

# Inhibition of Bubble Coalescence in Aqueous Solutions. 1. Electrolytes

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The inhibition of bubble coalescence has been studied in dilute 1:1 and 2:1 electrolyte solutions. All electrolytes studied were found to inhibit coalescence, with anions dominating the inhibition at concentrations below 0.01 M and cations dominating the inhibition at higher concentrations.

## Introduction

It has long been appreciated that moderate concentrations of electrolytes can have a strong effect on the coalescence of bubbles in aqueous solutions,<sup>1</sup> as is evidenced by the fact that in salt water waves can create substantial foam, whereas in fresh water this effect is minimal. The behavior of bubbles is of considerable importance to the chemical engineering community, and as such, the influence of electrolytes on bubble coalescence received considerable attention several decades ago.<sup>2–4</sup> Interest in this inhibition phenomenon has been reawakened in recent years.<sup>6–13</sup>

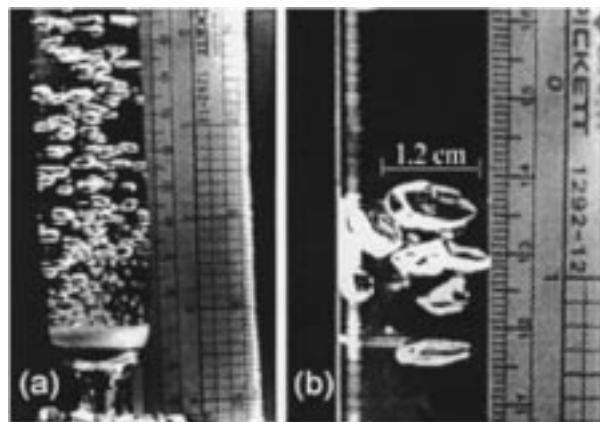
Much of this recent interest has been generated by the fascinating experiments of Craig, Ninham, and Pashley,<sup>7</sup> who used a scattering technique to study bubble coalescence in aqueous electrolyte solutions. These authors observed that a number of electrolytes switch from noninhibiting to inhibiting over a relatively narrow range of concentrations, whereas other electrolytes did not inhibit coalescence strongly at concentrations up to approximately 500 mM<sup>7</sup> (although many of these latter electrolytes do inhibit coalescence at higher concentrations<sup>11</sup>). Why similar electrolytes can have such disparate effects on bubble coalescence remains an open question. Indeed, there is of yet no consensus as to the reason that electrolytes inhibit coalescence at all, although a number of mechanisms have been proposed. Candidate causes for inhibition by electrolytes include decreased gas solubility,<sup>7,9,10</sup> double-layer effects,<sup>13</sup> changes in viscosity,<sup>4</sup> the Gibbs–Marangoni effect,<sup>2,6,11</sup> and other surface-tension effects.<sup>9,10</sup>

We have introduced a method<sup>14</sup> for measuring coalescence rates based on the technique of Craig, Ninham, and Pashley,<sup>7</sup> but designed to be sensitive to very small changes in coalescence rates. We have reported recently the use of this technique in the investigation of the effects of the identity of the sparging gas on bubble coalescence.<sup>14</sup> Here we apply this technique to the study of dilute aqueous solutions of alkali halides and alkaline earth halides. The concentrations used in the studies reported here are much lower than those at which complete inhibition of coalescence occurs.<sup>7</sup> However, all of the electrolytes investigated are found to have a measurable effect on coalescence even at concentrations in the range of 0.001 M. Furthermore, our data suggest that the inhibition of coalescence at low concentrations is dominated by a completely different mechanism than in the range of concentrations studied by Craig, Ninham, and Pashley.<sup>7</sup>

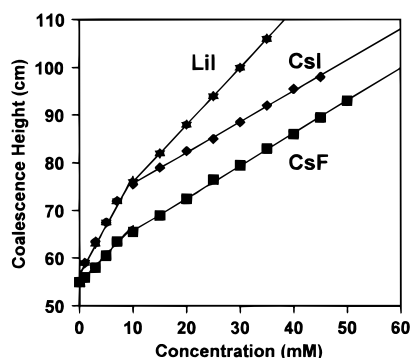
## Experimental Section

Our experimental apparatus is similar to that we have described previously.<sup>14</sup> High-purity helium gas is bubbled through a medium-porosity frit (pore size 30  $\mu\text{m}$ ) into a 140 cm tall glass column with an inner diameter of 18 mm. The column is filled with a set volume of aqueous electrolyte solution, which is prepared with ultrapure water (Millipore Milli-Q Plus) and analytical-grade salts that were oven-roasted overnight before use. The column is flushed with ultrapure water several times between samples; further cleaning of the apparatus was not found to affect the experimental results. Presumably any remaining surfactant organic impurities are quickly transferred to the top of the column when the gas flow is initiated.<sup>7</sup> Each solution is degassed by sonication under vacuum before being introduced into the apparatus, as this procedure enhances the coalescence rate and thereby increases the dynamic range of the experiment.<sup>14</sup> [Although the solution becomes saturated quickly with the sparingly soluble sparging gas, resaturation with air occurs on a time scale of hours, and so does not affect the measurements reported here.] The gas is flowed at a rate of 6.4 mL/s. To prevent the crystallization of electrolytes in the frit, the sparging gas is presaturated with water vapor. All measurements were made at 21 °C.

For electrolyte concentrations below 0.05 M, the size and shape of the bubbles at the frit do not change noticeably, in agreement with the results of Hofmeier, Yaminsky, and Christenson.<sup>12</sup> As the small bubbles produced at the frit rise through the column, they collide and coalesce (Figure 1a). For a given solution, we measure the “coalescence height”  $h_c$ , which is defined as the average height above the frit at which bubbles 1.2 cm in diameter form (Figure 1b). The coalescence height is first determined roughly by eye, and then the appropriate area of the column is videotaped for 5–10 min. There are generally 10–15 bubbles of the desired size in this quantity of video footage. The video is analyzed by stepping through frame by frame so that the heights of appearance of bubbles can be determined accurately. The standard deviation in the  $h_c$  so obtained is generally on the order of 2 cm. For bubbles that are overly large, bubble distortions and interactions with the walls of the column clearly affect the observed dynamics. However, for smaller bubble sizes the basic features of our data are not sensitive to the bubble size used for the determination of  $h_c$ . Furthermore, we obtain the same results in columns that are several times wider than the column used here. These observations suggest that bubble size and wall effects are



**Figure 1.** Close-up views of the experimental apparatus. (a) Bubbles emerging from the frit. (b) A representative bubble that would be used to determine  $h_c$ .



**Figure 2.** Dependence of  $h_c$  on concentration for several representative electrolytes. Lines are linear least-squares fits to the data. The uncertainty in each data point is approximately  $\pm 2$  cm.

unimportant in our measurements. We also obtain similar results at different flow rates. To test if surface charges in the apparatus were influencing the experimental results, the column and frit were silanized using chlorotrimethylsilane.<sup>15,16</sup> The data in the silanized apparatus were found to be identical to those in the untreated apparatus.

## Results and Discussion

The electrolytes studied include hydrochloric acid, sodium acetate, and sodium perchlorate; the fluorides, chlorides, bromides, and iodides of lithium, sodium, potassium, and cesium; and the chlorides of magnesium, calcium, barium, and strontium. The concentrations studied ranged from 1 mM up to the highest concentration at which  $h_c$  was still within the column (which was less than 100 mM for all electrolytes studied). As a measure of the sensitivity of our technique, we note that all electrolytes studied inhibited coalescence to a measurable extent, including HCl, NaClO<sub>4</sub>, and sodium acetate (none of which produced significant inhibition of coalescence in the studies of Craig, Ninham, and Pashley<sup>7</sup>).

Representative results for several electrolytes (CsF, CsI, and LiI) are plotted in Figure 2. A common feature of the data for all of the electrolytes studied is that  $h_c$  increases linearly with concentration until approximately 10 mM; we denote this range of concentrations region I. There is a discontinuity in the slope of the plot for each electrolyte at 10 mM, followed by a second region in which  $h_c$  increases linearly with concentration. For the electrolytes examined here, there is generally a concentration in the range 0.1–1.0 M at which  $h_c$  increases rapidly and nonlinearly with concentration. As was observed by Craig,

Ninham, and Pashley,<sup>7</sup> the concentration at which this occurs depends on the electrolyte. We denote the range of concentrations between 10 mM and the concentration at which  $h_c$  increases rapidly region II.

The change in  $h_c$  versus anion concentration in region I is listed in Table 1 for all of the electrolytes investigated. It is clear from this table that the change in the coalescence height in this range of concentrations is determined predominantly by the identity of the anion. Furthermore it is evident that the inhibitory power of the anions increases with atomic number. Similarly, Table 2 lists the change in  $h_c$  versus cation concentration in region II; in this case, the change in the coalescence height depends predominantly on the identity of the cation. The inhibitory power of the alkali metal cations decreases with increasing atomic number, whereas that of the alkaline earth cations increases with increasing atomic number. The dependence of the slopes in region I on the radius of the anions<sup>17</sup> and of the slopes in region II on the radius of the cations<sup>17</sup> is plotted in Figure 3. It is apparent that there is a strong correlation between the ionic radius and the degree of coalescence inhibition, although this correlation is positive for anions and divalent cations and negative for monovalent cations. Surprisingly, the degree of inhibition correlates more strongly with the crystalline ionic radius (Figure 3a) than with the Stokes (hydrated) radius (Figure 3b), despite the fact that the latter tends to be more highly correlated with the properties of ions in aqueous solution.

We can contrast the behavior that we have observed at low electrolyte concentrations and the results of other studies at higher concentrations.<sup>2,4,7</sup> In each of these previous studies, the concentration at which the transition to complete inhibition of coalescence occurs was determined. All studies, including the present one, indicate that multivalent ions have a greater effect on coalescence than do monovalent ions. Within a series of ions of the same valence the agreement is not consistent, however. The results of Craig, Ninham, and Pashley<sup>7</sup> indicate that the transition concentration decreases on going from KCl to KBr but increases on going from NaCl to KCl and from KBr to CsBr, which is consistent with larger halide ions having a greater inhibitory effect and with larger alkali metal ions having a lesser inhibitory effect. Lessard and Zieminski<sup>3</sup> observed the transition concentration to increase on going from LiCl to NaCl to KCl. However, they also observed an increase in the transition concentration on going from NaCl to NaBr, whereas this concentration was unaffected by going from MgCl<sub>2</sub> to CaCl<sub>2</sub>. Furthermore, Marrucci and Nicodemo<sup>2</sup> observed an increase in the transition concentration on going from KCl to KI. It would therefore appear that there is no direct relation between the phenomena that we observe and the complete inhibition of coalescence that can occur at higher concentrations, although due to differences in the techniques employed, these experiments may not be directly comparable with one another.<sup>18</sup>

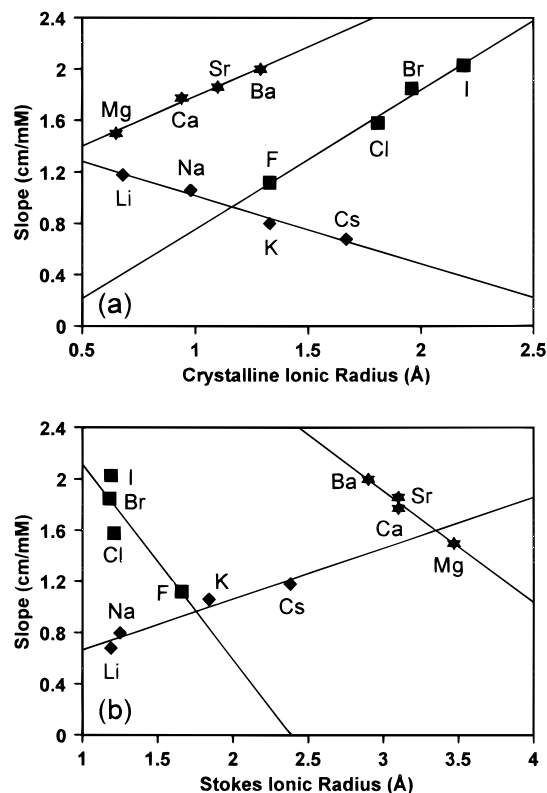
While the phenomenon that we have observed may not be related directly to the higher-concentration effects, we should still consider the same candidate mechanisms for inhibition mentioned above. We begin with the decreased gas solubility of electrolyte solutions over pure water, which was suggested by Craig, Ninham, and Pashley<sup>7</sup> and Weissenborn and Pugh<sup>9,10</sup> as a potential source of the inhibition of coalescence in electrolyte solutions. The intuitive picture is that dissolved gas can assist in the formation of a "bridge" between adjacent bubbles, leading to the rupture of the film that separates the bubbles. In support of this idea, recent experiments appear to indicate that dissolved gas can play a role in destabilizing oil/water emulsions, presumably by assisting in the coalescence of

**TABLE 1: Change in Coalescence Height as a Function of Anion Concentration (cm/M) in Region I. Numbers in Parentheses Are Approximate Standard Deviations**

	Li <sup>+</sup>	Na <sup>+</sup>	K <sup>+</sup>	Cs <sup>+</sup>	Mg <sup>2+</sup>	Ca <sup>2+</sup>	Ba <sup>2+</sup>	Sr <sup>2+</sup>	H <sup>+</sup>
F <sup>-</sup>	1110(10)	1150(20)	1120(20)	1100(20)					
Cl <sup>-</sup>	1580(10)	1600(20)	1560(30)	1570(30)	1510(30)	1560(20)	1610(40)	1680(30)	1570(50)
Br <sup>-</sup>	1830(30)	1820(30)	1820(30)	1850(30)					
I <sup>-</sup>	2080(20)	2040(30)	1970(40)	2040(30)					

**TABLE 2: Change in Coalescence Height as a Function of Cation Concentration (cm/M) in Region II. Numbers in Parentheses Are Approximate Standard Deviations**

	Li <sup>-</sup>	Na <sup>+</sup>	K <sup>+</sup>	Cs <sup>+</sup>	Mg <sup>2+</sup>	Ca <sup>2+</sup>	Ba <sup>2+</sup>	Sr <sup>2+</sup>	H <sup>+</sup>
F <sup>-</sup>	1200(20)	930(50)	800(50)	690(30)					
Cl <sup>-</sup>	1180(40)	1050(40)	770(40)	700(40)	1500(20)	1774(30)	1860(40)	2000(20)	1290(30)
Br <sup>-</sup>	1180(40)	1000(40)	780(20)	710(50)					
I <sup>-</sup>	1200(20)	1010(30)	780(40)	670(20)					

**Figure 3.** Dependence of the change of the coalescence height versus concentration of ions on (a) crystalline ionic radius and (b) hydrated ionic radius. Anion slopes are measured in region I and cation slopes in region II. Lines are linear least-squares fits.

the oil phase.<sup>19</sup> If dissolved gas does assist in coalescence, then by logical extension the more soluble a gas, the more quickly its bubbles would coalesce. However, we have demonstrated that the coalescence rate is not related directly to solubility and, if anything, decreases for more soluble gases.<sup>14</sup> Furthermore, as mentioned above, degassing the solution increases the rate of coalescence, which would appear to rule out this mechanism.

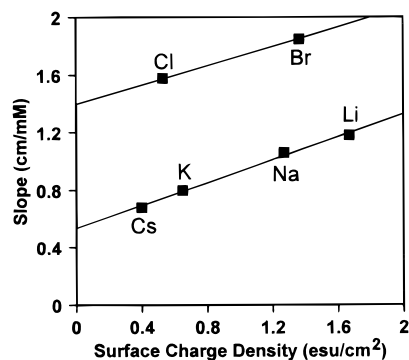
Zieminski and Whittemore have suggested<sup>4</sup> that electrolytes affect coalescence through changes in the microscopic structure of water (which in turn affects viscosity). The Jones–Dole viscosity *B* coefficient<sup>20</sup> is believed to be a direct measure of the structure-making or breaking ability of an ion. In general, the *B* coefficient, which describes the portion of the viscosity that is linear in the electrolyte concentration, is positive and increases in magnitude the smaller and more highly charged the ion;<sup>17</sup> thus, for instance, Li<sup>+</sup> is a strong structure maker and therefore would be expected to be the most potent coalescence inhibitor of the alkali metal ions. By the same token, however,

F<sup>-</sup> would be expected to be the most effective halide and Mg<sup>2+</sup> the most effective alkaline earth ion of those studied here in inhibiting coalescence. Furthermore, given large enough cations and anions, the viscosity of water decreases with increasing electrolyte concentration, which might be expected to enhance coalescence. It might be argued that at low concentrations the Jones–Dole *A* coefficient (which quantifies the contribution of the square root of the concentration to the viscosity) is a more appropriate parameter for discussing data at low concentrations. Indeed, *A* coefficients are generally positive,<sup>21</sup> and so all electrolytes might be expected to inhibit coalescence at low concentrations. However, the *A* coefficient also appears to decrease with increasing ionic radius for both anions and cations,<sup>21</sup> which is inconsistent with our data.

Stretching a film of electrolyte solution leads to a change in surface tension via changes in the adsorption excess. The elasticity of the film can thereby be shown to be proportional to the square of the adsorption excess. As a result, both positively and negatively adsorbed species increase the elasticity of the film and therefore can inhibit coalescence of bubbles; this is known as the Gibbs–Marangoni effect.<sup>2,6,11</sup> An electrolyte consisting of a negatively adsorbing ion and a positively adsorbing ion may, on balance, have a small adsorption excess and therefore not affect coalescence strongly. The net adsorption excess can be related to the change in surface tension with electrolyte concentration, and the square of this quantity has been shown to be related to the transition concentration in electrolytes that can inhibit coalescence completely.<sup>11</sup> Measured changes in surface tension with electrolyte concentration<sup>9,10</sup> for the substances studied here do not correlate with the observed inhibition efficiencies of anions in region I or cations in region II, suggesting that this effect is not important at low concentrations. We should note that detailed surface-tension measurements were not performed at concentrations of 0.01 M and below, however.<sup>9,10</sup>

Miklavcic has proposed that double-layer effects are involved intimately in the inhibition of coalescence by electrolytes.<sup>13</sup> In particular, Miklavcic pointed out that the liquid–vapor interface is a constant-potential surface, and therefore with increasing electrolyte concentration the length scale of double-layer repulsion between two bubbles decreases, but the total force increases. It is known that the film between two approaching bubbles in pure water ruptures at a distance on the order of 1000 Å,<sup>22</sup> which implies that only if the double-layer repulsion is of large enough magnitude at this distance could this force be primarily responsible for the inhibition of coalescence at low electrolyte concentrations. The model calculations of Miklavcic indicate that the double-layer force at this distance decreases with increasing electrolyte concentration up to at least 10<sup>-3</sup> M,<sup>13</sup>





**Figure 4.** Dependence of the change in coalescence height versus concentration of ions in region II as a function of the surface charge density of 4.92  $\mu\text{M}$  aqueous alkali chloride solutions and in region I as a function of half of the surface charge density of 4.92  $\mu\text{M}$  aqueous magnesium halide solutions (the factor of 0.5 is to make these data comparable to those of the alkali chlorides). The lines are linear least-squares fits to the data.

however, which appears to rule out double-layer forces as the sole source of inhibition in our experiments. However, it is feasible that double-layer effects influence coalescence in conjunction with other forces.

It would appear, then, that no single one of the mechanisms that have been advanced for explaining transition concentrations can account for our experimental observations. We do not yet have a model that can explain all of these observations, but we will sketch briefly an approach that we believe to be promising. It has been pointed out that when water is confined between two parallel hydrophobic surfaces, there is some separation between the two surfaces at which the surface/liquid and surface/gas free energy difference becomes greater than the vaporization energy of the confined liquid.<sup>23,24</sup> This critical separation is estimated to be on the order of 1500 Å for water confined between hydrocarbon surfaces at room temperature.<sup>24</sup> A water film between two hydrophobic surfaces will not always rupture at this separation due to the energetic cost of nucleating the vapor phase.<sup>24</sup> However, the vapor phase already exists when one of the interfaces is the surface of a bubble, so we can expect this 1500 Å to be a reasonable estimate of the film thickness at rupture. This number is well in line with measurements of the critical rupture distance of aqueous solutions confined between two hydrophobic surfaces.<sup>22,25–27</sup>

These same ideas should be applicable to bubble coalescence in electrolyte solutions. It is clear from this model that electrolytes can influence the coalescence of bubbles by altering either the free energy of the “bulk” liquid in the film or that of the vapor/liquid interface. The vaporization energy of a film of given thickness should increase with the electrolyte concentration, which would lead to a corresponding decrease in the coalescence rate. However, in the concentration range in which the electrolyte solutions are ideal, the change in the vaporization energy should be independent of the identity of the electrolyte, which implies that surface effects are important as well.

Because the dipole moment of water molecules at the liquid/vapor interface has a slight tendency to point into the liquid, anions can approach the interface more closely than can cations.<sup>28</sup> This idea is supported by the fact that bubbles have a negative electrophoretic mobility in pure water<sup>29,30</sup> and in dilute electrolyte solutions.<sup>31,32</sup> Since bubbles in dilute electrolyte solutions are negatively charged, it should not be surprising that anions dominate the inhibition of coalescence at the lowest electrolyte concentrations. Figure 4 illustrates the connection between surface charge density<sup>31</sup> and the inhibition

of coalescence. For the cations, the slope in region II is plotted versus the surface charge density of bubbles in a 4.92  $\mu\text{M}$  aqueous alkali chloride solutions. For the anions, the slope in region I is plotted versus half of the surface charge density of bubbles in 4.92  $\mu\text{M}$  aqueous magnesium halide solutions (the factor of a half is to account for the doubled concentration of halide ions compared to the alkali chloride solutions). The correlation between surface charge density and the rate of change of  $h_c$  with increasing concentration is strong, and it is particularly striking that the slopes of the anion and cation plots are identical to within experimental uncertainty. Surface potential measurements of electrolyte solutions<sup>33</sup> suggest that the alkaline earth ions will follow the same trend as the alkali metal ions of increasing surface charge density with decreasing size, but bubble surface charge density measurements on electrolytes containing these ions have not been reported.

Despite the good agreement between surface charge density and the observed coalescence inhibition behavior of ions, there is nothing in the surface charge density that explains the transition from domination of inhibition by anions to dominance by cations near 0.01 M. The effects of ions on the energy of the liquid/vapor interface become considerably more complex as the concentration is increased, and it is possible that a major change in the charge properties of bubbles occurs at a concentration near 0.01 M. Further investigations of surface charge density and the electrophoretic mobility of bubbles would be highly desirable to help resolve these issues.

## Conclusions

We have studied the effects of low concentrations of alkali halides and alkaline earth halides on the coalescence of bubbles in water. All of these electrolytes were found to inhibit coalescence, with the anions dominating the inhibition below 0.01 M and the cations dominating above this concentration. Our results cannot be reconciled with any single model that has been proposed to explain the influences of electrolytes at higher concentrations. We believe that our observations can be explained by a model that takes into account the influence of electrolytes on the vaporization energy of the solution and on the energy of the liquid/vapor interface.

The data presented here demonstrate that even a minimal concentration of electrolytes can have a measurable influence on the interactions between bubbles. We have observed similar effects in the presence of molecular solutes.<sup>34</sup> It is believed that the coalescence of bubbles is related directly to long-range hydrophobic interactions,<sup>7,14</sup> so elucidation of the underlying physics and chemistry of coalescence inhibition may have implications in the stability of colloids and the aggregation of proteins, among other areas. It therefore will be of great interest to study the effects of other parameters, such as temperature, on the coalescence rate of bubbles.

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