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Carbon dioxide absorption to rheologically complex liquid phases

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ABSTRACT

Aqueous solutions of hydrocolloid mixtures have been employed as liquid phases in gas–liquid absorption processes employing a bubbling stirred vessel as contactor. The influence of the liquid phase composition upon certain physical properties and, subsequently, upon the mass transfer and gas–liquid interfacial area, have been analysed taking into account the operating conditions in relation to the stirring rate, the gas flow-rate and the non-Newtonian behaviour of the liquid phases employed in the present work. The liquid phase viscosity shows an important role in mass transfer coefficient and in gas–liquid interfacial area and synergetic effects have been analysed to explain mass transfer behaviour.

1. Introduction

Most of the liquid phases used in gas–liquid processes, e.g. in food and pharmaceutical industries, are non-Newtonian fluids. Important number of studies [1,2] have analysed the effect caused by the presence of different polymers on the absorbent phase, with and without chemical reaction: for example, the oxygen transfers in non-Newtonian fermentation broths of different microorganisms. The values of the volumetric transfer coefficient ($k_L \cdot a$), obtained at various impeller speeds, air flow-rates and at different initial concentrations, were correlated with operational variables, the geometric parameters of the system and the physical properties of the broths, utilising rigorous techniques in order to obtain a set of reliable and accurate data.

As is well-known, the volumetric mass transfer coefficient, $k_L \cdot a$, is one of the most important parameters that govern the performance of gas/liquid reactors. Therefore, extensive efforts, in the last years, have been directed to estimate this coefficient [3,4]. However, most of them are based on empirical analyses. It is also important to determine the influence of the operational variables (physical properties, stirring rate, gas flow-rate, etc.) on $k_L \cdot a$ to optimise the absorption process.

The behaviour of these non-Newtonian liquids is commonly studied on the basis of the apparent viscosity variations. Some articles proved the important effect that the apparent viscosity of the liquid phase has upon the mass transfer [5,6]. For this reason, it is necessary to include the rheological behaviour in a serious study concerning gas–liquid absorption. The use of a stirred vessel with a non-Newtonian media as liquid phase has important characteristics [7]. A variation on the stirring rate also produces a change in the physical properties of the absorbent phase, since the viscosity of the non-Newtonian liquid is shear rate-dependent.

Previous studies of our research team have analysed the non-Newtonian rheological behaviour of CMC and ALG aqueous solutions, as well as the influence of the special characteristics of these kind of liquid phases upon the gas absorption process, using different contactors [8,9]. These studies have shown the great importance of the rheological behaviour upon the gas mass transfer to a liquid phase, due to the important connection between the viscosity and the mass transfer process [3]. In addition, when this kind of liquid phase is employed in certain equipments, the shear rate applied to the non-Newtonian liquid phase has great importance upon the mass transfer because the apparent viscosity value could be highly influenced by the shear rate [10].

Since it is possible to observe, the knowledge of the relation between rheological behaviour and mass transfer has great importance in these kind of systems. Then the presence of two polymers in the same liquid dispersion could produce changes on the rheological behaviour caused by synergetic effects and it influences on mass transfer processes. For this reason, the aim of this work is characterizing the gas/liquid mass transfer process, analysing the effect caused for different operational variables. These variables to be taken into account are the stirring rate and the gas flow-rate. It will also be taken into account the constitution of the liquid phase, a non-Newtonian one generated by the mixture, in different grades,

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Nomenclature

a	specific interfacial area (m^{-1})
C	CO_2 concentration ($mol\,L^{-1}$)
C^*	CO_2 solubility ($mol\,L^{-1}$)
d	stirrer diameter (m)
d_i	tank internal diameter (m)
d_{32}	Sauter mean diameter (m)
D	gas diffusivity ($m\,s^{-2}$)
e	minor axis of projected ellipsoid (m)
E	major axis of projected ellipsoid (m)
G	gas flow number ($\eta_{ap}\,U_{sg}\,\sigma^{-1}$)
h	tank height (m)
k	consistence index ($Pa\,s^n$)
k_L	liquid mass transfer coefficient ($m\,s^{-1}$)
$k_L{\cdot}a$	volumetric mass transfer coefficient (s^{-1})
k_s	constant corresponding to Eq. (2)
n	behaviour index
N	stirring rate (s^{-1})
Q_g	gas flow-rate ($m^3\,s^{-1}$)
Re	Reynolds number ($d^2\,N\,\rho\,\eta_{ap}{}^{-1}$)
Sc	Schmidt number ($\eta_{ap}\,\rho\,D^{-1}$)
Sh	Sherwood number ($k_L\,a\,d^2\,D^{-1}$)
t	operation time (s)
U_{sg}	superficial gas velocity ($m\,s^{-1}$)
Greek symbols	
γ	shear rate (s^{-1})
$\dot{\gamma}_{av}$	average shear rate (s^{-1})
ε	gas hold-up
η	absolute viscosity (Pa s)
η_{app}	apparent viscosity (Pa s)

of two polymers aqueous solutions that also show a non-Newtonian behaviour.

2. Experimental

Two polymers, CMC and ALG sodium salts, were supplied by Across Organics (CAS numbers: 9004-32-4 and 9005-38-3) with a low polymerisation degree. The solutions were prepared by mass using a balance with a precision of 10^{-7} kg. Bi-distilled water has been employed to prepare the absorbent phases.

Due to the special characteristics in relation to the rheological behaviour and the apparent viscosity value, we have carried out absorption experimental studies on CMC and ALG mixtures with different ratios. Two kinds of hydrocolloid mixtures have been employed in the present work. The first one (mixture A) consists in the mixture of a CMC aqueous solution of 3 $g\,L^{-1}$ with another one of 1.25 $g\,L^{-1}$ of ALG. These aqueous solutions were blended over all the compositions range, varying from pure CMC aqueous solutions to pure ALG ones. We chose this mixture because the values corresponding to apparent viscosity of individual aqueous solutions of CMC and ALG are similar.

On the other hand, the other mixture analysed (mixture B) was obtained from individual aqueous solutions of CMC and ALG with very different values of apparent viscosity. The mixture consists in blending a 10 $g\,L^{-1}$ CMC aqueous solution with a 1 $g\,L^{-1}$ ALG one.

Rheological studies have been 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 CMC aqueous solutions between the range of 0–10 $g\,L^{-1}$, and for ALG ones between 0 and 1.5 $g\,L^{-1}$.

Mass transfer measurements have been carried out using an aerated stirred vessel used in previous studies [9]. The gas/liquid contactor consists in a cylindrical bubbling stirred vessel made in perpex. The volume of the reactor was 2.5 L and the typical geometric characteristics were: internal diameter, $d_i = 14$ cm, height, $h = 25$ cm. Four baffles were placed on its internal wall from top to bottom in order to improve the mixing and prevent vortex formation during the experiment. The absorption processes have been carried out at 25 °C using a thermostat with a precision of ±0.1 °C. A six blade Rushton turbine, made in metal, was used to stir the liquid in the range of 200–600 rpm.

The gas to be absorbed, carbon dioxide, was passed through two humidifiers at 25 °C to prepare the gas phase and to prevent equimolecular contra diffusion. The gas flow-rate introduced in the bubble contactor was measured and controlled with a mass flow controller (5850 Brooks Instruments), while the flow-rate of the non-absorbed gas phase was determined using a ADM 2000 flow metre, supplied by Agilent Technologies. The pressure drop was measured between the inlet and outlet of the reactor using a Testo 512 digital manometer.

The gas phase was introduced into the contactor through a porous plate (pore diameter ~120 μm) of 4 cm in diameter, placed at the bottom of the vessel. The operational regime was continuous in relation to the gas phase and batch in relation to the absorbent one.

The volumetric mass transfer coefficient has been determined based on the measurements of the amount of gas absorbed per unit time and per unit of liquid phase volume, according to the following equation [11]:

$$\frac{dc}{dt} = k_L \cdot a \cdot (C^* - C) \qquad (1)$$

where C^* is the interfacial concentration of gas at equilibrium (i.e. the gas solubility in the liquid phase), and C (the gas concentration in the bulk liquid) is calculated from the experimental absorption rate data. To evaluate the validity of the methodology employed by means of the use of Eq. (1) for volumetric mass transfer coefficient determination, the above-mentioned calculation has been realized according to the method described by Kordač and Linek [12], and the compared results indicate that both methods agree in the values of volumetric mass transfer coefficient.

Due to the fact that there are differences on the shear rate value both in the contactor and in the rotational viscosimeter, it is necessary to use an average shear rate (γ_{av}). Cropper et al. [13], who studied several models to calculate the average share rate in non-Newtonian liquid phases, concluded that the best model for this kind of systems was the equation proposed by Metzner and Otto [7]. According to Metzner and Otto, the equation employed to calculate the average shear rate is:

$$\dot{\gamma}_{av} = k_s \cdot N \qquad (2)$$

where N, is the stirring velocity and k_s is an impeller constant determined by the impeller geometry. For a Rushton turbine the value proposed by Metzner was 11.5.

The bubble diameter was measured using a photographic method consisting in taking images of the bubbles into the stirred tank contactor. 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, as well as for each gas flow-rate which has been used. The Image Tool v2.0 software has been used to carry out the necessary measurements of the bubbles' geometric characteristics.

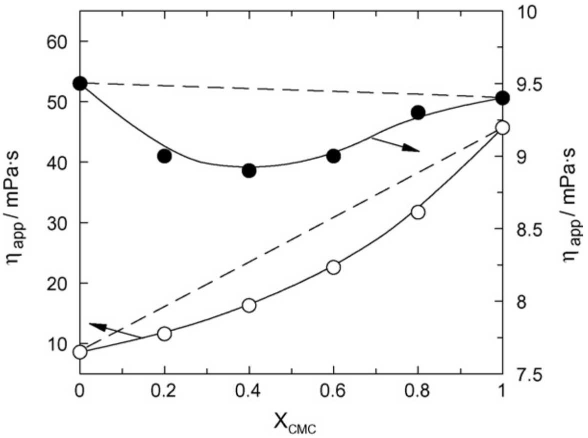

Fig. 1. Influence of mixture composition upon apparent viscosity value. (●) Mixture A at $\gamma = 165\,s^{-1}$; (○) mixture B at $\gamma = 106\,s^{-1}$.

The images we obtained of the bubbles in the liquid phase employed show an ellipsoid shape. For this reason, major (E) and minor (e) axes of the projected ellipsoid (in two dimensions) have been determined. The diameter of the equivalent sphere (Eq. (3)) was taken as the representative bubble dimension.

$$d = \sqrt[3]{E^2 \cdot e} \tag{3}$$

Different authors recommend to use the Sauter mean diameter (d_{32}) [14], which can be determined using the data calculated for the equivalent diameter.

$$d_{32} = \frac{\sum_i \left(n_i \cdot d_i^3\right)}{\sum_i \left(n_i \cdot d_i^2\right)} \tag{4}$$

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

The Sauter mean diameter (d_{32}) and the gas hold-up (ε) values allow the calculation of the specific interfacial area using the following equation [15]:

$$a = \frac{6 \cdot \varepsilon}{d_{32} \cdot (1 - \varepsilon)} \tag{5}$$

3. Results and discussion

Previous studies of our research team have shown that the mixture of two CMC and ALG aqueous solutions has a characteristic behaviour in relation to the mixture composition and the viscosity value [16]. Fig. 1 shows an example of the observed behaviour. The viscosity takes in all cases lower values for the different mixtures analysed than the linear trend. This behaviour is assigned to low interactions between two polymers in aqueous solutions [17], which produce a decrease in viscosity. This decrease in the value of the apparent viscosity was detected at all shear rate values applied to the liquid phase and at different temperatures. Both mixtures analysed in present work have showed negative deviations in relation to the apparent viscosity value, regarding linear trend.

With regard to the mass transfer in the absorption process, previous studies [8,9] have shown that liquid phase viscosity has great influence on the absorption rate to the liquid phase because it produces a clear decrease when the concentration of both polymers increases in the liquid phase. This reduction caused by the increase in viscosity was compensated partly by means of an increase in the stirring power to the liquid phase by the mechanical stirrer. In these studies, the operational variables such as composition, stirring power and gas flow-rate were employed in typical correlation in absorption studies and reaching the conclusion that the dimensionless number correlation allowed the calculation of volumetric mass transfer coefficient with low deviations compared with the experimental values.

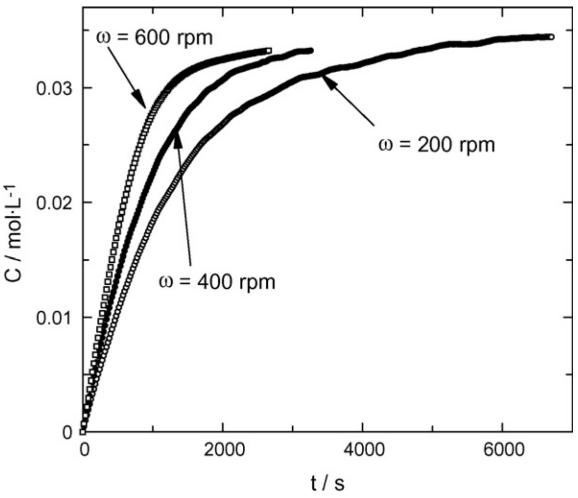

Fig. 2. Influence of stirring rate upon absorption kinetics for mixture A at 80% of CMC solution. $Q_g = 10\,L\,h^{-1}$.

The gas concentration value absorbed in the liquid phase along the operation time has been calculated using the experimental data of gas flow-rate, determined before and after the gas phase was put in contact with the liquid phase in the contactor. The difference between both gas flow-rates allows the calculation of the absorbed gas flow-rate and then, the gas concentration in the liquid phase. Fig. 2 shows the shape adopted by the evolution of carbon dioxide concentration in the liquid phase, with a fast increase at low operation time due to the high driving force. When the liquid phase increases the gas concentration, the driving force decreases its value, and then the absorption rate decreases to a zero value when the solubility value was reached in the liquid phase.

Integrating Eq. (1) and plots of $\ln[C^*/(C^* - C)]$ against time, they were well fitted by straight lines through the origin, showing that $k_L{\cdot}a$ was independent of time under this work conditions (see Fig. 3).

Concerning the influence of operational variables commonly analysed (stirring rate and gas flow-rate) upon the absorption

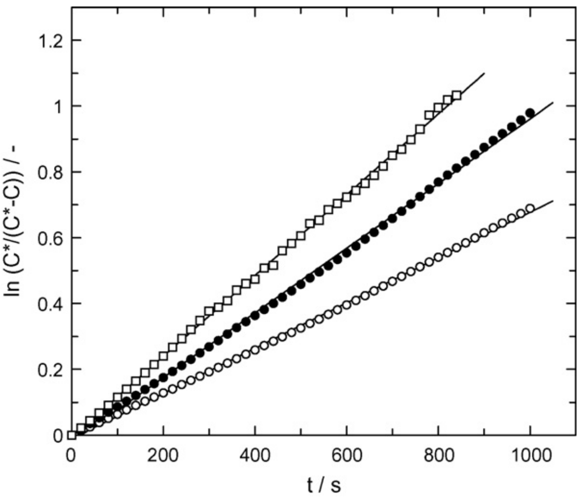

Fig. 3. Volumetric mass transfer determination using Eq. (1). Mixture A. $Q_g = 10\,L\,h^{-1}$. (○) $\omega = 200$ rpm; (●) $\omega = 400$ rpm; (□) $\omega = 600$ rpm.

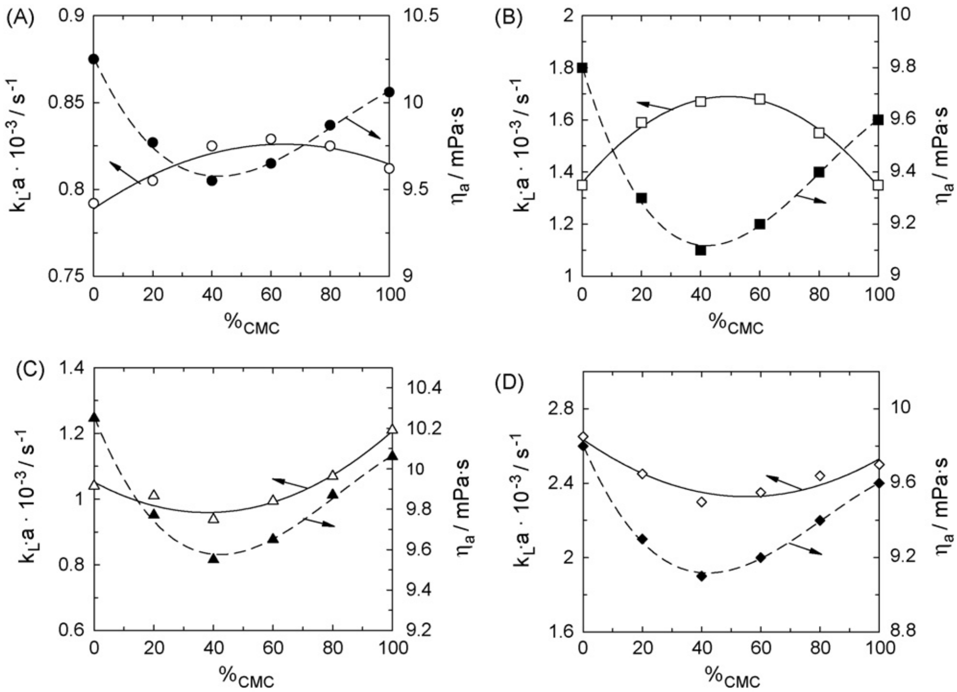

Fig. 4. Influence of liquid phase composition upon apparent viscosity and volumetric mass transfer coefficient. Mixture A. (A) $\omega = 200$ rpm and $Q_g = 10\,\mathrm{L\,h^{-1}}$; (B) $\omega = 600$ rpm and $Q_g = 10\,\mathrm{L\,h^{-1}}$; (C) $\omega = 200$ rpm and $Q_g = 20\,\mathrm{L\,h^{-1}}$; (D) $\omega = 600$ rpm and $Q_g = 20\,\mathrm{L\,h^{-1}}$.

rate, Fig. 2 shows examples of the absorption kinetics obtained for different experimental conditions. The effects observed for both operational variables are similar to previous studies. These indicate that the increase in the stirring rate and the gas flow-rate produces a higher absorption rate, which develops a fast saturation of the liquid phase. This behaviour has a direct influence upon the value of the volumetric mass transfer coefficient, since a high absorption rate produces an increase in the slope of linearized Eq. (1).

The main part of the present work studies the influence caused by the use of mixtures of CMC and ALG aqueous solutions upon the absorption rate and then, upon the volumetric mass transfer coefficient. Subsequently, it also studies the connection with the apparent viscosity value, due to the great influence of this physical property upon the mass transfer [5].

Absorption experimental studies have been carried out varying the operational variables previously commented (stirring rate and gas flow-rate) as well as using liquid phases that cover all the composition range of mixtures employed. Fig. 4 shows experimental results of the volumetric mass transfer coefficient obtained under different experimental conditions. Viscosity data and the influence of the mixture upon its value have also been included in Fig. 4, so that the obtained behaviour can be understood.

The liquid phases employed in this work have a non-Newtonian behaviour [17] and then, the apparent viscosity is shear rate-dependent. This characteristic implies the knowledge of the shear rate applied to the liquid phase into the contactor, as well as the rheological parameters that relate the apparent viscosity and the shear rate by means of a model, i.e. the Ostwald model.

As well as the analysis of the influences caused by the stirring rate and the gas flow-rate, another study has been developed taking into account the influence of the polymer mixtures composition upon the absorption rate and the mass transfer. Fig. 4 shows the experimental results obtained for the volumetric mass transfer coefficient for different mixtures, under different operational conditions. This figure also shows the influence of the mixture composition upon apparent viscosity. In relation to the value of apparent viscosity, for these kind of fluids (non-Newtonian), this physical property is shear rate-dependent and then, the viscosity into the contactor depends on the shear rate applied for the mechanical stirrer.

The knowledge of the shear rate applied to the contactor by the mechanical stirrer (Eq. (2)) is necessary to calculate the apparent viscosity of the liquid phase, as well as to analyse the absorption experimental data. The liquid phase viscosity was calculated using Eq. (6), similar to Oswald's equation. The main difference is the inclusion of the average shear rate present inside the vessel.

$$\eta_{\mathrm{app}} = k \cdot \dot{\gamma}_{\mathrm{av}}^{n-1} \tag{6}$$

Table 1 includes the determined values of rheological parameters corresponding to Eq. (6) for the liquid systems employed such as absorbent phase in present work.

The experimental results shown in the graphs included in Fig. 4 reveal two very different behaviours in relation to the influence of the mixture upon the volumetric mass transfer coefficient, related as well with the absorption rate. The experimental results obtained at the lowest gas flow-rate under different stirring rate values, and then under different shear rates, show a maximum in the value of volumetric mass transfer coefficient with a clear positive deviation from linearity. This behaviour could be expected on the basis of the influence of a mixture ratio upon the value of the apparent viscosity,

Table 1
Values for flow index, *n*, and consistency index, *k*, for both mixtures.

% CMC	A mixture		B mixture	
	n	*k*	*n*	*k*
0	0.915	14.70	0.903	14.16
20	0.930	12.76	0.917	15.48
40	0.941	12.09	0.924	22.86
60	0.946	11.28	0.926	32.07
80	0.954	10.87	0.927	42.89
100	0.960	10.44	0.922	65.44

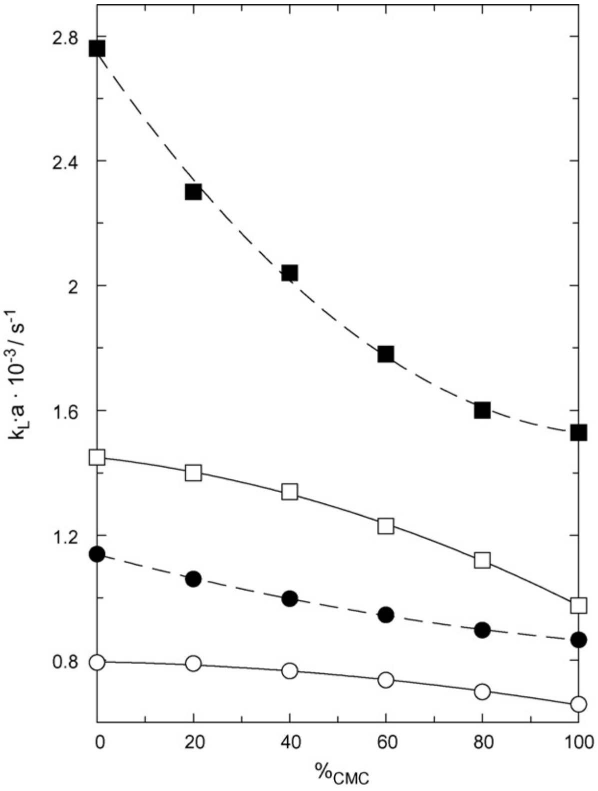

Fig. 5. Influence of liquid phase composition upon volumetric mass transfer coefficient. Mixture B. (○) ω = 200 rpm and Q_g = 10 L h^{-1}; (●) ω = 200 rpm and Q_g = 20 L h^{-1}; (□) ω = 600 rpm and Q_g = 10 L h^{-1}; (■) ω = 600 rpm and Q_g = 20 L h^{-1}.

since it produces a decrease in it. At the same time, important number of studies [18] have shown the negative effect of the viscosity upon the absorption rate.

On the other hand, the experimental results of volumetric mass transfer coefficient determined at high gas flow-rate show an opposite behaviour to the abovementioned. Under this high value gas flow-rate, the behaviour obtained presents a minimum in the value of volumetric mass transfer coefficient around a ratio of 0.4 of CMC solution. In this case, a decrease in viscosity corresponds with a decrease in mass transfer coefficient, too.

A similar trend has been observed when experimental results obtained for other mixture (mixture B) under the same experimental conditions of stirring rate and gas flow-rate were analysed. Positive deviations in volumetric mass transfer coefficient were also obtained for the experiments carried out at low gas flow-rate, but the character of deviations changes to negative values when the gas flow-rate is increased (see Fig. 5).

The stirring rate does not show any influence upon the deviations' character for both mixtures analysed in this work, but the magnitude of the deviation is related to the stirring rate, producing an increase in the deviation when the stirring rate value increases. Figs. 4 and 5 do not include experimental data at 400 rpm to remove difficulties to understand the figures and because the effect caused by the stirring rate upon the volumetric mass transfer coefficient has a linear trend.

The behaviour previously analysed on the basis of the experimental data shown in Figs. 4 and 5 could be explained by the combined effect of operational conditions upon both parameters that forms the volumetric mass transfer coefficient. Due to the important effect caused by the gas flow-rate, since it changes the influence of the liquid phase composition upon the volumetric mass transfer coefficient when the gas flow-rate is varied from low to high values, this behaviour must be explained taking into account the effect of the gas flow-rate upon the two parameters: mass transfer coefficient and interfacial area.

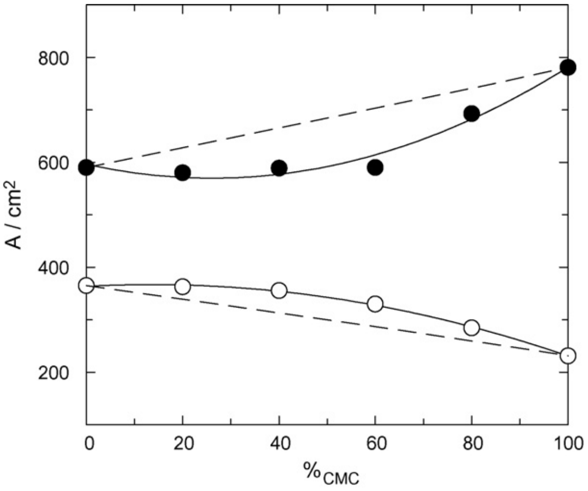

Fig. 6. Influence of mixture ratio and gas flow-rate upon gas–liquid interfacial area. Mixture B. ω = 600 rpm. (○) Q_g = 10 L h^{-1}; (●) Q_g = 20 L h^{-1}.

The decrease in viscosity for mixtures of CMC and ALG aqueous solutions at 40% of CMC aqueous solutions could produce an increase in the mass transfer coefficient, k_L, due to the fact that the mass transfer resistance decreases, as many different previous studies have concluded [5]. This phenomenon explains the behaviour observed in Figs. 4 and 5, where an increase in the volumetric mass transfer coefficient was observed (at low gas flow-rate value) with a corresponding decrease in viscosity. On the other hand, a decrease in mass transfer when a high value of gas flow-rate is employed is assigned to the effect of this variable upon the interfacial area, *a*, and the influence of this hydrodynamic parameter upon the global mass transfer process. A higher value of gas flow-rate fed to the stirred tank produces an increase in mass transfer due to an increase in the interfacial area, as well as turbulence in the bulk of the liquid phase. In this work, the mixture of aqueous solutions of both polymers produces a decrease in viscosity that has a negative effect upon the interfacial area. This negative effect is related to the increase in the effectiveness of bubbles coalescence along the liquid phase, producing a decrease in the interfacial area in relation to systems with higher viscosity values, such as individual polymers solutions. This decrease affects directly upon the volumetric mass transfer coefficient compensating the positive effect of viscosity decrease upon the mass transfer coefficient.

To confirm the previous hypothesis, different studies have been carried out based on the gas–liquid interfacial area generated into the bubble contactor, employing the experimental methodology commented in Section 2.

Fig. 6 shows the results obtained as regards the influence of the liquid phase composition, and therefore, as regards the liquid viscosity and the gas flow-rate upon the gas–liquid interfacial area produced inside the contactor. When the lowest gas flow-rate is employed, positive deviations have been observed as regards the linearity between pure polymer solutions. This behaviour corresponds with the negative deviations also observed for viscosity in the mixtures employed. A decrease in viscosity allows the produced bubbles to have a minor size, and this characteristic implies a positive effect upon the interfacial area. On the other hand, an increase in gas flow-rate produces an increase in the interfacial area due to the gas hold-up increase, as regards the results observed at low values in gas flow-rate. However, it produces a change in the charac-

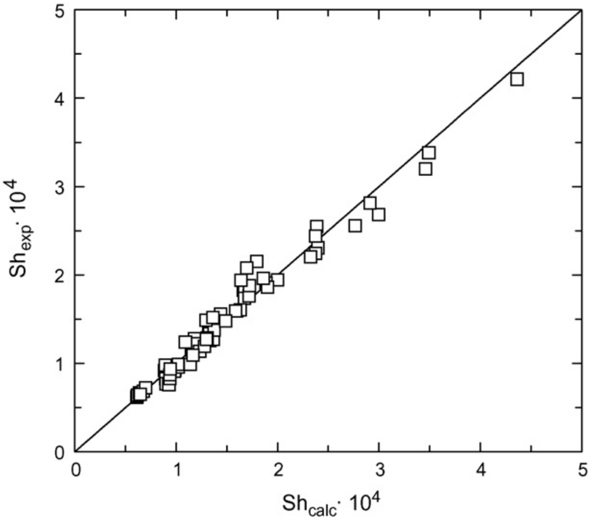

Fig. 7. Comparison between experimental values and calculated ones of Sherwood numbers employing Eq. (7).

ter (negative) of the deviations as regards linearity. This behaviour is due to minor values of viscosity, that combined with high gas flow-rates produce the effective collisions and fusion of bubbles, increasing the size and then producing a decrease in the interfacial area.

A common equation based on dimensionless numbers has been used to fit the volumetric mass transfer coefficient experimental data. Operational variables and physical properties are included in the dimensionless numbers corresponding to this equation.

$$Sh = \alpha \cdot Re^{\beta} \cdot Sc^{\gamma} \cdot G^{\delta} \tag{7}$$

where *Sh*, *Re*, *Sc*, are the numbers of Sherwood, Reynolds and Schmidt, respectively, and *G* is a gas-flow number. Eq. (7) introduces several physical properties like viscosity and density of the absorbent phase, as well as the diffusivity of the gas into the polymer aqueous solutions.

As regards the diffusivity of carbon dioxide in polymer aqueous solutions, the results contributed in literature show different trends in relation to the polymer concentration effect upon the diffusivity value. Certain authors have proposed that the diffusivity decreases when the concentration of several polymers increases [19], while other researchers, for carboxymethyl cellulose aqueous solutions and other polymers, have not found a clear behaviour [20]. In the present work, a typical equation for carbon dioxide diffusivity has been employed [21].

$$\left(\frac{D_{\text{solution}}}{D_{\text{water}}}\right) = \left(\frac{\eta_{\text{water}}}{\eta_{\text{solution}}}\right)^{0.8} \tag{8}$$

where *D* and η are diffusivity and viscosity.

Employing Eq. (8), gas diffusivity was calculated on the basis of the liquid phase viscosity and the corresponding data for till water, and then Eq. (7) could be employed to fit experimental Sherwood data.

Fig. 7 shows the comparison between experimental and calculated data from Eq. (7) for Sherwood number. Fit parameters corresponding to Eq. (7) have been determined employing all experimental data under different experimental conditions of composition, stirring rate and gas flow-rate. The determined values for α, β, γ and δ parameters were 72.6, 0.55, 0.33 and 0.54, respectively. The exponents determined for the dimensionless numbers agree with those previously suggested by certain authors through the microconvection model for mass transfer [22].

4. Conclusions

The present paper analyses the gas–liquid mass transfer process of a pure gas phase to complex phase formed by a ternary system (water + CMC + ALG) in a bubble stirred tank. The rheological characterization of the aqueous solutions has been carried out observing clear deviations from linearity for apparent viscosity. The influence of these deviations upon gas absorption has been the central study of present work, but the high importance of gas flow-rate upon the behaviour of volumetric mass transfer coefficient indicates that the gas–liquid interfacial area plays an important role in the global mass transfer process. Studies regarding interfacial area determination under the different experimental conditions (liquid phase composition, gas flow-rate and stirring rate) has been performed and confirms that the use of high gas flow-rate values produces important changes in the behaviour of absorption process because enhance bubble coalescence that implies a reduction in interfacial area and also in absorption rate.

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