

Interfacial Area Generated in Aqueous Electrolyte Solutions on a Sieve Plate

A light depolarization method was used to measure the interfacial area of froth on a sieve plate. The effects of hydrodynamics and the presence of electrolytes on the magnitude of the interfacial area were studied. The results obtained for pure water and for two different aqueous solutions of inorganic salts were compared. In the case of aqueous electrolyte solutions the size distributions of ionic bubbles and the fraction of interfacial area contributed by these bubbles were determined by a photographic method. It was found that the interfacial area generated in aqueous electrolyte solutions was considerably (about 30%) greater than the interfacial area generated in pure water, and that about 45% of the total interfacial area in the former case was contributed by ionic bubbles which formed a separate population.

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SCOPE

Interfacial areas were measured in froths generated on a sieve plate. Measurements were carried out using a light depolarization method by which it is possible to determine local values of interfacial area along the axis of the column. The method requires previous calibration with dispersions of known interfacial area and given optical properties. Measurements of interfacial area were carried out for pure water and for water solutions of three salts: NaOH, Na₂CO₃, and Na₂SO₃. The hydrodynamic conditions varied in the ranges: $h_L = 5$ –13 cm, $U_K = 0.5$ –1.6 m/s.

The presence of an electrolyte in water causes generation of a great number of small bubbles referred to as "ionic" bubbles. The size distributions of these ionic bubbles were determined for the three salt solutions. These measurements were carried out using a photographic technique. On the basis of the measurements it was possible to estimate the contribution of the ionic bubbles to the total interfacial area on the sieve plate.

CONCLUSIONS AND SIGNIFICANCE

Gas-liquid bubbling systems are used in many mass transfer processes. In order to design appropriate equipment it is necessary to know the factors which, among others, determine the conditions of mass transfer: mass transfer coefficients, interfacial area, and hold-ups of both phases. In the case of the physical mass transfer process it is sufficient to know the values of the volumetric mass transfer coefficients (defined as the products of interfacial area and respective mass transfer coefficients). If, however, a reaction is taking place in the liquid phase, it is usually necessary to know separately the values of the interfacial area and the liquid-side mass transfer coefficient.

In this work, interfacial areas were measured in froths generated on a sieve plate. From the measured local values of interfacial area along the column axis, the mean values of interfacial area in froths were calculated as a function of the plate operating parameters. Interfacial area determined by the optical method used can be considered as the total geometrical interfacial area generated on the sieve plate.

Comparison of the results obtained for pure water with those

obtained in the presence of electrolytes has shown that in the latter case the interfacial area is about 30% greater. Furthermore, the effect of gas velocity on the magnitude of interfacial area is in both cases different. The structure of electrolyte froth is different from that made of pure water. Direct comparisons of the values of interfacial area obtained by means of physical methods for pure water with those measured by chemical methods using aqueous electrolyte solutions, often attempted in earlier works, are therefore misleading.

The bubble size distributions observed for aqueous electrolyte solutions show that ionic bubbles may be considered as a separate bubble population. These bubbles, their diameters not exceeding 0.6 mm, contribute nearly half of the total interfacial area in the froth.

It is seen that determination of the contribution of these bubbles to the mass transfer is very important. With the decrease of bubble diameter the ratio of bubble surface area to bubble volume increases. In the case of fast absorption processes the ionic bubbles may become very quickly exhausted of the gas component transferred to the liquid phase, whereupon they cease participating in mass transfer. A similar situation may occur in the case of evaporation of water from an electrolyte solution to an inert gas (this process has often been used to

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measure mass transfer coefficients in the gas phase). The rate of the evaporation process is controlled by gas-phase resistance only, and the practical saturation of ionic bubbles may be reached within a considerably shorter time than the residence time of the bubbles in the froth. In such cases the interfacial

area active for mass transfer may be much smaller than the total interfacial area.

The results described here are therefore of importance in absorption, fermentation, gas-liquid reactions, and other gas-liquid mass transfer processes.

INTRODUCTION

When a gas is bubbled through a layer of an aqueous electrolyte solution, a great number of small bubbles is observed in the froth. These so-called ionic bubbles are not observed if pure water is used as the liquid phase (Braulick et al., 1965; Vinter et al., 1972). Ionic bubbles usually have diameters less than 1 mm.

The small sizes of the bubbles (considerably less than the orifice diameter) show that ionic bubbles are not primary ones, but are formed by the breaking of larger bubbles during their passage through the dispersion layer.

The structure of froths on sieve plates has been studied mainly for an air-water system. Ashley and Haselden (1972) took photographs of the dispersion vertically from above the plate. They concluded that two distinct bubble populations existed in the froth on a sieve plate. The first population, referred to as "vapor voids," consisted of large bubbles transporting the larger fraction of the gas through the froth and breaking up at the surface of the froth. The diameters of these bubbles were found to lie between 4 and 8 cm. Porter et al. (1967), on the other hand, measured the frequency of bubble formation at plate orifices. They determined the diameters of the bubbles within the limits 1.5-3 cm. Comparison of the results of both works shows that the sizes of the bubbles of the first population increase as the bubbles pass through the froth layer. The second population consists of bubbles having diameters of 4 to 8 mm, which may circulate in the froth with the liquid phase. The bubbles of this population form a thin curtain near the wall of the column and for this reason photographs taken through the transparent wall may not be representative of the mean bubble size in the bulk of the froth. As the diameters of bubbles generated at the plate orifices are between 1.5 to 3 cm, the bubbles belonging to the second population must be generated by breaking of the bubbles belonging to the first population. It is also evident that bubbles of the first type both coalesce and break up in the froth.

The presence of the great number of very small bubbles in aqueous electrolyte solutions can be explained in the light of the work of Lessard and Ziemiński (1971), and Ziemiński and Whitemore (1971). The authors investigated the effect of salt concentration on bubble coalescence in aqueous solutions and found that the addition even of a small amount of an inorganic salt considerably decreases the probability of bubble coalescence. The disappearance of the bubbles of the second population is probably due mainly to the coalescence of these bubbles with the great bubbles of the first population (the contact of small bubbles with the surface of the froth is less probable). The differences between the second bubble population in pure water and the second bubble population in electrolyte solutions are thus caused by the hindering effect of the ions on the coalescence.

The main purpose of this work was to find out what part of the total interfacial area in froth is contributed by ionic bubbles. It was therefore necessary to determine the total interfacial area in the froth as well as the number and size distribution of ionic bubbles.

DETERMINATION OF TOTAL INTERFACIAL AREA

The total interfacial area of the froth was determined by means of the light depolarization method proposed originally by Rodionov and Ulyanov (1967). A beam of initially linearly polarized light is passed through a dispersion layer, and the intensities of the light components polarized in the planes parallel and perpendicular to the initial polarization plane are measured behind the layer. The value of the interfacial area is correlated to the ratio of the intensities of both components: $D = I_{\perp}/I_{\parallel}$.

Determination of the values of the interfacial area in froths from the measured values of D required prior calibration, i.e., establishing the relationship of D to the interfacial area and other optical properties of the system. This was done experimentally, as light transmission through the dispersions of high concentration has not been described theoretically yet.

Experimental calibration required using dispersion models of known interfacial areas and the same optical properties as the bubble layer. However, the original calibration method used by Rodionov and Ulyanov (1967) did not conform to the latter condition. In the bubble dispersions the refractive index of the dispersed phase is lower than the refractive index of the continuous phase. The same optical situation may be produced by using a layer of glass spheres submerged in a liquid having a refractive index higher than the refractive index of the glass, or a layer of hollow glass spheres submerged in a liquid of the same refractive index as that of the glass. In this work both methods were applied; thus it was possible to determine the dependences of D on the interfacial area and the ratio of the refractive indices N_c/N_D . Rodionov and Ulyanov (1967) used layers of glass spheres submerged in liquids having refractive indices lower than that of the glass, i.e. systems optically reversed with respect to the froth.

The scheme of the optical arrangement used in this work is shown in Figure 1. A beam of light from a light source 1 passes through a condenser 2, a color filter 3, and is polarized linearly by a polarizing filter 4. The diameter of the light beam, p , is limited by screens 5 and 7. Upon passing through the dispersion layer, 6, the beam is analyzed using two polarizing filters, 8 and 9, and a photovoltaic element, 10. This is done as follows. First polarizing filters 8 and 9 are set so that their polarization planes are perpendicular to that of filter 4, and the light intensity is then measured. Afterwards, filter 8 is set so that its polarization plane is parallel to that of filter 4, and filter 9 is turned until the same light

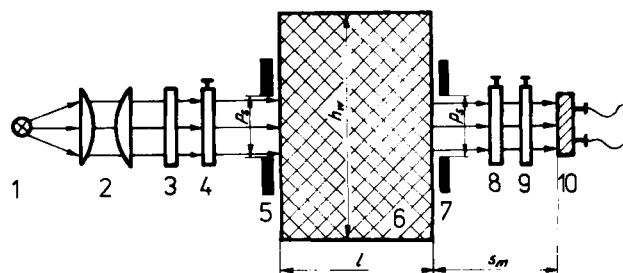


Figure 1. The experimental optical arrangement.

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|--------------------------|--------------------------|
| 1. Light source | 2. Condenser |
| 3. Color filter | 4. Polarizing filter |
| 5. Optical screen | 6. Dispersion layer |
| 7. Optical screen | 8, 9. Polarising filters |
| 10. Photovoltaic element | |

intensity is obtained as in the former arrangement. The value of D is then calculated from the expression.

$$D = \frac{(H_0 - H_{90}) \cos^2 \theta + H_{90}}{H_0} \quad (1)$$

where θ is the angle between the polarization planes of filters 8 and 9 in the second arrangement, H_0 is the optical transmittance of two filters set parallel, and H_{90} is the transmittance of two crossed filters. This measuring technique does not require calibration of the photovoltaic element. The value of D depends on the interfacial area of the dispersion, on the ratio of the refractive indices of both phases, and also on the characteristic dimensions of the optical system used. For the dispersed phase formed of spherical particles, and the experimental arrangement used in this work, the following general relationship can be written

$$D = \frac{I_{\perp}}{I_{\parallel}} = f \left(a_f l, N_D, N_C, \alpha_D l, \alpha_C l, \frac{p_s}{l}, \frac{s_m}{l}, \frac{h_w}{l}, \frac{b_w}{l} \right) \quad (2)$$

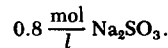
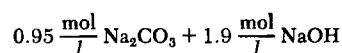
The effect of light absorption in both phases on the value of D is significant only for large values of the absorption coefficients α_D , α_C . Test measurements have proved that this effect was negligible in all the experiments carried out in this work. In this case and for the given geometry of the optical arrangement, the ratio I_{\perp}/I_{\parallel} depends only on the product $a_f l$ and the ratio N_C/N_D (this can also be shown using the Fresnel equations).

The following geometrical relations were fixed in the optical arrangement used: $p_s/l = 0.5$; $s_m/l = 1.1$. All measurements were carried out for $h_w/l > 1.2$ and $b_w/l > 1.2$, as in this situation no influence of h_w and b_w on the value of D was observed. Finally, it was only necessary to determine the relationship of the value of D and the product $a_f l$ for different values of the ratio N_C/N_D . The results of the calibration are shown in Figure 2. The relationship for $N_C/N_D = 1.474$ was obtained using hollow glass spheres submerged in a liquid of the same refractive index as that of the glass. Five other groups of measurements were carried out for systems consisting of glass spheres and of a liquid with a refractive index greater than that of the glass. The ratio N_C/N_D in the experiments ranged from 1.12 to 1.32. The results for greater values of N_C/N_D were obtained using so called West's liquids (mixtures of diiodomethane, sulfur, and white phosphorus).

The ratio I_{\perp}/I_{\parallel} was also measured for solid glass spheres in air. This system may be considered as optically reversed with respect to the system of hollow glass spheres submerged in a liquid of the same refractive index as the refractive index of glass. The relationship obtained for this system is shown in Figure 2 by the dotted line ($n = 0.677$). These measurements show that the procedure used by Rodionov and Ulyanov (1967) leads to the values of interfacial area about 80% greater than the real ones.

The measurements of interfacial area on the sieve plate were based on the relationships shown in Figure 2. The measurements were carried

out for the following two water solutions of inorganic salts:



The salts employed are those commonly used in the chemical method of interfacial area measurements.

The experiments were carried out with no liquid flow on the plate. A rectangular cross-section column 80×130 mm with a single sieve plate was used. The diameter of the holes in the plate was 2.4 mm. There were 89 holes, drilled 9.6 mm apart on vertices of equilateral triangles. The superficial velocity of air in the column ranged from 0.5 to 1.6 m/s.

MEASUREMENTS OF THE INTERFACIAL AREA FORMED BY IONIC BUBBLES

The measurement method is based on the fact that the rising velocity of the ionic bubbles is not great, so that when the gas flow is stopped suddenly, the ionic bubbles remain in the liquid layer for several seconds. A flap valve installed below the plate in the column allowed rapid stopping of the gas flow and prevented liquid leakage.

When the gas flow is stopped, the froth layer disappears rapidly. The bubbles of diameters greater than about 1 mm were not observed in the liquid one second after stopping the gas flow. Gas hold-up in the layer decreased within the first second to 1-3%. Determination of the size distribution of ionic bubbles was based on photographs taken through the transparent wall of the column. To fix the liquid volume photographed, a small black plate serving as the background was mounted vertically in the column 7.5 cm above the sieve plate. It was thus possible to determine the number of ionic bubbles per unit volume of the liquid and calculate the interfacial area formed by the bubbles. The photographs were taken at different time intervals after stopping the gas flow. The first photographs were taken one second and the last photographs ten seconds after stopping the flow. Figures 3 and 4 are examples of the photographic results. Only bubbles having diameters less than 0.6 mm were taken into consideration in the calculations. Because of the small sizes of the ionic bubbles, it could be assumed that at the moment the gas flow was stopped the concentration of the ionic bubbles was the same at every point in the liquid.

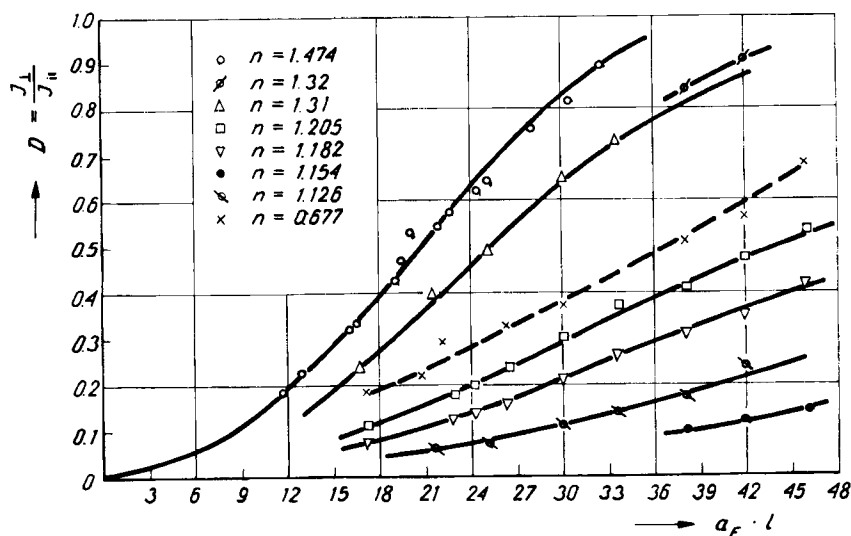


Figure 2. Calibration curves for the light depolarization method.

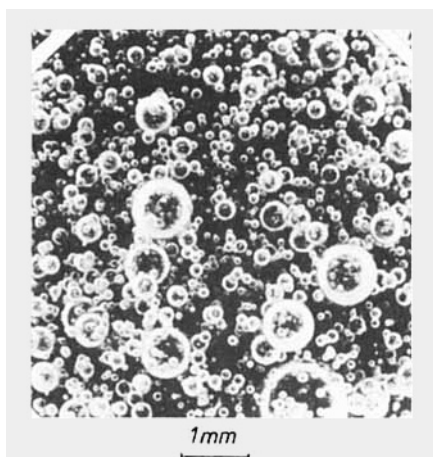


Figure 3. Ionic bubbles; photograph taken 1 s after stopping gas flow.

The counting of the bubbles was performed on photographs enlarged 30X. Several photographs were usually used in the calculations.

In all experiments the height of the clear liquid head in the column was $h_L = 13$ cm. The measurements were carried out for three different water solutions:

$$3.8 \frac{\text{mol}}{\text{l}} \text{NaOH}$$

$$1.9 \frac{\text{mol}}{\text{l}} \text{NaOH} + 0.95 \frac{\text{mol}}{\text{l}} \text{Na}_2\text{CO}_3$$

$$0.8 \frac{\text{mol}}{\text{l}} \text{Na}_2\text{SO}_3.$$

RESULTS

By means of the light depolarization method the local values of the total interfacial area were measured at different levels above the sieve plate. The results obtained for pure water at the level 7.5 cm above the sieve plate are shown in Figure 5. The values are 2 to 4 times less than those measured in earlier works

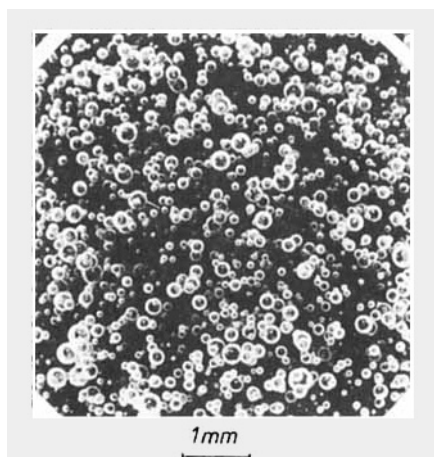


Figure 4. Ionic bubbles; photograph taken 3 s after stopping gas flow.

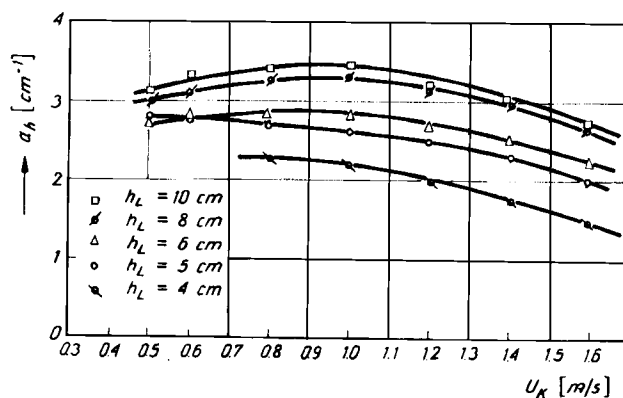


Figure 5. Local values of interfacial area measured 7.5 cm above sieve plate, air-water system.

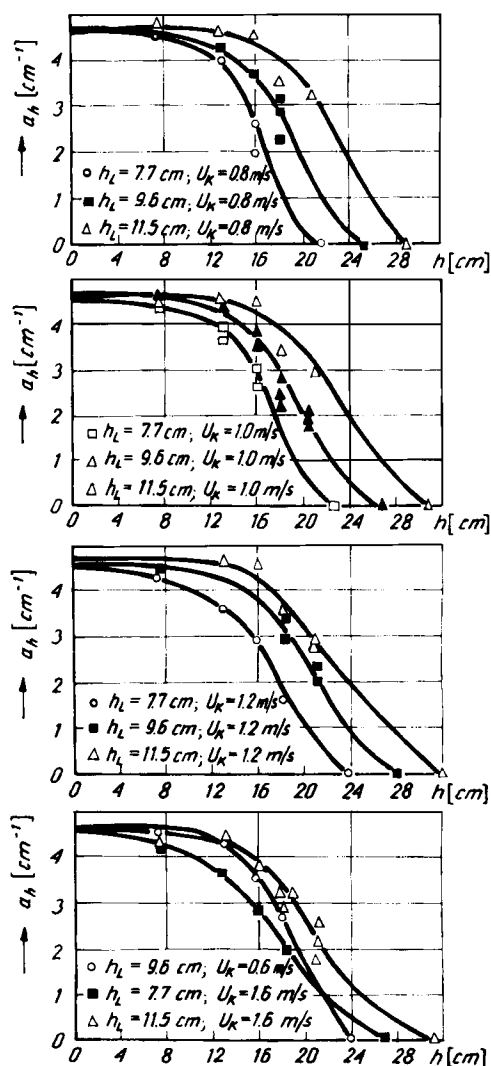
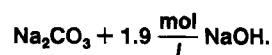


Figure 6. Variation of local values of interfacial area along column axis above sieve plate; water solution, $0.95 \frac{\text{mol}}{\text{l}}$



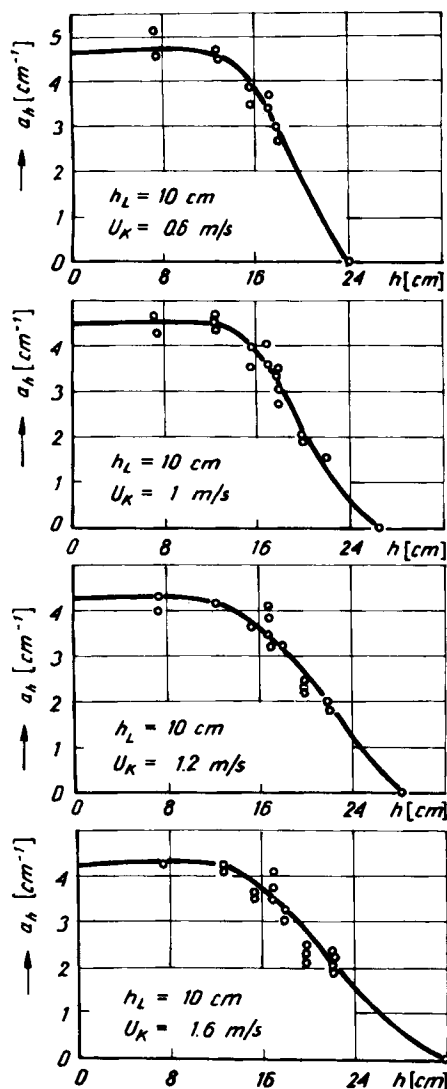


Figure 7. Variation of local values of interfacial area along column axis above sieve plate; water solution, $0.8 \frac{\text{mol}}{\text{l}} \text{Na}_2\text{SO}_3$.

using optical and photographic techniques. In most cases this is probably caused by the fact that in the earlier works the interfacial area of the bubbles forming the curtain near the column wall rather than the actual interfacial area of froth on the plate was measured (Calderbank, 1958, 1959; Calderbank et al., 1960; Calderbank and Moo-Young, 1960, 1961; Calderbank and Rennie, 1962; Rennie and Evans, 1962).

In the case of earlier works using the light depolarization method the difference is caused by erroneous calibration, as explained above (Rodionov and Ulyanov, 1967; Rodionov and Shabdanbekov, 1970).

Good agreement is noted between the results obtained in this work and the data reported by Burgess and Calderbank (1975a,b), who have applied an improved conductivity probe technique. The technique made it possible to measure local values of interfacial area and thus the results obtained by Burgess and Calderbank may easily be compared with those obtained in this work. At a level 7.5 cm above the plate the probe technique gave an interfacial area value of 2.3 cm^{-1} for a superficial gas velocity of 0.5 m/s , and a value of 3.1 cm^{-1} for a velocity of 0.7 m/s . In Burgess and Calderbank's (1975b) ex-

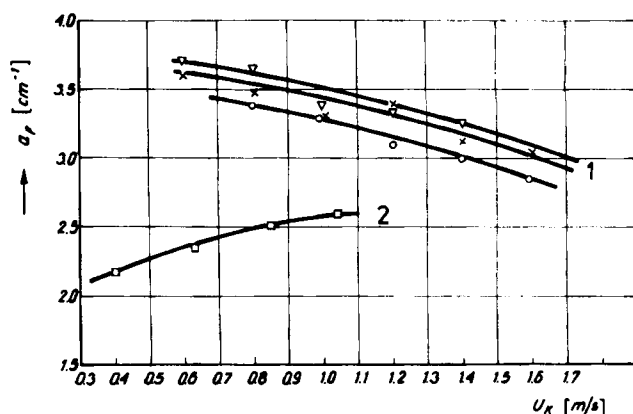


Figure 8. Dependence of averaged interfacial area on superficial gas velocity in the column.

periments the clear liquid head on the plate may be estimated to be 7 cm. Almost exactly the same values of interfacial area may be found from Figure 5. The agreement between the results obtained by both methods is seen to be excellent.

For the water solutions of electrolytes the variations of interfacial area with the vertical distance above the sieve plate at different plate running conditions are shown in Figures 6 and 7. The values of interfacial area vary considerably in the upper part of the froth (the points situated on the axis were found from the measurements of froth height using a cathetometer). In the lower part of the froth layer the values of the interfacial area are almost constant. The last statement, however, may not be true in the vicinity of the sieve plate where the bubbles are formed at the holes. Unfortunately, the optical method used did not permit the measurement of the interfacial area near the plate, because the condition $h_w/l \geq 1.2$ in a column 8 cm wide required a distance greater than 4.8 cm between the optical axis and the plate.

The averaged values of interfacial area per unit froth volume, obtained by integration of the local value profiles, are shown in Figures 8 and 9. It is seen that the values obtained for both the electrolyte solutions used are almost the same. However, the

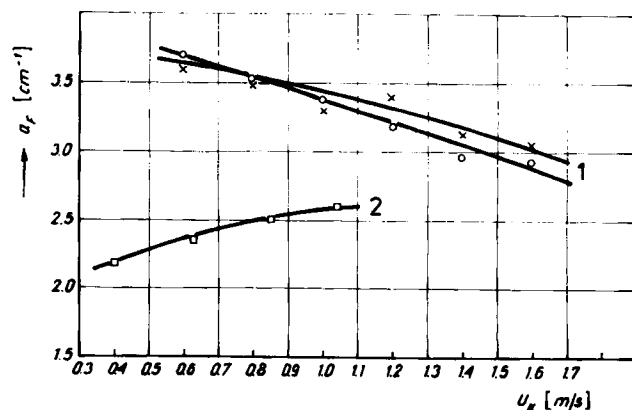


Figure 9. Comparison of averaged interfacial area values obtained for different aqueous salt solutions.

1. $\circ 0.8 \frac{\text{mol}}{\text{l}} \text{Na}_2\text{SO}_3$; $\times 0.95 \frac{\text{mol}}{\text{l}} \text{Na}_2\text{CO}_3 + 1.9 \frac{\text{mol}}{\text{l}} \text{NaOH}$;
2. \square pure water, $h_L = 10 \text{ cm}$

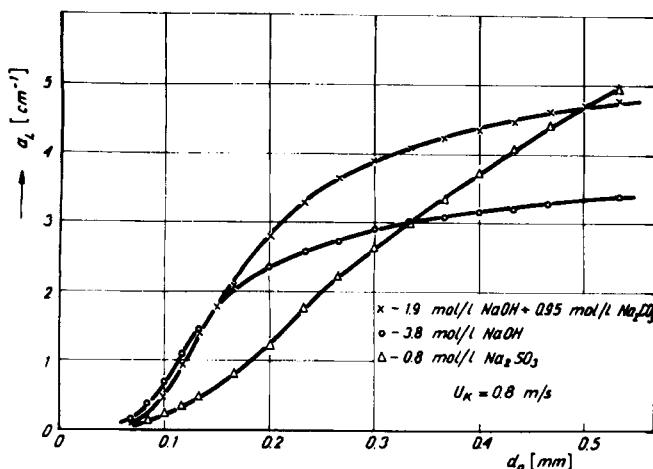


Figure 10. Interfacial area of ionic bubbles for different aqueous salt solutions.

decrease of the interfacial area with an increase of gas flow rate was greater in Na_2SO_3 solutions.

Considerable differences are observed between the results obtained for electrolyte solutions and those for pure water. Comparison of local values of interfacial area shown in Figure 5 with corresponding values in Figures 6 and 7 demonstrates that in the case of the electrolyte solutions the interfacial areas are about 30% greater. Similar differences are observed between averaged values of interfacial area in the froth. It is seen that the differences do concern not only the values of interfacial area but also the changes of these values with changing gas flow. The averaged values obtained for pure water increase with increasing gas flow. Again, the results obtained for pure water agree very well with those of Burgess and Calderbank (1975b).

The results of measurements of interfacial area formed by ionic bubbles, obtained by using the previously described photographic technique, are shown in Figures 10 through 13 per unit liquid volume. Figures 10 and 11 show cumulative integral curves, i.e., the value of the interfacial area formed by all bubbles having sizes less than that indicated on the abscissa. Figures 12 and 13 illustrate the variations of the derivatives of those relationships (differential curves). It is evident that both the number and the size distribution of ionic bubbles are strongly dependent on the kind of salt dissolved in water. For

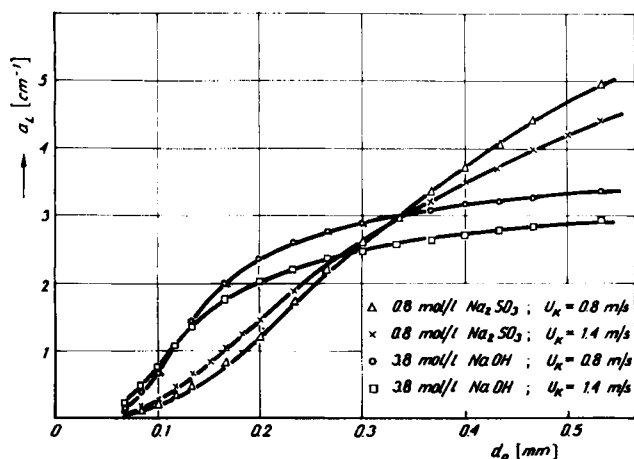


Figure 11. Effect of superficial gas velocity on interfacial area of ionic bubbles.

Na_2SO_3 solution the maxima of the differential curves appear at greater values of bubble diameter. The differential curves for NaOH and Na_2CO_3 solutions exhibit very narrow peaks, and they suggest that a second maximum exists in the range of greater diameters. The ionic bubbles provide only a part of the total interfacial area in the froth, and with such a sharp decrease of the value of derivative da_L/dd_p it would not be possible to achieve the total value of the interfacial area without another maximum. The presence of two separate bubble populations with two distinct peaks of bubble size distribution curves was also observed by Burckhart and Deckwer (1975) in a bubble column at low gas flows (about 3 cm/s) and large liquid head (about 7 m). The first population (first generation) was produced by the bubbling device itself, the second population (second generation) was formed by coalescence of the bubbles of the first generation. The situation was not the same in this work, because in our case the second generation of bubbles is not created by coalescence, but by the breaking of the bubbles of the first generation. Nevertheless, the work cited above has demonstrated that two separate bubble collectives can exist when two different mechanisms of bubble creation are operating in a bubble dispersion.

The optical (depolarization) method gives the values of the total interfacial area per unit volume of froth. Using the relation $a_L = a_F h_F / h_L$, the results obtained may be expressed per unit volume of liquid and then compared with the values of interfacial area formed by the ionic bubbles (expressed in the same units). The comparison leads to the conclusion that the ionic bubbles contribute about 45% of the total interfacial area in the froth, the latter amounting to about 9-10 cm^{-1} per unit volume of liquid. The question of the contribution of these bubbles to the mass transfer is thus of primary importance.

With the decrease of bubble diameter the ratio of bubble surface area to bubble volume increases. In the case of fast absorption processes the ionic bubbles may be very quickly exhausted of the gas component transferred to the liquid phase, whereupon they cease participating in mass transfer. A similar situation may exist in the evaporation of water from an electrolyte solution to an inert gas (this process has often been used to measure mass transfer coefficients in the gas phase). The rate of the evaporation process is controlled by gas-phase resistance only and the practical saturation of ionic bubbles may be reached within a considerably shorter time than the residence time of the bubbles in the froth. In such cases the interfacial area active for mass transfer may be much smaller than the total interfacial area.

The results described here are therefore of importance in absorption, fermentation, gas-liquid reactions, and other gas-liquid mass transfer processes.

ACKNOWLEDGMENT

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NOTATION

- a_F = total interfacial area per unit volume of dispersion
- a_h = local interfacial area at level h above the plate, per unit volume of dispersion
- a_L = interfacial area per unit volume of liquid
- b_w = width of the dispersion layer
- D = ratio of the intensities of both light polarization components
- d_p = bubble diameter
- H_0 = transmittance of two polarization filters set parallel

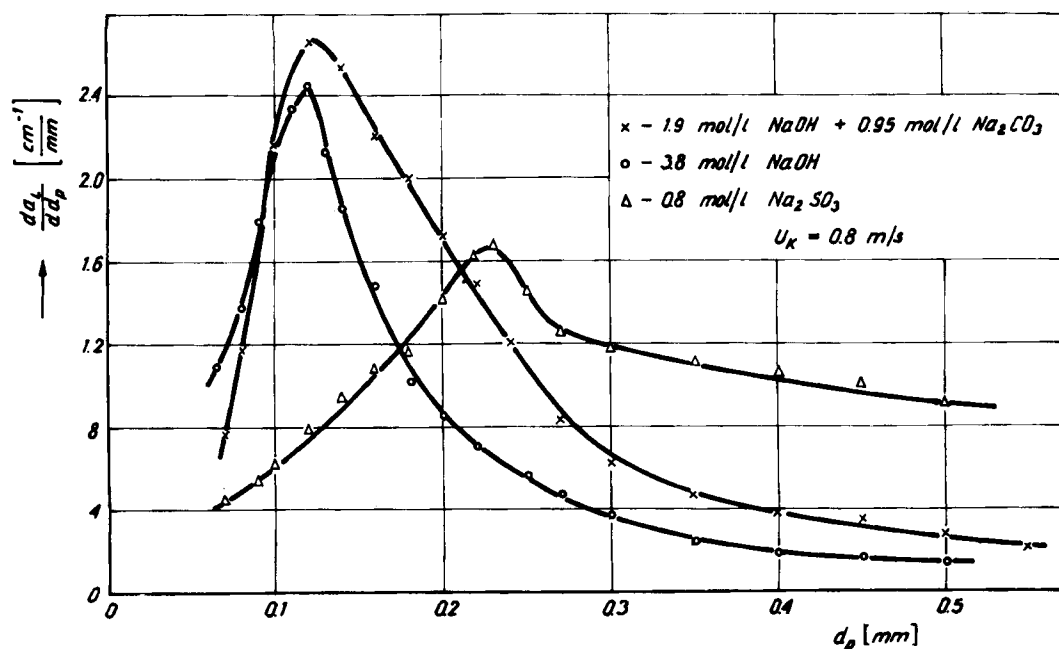


Figure 12. Interfacial area distribution for different aqueous salt solutions.

H_{90} = transmittance of two crossed polarization filters
 h = distance between the plate and the optical axis of the measuring arrangement
 h_F = height of the froth
 h_L = height of the clear liquid head
 h_w = height of the dispersion layer used for calibration
 $I_{\perp}(I_{\parallel})$ = intensity of the light component polarized in the plane perpendicular (parallel) to the initial polarization plane
 l = optical path length
 $N_C(N_D)$ = refractive index of the continuous (dispersed) phase
 $n = N_C/N_D$

p_s = hole diameter of the optical screens
 s_m = distance between the dispersion layer and photo-voltaic element
 U_K = superficial velocity of gas

Greek Letters

α_C = light absorption coefficient in the continuous phase
 α_D = light absorption coefficient in the dispersed phase
 θ = angle between the polarization planes of the analyzing polarization filters

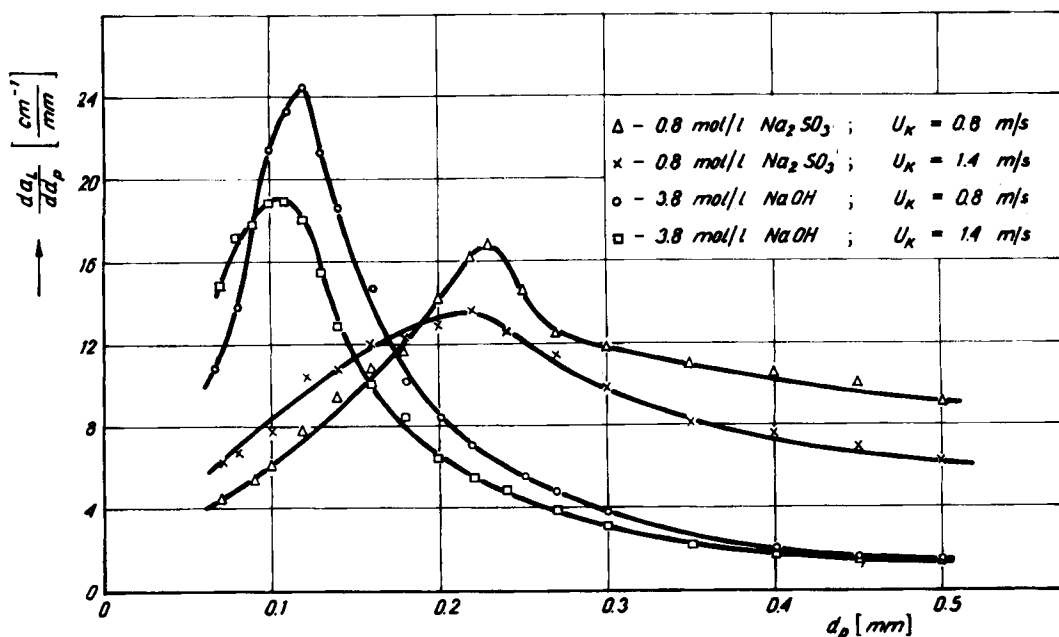


Figure 13. Effect of superficial gas velocity on interfacial area distribution.

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