

Effects of Surfactants and Electrolytes on the Nucleation of Bubbles in Gas-Supersaturated Solutions

Edvard A. Hemmingsen

Physiological Research Laboratory, Scripps Institution of Oceanography, University of California,
San Diego, La Jolla, California 92093

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Various solutions of surfactants and electrolytes were equilibrated with argon at gas pressures up to 202 atm and then decompressed to atmospheric pressure while observed visually or cinemicrographically for occurrence of bubbles. In water, the threshold gas supersaturations for spontaneous nucleation of bubbles at the glass-water interface and in the bulk liquid were 135 atm and 155 atm, respectively. Among the surfactants, only those of molecular weight less than about 330 daltons significantly decreased the cavitation thresholds; those of higher weights had no effect. The electrolytes either had small to moderate or no effect on the threshold, although the dissolved gas concentrations resulting from the solubility changes were greatly decreased. The data indicate that changes in the structure of the solvent water may affect the nucleation process.

Introduction

The occurrence of bubbles in water and other liquids subjected to heating, tensile stress, and gas supersaturation usually is due to microscopic bodies of gas trapped either in crevices of the container or in motes suspended in the liquid. However, in all of these systems, bubbles also may form spontaneously at sufficiently high degrees of "metastability" unaided by such nuclei. In the cases of superheating [2–4] and tensile stress [5–7], the threshold conditions required for spontaneous nucleation of bubbles are determined by kinetic processes involving only the liquid molecules. Attempts to define these conditions either on theoretical or empirical grounds have not led to satisfactory results, and large discrepancies exist between results obtained by various approaches [8].

For gas-supersaturated systems [9–15], in which the nucleation of bubbles occurs as a result of various interactions between the liquid and the dissolved gas, a quantitative reconciliation of the nucleation thresholds of gassupersaturation indicated by empirical observations and those which may be anticipated from known macroscopic physical properties of the liquids and the liquid/gas interfaces appears to be even more distant. In fact, a comprehensive theoretical approach which would contribute to the solution of this problem has yet to be put forward. One of the difficulties is that the

nucleation events no doubt are triggered by energy and density fluctuations on the molecular level not readily predictable from the continuum macroscopic equilibrium properties of the liquid-gas system.

In recent studies [13–15], the approximate supersaturation thresholds for spontaneous nucleation of bubbles in water have been experimentally determined for several gases. With argon, for example, the onset of nucleation in bulk water occurred after equilibration and decompression from about 160 atm gas pressure to ambient atmospheric pressure; the values for various other gases ranged from about 115 atm for methane to about 350 atm for helium. These values would require an initial, critical bubble diameter of at least 10^{-6} cm, if the normal, macroscopic surface tension properties of the water were retained in the formation and early growth of the bubbles. This is far larger than what may be reasonably envisioned for such spontaneously formed nuclei. Indeed, these experimental data strongly indicate that the normal concept of surface tension may not be applicable to submicroscopic bubble dynamics and must be reevaluated.

These facts led to the need to determine how solutes with strong surfactant or other water-interacting properties may affect the nucleation of bubbles, since this information would shed further light on the kinetic nature of the process. The present report describes the results obtained with argon supersaturated water which contains surfactants that affect the liquid-gas interface properties, or electrolytes that presumably affect the structural properties of the water

Reprint requests to Dr. E. A. Hemmingsen, Scripps Institution of Oceanography, University of California, San Diego, La Jolla/California 92093 U.S.A.

Methods

Nucleation of bubbles at the glass-water interface appear to differ only by degree from that in bulk water as the gassupersaturations required for nucleation in the two situations are similar [13–15]. Cavitation behavior at the interface was chosen as the primary mode of observation because it offered the most sensitive method to detect changes in the nucleation thresholds; however, in some cases cavitation in bulk solutions also was monitored in order to confirm typical effects observed at the interface.

The methods used have been described elsewhere [13–15]. In principle, the solutions were equilibrated with gas at high pressures (50 to 202 atm), transferred under pressure to either a Pyrex glass capillary (0.11 cm inside diameter), or, for 16-mm cinemicrography, a chamber formed between two Pyrex windows separated by a flat stainless steel ring (0.30 cm thick, 1.40 cm inner diameter). The solution then was decompressed to ambient pressure (1 atm absolute) over a period of 2–3 seconds while it was observed visually or photographically for appearance of bubbles. The degree of gas-supersaturation in each case was defined as the equilibration pressure minus one atmosphere. All experiments were carried out at room temperature, which usually ranged from 21–23°C.

Solutions were made up from glass double-distilled water and passed through Millipore filters of 0.45 µm pore size. Argon gas of better than 99.99% purity was used in all experiments. Reagent grade electrolytes and sucrose were used. Purified sodium dodecyl sulfate and sodium decyl sulfate were obtained from Sigma Chemical Company, Saint Louis, Missouri. The remaining surfactants were of technical or commercial grades and were used as supplied by the manufacturers. *Alfonic* surfactants were supplied by Conoco Chemicals, Ponca City, Oklahoma; *Plurafac* and *Pluronic* surfactants by BASF Wyandotte Corporation, Wyandotte, Michigan; and *Triton* and *Priminox* surfactants by Rohm and Hass, Philadelphia, Pennsylvania.

The approximate surface tensions and viscosities of the various solutions were obtained from the literature [16, 17]; or were provided by the manufacturers. In a few cases where values were not available they were determined by standard methods [18, 19] in our laboratory. The solubility of argon at 150 atm gas pressure in the solutions was

determined with a method [20] which in principle was similar to that described by Wiebe et al. [21] and Schröder [22].

Results

A) Cavitation in Pure Water

With the capillary system, argon supersaturations of less than 130 atm rarely produced bubbles upon decompression, either in the bulk water or at the glass-water interface; waiting periods of several minutes did not alter this stability [13]. At increasing supersaturation, bubbles formed at the interface in increasing numbers. For example, at 135 atm supersaturation, a few bubbles formed consistently in the capillary; at 145 atm, several bubbles formed per mm length of the capillary. At supersaturations higher than 150 atm, there was a massive cavitation throughout the capillary, with some bubbles clearly originating in the bulk water [13, 14]. Figure 1 shows the results obtained with water in one typical series of experiments; the cavitation behavior of some other solutions is illustrated here for comparison purposes. A most notable characteristic was the relatively small difference that generally existed between the maximum supersaturations which could be maintained without any bubbles forming and those which produced bubbles massively throughout the capillary.

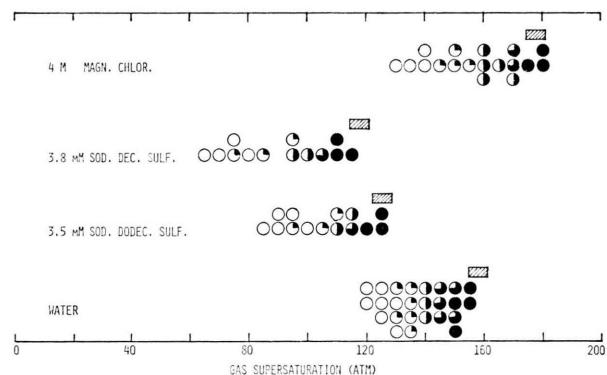


Fig. 1. Cavitation behavior of water and some solutions (sodium dodecyl sulfate, sodium decyl sulfate and magnesium chloride) as a function of argon supersaturation. Each circle represents one experiment with the capillary method. The degrees of cavitation were classified as follows: ○ no cavitation; ○ less than one bubble per cm length of glass capillary (*light cavitation*); ○ 1–20 bubbles per cm; ● 20–100 bubbles per cm; ● more than 100 bubbles per cm (*massive cavitation*). Horizontal hatched bars signify the lowest range of supersaturations for which bubbles clearly originated in the bulk liquid rather than at the glass interface. These latter data were obtained from cinemicrographic recordings made during decompression.

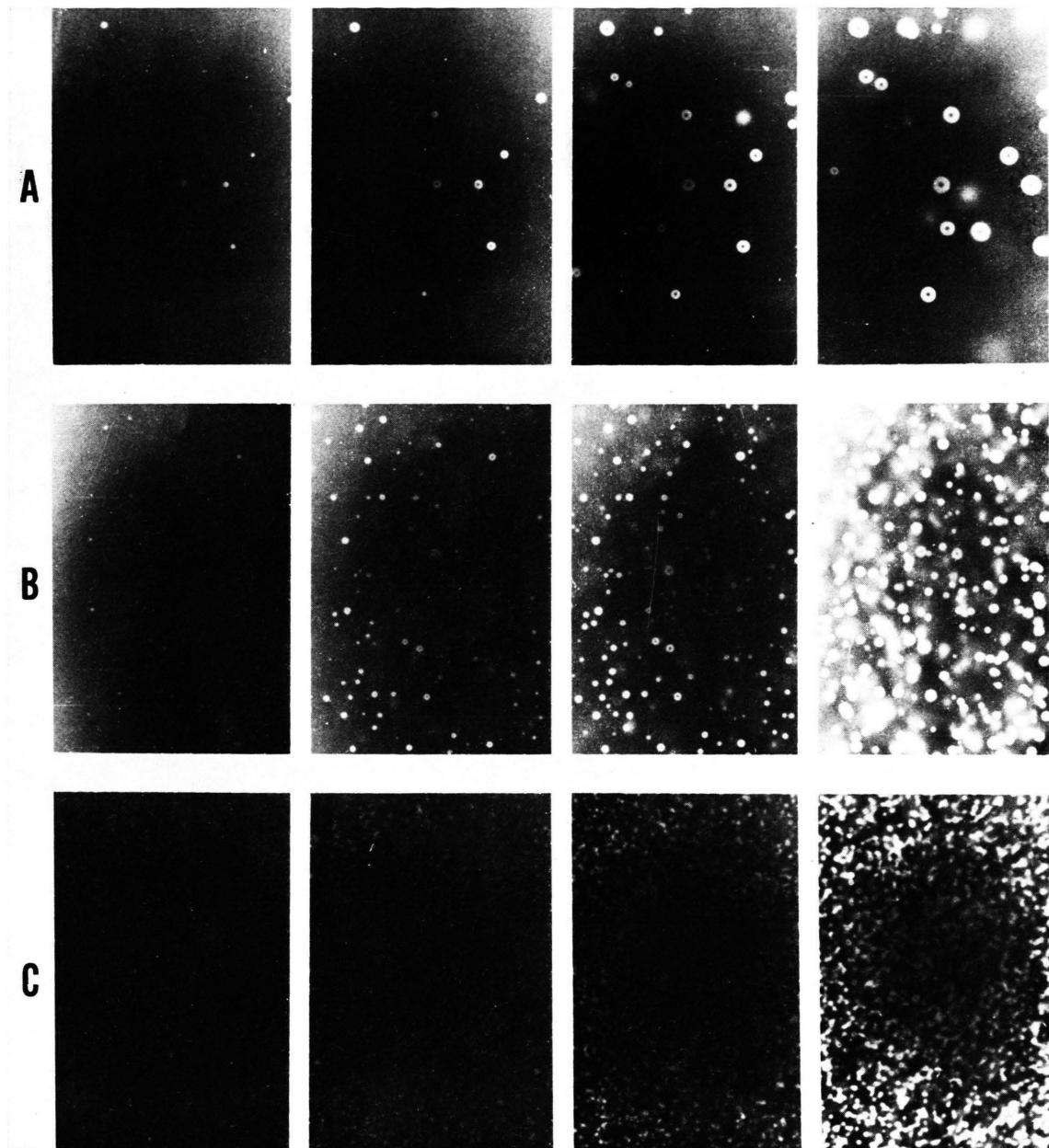


Fig. 2. Examples of data obtained by the cinemicrographic method. The appearance of gas bubbles during the late stages of decompression of water saturated with argon at (A) 158 atm, (B) 177 atm, and (C) 202 atm gas pressure; at 0.1 seconds intervals from left to right. Bubbles forming on the cuvette window are distinguished from those in the water by being in sharper focus and in the same position from frame to frame. Each frame represents an area of $0.08 \text{ cm} \times 0.12 \text{ cm}$. Negative prints from 16 mm movie film (Kodak Plus-X Reversal), recorded at 64 frames per second.

Cavitation in the bulk water was examined more closely by means of the cinemicrographic method, in which a cuvette area of $0.14 \text{ cm} \times 0.10 \text{ cm}$ (and a volume of about $0.3 \mu\text{l}$) was monitored during the decompression. Bubbles with diameters down to $5 \mu\text{m}$ could be detected on the film. Bubbles de-

veloping in the liquid could be distinguished readily from those on the cuvette window by their slight movement from film frame to film frame [15]. Examples from the cinemicrographic recordings are shown in Figure 2. The lowest supersaturation at which bubbles clearly formed in the bulk water,

Table 1. Cavitation thresholds (in atm) for surfactant solutions. A: the maximum argon supersaturations without any bubbles forming; B: the supersaturation required for more than ten bubbles per cm length of glass capillary.

Solution	Molecular weight	Concentration		Surface tension dynes/cm	Threshold (atm)	
		Wt.%	mMolar		A	B
Water				72	130	140
Sodium decyl sulfate	260	0.1	3.84	66	130	135
		1		38	75	95
Sodium dodecyl sulfate	288	0.1	3.47	43	105	125
		1		34	90	115
Sodium oleate	304	0.1	3.28	27	110	130
		1		25	105	120
Alfonic 1412-40	330	sat.			135	140
Plurafac A-24	350	sat.		29	130	140
Cetyltrimethyl ammonium bromide	365	0.1	2.74		125	135
		1			130	140
Alfonic 1012-60	378	0.1	2.65	26	130	140
		1			125	135
		10			130	140
Triton X-45	426	sat.		28	130	
Alfonic 1412-60	524	0.1	1.91	28	130	140
		1			125	140
		10			130	140
Triton X-100	628	0.1	1.59	30	125	140
		1			125	135
Plurafac C-17	770	0.1	1.30	34	125	
		1			125	135
		10			130	145
Priminox R-15	832	0.1	1.20	31	125	140
		1			130	135
Triton X-165	910	0.1	1.10	35	130	140
		1			130	140
Plurafac D-25	950	0.1	1.05	34	130	140
		1			125	135
Plurafac B-26	1050	0.1	0.95	36	130	140
		1			130	140
		10			130	145
Triton X-305	1526	0.1	0.66	38	130	140
		1			130	140
Pluronic L64	2900	0.1	0.35	43		
		1			130	140
Pluronic P104	5850	0.1	0.17	33	130	140
		1			125	140

away from the glass interface, ranged from 155 to 160 atm. At 165 atm, several bubbles consistently formed within the monitored volume of water. The number of bubbles rapidly increased with increasing supersaturations, and amounted to several thousand per μl at 200 atm.

The bulk cavitation threshold established by this method was in satisfactory agreement with that obtained by the capillary method. This agreement was confirmed for some selected solutions with cavitation stabilities different from water (Figure 1).

In each of these cases, the bulk cavitation threshold determined by cinemicrography was equal to, or just detectably higher than, that for onset of massive cavitation in the glass capillaries, in spite of the substantial difference in liquid volumes monitored in the two approaches.

B) Effects of Surfactants

In one series of experiments, the effect which surfactants of various molecular weights and surface tension properties may have on the cavitation

stability of water was tested. For each substance, the supersaturation required for onset of both light and moderate cavitation at the glass-water interface was determined, using the capillary method. The results are given in Table I. For most of the solutions, the cavitation thresholds were identical to that of pure water, even though the surface tension was much decreased. Only surfactants of molecular weights of less than about 330 daltons had a clear effect; they decreased the cavitation thresholds substantially. The lower concentration of sodium dodecyl sulfate was an exception; but in this case the surface tension of the solution also was relatively little decreased.

This decrease in cavitation stability was examined in more detail using various concentrations of purified as well as of technical grade sodium dodecyl sulfate (Figure 3). In all of these solutions, the overall stability was considerably less than in the pure water. With the purified substance, the degree of stability change was dependent on the concentration. However, the change could not be directly related in a simple way to concentration or to the prevailing surface tension of the solutions. For example, the smallest change occurred at an intermediate concentration and a low surface tension. The concentration dependence was less obvious with the unpurified substance as the decrease here remained relatively constant for all of the concen-

trations used. For both grades, the range of supersaturations spanning from the onset of light cavitation to that of massive cavitation was wider in general than for water. Most of this expanded range generally was due to lower thresholds for light, incidental cavitation and may not represent real stability changes with reference to the bulk cavitation.

The bulk cavitation thresholds were decreased by the sodium dodecyl sulfate and the sodium dodecyl sulfate to the same degree as were the glass-water interface cavitation thresholds for the same substances (Figure 2). With 1% (i.e., 3.8 and 3.5 mM) solutions of these substances, bulk cavitation clearly occurred at supersaturations as low as 115 atm and 123 atm, respectively. Although the cavitation behavior in the bulk solution could not be readily quantified by the technique used, it was noted that the increase in number of bubbles with increasing saturations was substantially smaller for the two surfactant solutions than it was in the pure water for either the equivalent absolute or relative gas-supersaturation increases.

C) Effects of Solutes

Salts decrease the solubility of gas in water and may also greatly affect its viscosity. Presumably, the increase in viscosity caused by magnesium salts, for example, reflects a stabilization of the solvent water structure, and the decrease caused by potassium salts, a destabilization of the structure [23]. Figure 4 shows the cavitation properties of solutions of such salts, together with their viscosities and gas solubilities. A clear effect was obtained only with the 4 M solution of magnesium chloride, which showed a substantial increase in the thresholds for both light and heavy cavitation. No cavitation occurred at saturations of 140 atm; the onset of heavy cavitation occurred at 170–175 atm, which is about 25 atm, or about 15%, higher than for water. The cavitation behavior of the 4 M magnesium chloride solution was examined also with the cinemicrographic method. These experiments confirmed that bubbles originated in the bulk of the solution only at supersaturations higher than 175 atm (Fig. 2).

A detectable increase of the cavitation stability also was obtained with 2 M magnesium sulfate, 2 M magnesium chloride, and 4 M sodium chloride solutions. With 2 M potassium chloride solution, there

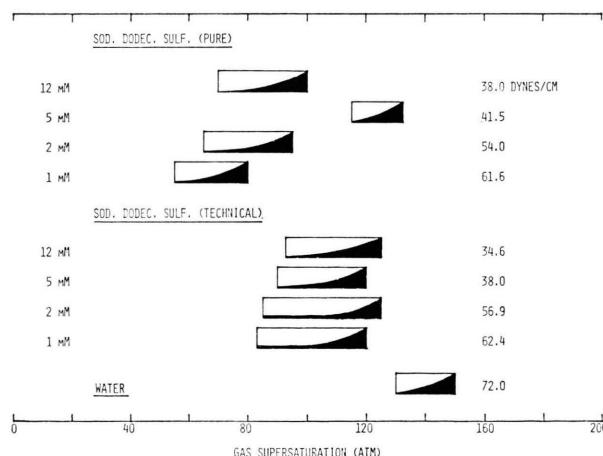


Fig. 3. Cavitation in argon supersaturated solutions of sodium dodecyl sulfate, purified and technical grade. The horizontal bars cover the range from onset of very light cavitation (no shading) to that of massive cavitation (fully shaded) in the glass capillaries. Each bar is a summary of data obtained with the capillary method (see Figure 1). The surface tension is indicated for each of the solutions.

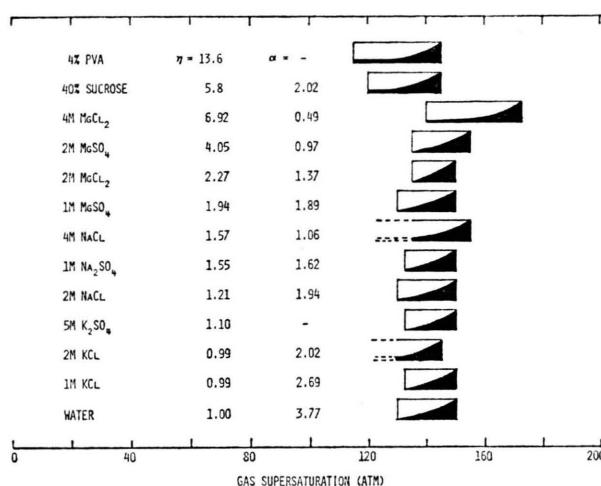


Fig. 4. Cavitation in various argon supersaturated solutions. Bars represent the range from onset of very light cavitation (no shading) to that of massive cavitation (fully shaded). The numbers represent the relative viscosities at 25°C (η), and the argon solubilities, in ml gas per ml liquid at 150 atm, 25°, normalized to 0° (α).

was a slight decrease in the stability. In the latter case, in particular, the effect may not be of real significance. Although it was discernable in the data it was not clearly beyond the accumulated experimental uncertainties and artifact interferences.

Since it was possible that the threshold changes observed in these experiments could be related to the increase in viscosity *per se*, two other solutions (polyvinylalcohol and sucrose) of moderately high viscosity were included in this experimental series (Figure 4). However, the cavitation stabilities of these solutions were similar to that of water, or were even slightly decreased.

Discussion

The experimental evidence which has accumulated in our previous and present investigations strongly indicates that the bubbles which form both at the glass interface and in the bulk water are generally of spontaneous origin [13–15]. In some cases pre-formed gaseous nuclei have appeared to be present to disrupt the supersaturation stability of the experimental systems, but their numbers are few and their occurrence incidental.

Although we do not yet have the empirical foundation to attempt a quantitative description of the nucleation process, we may speculate on the sequence of events. It is generally accepted that water undergoes very rapid fluctuations in its mole-

cular density and structural configurations, leading to a continuous formation and collapse of molecular voids, or holes, throughout it [11, 23]. It is likely that such voids serve as the foci for the initial cavities. Normally, the gas molecules dissolved in water may occupy some of these structural voids [22]. However, successful bubble nucleation depends upon a number of additional gas molecules entering into the void, or cavity, during a phase when there is a statistical net kinetic motion of the surrounding water molecules outward. During the phase of net inward motion of the water molecules, a sufficient number of gas molecules may be retained in the cavity to prevent its immediate collapse. Subsequent "pulsations" in the adjacent water structure may allow further incremental gains in volume of the cavity and the number of gas molecules captured within it until the cavity has attained an *equilibrium* stability, at which time the structural fluctuations in the water cease to be of further consequence. Such a scheme requires that the gas have a higher dynamic tendency to enter from the water to the "gas phase" by diffusion than in the reverse direction. On a macroscopic level, this type of non-equilibrium net diffusion (or "rectified diffusion") has been found to play a significant role for bubble growth during hydrostatic pressure oscillations [24]. It is reasonable to postulate that this type of process also could be operable on a lower submicroscopic and molecular level.

In such a nucleation process, the initial cavities are fundamentally a result of the liquid properties; the participation of the dissolved gas is in a sense more "passive" in nature but, nevertheless, its concentration and diffusion behavior are crucial in the stabilization and growth phase. The other possibility, that the initial cavities may be formed by simultaneous aggregation of dissolved gas molecules which secondarily disrupt the water structure, appears not to warrant serious consideration due to the relatively low number of gas molecules present at the established threshold conditions [13, 15].

The relatively small difference between the threshold for interface cavitation and that for bulk cavitation is not unexpected in view of the similarities in molecular structure and bonding forces of the water and the glass. At the surface of the glass strong polar bondings will exist between the oxygen of the water and the silicon of the glass, and conversely between the oxygen of the silicon dioxide

and the hydrogen of the water [25]. These bondings would be of the same magnitude, or larger, than cohesive bonding between the water molecules. Ideally, therefore, there is a continuous transition between the two phases, without any abrupt discontinuities. Impurities other than the highly polar metal oxides may tend to weaken the adhesive forces at the interface, and thereby create definite spots for nucleation at supersaturations which are somewhat lower than for either the normal interfacial water or the bulk water, but they would hardly lead to any major structural disruptions in the interfacial zone.

A most striking characteristic of the cavitation behavior of water observed here is the small effect which the addition of solutes in general has on the cavitation stability. In the case of surfactants, this reveals some particularly interesting facts about the nucleation process, and also further emphasizes the minor role of pre-formed nuclei. If such nuclei were present in, for example, crevices of the glass or in motes, they would have been stabilized to a certain size during the equilibration, and the surface tension at the gas interface would be the equilibrium value. Any significant decrease in the surface tension, irrespective of its cause and its rate of change would increase the stable critical size of the nuclei, and hence, decrease the gas-supersaturations required to expand them after the release of the hydrostatic pressure. This was clearly not the case; most of the surfactants had no effect on the cavitation thresholds even though the surface tension of the solutions generally was decreased to one-third of the normal value for pure water.

This absence of an effect on the cavitation threshold by some surfactants, and the presence of an effect by others, are more readily accounted for in a process of spontaneous nucleation. In such a process, all nuclei formed by fluctuations in the water structure will collapse at supersaturations below the normal cavitation threshold value. However, if a "shell" of surfactant molecules can be established around a nucleus, its collapse may be inhibited or much retarded. In order for this to occur, the time of aggregation and orientation of surfactant molecules around the nucleus must be shorter than its normal lifetime. Only then will the surfactant aid its expansion and growth into true gas bubbles of macroscopic size; at longer aggregation times, the nucleus recollapses into solution.

The critical surfactant size appears to be at a molecular weight between 305 and 330 daltons. With larger, slowly diffusing molecules, the nucleation thresholds remained identical to that of pure water; with smaller molecules, that diffuse faster, a clear decrease of the thresholds was obtained in most of the cases (Table 1). The lower concentration of sodium decyl sulfate was a notable exception. However, in this case the concentration also was insufficient to lower the equilibrium surface tension of the free solution surface. Thus, it would appear that with sufficiently small surfactant molecules, there is a lower "capture efficiency" for molecules arriving at the nuclei's interface.

Although the concentration of surfactant may determine to some extent the aggregation time, in most of the cases, the surfactant efficiency even at equilibrium is not directly related to concentration since the formation of multimolecular micelle aggregates becomes an important factor at certain concentrations. For example, with pure sodium dodecyl sulfate, the critical concentration for formation of micelles has been placed at about 0.2%; however, many solution properties relating to surface tension and solute mobility show aberrations below as well as above this concentration [26, 27]. This aggregation tendency of the surfactant molecules makes it difficult to quantify their rates of diffusion and their dynamic behavior at the interface. These difficulties are exaggerated by the great uncertainties which exist with respect to the critical size of the initial cavities. Nevertheless, it would appear that further detailed studies of the cavitation thresholds as a function of the surfactant properties will provide valuable information on the dynamic characteristics of the nucleation events.

The results obtained with the electrolytes and the sucrose add information about some other factors involved in the nucleation process. Despite the considerable decrease in the gas solubility, and hence, in the concentration of dissolved gas, caused the addition of electrolytes, the cavitation threshold essentially remained unchanged. Only with the most concentrated solution of magnesium chloride, in which the concentration of gas was decreased to about one-eighth that in pure water, was there a clearly significant change in the cavitation threshold, although also in this case the magnitude of the effect was small compared with the concentration change.

This near lack of an effect by solute-induced changes in gas concentration is in apparent contrast to the effect observed with gases of different solubilities in pure water [13, 14]. In this latter situation, solubility differences of equivalent magnitude as those caused by the solutes greatly affected the cavitation thresholds, which increased with decreased solubility. This problem is further amplified by the possibility that even the small effect obtained with the salts in part may be ascribed to other solute-water interactions, such as those leading to changes in the water structure. Indeed, the data indicate that this factor may be of significance. A prevailing concept is that salts of magnesium in particular but also of sodium increase the ordering or structure of the solvent water, while salts of potassium have a disordering effect [28]. In correspondence with such a pattern, the most concentrated solutions of magnesium salts and, to a lesser extent, sodium chloride, tended to have an increased cavitation stability, and that of potassium

chloride a perceptible decreased stability. An influence of water structuring on the nucleation process also was indicated earlier by the effect of temperature on the cavitation stability [13]. The sharp increase that occurred in the stability near 0°C hardly can be ascribed to factors other than the increased molecular ordering that probably occurs in the water near its freezing point [29].

This structuring influence may come about by one or more different types of action; increased structuring may lead to, for example, (1) a reduction of the density fluctuations in the water, leading to fewer structural "voids", (2) a decrease of the expansion rate of the subsequent cavities, preventing them from reaching the necessary critical dimensions, and (3) an impediment of the diffusive gas flux into the pre-stable cavities, thereby increasing their chance for recollapse. Any speculation as to which of these mechanisms, or even other ones, may be important is best deferred until further experimental data become available.

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