. For this reason, it is necessary to include the rheological behaviour in a serious study concerning the modelling of the absorption process. A variation on the stirring rate and/or the gas flow-rate could also produce a change in the physical properties of the absorbent phase, since the viscosity of the non-Newtonian liquid is a shear rate-dependent [7,8].

Parameters of gas–liquid contactors such as mass and heat transfer coefficients are usually correlated in relation to viscosity by means of using the apparent viscosity. The local shear rate in such systems cannot be calculated. Therefore, it is necessary to use an effective shear rate with a corresponding effective viscosity. These liquids (polymers aqueous solutions) are usually shear thinning and they have rheological properties similar to real industrial media, like fermentation broths and liquid suspensions of small particles. Also nowadays certain studies have proposed the use of polymers to capture carbon dioxide in gas–liquid systems [9].

2. Materials and methods

The aqueous solution employed as liquid absorbent phase has been produced using different quantities of κ -carrageenan supplied by Fluka (CAS number 11114-20-8). The solutions were prepared by mass using a balance with a precision of $\pm 10^{-7}$ kg. Bi-distilled water has been employed to prepare the absorbent phases.

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Nomenclature

a	specific interfacial area (m^{-1})
c	polymer concentration (g L^{-1})
C	carbon dioxide concentration (mol L^{-1})
C^*	carbon dioxide solubility (mol L^{-1})
d	bubble column diameter (m)
k	consistence index (Pa s^n)
k_L	liquid side mass transfer coefficient (m s^{-1})
M_w	molecular weight (kDa)
n	flow index
Q_g	gas flow-rate ($\text{m}^3 \text{s}^{-1}$)
t	operation time (s)
U_g	superficial gas velocity (m s^{-1})
Re	Reynolds number, $Re = d\rho U_{sg}\eta_{ap}^{-1}$
Sc	Schmidt number, $Sc = \eta_{app}\rho^{-1}D^{-1}$
Sh	Sherwood number, $Sh = k_L ad^2 D^{-1}$

Greek symbols

γ	shear rate (s^{-1})
γ_{av}	average shear rate (s^{-1})
η	dynamic viscosity (Pa s)
$[\eta]$	intrinsic viscosity (L g^{-1})
η_{app}	apparent viscosity (Pa s)
η_r	relative viscosity
η_s	solvent viscosity (Pa s)
η_{sp}	specific viscosity
ν	kinematic viscosity ($\text{mm}^2 \text{s}^{-1}$)
ρ	density (g cm^{-3})
τ	shear stress (Pa)

The average molecular weight of κ -carrageenan has been determined by the use of the intrinsic viscosity, which has been calculated employing the Huggins and Kramer equations. This procedure has been employed successfully by different authors [10,11] for other polymers. The experimental set up to carry out these experiments was a capillary viscometer (Schott Gerate AVS 350), which was immersed in a thermostated bath with a precision of $\pm 0.1^\circ\text{C}$. The solutions were prepared in a $\text{NaCl } 0.1 \text{ mol L}^{-1}$ aqueous solution. The Mark–Houwink constants were determined using this solvent to prevent the polyelectrolyte expansion in solution when using size exclusion chromatography. Different concentrations were prepared to obtain relative viscosities in a range of 1.2–1.6 to assure good accuracy and linearity of extrapolation at zero concentration. The kinematic viscosity was measured at $25.0 \pm 0.1^\circ\text{C}$.

Rheological studies were performed using an Anton Paar DV-1P digital thermostated rotational viscosimeter based in two coaxial cylinders. The polymer concentrations employed in the present study have been included, for polymer aqueous solutions, in a range of $0\text{--}10 \text{ g L}^{-1}$.

The studies of carbon dioxide mass transfer to liquid phases were carried out using the experimental set-up employed in previous works (Fig. 1) related to absorption processes. The gas–liquid contactor used in these studies has been a cylindrical bubble column (internal diameter = 7 cm; height = 100 cm), made of methacrylate with a volume of 2.4 L.

The absorption process has been carried out at 25°C . The gas to be absorbed, carbon dioxide, was passed through two water flasks at work temperature to prepare the gas phase. This procedure removed other resistances to mass transport and it only allowed the evaluation of the liquid phase resistance to the gas transfer. Water was placed into the water flasks. The gas flow-rate was mea-

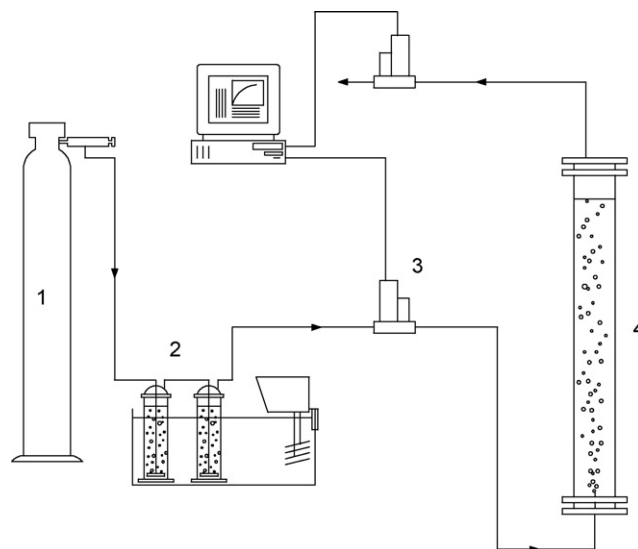


Fig. 1. Scheme of experimental set-up employed to carry out absorption studies: (1) gas cylinder, (2) water flasks, (3) gas flow-meter/controller and (4) bubble column.

sured and controlled with two mass flow controllers (5850 Brooks Instruments) which have been calibrated by the supplier for the gas flow-rate and pressures used in the present work, in a range of $18\text{--}30 \text{ L h}^{-1}$.

The pressure drop was measured between the column's inlet and outlet using a Testo 512 digital manometer. The regime employed was continuous in relation to the gas phase and batch as regards the absorbent liquid.

The procedure employed to determine the bubble size distribution in the experimental contactor has been based on a photographic method [12,13]. The bubble diameter was measured using a photographic method based on taking images of the bubbles along the height of the column, from bottom to top. A Sony (DCR-PC330E) video camera was used to obtain the images. A minimum number of 50 well-defined bubbles along the bubble column were used to evaluate the size distribution of bubbles in the liquid phase employed, and for each gas flow-rate which has been used. We used the Image Tool v2.0 software to carry out the necessary measurements of the geometric characteristics of the bubbles.

3. Results and discussion

The molecular weight of the κ -carrageenan employed in the present study has been determined using the dynamic viscosity (η) values determined by the product of density (ρ) and kinematic viscosities (ν).

$$\eta = \nu\rho \quad (1)$$

The average molecular weight has been calculated employing the intrinsic viscosity obtained by a combined application of Huggins and Kramer's equations:

$$\frac{\eta_{sp}}{c} = [\eta] + k_1[\eta]^2 c \quad (2)$$

$$\ln \eta_r = [\eta] + k_2[\eta]^2 c \quad (3)$$

where η_{sp} is the specific viscosity, η_r is the relative viscosity, c is the polymer concentration, $[\eta]$ is the intrinsic viscosity, and k_1 and k_2 are fit constants corresponding to Huggins and Kramer equations.

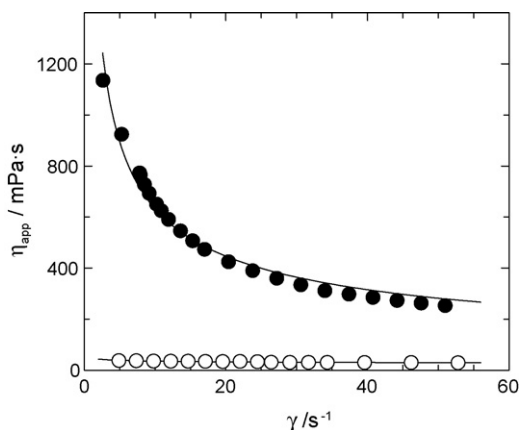


Fig. 2. Influence of polymer concentration upon the rheological behaviour and Ostwald model (solid line): (○) $c = 6 \text{ g L}^{-1}$ and (●) $c = 10 \text{ g L}^{-1}$.

Eqs. (2) and (3) employ the specific (η_{sp}) and relative viscosity (η_r). Both variables are defined in the following equations:

$$\eta_{sp} = \frac{\eta - \eta_s}{\eta_s} \quad (4)$$

$$\eta_r = \frac{\eta}{\eta_s} \quad (5)$$

where η and η_s are the viscosities of the solution and the solvent, respectively.

The molecular weight was determined using the corresponding Mark–Houwink equation (Eq. (6)). The Mark–Houwink constants for these polymers were obtained from the literature [14].

$$[\eta] = K_{MH} M_w^{a_{MH}} \quad (6)$$

where M_w is medium molecular weight and K_{MH} and a_{MH} are Mark and Houwink constants for κ -carrageenan.

The value of the intrinsic viscosity, determined employing the Huggins and Kramer equations, was 6.03 L g^{-1} . This value was employed in Eq. (6) to determine the average molecular weight of the polymer used in the present work. The corresponding values for K_{MH} and a_{MH} parameters in Eq. (6) are $8.84 \times 10^{-5} \text{ L g}^{-1}$ and 0.86, respectively [14]. The use of the data shown in this paragraph allowed the determination of the κ -carrageenan medium molecular weight; that is, 417.6 kDa.

Few researchers have studied the rheological behaviour of different kinds of aqueous dispersions of hydrocolloids [15], and these studies have concluded that these solutions show a non-Newtonian and pseudoplastic behaviour in almost all the cases. Similar experiments have been carried out in the present work. An example of the experimental data is presented in Fig. 2.

In our case, the polymer solutions studied show a shear thinning. This behaviour corresponds to a non-Newtonian and pseudoplastic fluid. The viscosity decreases when the shear rate increases, and the same behaviour is observed for all the concentrations. The experimental data were fitted to Ostwald's law (Eq. (7)). Suitable fits have been observed (see Fig. 2) and the experimental results show that the behaviour index, n , for κ -carrageenan aqueous dispersions, has values minor than 1. The behaviour observed for these parameters is typical for this kind of aqueous solutions. An increase in the polymer concentration produces a higher deviation from the Newtonian behaviour ($n = 1$), as well as a clear increase in the magnitude of viscosity, due to the increase in the consistency value. Therefore, we can confirm that these solutions show a pseudoplastic behaviour. The values obtained for the Ostwald model (n

Table 1
Ostwald model rheological parameters for aqueous solutions of κ -carrageenan

$[\kappa\text{-Carr}] (\text{g L}^{-1})$	n	$k (\text{mPa s}^n)$
0	1.000	1.0
2	0.948	16.8
4	0.918	33.4
6	0.656	311
8	0.435	1880
10	0.271	7320

and k) are listed in Table 1 for different aqueous solutions of both polymers.

$$\eta_{app} = k\gamma^{n-1} \quad (7)$$

The application of the Ostwald model to the experimental data shown in Fig. 1 allows a suitable calculation of the viscosity value at different shear rate values.

The liquid phase viscosity value depends significantly on the shear rate (*vide supra*) and then, in the bubble column, the shear rate and viscosity must be calculated for each experimental conditions.

The procedure to determine the volumetric mass transfer coefficient is based on the measurement of the amount of gas absorbed per time unit time and volume unit according to the following equation [16]:

$$\frac{dC}{dt} = k_L a (C^* - C) \quad (8)$$

where C^* is the interfacial concentration of gas compound in the liquid phase at equilibrium (i.e. the solubility of the gas in the liquid phase) and C is the concentration of carbon dioxide in the bulk liquid, that is calculated from the experimental absorption rate data.

$$\ln \left(\frac{C^*}{C^* - C} \right) = k_L a t \quad (9)$$

Integrating Eq. (8), Eq. (9) was obtained. Then, plots of $\ln[C^*/(C^* - C)]$ against time showed straight lines passing the origin showing $k_L a$ was independent of time under the conditions of this work. The carbon dioxide concentration in the bulk of the liquid increases with time until the liquid phase is saturated. The experimental results show that a higher presence of polymer in the liquid phase, as well as an increase of the polymer concentration, produces a decrease in the slope of the straight line (corresponding to a lower volumetric mass transfer coefficient). Similar results have been obtained in previous studies [17,18] employing different systems formed by aqueous solutions of polymers. Two opposite effects or mechanisms (slip and elasticity effects) have been described in literature for different systems when polymer solutions are present and these effects act simultaneously. The slip effects produce an increase of absorption rate, whereas elasticity produces a decrease of absorption rate. According to Mashelkar [19], the overall effect seems to be the reduction on the mass transfer rate. In our work, the elasticity effect is more important and a clear reduction was observed in the mass transfer rate, depending on the kind of contactor employed.

As regards the effect of the gas flow-rate upon the value of the volumetric mass transfer coefficient, Fig. 3 shows the behaviour observed for this system. Fig. 3 shows a complete vision of the influence of polymer concentration and gas flow-rate upon the volumetric mass transfer coefficient. The increment of both variables produces opposite effects: positive for gas flow-rate and negative for polymer concentration. Increasing the value of the gas flow-rate produces an increase on the mass transfer rate. This figure shows that this increase is observed for all the experimental conditions. In the same time, the previously observed influence of viscosity

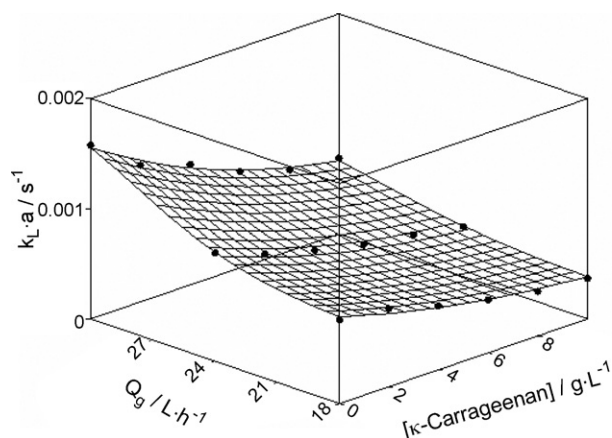


Fig. 3. Effect of polymer concentration and gas flow-rate upon experimental values of volumetric mass transfer coefficient.

(related to the polymer concentration in the liquid phase) upon the volumetric mass transfer coefficient is corroborated by the values obtained for the mass transfer coefficient at different gas flow-rates.

The influence of the presence of the polymer upon the global absorption process it is necessary to take into account the value of the specific area, so that we can draw a conclusion about the way the κ -carrageenan affects on the carbon dioxide absorption in the bubble column.

The bubble images we obtained show an ellipsoid shape. For this reason, major (E) and minor (e) axes of the projected ellipsoid (in two dimensions) were determined. The diameter of the equivalent sphere (Eq. (10)) was taken as the representative bubble dimension.

$$d = \sqrt[3]{E^2 e} \quad (10)$$

Different authors recommend to use the Sauter mean diameter (d_{32}) [20], which can be calculated using the following equation:

$$d_{32} = \frac{\sum_i (n_i d_i^3)}{\sum_i (n_i d_i^2)} \quad (11)$$

where n_i is the number of bubbles which have an equivalent diameter (d_i) [20].

The Sauter mean diameter (d_{32}) and the gas hold-up (ε) values allowed the calculation of the specific interfacial area using the following equation [21]:

$$a = \frac{6\varepsilon}{d_{32}(1-\varepsilon)} \quad (12)$$

Fig. 4 shows the experimental results and the effects of the polymer concentration in the liquid phase and the gas flow-rate fed to the bubble column. This figure shows that an increase of the polymer concentration in the liquid phase causes a decrease of the gas–liquid interfacial area. This decrease is related to the difficult for the bubble to ascend in the liquid phase that increases with the liquid viscosity and then with the polymer concentration. An example of the photographs analysed to obtain information about bubble size and interfacial area is shown in Fig. 5. These photographs demonstrate the effect caused by an increase in the polymer concentration, producing an increment of bubble size and then a clear decrease of the interfacial area value.

On the other hand, an increase of the gas flow-rate drives to an increase of the gas–liquid interfacial area. Even if a higher gas flow-rate produces large bubbles such as the photographs have shown, the global behaviour indicates an increase in the interfacial area caused by an increase in the gas hold-up.

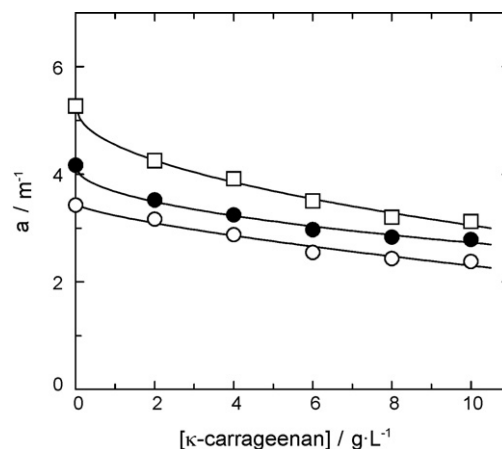


Fig. 4. Influence of polymer concentration upon gas–liquid specific interfacial area: (○) $Q_g = 18 \text{ L h}^{-1}$, (●) $Q_g = 24 \text{ L h}^{-1}$ and (□) $Q_g = 30 \text{ L h}^{-1}$.

The values determined for the interfacial area under the experimental conditions employed in present work have been employed to calculate the values of mass transfer coefficient corresponding to the liquid phase, k_L , by means the division of volumetric mass transfer coefficient (obtained from Eq. (9)) and specific interfacial area (using Eq. (12)). This allowed to remove the influence caused by the nature of the liquid phase and operating conditions upon the interfacial area and then is possible obtain only the influence caused by these variables upon the mass transfer coefficient, k_L .

Fig. 6 shows that the behaviour observed for the liquid side mass transfer coefficient, k_L , is similar to the one of the volumetric mass transfer coefficient because a decrease caused by the increment in polymer concentration in the liquid phase. Nevertheless, for k_L , the observed trend was linear. This decrease in the mass transfer coefficient value is due to the increase in mass transfer resistance caused by the increase in viscosity that produces a lower mass transfer rate.

Regarding the influence of the gas flow rate, it produces a positive effect upon the value of liquid side mass transfer coefficient; indeed, to an increase in gas flow-rate implies an increase in terminal bubbles velocity and this phenomenon produces an increase in turbulence near the bubble interface and then an increase in mass transfer rate.

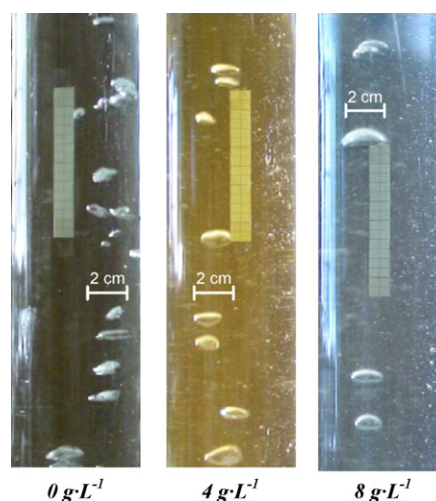


Fig. 5. Bubbles photographs corresponding to the absorption of carbon dioxide at $Q_g = 24 \text{ L h}^{-1}$ at the middle section of the bubble column.

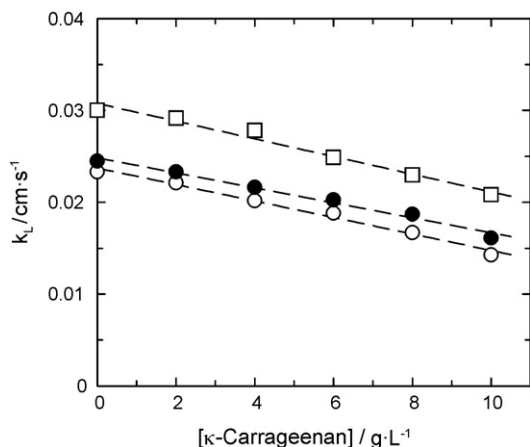


Fig. 6. Influence of polymer concentration in the liquid phase and gas flow-rate upon liquid side mass transfer coefficient: (○) $Q_g = 18 \text{ L h}^{-1}$, (●) $Q_g = 24 \text{ L h}^{-1}$ and (□) $Q_g = 30 \text{ L h}^{-1}$.

The last point of this study has been to fit experimental data of volumetric mass transfer coefficient with different correlations found in literature. In order to calculate the effective viscosity of a non-Newtonian fluid in the bubble column, it is necessary to know the effective shear rate in the bubble contactor. Several studies [22,23] used the relation proposed by Nishikawa et al. [24] to estimate it.

$$\gamma_{av} = 5000U_g \quad (13)$$

where γ_{av} and U_g are the average shear rate and gas superficial velocity, respectively.

The knowledge of the average apparent viscosity of liquid phase into the bubble column allows us to employ this variable to fit the experimental values of volumetric mass transfer coefficient. The equation employed based on experimental variables has been used for different studies to correlate the influence of these variables upon the global gas mass transfer [25,26].

$$k_L a = 0.85U_g^{1.2}\eta_{app}^{-0.1} \quad (14)$$

Fig. 7 shows the comparison between the experimental values of volumetric mass transfer coefficient and the ones calculated with Eq. (14). This equation gives suitable values for the volumetric mass transfer coefficient. In relation with the value of the fit parameters, similar results have been obtained by other authors [18,25,26]. The negative effect of the liquid phase viscosity (polymer concentra-

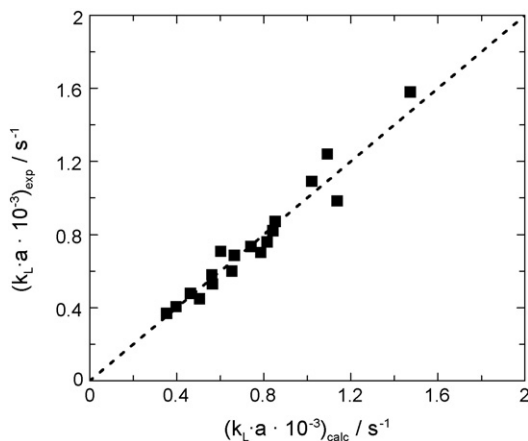


Fig. 7. Comparison between experimental and calculated values employing Eq. (14).

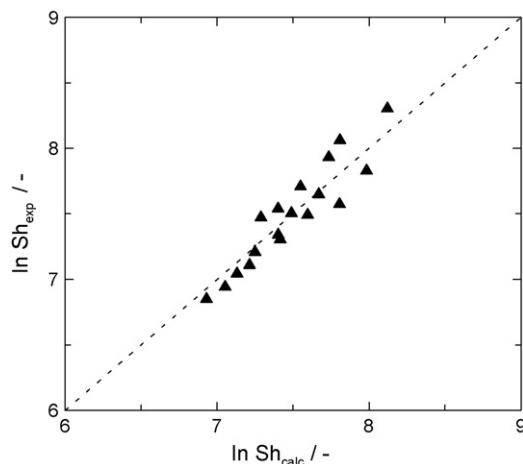


Fig. 8. Experimental and calculated values of Sherwood number by Eq. (15).

tion in the liquid phase) is observed with a negative value of the fit parameter (exponent of apparent viscosity). On the other hand, the superficial gas velocity (gas flow-rate) shows great importance upon the mass transfer (see Eq. (14)). The sign of exponents in Eq. (14) indicates the effect caused by each variable; i.e. the viscosity produces a decrease in liquid side mass transfer coefficient while gas flow-rate (gas superficial velocity) produces an increase.

Another typical expression employed to fit experimental data of volumetric mass transfer coefficient in this kind of systems is based on the use of dimensionless numbers. Several studies have used complex expressions that include numerous dimensionless numbers, such as Schmidt, Froude, Reynolds, Bond, etc. [8,25,26]. The expression employed in present work is simpler than the previously commented ones, using two dimensionless numbers to correlate the Sherwood number values.

$$Sh = 6.7Re^{0.6}Sc^{0.5} \quad (15)$$

The expression, shown in Eq. (15), have fit parameters with similar values than the previous ones obtained by other studies [25,26] that employ non-Newtonian fluids as absorbent phase. The results contributed for Eq. (15) are suitable and similar to the previous ones obtained using Eq. (14) (Fig. 8).

4. Conclusions

The present paper allowed the characterization of the gas–liquid mass transfer process using non-Newtonian media as liquid phase. The effect of different operating variables (gas flow-rate and polymer concentration) has been studied. In a previous step, polymer characteristic parameters such as intrinsic viscosity and average molecular weight, have been determined using a viscosimetric technique. The polymer solutions employed as liquid phase in the absorption studies have been characterized in relation to the rheological behaviour, and all the aqueous solutions exhibited a non-Newtonian and pseudoplastic behaviour.

The mass transfer process showed an increase of the gas transfer rate to the liquid phase when the gas flow-rate increased. A different trend was observed when the presence of the polymer grows of the absorbent phase. Its presence in the liquid phase produces an increase in the viscosity and then, the resistance to the transport also increased. Gas–liquid interfacial area in the bubble column has also been determined to calculate liquid side mass transfer coefficient and to analyse the effect of operating variables.

The mass transfer behaviour has been analysed employing typical equations based on operational variables or dimensionless

groups. Nearly all the cases have been successful, being known the difficulty generated by this kind of liquid phases to modelize different chemical and engineering processes.

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