Correlation for Volumetric Mass Transfer Coefficient in Mechanically Agitated Aerated Vessel for Oxygen Absorption in Aqueous Electrolyte Solutions

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Empirical equations used so far for computing $k_l a$ in mechanically agitated aerated vessels agree well with the measured dependence of $k_l a$ on the power input of the stirrer and the superficial velocity of the gas. However, they do not fit the dependence of $k_l a$ on the physical properties of the liquid batch. From the data published so far it can be deduced that the equations suitable for the description of the dependence of $k_{mol} a$ on the liquid physical properties must include some independent variable, which characterizes the rate of coalescence of the bubbles. About 400 experimental values of $k_l a$ were compared with those calculated from different relations. This comparison shows that the rate of coalescence for the aqueous electrolyte solutions is well described on the basis of the Gibbs theory of adsorption of the solute in the surface layer of the solution.

INTRODUCTION

The correlations for the calculation of $k_l a$ published so far are mostly of the form

$$k_l a = C e^{n_1} v_s^{n_2} \tag{1}$$

This equation expresses satisfactorily the influence of the specific power dissipated in the liquid phase and of the gas superficial velocity on the value of $k_l a$ in the apparatus of a given geometry for one type of liquid phase. However, it is not able to describe the $k_l a$ data obtained in liquids of different physical properties.

The aim of this work is to find such an equation, which would express the dependence of $k_l a$ for oxygen absorption in aqueous inorganic electrolyte solutions on the physical properties of the liquid phase.

One of the reasons published equations do not adequately describe the dependence of $k_l a$ on the liquid properties is the lack of sufficiently large sets of consistent experimental data. Such large sets are usually measured using dynamic methods, where the dissolved oxygen concentration is mostly measured with the oxygen probe. Linek et al.[1–4] have shown that the majority of the data found by this method are incorrect. The usual causes of the errors are the assumption of ideal mixing of the gas in the vessel when it

is not justified (e.g., for noncoalescent batches such as electrolyte solutions) and incorrect interpretation of the oxygen probe data. Attempts to construct empirical equations from incorrect data lead not only to incorrect values of numerical coefficients in these equations, but also to an unsuited form of the correlations and to incorrect choice of independent variables. To illustrate these assertions, Figure 1 shows the dependence of $k_l a / v_s^{0.4}$ on e for noncoalescent electrolyte solutions. The experimental points fitted by the full straight line were obtained by the correct variant of the dynamic method as that used in this work. The dashed line corresponds to the data obtained by the incorrect dynamic method under the same experimental conditions, where $k_l a$ was evaluated from the response of the system on the nitrogen–oxygen exchange in the entering gas while the impeller speed remained constant. This procedure is very often used in the literature. The data shown in Figure 1 are taken from the work of Linek et al.,[4] where it is explained that the error of the incorrect variant mentioned (designated by Linek et al.[3,4] as procedure A, $N_2 \rightarrow O_2$) was caused by an incorrect assumption of the ideal mixing of the gas in the vessel. Figure 1 illustrates how the incorrect method distorts the character of the dependence of $k_l a$ on e. When the exchange air–nitrogen was used, the results were even worse. For e of about 2000 W/m³, the incorrect method yields values of $k_l a$ about 60% lower, and with increasing values of e the error increases. Data presented in this article were measured with the correct variant of dynamic method,[3] which on the basis of present knowledge[3,4] can be taken as reliable, producing $k_l a$ data consistent with those measured with the independent steady-state methods.

PRELIMINARY CONSIDERATIONS ON FORM OF CORRELATION FOR $k_l a$ DATA

It follows from the previous section that many of the published data on $k_l a$ should be approached critically, but in spite of that it is possible, on the basis of the earlier
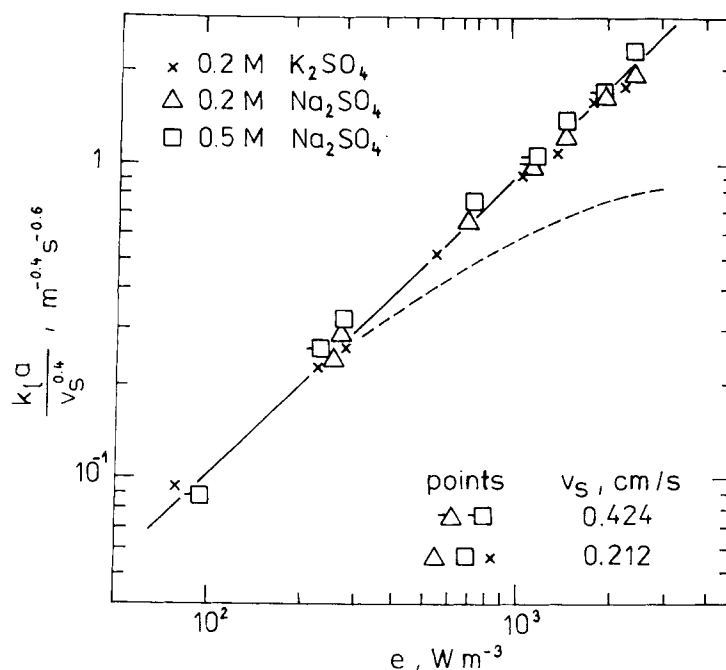
Figure 1. Distortion of k_1a dependence on e caused by unsuitable method of k_1a measurement.

works, to gain the idea of the adequate form of the empirical equation for the correlation of k_1a data.

First, it is necessary to decide whether to use separate relations for the specific interfacial area and for the mass transfer coefficient or to use only one relation for the product k_1a. When the quantities a and k_1 are treated separately, the usual procedure is to measure k_1a and by an optical or chemical method to measure a. Hassan and Robinson[5] show that the geometric surface found by the optical method can significantly differ from the surface taking part in the mass transfer. As far as the chemical methods are concerned, Figure 2 on page 1286 of the work[5] cited shows that the results of determination of a from the CO_2 absorption in the aqueous inorganic electrolyte solutions with the addition of KOH may differ up to 70 times, depending on assumptions made to determine the mean value of the driving force for absorption. These facts lead to conclusion that, so far, it is necessary to use k_1a.

It would be advantageous to have the relation for k_1a in the dimensionless form. The results of Figueiredo and Calderbank[6] suggest, however, that the dimensionless parameters should contain a characteristic length of the system. To evaluate such a relation, it is necessary to have data measured on reactors of different volumes. A sufficiently large set of consistent data of this kind is not yet available, and therefore we shall limit ourselves to dimensional equations for k_1a.

During the study of the behavior of the gas holdup it was convenient[7] to use those independent variables that are suitable for the description of the dependence of the holdup on the intensity of stirring and aeration and to add further independent variables that characterize the dependence of the holdup on the physical properties of the liquid. This

approach will be used here too. It is then possible to expect, that the influence of the intensity of stirring and aeration on k_1a for the given liquid and geometry of the system will be sufficiently characterized by the values of the quantities e and v_s.[6,8–15] Zlokarnik[16,17] suggests the use of $\dot{V}_g/V_1$ instead of v_s, but Judat[18] shows that it is not advantageous. As the dependence of k_1a on the physical properties of the batch is concerned, when pure liquids were aerated, it follows from the work of Moo-Young and Blanch[11] that if the bubbles are taken as rigid, the correlation should contain the parameter

$$S_1 = (\rho_1^{0.533} D^{2/3})/(\sigma^{0.6}\mu_1^{1/3}) \tag{2}$$

and for bubbles with the unhindered surface the parameter should be

$$S_2 = (\rho_1^{0.367} D^{0.5})/(\sigma^{0.6}\mu_1^{1/6}) \tag{3}$$

Linek et al.[4] show that for aqueous glycerine solutions the parameter S_2 is more adequate.

Marrucci and Nicodemo[19] found that the change of ρ_1, σ, and μ_1 of 8% at most for electrolyte solutions changes the average diameter of the bubbles in the dispersion up to 8 times. For other solutes the situation is analogous.[20] However, in case of pure liquids containing no solute, little influence of the kind of liquid on the bubble size was found, even for large changes of physical properties.[20] One should not expect, therefore, that any expressions containing only quantities ρ_1, μ_1, σ, and eventually D would describe the dependence of k_1a on the concentration and kind of solute properly. Koetsler and Thoenes[21] measured the coalescence frequency of gas bubbles and the dependence on the concentration of aqueous NaCl solutions and found that the salt concentration does not influence the efficiency

of the initial dispergation, but it significantly lowers the frequency of bubble coalescence in batch. As long as the initial dispersion is sufficiently fine, the small dimensions of the bubbles remain preserved at higher concentrations of the salt, which prevent bubble coalescence. At the same gas holdup, the values of interfacial area will be much higher for noncoalescent than for coalescent batches. A correlation for $k_l a$ should therefore contain a quantity that characterizes the ability of the liquid to slow down the bubble coalescence, which we shall call the coalescence parameter:

$$k_l a = f(e, v_s, S_2, S) \tag{4}$$

where S is the coalescence parameter. Judat[16] and Zlokarnik[17] came to the same conclusion.

COALESCENCE PARAMETER

For the following considerations it is not necessary to limit oneself to the data on mechanically stirred equipment for gas–liquid contact. Keitel and Onken[22] compared the influence of the properties of liquid on the character of bubble dispersion in a bubble column with that in a stirred reactor and found that it was almost identical.

Since 1960 several papers were published in which the coalescence parameter was searched either empirically or on a theoretical basis. Lessard and Zieminski[23] tried to use for the coalescence parameter such quantities as the viscosity of the experimental solution relative to the viscosity of water, the entropy of a solution, the self-diffusion parameter of water, and the ionic strength of the solution. Their results show that none of these quantities was successful. The dependence of the ability of the liquid to slow down the coalescence on ionic strength was studied by several authors.[10,12,22,23] Keitel and Onken[22] show in Figure 1 of their work the dependence of the bubble Sauter diameter on the ionic strength for solutions of NaOH, Na_2SO_4, and $Al_2(SO_4)_3$. For each of these electrolytes the experimental points lie on a separate curve. This confirms the opinion that the ionic strength is not sufficiently suitable as a coalescence parameter even for aqueous electrolyte solutions. The main disadvantage of ionic strength as a coalescence parameter is, of course, the impossibility to apply it to solutions of nonelectrolytes.

The authors, who tried to deduce the coalescence parameter from theoretical analysis, started from the following considerations. For two bubbles to coalesce, it is necessary to push one to the other, which is done by some external forces. The thin liquid film separating the bubbles must burst while the two bubbles are in contact. As long as we compare systems with external forces of about the same character (e.g., mechanically stirred aerated reactors), the intensity of coalescence is determined by the resistance of the film against rupture. Authors who studied experimentally the influence of solute concentration on the bubble dispersion behavior found that increasing the salt concentration decreased the coalescence until a limiting concentration (generally different for different solutes) was

reached, above which coalescence remained practically independent of solute concentration.[5,7,10,11,19,20,22,23,25–29] This supports the view that the stability of the film could be connected with the adsorption of the solute on the interface. The form of the expression for the coalescence parameter in the works published so far was based on the Gibbs formula for the adsorption of the solute on the solution surface. Marrucci and Nicodemo[19] initially assumed that the ability of the solute to slow down coalescence was directly proportional to its Gibbs relative adsorption with respect to the solvent. This is the basis of the following coalescence parameter

$$Y = \frac{1}{2} nc \frac{d\sigma}{dc} F \tag{5}$$

where

$$F = \left(1 + \frac{d \ln \sigma}{d \ln c}\right)^{-1} \tag{6}$$

is a correction to the nonideality of the solution. The use of the parameter Y was unsuccessful.[7,25] Andrew[20] starts from the consideration that during the fast and steady expansion of the liquid film between the bubbles (caused by external forces) arises the difference of the solute concentrations in the bulk of the liquid and in the liquid in the very short distance (a few molecules) from the interface, called the subsurface region. This difference is caused by the solute adsorption on the increasing interfacial area. He assumes instantaneous equilibrium between surface and subsurface concentrations so that the fast change in interfacial area induces the concentration change in the subsurface region only, while the solute diffusion and convection from the bulk of the liquid is not fast enough to balance the concentration difference so formed. The liquid film is then stabilized by the surface tension gradient resulting from the surface tension dependence on the solute concentration. Andrew[20] suggests two mechanisms leading to film rupture. Either the external forces are large enough to break the film, even if it is stabilized, or the contact time of the bubbles is long enough, so that the concentration difference and hence the stabilizing effect of the surface tension gradient cancels because of solute diffusion into the subsurface layer. For both cases he derives (under the assumption of ideal solution) the same coalescence parameter:

$$X_1 = g(x) \left(\frac{d\sigma}{dx}\right)^2 \tag{7}$$

where $g(x)$ depends on concentration only. Lee and Meyrick[25] neglected the diffusion contribution in the mechanism proposed by Andrew and assumed purely convective solute transfer from the bulk to the subsurface layer of the liquid film. They took into account solution nonideality. They proposed the coalescence parameter

$$X_2 = c \left(\frac{d\sigma}{dc}\right)^2 F \tag{8}$$

assuming that the quantities $c(d^2\sigma/dc^2)$ and $c(d\sigma/dc) \cdot (dF/dc)$ were negligible compared with $(d\sigma/dc)F$. Table I

Table I. Verification of validity of assumptions of Lee and Meyrick.[a]

Electrolyte	c (kmol/m^3)	$10^3 \cdot F \dfrac{d\sigma}{dc}$ (kg m^3/s^2 kmol)	$10^3 \cdot Fc \dfrac{d^2\sigma}{dc^2}$ (kg m^3/s^2 kmol)	$10^3 \cdot c \dfrac{d\sigma}{dc} \dfrac{dF}{dc}$ (kg m^3/s^2 kmol)
Na$_2$SO$_4$	0.05	3.47	1.01	0.24
Na$_2$SO$_4$	1.00	4.76	0.35	−1.65
KI	0.25	0.94	0.02	−0.007
KI	2.00	0.88	0.19	−0.28
NaCl	0.175	1.79	0.01	−0.02
NaCl	1.00	2.67	0.12	−0.32

[a] Reference 25.

contains results of calculation showing that the assumption was not fulfilled and the mechanism was too simplified. Yang and Maa[28] derive their coalescence parameter from the assumption that the stability of the liquid film is proportional directly to the value of the Gibbs elasticity of the film, which they express together with Rosen[30] by means of the corrected form of Sheludko's equation. Under the assumption that the surface concentration of the solute approaches the saturation concentration, the equation can be written in a simplified form:

$$E = \frac{4RT}{h_b c} \Gamma^2 \qquad (9)$$

For a suitable choice of the Gibbs dividing surface and ideal solution ($F = 1$) we have

$$\Gamma = -\frac{c}{RT} \frac{d\sigma}{dc} \qquad (10)$$

and finally we get the coalescence parameter X_2 (it is implicitly assumed that h_b is independent of concentration). In the derivation of the Sheludko equation,[30] it is assumed that the dependence of the equilibrium surface concentration on the subsurface concentration has the form of the Langmuir isotherm. Marrucci[27] adds to the factors suggested by Andrew as determining the film stability yet the Hamaker–London forces, Saggert and Quinn[31] add further the electrostatic forces. They compare the theoretical calculations with experimental data and come to the conclusion that all the factors mentioned may be important, and depending on the conditions of the experiment, any of them may prevail.

It can be concluded from the preceding considerations that the general form of the coalescence parameter can be written as

$$X = g(z) \left(\frac{d\sigma}{dz}\right)^2 F(z) \qquad (11)$$

where z is the solute concentration and g some function of it. By measuring different quantities dependent on coalescence intensity (e.g., height of froth,[20,28] diameter of bubbles,[25,32] coalescence time[28]), it was experimentally confirmed that X was a suitable coalescence parameter independent of the form of function $g(z)$. It follows from the review of the relations given by Adamson[33] (pp. 78–87) that the form of

the function $g(z)$ depends only on the choice of the Gibbs dividing surface for the calculation of the relative adsorption Γ and on the kind of concentrations used. There are no physical reasons for any specific choice of the Gibbs dividing surface, and so we may choose one that yields the simplest form of $g(z)$. Therefore, we shall use the coalescence parameter X_2 defined by equation (8).

EXPERIMENTAL

The detailed description of the apparatus and of the experimental method was already published.[3] The experimental equipment consisted of a cylindrical vessel from organic glass, its diameter was 0.29 m, it was provided with four baffles, and the height of the liquid was equal to the vessel diameter. The open six straight-blade turbine impeller had a diameter equal to one-third of the vessel diameter and was placed 0.1 m above the vessel bottom. Gas was introduced under the impeller. The power input to the impeller was measured tensometrically and the gas holdup by the method of contact tip. Both methods are described in detail elsewhere.[7] (In Figure 1 of ref. 7 the value 200 mm for the vessel diameter should be corrected to 290 mm). The experimental conditions are summarized in Table II. The volumetric mass transfer coefficient was measured using a dynamic method consisting of the sudden introduction of pure oxygen in the liquid, previously degassed in vacuum. The oxygen concentration was measured by two oxygen probes covered by a 12-μm polypropylene membrane and located on the bottom of the reactor. The method of $k_l a$ evaluation is described by Linek et al.[4]

RESULTS AND DISCUSSION

The experimental values of $k_l a$ for each electrolyte solution were treated according to equation (1). As there were only two levels of v_s values, the exponent n_2 was not evaluated. Published results[5,9,10,13,15] can be taken as sufficient proof that the value of n_2 for the given geometry should be 0.4 and that it is independent of the physical properties of the batch. Figure 2 shows an example of such a relation for aqueous KNO$_3$ solutions. It follows from the figure that the dependence of $k_l a$ on e and v_s is represented well by equation (1) and that the exponent n_1 is for one solute a

Table II. Experimental conditions.[a]

Solute	Concentrations (kmol/m³)			
NaCl	0.175	0.325	0.500	1.000
KCl	0.100	0.250	0.500	1.000
KNO₃	0.150	0.300	0.600	1.000
KI	0.250	—	—	—
NaSCN	0.200	0.500	—	3.000
MgCl₂	—	0.350	0.500	1.000
Na₂SO₄	0.050	0.200	0.500	1.000
K₂SO₄	0.050	0.100	0.200	0.500
MgSO₄	—	0.200	0.500	1.000
Al₂(SO₄)₃	0.050	0.100	0.200	—
H₂O	—	—	—	—

[a] Experimental conditions: v_s (m/s): 0.00212, 0.00424; stirrer speed (s⁻¹): 4.167, 5.833, 7.500, 9.167, 10.833; temperature, 20°C for all experiments.

monotonous function of solute concentration. This was observed before by Hassan and Robinson[10] and by Robinson and Wilke,[12] who correlated the dependence of n_1 on ionic strength. For the reasons given in the preceding, we prefer the coalescence parameter X_2. It is difficult to decide on the basis of the earlier data of the authors, who used the parameter X_2,[20,25,28,32] whether a correction for the nonideal behavior of the solution was necessary. Therefore, the quantity n_1 was correlated as a function of X_2 both for $F = 1$ (ideal solution) and for F given by

$$F = \frac{1}{n}\left(1 + \frac{d \ln \gamma_\pm}{d \ln c}\right)^{-1}$$ (12)

which differs from equation (6) only in the use of the mean activity coefficient. The values of the activity coefficients were taken from Robinson and Stokes[34] and the remaining physical data from Horvath.[24] The values of n_1 are plotted

against X_2 with $F = 1$ in Figure 3 and against X_2 with F from equation (12) in Figure 4. The dependence of n_1 on X_2 was found empirically in the form

$$n_1 = \frac{aX_2}{1 + bX_2} + q$$ (13)

The values of the constants a, b, q found by nonlinear regression for $F = 1$ yield

$$n_1 = \frac{0.85(X_2 \cdot 10^6)}{1 + 1.75(X_2 \cdot 10^6)} + 0.56$$ (14)

The mean quadratic deviation of n_1 was 0.05. For F calculated from (12) the relation is

$$n_1 = \frac{1.50(X_2 \cdot 10^6)}{1 + 3.05(X_2 \cdot 10^6)} + 0.56$$ (15)

with a mean quadratic deviation for n_1 of 0.042. Therefore, the nonideality correction is significant. Values of X_2 in (14) and (15) are in kg² m³/s⁴ kmol.

The values of n_1 calculated from (14) and (15) are compared with those computed from experimental data in Figures 3 and 4. We did not include the parameter S_2 in our correlation because its change corresponded roughly to the dispersion of the data from which it was calculated. However, for aqueous glycerine solutions in a large range of concentrations with significant changes of D and μ_1, the influence of the parameter S_2 is not generally negligible.[4] If we express the dependence of n_1 on the ionic strength, as suggested by Hassan and Robinson[10] and Robinson and Wilke,[12] we get
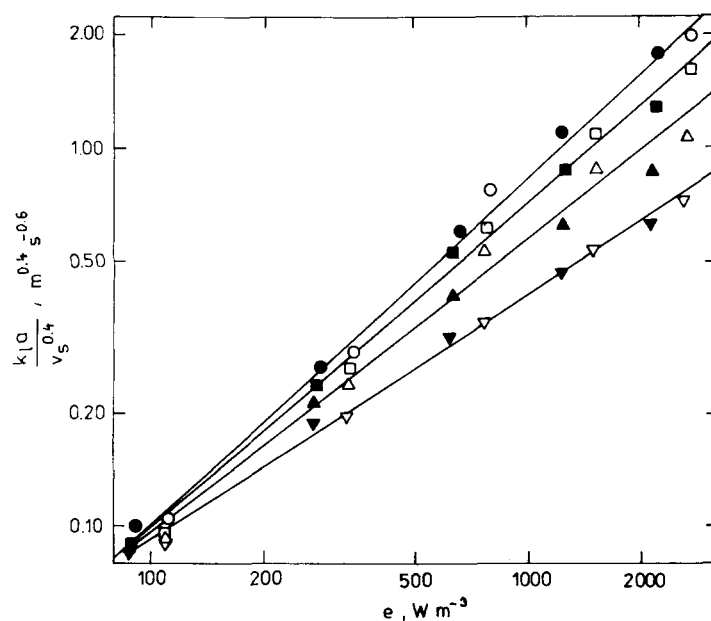
$$n_1 = \frac{1.35I}{1 + 2.7I} + 0.554$$ (16)

Figure 2. Dependence of n_1 on KNO₃ concentration: ▽▼, 0.15M; △▲, 0.30M; □■, 0.60M; ○●, 1.0M. Empty symbols: $v_s = 0.212$ cm/s. Full symbols: $v_s = 0.424$ cm/s.
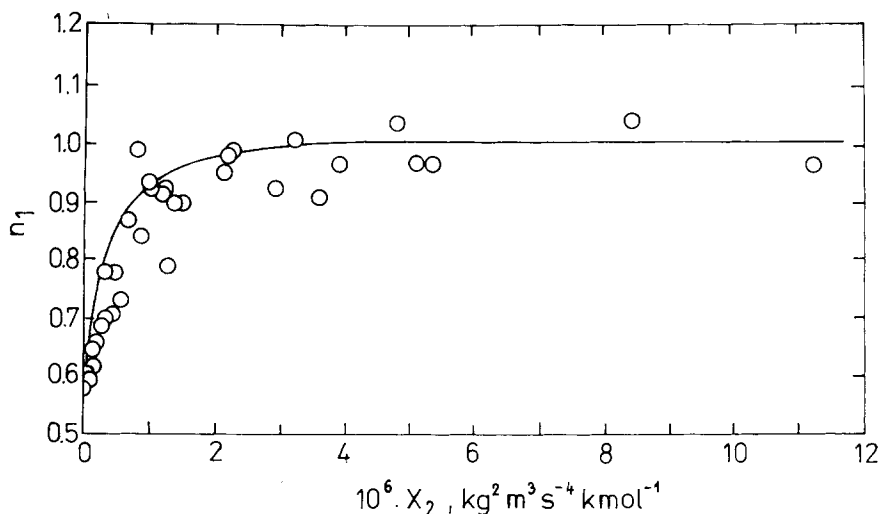
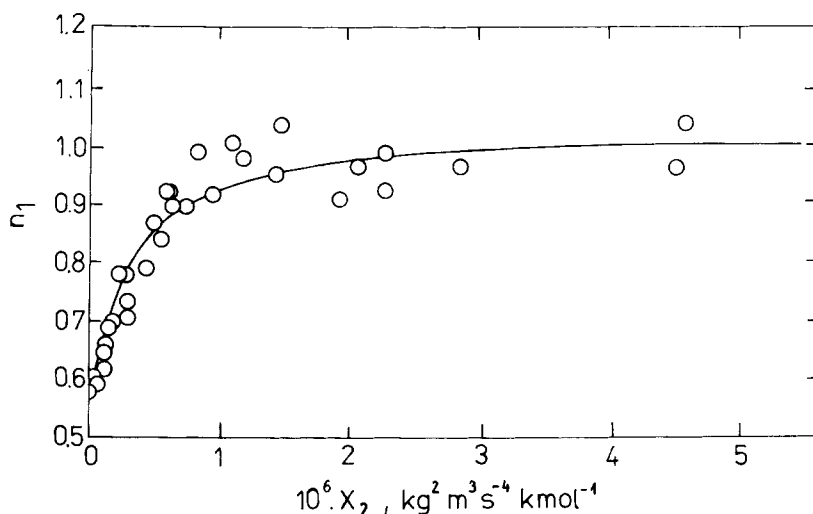**Figure 3.** Dependence of n_1 on X_2 with $F = 1$; curve corresponds to (14).

Figure 4. Dependence of n_1 on X_2 with F calculated from eq. (12); curve corresponds to (15).

In Figure 5 the values of n_1 calculated from (16) are compared with those from experimental data. The mean quadratic deviation in this case is 0.077. It is significantly higher than that for the equations (14) and (15).

Further, it was found that the parameter C in equation (1) depends on n_1. Figure 6 shows that this dependence can be approximated by

$$\log C = -1.81 n_1 - 1.19 \qquad (17)$$

After rearrangement, we finally have the equation for $k_1 a$:

$$k_1 a = \exp(-4.18 n_1 - 4.36) e^{n_1} v_s^{0.4} \qquad (18)$$

where n_1 is given by (15). Equation (18) fits the whole set of experimental data with the mean relative error of 15% and a maximum error of 53%. The measured and calculated values of $k_1 a$ are compared in Figure 7. The advantage of relation (18) is its ability to describe the behavior of coalescent (H_2O, aqueous solutions of NaSCN and KI), noncoalescent [aqueous solutions of K_2SO_4, Na_2SO_4, $MgSO_4$,

$MgCl_2$, $Al_2(SO_4)_3$], and transient (aqueous solutions of NaCl, KCl, and KNO_3) batches at the same time. It would be desirable to include the influence of μ_1, ρ_1, σ, and D in the correlation for the estimation of $k_1 a$, e.g., in terms of parameter S_2. That would enable us to use it for highly viscous media. Unfortunately, the $k_1 a$ data for such batches that can be found in the literature have been measured by erroneous variants of the dynamic methods. The values of $k_1 a$ based on correct and incorrect variants of the dynamic method[4] are compared in Figure 8. They apply to 1% mass solution of CMC in $0.5M$ aqueous solution of Na_2SO_4 (a noncoalescent batch with high viscosity). Figure 8 shows that for the incorrect dynamic method the distortion of the dependence of $k_1 a$ on e is even greater than for noncoalescent electrolyte solutions (see Fig. 1). A correlation that would be valid for a large extent of μ_1, ρ_1, σ, and D must be based on new experimental data on $k_1 a$ in the highly viscous media.
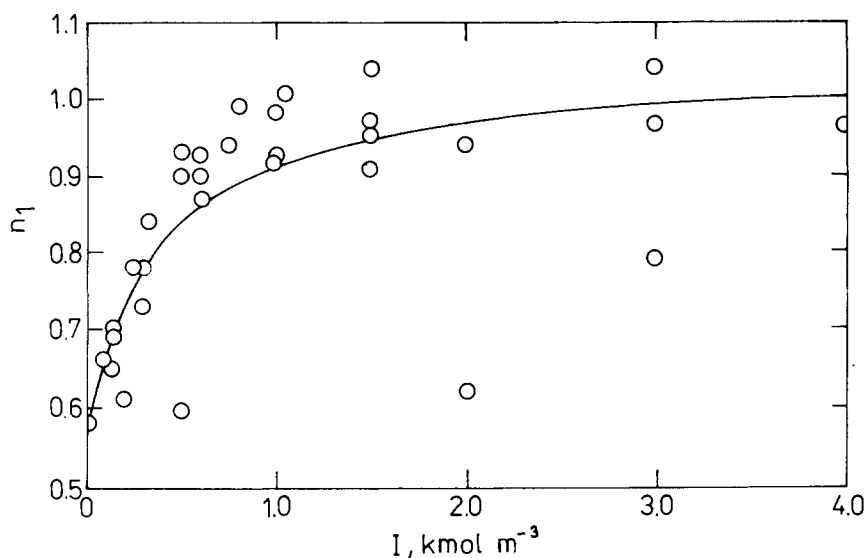
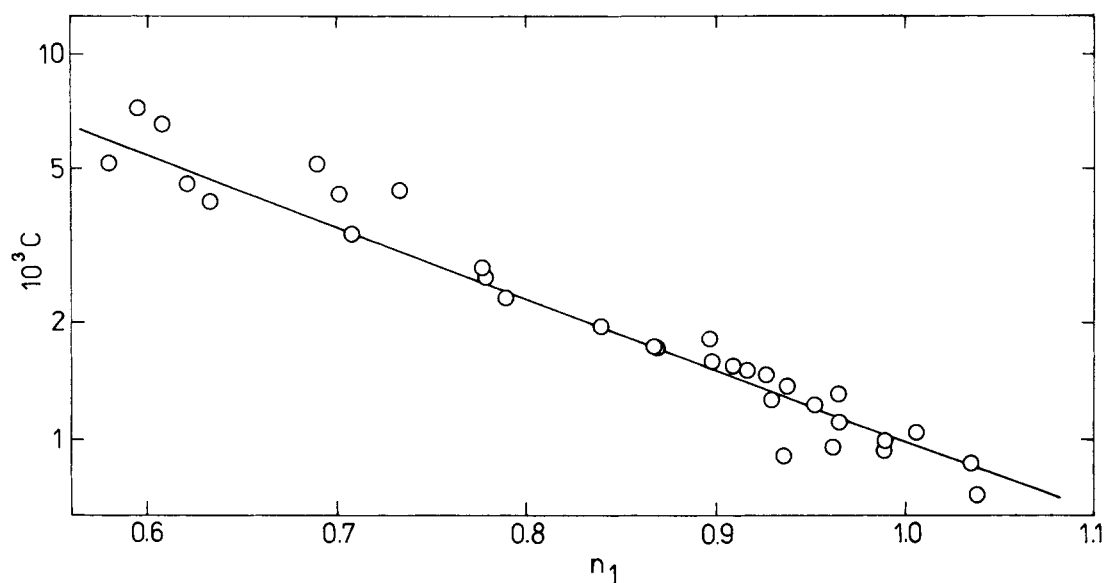**Figure 5.** Dependence of n_1 on I; curve corresponds to (16).

Figure 6. Dependence of C on n_1; curve corresponds to (17).

NOMENCLATURE

a	interfacial area per unit volume of liquid phase
c	molarity
C	constant in eq. (1)
D	diffusivity of O_2 in liquid phase
e	power input dissipated per unit liquid volume
E	Gibbs elasticity
F	correction for nonideality of solution defined by eq. (6)
$g(z)$	concentration function in eq. (14)
h_b	layer thickness of liquid bulk adjacent to interface
I	ionic strength
k_1	mass transfer coefficient
k_1a	volumetric mass transfer coefficient
n	number of ions to which molecule of electrolyte splits in aqueous solution
n_1	exponent of e in eqs. (1) and (18)
n_2	exponent of v_s in eqs. (1) and (18)
R	universal gas constant

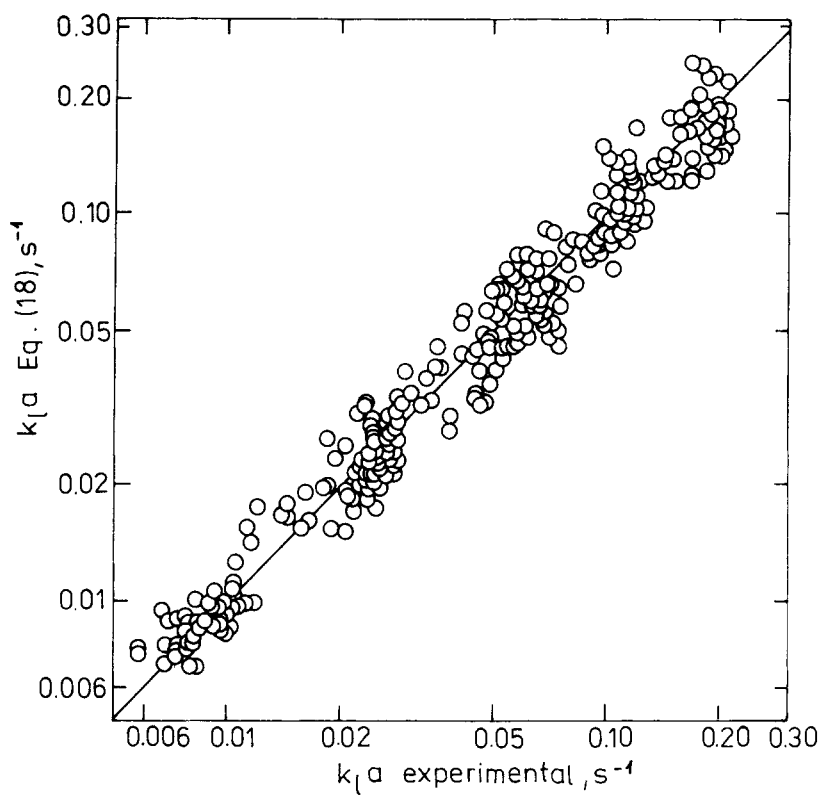|	
S	general coalescence parameter in eq. (4)
S_1	quantity defined by eq. (2)
S_2	quantity defined by eq. (3)
T	absolute temperature
v_s	superficial gas velocity
V	volume of liquid charge
$\dot{V}_g$	volumetric gas flow rate
x	mole fraction
X	coalescence parameter in eq. (14)
X_1	coalescence parameter defined by eq. (7)
X_2	coalescence parameter defined by eq. (8)
Y	coalescence parameter defined by eq. (5)
z	concentration
γ	activity coefficient
$\gamma_\pm$	mean activity coefficient
Γ	relative Gibbs adsorption
μ	dynamic viscosity
ρ	density
σ	surface tension

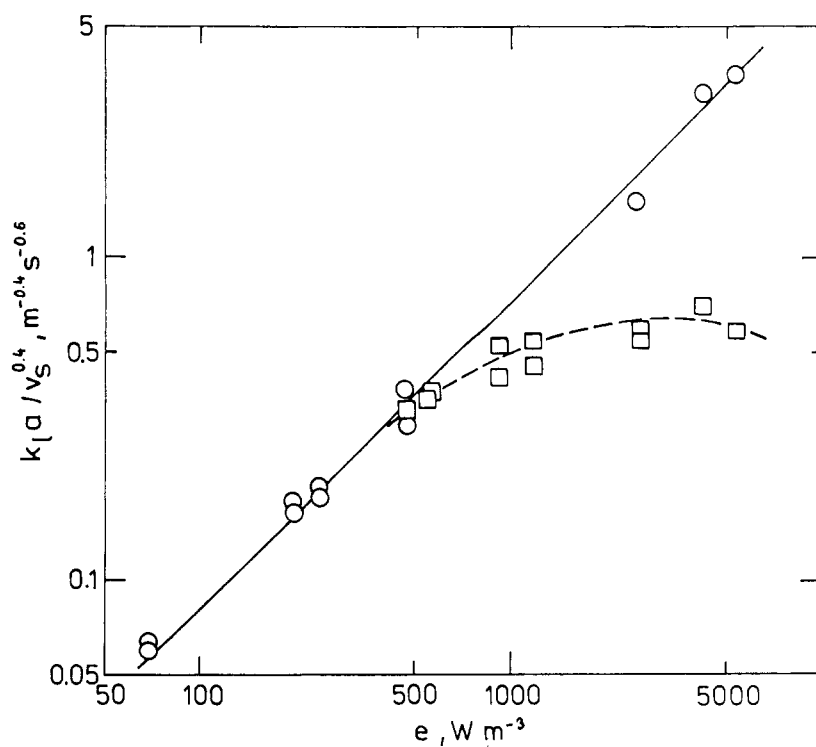**Figure 7.** Comparison of $k_l a$ calculated from eq. (18) with experimentally determined values.

Figure 8. Comparison of $k_l a$ values obtained by correct (○) and incorrect (□) variant of dynamic method; 1% (wt) CMC in $0.5M$ aqueous Na_2SO_4.

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