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EFFECT OF DISSOLVED METAL SULPHATES ON GAS–LIQUID OXYGEN TRANSFER IN AGITATED QUARTZ AND PYRITE SLURRIES

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ABSTRACT

A previous study on mass transfer in agitated three phase systems showed that for quartz slurries the volumetric oxygen transfer coefficient k_La *[*s^{-1}*] decreases with increasing solids fraction, whereas pyrite particles increase the* k_La *value. The present study was conducted in continuation of these results and attempts to explain why the addition of pyrite particles resulted in an increase in* k_La*. For this purpose, the effect of ionic strength, mineral density and oxygen consumption due to homogeneous reactions was examined. Neither the high density of pyrite nor the oxygen consumption could offer an explanation for the increased* k_La *values obtained for pyrite slurries. The variable mineral density did not affect* k_La *at all, while the consumption of oxygen was not large enough to cause an enhancement of* k_La*. However, by reducing bubble coalescence frequency and thus bubble size, the ionic strengths found for pyrite slurries could partially explain the increase in* k_La*. For 15 vol.% pyrite slurries, the ionic strengths proved to account for at least 60% of the enhancement. The variable(s) and/or mechanism(s) causing the remaining 40% of the enhancement could not be identified (yet). To demonstrate the effect of ionic strength as such, various electrolytes (viz.* $CuSO_4$*,* $FeSO_4$*,* $ZnSO_4$ *and* $Al_2(SO_4)_3$*) were added to a 15 vol.% quartz slurry. For all metal sulphates, increasing the ionic strength up to about 0.25 mol/l resulted in an increase of* k_La *by up to a factor 2.5. © 2000 Published by Elsevier Science Ltd. All rights reserved.*

Keywords
Sulphide ores; gold ores; oxidation; biooxidation

INTRODUCTION

Although the influence of solids loading on the volumetric oxygen transfer coefficient k_La has been studied extensively (Albal *et al.*, 1983; Garchia-Ochoa *et al.*, 1997; Oguz *et al.*, 1987), the effect of suspended particles on the k_La value is not clear yet. All three possible effects have been reported: a decrease in k_La value (Mills *et al.*, 1987; Oguz *et al.*, 1987), an increase (Derksen *et al.*, 2000; Mehta *et al.*, 1971) and a negligible influence (Oguz *et al.*, 1987). A general trend suggested by Lee and Foster (1990) is that a small volume of relatively small sized solids (up to 2–3 vol.%) has little effect on k_La. As more solids are added (up to 7–9 vol.%), the k_La value increases and at higher solids fractions (above 10 vol.%) k_La decreases significantly. A possible explanation offered by Lee and Foster (1990) for this trend is that a small amount of solids does not change the viscosity of the liquid, but enhances surface renewal rate and mobility thereby increasing k_La, whilst a large amount of solids increases the viscosity of liquid, thereby decreasing the surface renewal rate and hence k_La.

One of the exceptions to the trend proposed by Lee and Foster (1990) can be found in the work of Derksen *et al.* (2000) on oxygen transfer in agitated quartz (SiO_2) and pyrite (FeS_2) slurries. For quartz slurries, the results of Derksen *et al.* (2000) confirm the suggested trend, viz. a negligible effect or a slight increase of k_La at 5 vol.% and a significant decrease at higher solids fractions. However, pyrite particles were found to have an opposite effect on the k_La value compared to quartz particles: the addition of pyrite particles (up to 12.7 vol.%) resulted in a significant increase in k_La value. Derksen *et al.* (2000) did not offer an explanation for the difference in effect.

The understanding of the mechanisms influencing the oxygen transfer rate is important, however. By influencing the availability of dissolved oxygen, the transfer rate affects the kinetics of oxidation processes such as waste water treatment and pressure oxidation of sulphide concentrates. In the case of bio-oxidation of refractory gold-bearing sulphide concentrates, the oxygen transfer rate even proved to be the key factor limiting the process kinetics at high solids fractions (Bailey *et al.*, 1994).

In a continuation of the work done by Derksen *et al.* (2000), the present study was conducted to gain an understanding of the reason(s) why the addition of pyrite particles results in an increase in k_La. Obviously, (possible) differences in physicochemical properties of pyrite and quartz slurries provide possible explanations for the different effect of pyrite or quartz addition on k_La. These (possible) differences are viscosity, surface tension and ionic strength of the slurry, oxygen consumption and oxygen adsorption in the slurry and hydrophobicity, density and zeta potential of the solids.

On the basis of theory and/or literature data, the variables viscosity, surface tension, oxygen adsorption, hydrophobicity and zeta potential were excluded as possible explanations. Either the difference was estimated to be not significant (viscosity and oxygen adsorption), or the difference was not presumed to cause an improvement in the oxygen transfer rate (surface tension (Craig *et al.*, 1993; Machon *et al.*, 1997), hydrophobicity (Siegel *et al.*, 1988; van Weert *et al.*, 1995) and zeta potential). The following parameters were identified as possibly or presumably related to the enhanced k_La values and their influence on k_La was experimentally investigated:

- Mineral density
- Oxygen consumption due to homogeneous reactions (Beek *et al.*, 1991)
- Ionic strength of the slurry (Craig *et al.*,1993; Zieminski *et al.*, 1971)

Mineral density

Little is known about the effect of mineral density on oxygen transfer. In theory, mineral density is not expected to influence k_La since for slurries the bubble coalescence frequency, and thus k_La, was found to depend on the local energy dissipation rate [W/kg] (Nagaraj *et al.*, 1987), which itself is independent of mineral density. Nevertheless the influence of mineral density was examined in this study, since it could not be excluded as a possible cause beforehand.

Oxygen consumption due to homogeneous reactions

When aerating a pyrite slurry, oxygen could be consumed if pyrite- or ferrous iron oxidation reactions would take place (Evangelou, 1995). Oxygen transfer can be significantly enhanced by homogeneous oxidation reactions, but only when the Hatta number (Ha $= \sqrt{Dk_r / k_L^2}$) is larger than 0.3 (Beek *et al.*, 1991).

Ionic strength of the slurry

Pyrite's tendency to oxidise could lead to the presence of dissolved ferrous (Fe^{2+}), ferric (Fe^{3+}) and sulphate (SO_4^{2-}) ions in the pyrite slurries. The ions can be produced either directly by the in-situ occurrence of pyrite oxidation reactions (Evangelou, 1995), or indirectly by dissolution of the (ex-situ developed) superficial oxidation layer of pyrite particles.

It is well-known (Lee *et al.*, 1970; Lessard *et al.*, 1970; Linek *et al.*, 1987), that addition of electrolytes to a gassed, aqueous system could lead to a reduction in bubble coalescence frequency, up to the so-called critical concentration beyond which a further increase in electrolyte concentration no longer has an effect on coalescence frequency. A reduction in bubble coalescence frequency will result in a decrease in bubble size and, as a consequence, an increase in k_La. To what extent the addition of an electrolyte will cause a reduction in bubble size depends, given a certain initial bubble size, on the type of electrolyte. Certain electrolytes are effective at lower concentrations than others (Lessard *et al.*, 1970), while some have little or no effect at all (Craig *et al.*, 1993).

Numerous studies have been conducted on the effect of electrolytes on bubble size (Barigou *et al.*, 1992; Machon *et al.*, 1997) and on the effect of electrolyte type on bubble coalescence frequency ((Craig *et al.*, 1993; Deschenes *et al.*, 1998; Pashley *et al.*, 1997). (Only few experimental studies paid attention to the effect of electrolytes on k_La values (Linek *et al.*, 1988; van 't Riet, 1979)). Although the impact of effective dissolved ions on bubble size is clear, viz. reduction, the mechanisms by which these ions hinder bubble coalescence are still in discussion.

A clear relation between coalescence rate and ionic strength I [mol/l] of an aqueous system has been established (Craig *et al.*, 1993; Keitel *et al.*, 1981; Zieminski *et al.*, 1971). As a general rule of thumb, 3–2 electrolytes (e.g. $Al_2(SO_4)_3$) will have a more pronounced influence on k_La than 3–1 and 2–2 combinations, which in turn are more effective than 2–1 and 1–2 combinations. However, several authors demonstrated that other factors besides ionic strength also play a role in hindering bubble coalescence (Christenson *et al.*, 1995; Linek *et al.*, 1988; Prince *et al.*, 1990). Overall, there is no unambiguous relation available in literature describing the dependence of bubble coalescence frequency (and thus k_La) on the concentration and type of dissolved ions. In this study, we used the parameter ionic strength to relate the influence of the dissolved ions to k_La.

EXPERIMENTAL

Mineral density

The effect of mineral density on k_La was investigated by conducting oxygen transfer experiments in quartz, pyrite and specular hematite (Fe_2O_3) slurries. The density of hematite is similar to that of pyrite and almost twice as high as that of quartz. The particle size distributions of all three mineral fractions were equivalent.

It is pointed out that the k_La values obtained in the oxygen transfer measurements, besides being dependent on solids fraction, are dependent on time as well: the k_La values obtained for a specific solids fraction increase in time. Therefore, ideally, the k_La values should be measured at time $t=0$, directly after preparation of the specific solids fraction. However, this was practically not feasible. To ensure a stable value of C^*, all measurements had to be made at times t larger than zero, however never exceeding 15 minutes.

Oxygen consumption due to homogeneous reactions

The influence of oxygen consumption due to homogeneous reactions on the obtained k_La values is investigated by measuring the oxygen consumption in a 15 vol.% pyrite slurry after 1 and 11 hours of aeration and calculating the accompanying Hatta number.

Ionic strength of the slurry

To investigate the influence of ionic strength on the k_La values obtained for pyrite slurries, first the ionic strength of a pyrite slurry was measured as a function of the solids fraction. Besides being dependent on the solids fraction, the ionic strength proved to be dependent on time as well: it increased with time. However, all measurements were conducted as fast as possible, viz. within 15 minutes after preparation of the specific solids fraction. The ionic strength obtained in this first measurement showed to have approximately the same value as the ionic strengths obtained after several hours.

Secondly, the quantitative contribution of ionic strength was examined by (a) adding quartz particles (up to a volume fraction of 15%) to a leach solution of a 12 hours aerated 15 vol.% pyrite slurry and (b) adding $FeSO_4$ to a 15 vol.% quartz slurry up to similar ionic strengths.

To demonstrate the effect of ionic strength on k_La, various metal sulphates (viz. $CuSO_4$, $ZnSO_4$, $FeSO_4$ and $Al_2(SO_4)_3$) were added to a 15 vol.% quartz slurry. The ionic strengths of aqueous quartz slurries were assumed to be equal to that of tap water.

Experimental set-up

The experiments were carried out in a sparged, stirred Perspex tank of 0.14 m in diameter, which can be closed airtight. Under ungassed conditions, the liquid level of the batch was equal to the diameter of the vessel. Four baffles (width = 1/10 of tank diameter) prevented solid body rotation of the mixing phase. The baffles did not extend all the way down to the bottom of the tank to avoid sedimentation of the solids in the wake of the baffles. Air was introduced into the vessel through a point sparger (1 mm internal diameter) located below the impeller. A Rushton turbine (0.35 times tank diameter) of standard proportions was used for agitation. The superficial gas velocity used was 0.65 cm/s and the estimated mechanical power input per slurry mass under gassed conditions was 3 W/kg. The offgas was passed through a condensor to minimise water losses. A polarographic electrode (Schott Geräte CG 867) was used for measuring the dissolved oxygen concentration of the liquid phase. The probe was located behind a baffle to protect the membrane from the abrasive action of the quartz particles in particular. The membrane of the probe was placed at the same height as the impeller.

Materials

The three minerals used, viz. quartz, pyrite and hematite, all had similar particle size distributions: a d_{90} of 35 μm and a d_{50} of 10 μm. Both the pyrite and the hematite ore had to be crushed (jaw crusher) and milled (ball mill) before use, whereas the quartz particles were already sufficiently small as obtained. For pyrite, the chemical and mineralogical composition was determined. X-Ray Fluorescence (XRF) analysis revealed the presence of minor elements such as silicon, calcium, aluminium and copper in the pyrite. X-Ray Diffraction (XRD) analysis identified the presence of α-quartz, muscovite and/or illite and possibly anhydrite ($CaSO_4$).

The electrolytes used, viz. $CuSO_4$, $ZnSO_4$, $FeSO_4$ and $Al_2(SO_4)_3$, were of analytical grade and were added in hydrated form. Distilled water was used for all experiments in which electrolytes were added. The pH of the distilled water was lowered with sulphuric acid to pH values of 1.5 to 4 in order to prevent the dissolved cations from precipitating as metal hydroxides. Tap water (pH ~ 8.5) was used for the other tests.

Determination of k_La values

Volumetric oxygen transfer coefficients can be determined by a large variety of dynamic or steady-state methods. In this study, the nitrogen gas → air interchange method (a dynamic gas–liquid absorption variant) was chosen to determine k_La values (Derksen *et al.*, 2000).

The $N_2 \rightarrow$ air interchange method is a simple and reliable way to determine k_La values in coalescent systems such as aqueous slurries (provided that both the probe response time τ_p [s] and the gas phase residence time τ_G [s] are significantly smaller than the mass transfer response time of the system, $1/k_La$). In the experiments performed by the authors, these two restrictions on applicability of the $N_2 \rightarrow$ air interchange method were met.

However, for non-coalescent systems such as ionic slurries, gas interchange variants could underestimate k_La values. An alternative method, which is assumed not to encounter this problem, is the $Na_2SO_3 \rightarrow O_2$ degassing variant (Linek *et al.*, 1982). The latter method is however not suitable for three phase systems, since sedimentation of solids will occur during the time agitation is stopped for degassing.

The k_La value can be determined from a dynamic oxygen transfer measurement by least squares fitting the probe response (i.e. the measured dissolved oxygen concentration in liquid, C_{lm}, as a function of time t) to Equation 1.

$$C_{lm} = C^* \cdot \left(1 - \frac{1}{k_L a \cdot \tau_p - 1} \cdot \left(k_L a \cdot \tau_p \cdot \exp(-\frac{(t-t_0)}{\tau_p}) - \exp(-k_L a \cdot (t-t_0))\right)\right) \quad (1)$$

In this equation, C^* [mol/m^3] is the dissolved oxygen concentration in the liquid in equilibrium with the gas bulk concentration; t_0 [s] is a variable correcting for the time necessary to build up the oxygen concentration gradient at the start of a dynamic oxygen transfer measurement. The value of t_0 was, just like the value of k_La, determined by fitting the probe response to Equation 1.

The values of variables C^* and τ_p in Equation 1 were estimated. C^* was estimated directly from the probe response curve of a dynamic oxygen transfer measurement; the probe response time τ_p was determined by fitting the probe response to a step change in dissolved oxygen concentration to Equation 2.

$$C_{lm}(t) = C^* \left(1 - \exp\left(\frac{-t}{\tau_p}\right)\right) \quad (2)$$

Equation 1 is derived from the second order differential equation obtained when combining the oxygen mass balance for the liquid phase during a dynamic oxygen transfer measurement (Eq. 3) with Equation 4, to account for the probe response time. The boundary conditions used to solve the second order differential equation are given in Equation 5.

$$\frac{dC_l}{dt} = k_L \cdot a \cdot (C^* - C_l) \quad (3)$$

$$\frac{dC_{lm}}{dt} = \frac{1}{\tau_p} \cdot (C_l - C_{lm}) \quad (4)$$

$$C_{lm}(0) = 0 \; ; \frac{dC_{lm}(0)}{dt} = 0 \quad (5)$$

Equation 2 is derived from solving Equation 4 with the assumption of $C_l = C^*$ and with the boundary condition $C_{lm}(0) = 0$.

All measured k_La values were converted to values at a reference temperature of 20°C, using the empirical equation (Eq. 6) of Oosterhuis *et al.* (1983).

$$(k_L a)_{20} = \frac{(k_L a)_T}{1.020^{(T-20)}} \quad (6)$$

Determination of oxygen consumption due to homogeneous reactions

The rate of oxygen consumption due to homogeneous reactions was measured according to the following procedure. After saturating the slurry with air, the air supply was stopped and the dissolved oxygen concentration in the airtight vessel was monitored as a function of time. The reaction rate constant required to calculate the Hatta number was determined from the probe response; the mass transfer coefficient in the liquid phase, k_L, was estimated to have a value of approximately 1×10^{-4} m/s (Calderbank *et al.*, 1961).

Determination of chemical composition of liquid slurry phase

The chemical composition of the liquid slurry phase was determined by XRF spectrometry. In addition, the valence of the dissolved iron (viz. 2+ or 3+) was determined with a colorimetric technique and it was assumed that all sulphur present in the solution occurred in the form of sulphate ions.

RESULTS AND DISCUSSION

Effect of mineral density on k_La

From Figure 1 it can be observed that mineral particles of different density, viz. hematite and quartz particles, affect the oxygen transfer rate in a similar manner. That is, they both reduce the k_La value to the same extent. It can therefore be concluded that mineral density has no influence on the volumetric oxygen transfer coefficient. The relatively high density of pyrite cannot provide an explanation for the increased k_La values obtained for pyrite slurries.

The respective effects of quartz and pyrite particles on k_La values were also observed by Derksen *et al.* (2000).

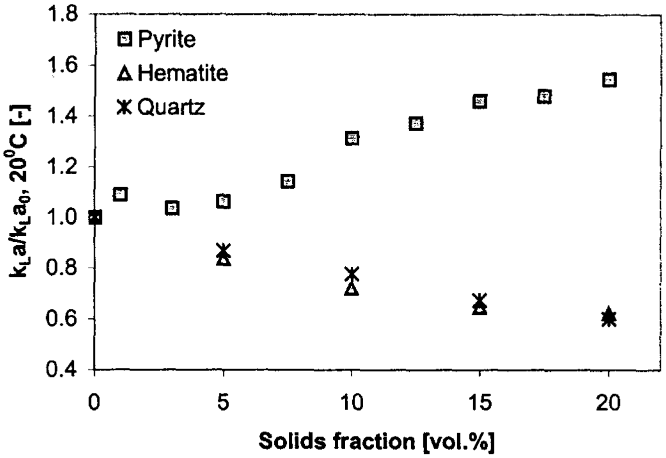

Fig. 1 Volumetric oxygen transfer coefficients relative to the value in water, k_La/k_La_0, as a function of slurry solids fraction for minerals of different density, viz. 5.02 (Pyrite), 5.25 (Hematite) and 2.65 (Quartz) kg/m^3; $k_La_0 \sim 0.08$ s^{-1}.

Oguz *et al.* (1987) found similar results for hematite (Fe_2O_3) slurries, viz. a decreasing k_La with an increasing hematite solids fraction. The k_La value decreased by 40% when increasing the solids fraction from 2.5 to 10 vol.%. The authors (who performed their tests in a stirred tank of standard geometry) examined the effect of sand particles as well, comparing sea sand and kieselguhr. Despite the fact that both types of sand have similar densities as quartz particles, only kieselguhr showed the same trend as quartz particles. Sea sand, which is slightly coarser than kieselguhr, was found to have no effect on k_La at all (perhaps because of the effect of sea salt ions).

The results shown in Figure 1 agree only partly with those of Garcia-Ochoa *et al.* (1997). Their experimental work in bubble columns showed that k_La values obtained for glass bead slurries are much smaller than those obtained for pyrite slurries. Since the average density of glass beads is similar to that of quartz, these results are analogous. However, contrary to the results presented in this paper, no increase in k_La values was observed for pyrite slurries relative to water. Garcia-Ochoa *et al.* (1997) obtained for all gas–liquid–solid systems investigated by them k_La values definitely smaller than those of water. This may result from particles directly influencing sparging in bubble columns, leading to increased coalescence.

Effect on k_La of oxygen consumption due to homogeneous reactions

The oxygen consumption due to homogeneous oxidation reactions was measured in a 15 vol.% pyrite slurry after 1 and 11 hours of aeration. It is assumed that no oxygen is consumed in the quartz slurries. The results showed that the maximum Hatta number was 0.03, which is significantly smaller than the 0.3 required for an enhancement of the oxygen transfer rate. It is, therefore, concluded that the oxygen consumption due to homogeneous reactions is not large enough to cause any enhancement of the k_La value.

Effect of ionic strength on k_La

The ionic strength of pyrite slurries was measured as a function of solids fraction. The results show that the ionic strength of a pyrite slurry increases with increasing solids fraction, viz. from 0.07 mol/l at 5 vol.% to 0.23 mol/l at 15 vol.%. (after 15 minutes). The increase in k_La with increasing pyrite volume fraction clearly corresponds to an increase in ionic strength. The dissolved ions (primarily sulphate, ferrous iron and calcium ions) proved to originate mainly from dissolution of the superficial oxidation layer of pyrite particles. This layer formed prior to the use of the pyrite particles in the slurry.

Subsequently, the quantitative contribution of ionic strength was examined by (1) adding quartz particles (up to a volume fraction of 15%) to a leach solution of a 12 hours aerated 15 vol.% pyrite slurry and (2) adding $FeSO_4$ to a 15 vol.% quartz slurry up to similar ionic strengths.

The k_La value obtained for the "pyrite leach solution+quartz" slurry was 40% lower than the one obtained for the pyrite slurry itself, respectively 0.10 and 0.16 s^{-1}.

The results of adding $FeSO_4$ to a quartz slurry are shown in Figure 2. Plotted in the same figure are the k_La values obtained for pyrite slurries after various aeration times (0.25 to 44 hours) resulting in an increase of ionic strength. Note that the quartz and pyrite slurries are respectively artificially ionic (adding salts) and naturally ionic (dissolution of outer oxidation layer).

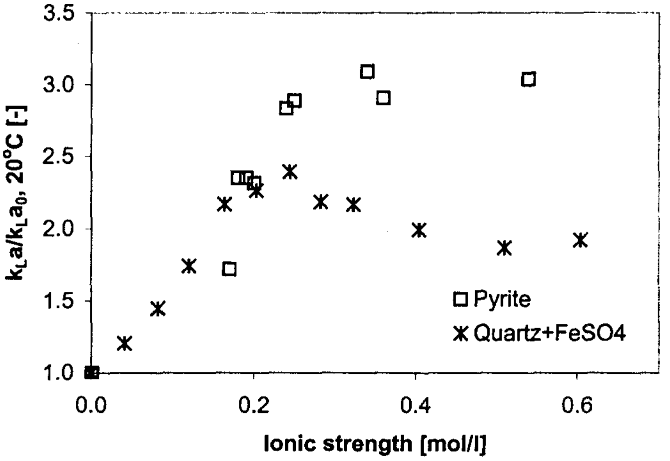

Fig. 2 Volumetric oxygen transfer coefficients relative to the value in water, k_La/k_La_0, as a function of ionic strength for pyrite (naturally ionic) and quartz (artificially ionic) slurries; Solids fraction of both slurries: 15 vol.%.

In addition to the results of the "pyrite leach solution" experiments, the data in Figure 2 show that the ionic strengths present in the 15 vol.% pyrite slurries account for (at least) 60 % of the measured enhanced k_La values. Apparently, pyrite slurries behave similar to ionic slurries up to an ionic strength of 0.25 mol/l, but are more efficient in increasing k_La at higher values.

It can also be observed in Figure 2 (and Figure 3) that, in the case of adding salt to a quartz slurry, k_La values dropped after reaching their maximum value and seemed to reach a constant value for high ionic strengths. No adequate explanation can be given for this phenomenon at this stage.

In addition to investigating the effect of minerals on k_La, the effect of adding various metal sulphates, viz. $CuSO_4$, $ZnSO_4$, $FeSO_4$ and $Al_2(SO_4)_3$, to a 15 vol.% quartz slurry was examined. The results are depicted by Figure 3.

In Figure 3 it can be seen that the addition of various metal sulphates up to an ionic strength of about 0.25 mol/l resulted in an increase of the k_La value up to a factor 2.5.

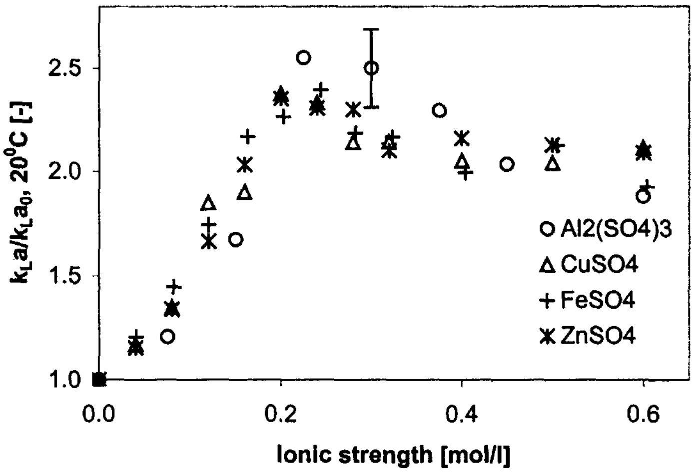

Fig. 3 Volumetric oxygen transfer coefficients relative to the value in water, k_La/k_La_0, as a function of ionic strength for the addition of various metal sulphates to a 15 vol.% quartz slurry.

CONCLUSIONS

1. The variable mineral density had no influence on the volumetric oxygen transfer coefficient. The high density of pyrite can therefore not explain the increased k_La values obtained for pyrite slurries.

2. The k_La values obtained for pyrite slurries are not enhanced by homogeneous oxidation reactions.

3. The ionic strengths found for pyrite slurries (originating mainly from dissolution of the superficial oxidation layer) can partly explain the enhanced k_La values. For 15 vol.% pyrite slurries, the ionic strength accounts for at least 60% of the increase in k_La.

4. The mechanism(s) causing the remaining 40% of the enhancement was (were) not identified.

5. Up to an ionic strength of about 0.25 mol/l, the addition of various metal sulphates to a 15 vol.% quartz slurry increases the k_La value by up to a factor 2.5.

PRACTICAL IMPLICATIONS

For industries working with ionic strengths below approximately 0.25 mol/l, the implications of the results obtained in this work could be significant. In the region $I < 0.25$ mol/l, the k_La value can be enhanced by up to a factor 2.5 by increasing the ionic strength. Depending on the actual situation, this option may be practically and/or economically feasible for industrial plants.

NOMENCLATURE

a	specific interfacial area	m^2/m^3
c_i	concentration of ionic species i	mol/l
C^*	equilibrium oxygen concentration	mol/m^3
C_l	oxygen concentration in bulk of liquid	mol/m^3
C_{lm}	measured dissolved oxygen concentration in liquid	mol/m^3
D	diffusion coefficient	m^2/s
k_L	mass transfer coefficient in liquid phase	m/s
k_r	reaction rate constant	s^{-1}
k_La	gas–liquid volumetric oxygen transfer coefficient	s^{-1}
t	time	s
t_0	fit parameter	s
z_i	valence of ionic species i	—
T	temperature	°C
τ_p	probe response time constant	s
τ_G	gas phase residence time	s
Ha	Hatta number [-]	$\sqrt{Dk_r / k_L^2}$
I	Ionic strength [mol/l]	$\frac{1}{2} \Sigma c_i z_i^2$

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