

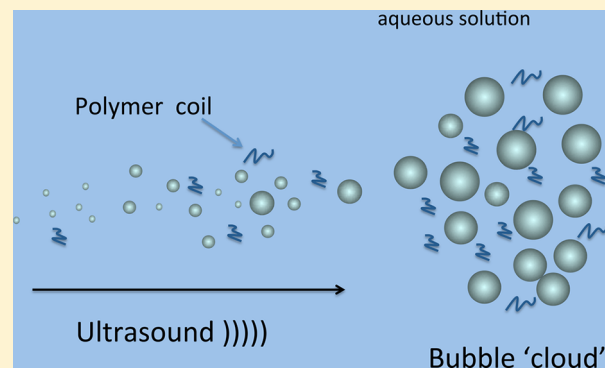
# The Behavior of Acoustic Bubbles in Aqueous Solutions Containing Soluble Polymers

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**ABSTRACT:** The effects of the water-soluble polymer polyvinylpyrrolidone (PVP) on the multibubble sonoluminescence (MBSL) intensity generated in aqueous solutions exposed to ultrasound at the two ultrasound frequencies of 20 and 363 kHz have been examined. In both cases, the presence of PVP, at concentrations of up to 2 g/100 mL, was found to enhance the MBSL intensity emitted from the solutions. On the basis of the intensity behavior of the SL observed from aqueous solutions containing PVP/surfactant and PVP/alcohol mixtures, it is suggested that PVP enhances MBSL by increasing the number of active bubbles in the system by hindering bubble–bubble coalescence processes and probably also by changing the structure of the bubble “clouds” formed at the acoustic antinodes in solution.

The influence of PVP on bubble–bubble coalescence rates was also measured to support the interpretation of the MBSL emission experiments.



## INTRODUCTION

Sonoluminescence (SL) is one of the “byproducts” of the complex processes that ensue from the high temperatures generated on the collapse of cavitation bubbles that are produced when high intensity ultrasound passes through a liquid.<sup>1,2</sup> The intensity of the SL generated is dependent on a number of parameters, e.g., ambient temperature, gases dissolved in the fluid, ultrasound frequency and power, solutes, etc. As SL is sensitive to several physicochemical conditions of a solution system, it can serve as a useful diagnostic indicator of the behavior of acoustic bubbles and so provide insight into the processes that control sonochemistry.<sup>3</sup>

A number of studies have examined the effects of surface-active solutes on multibubble sonoluminescence (MBSL) generated in aqueous solutions exposed to ultrasound.<sup>4–8</sup> Depending on the nature of the solute in solution and the type of cavitation generated by the ultrasound, either suppression or enhancement of the MBSL signal can be observed.<sup>8,9</sup> In general, in aqueous systems exposed to high frequency ultrasound, volatile surface-active solutes cause SL quenching. This quenching of SL has been linked to the thermal decomposition of solutes and the accumulation of decomposition products inside relatively long-lived bubbles undergoing “repetitive transient” cavitation.<sup>5,10–13</sup> At lower frequency (e.g., 20 kHz), the same degree of quenching is generally not observed, reflecting that SL emission is predominantly from bubbles with a lifetime of only a few acoustic cycles.<sup>7,9,14</sup> MBSL enhancement by charged surface active species has been attributed to either electrostatic interactions between charged bubbles increasing the number of bubbles that are directly affected by the acoustic field<sup>4,10</sup> or surfactant adsorption onto the bubble surface acting to retard bubble coalescence, resulting

in more bubbles of the size capable of generating SL.<sup>15</sup> Recent work suggests both processes can be in operation.<sup>16</sup>

The present study reports on the effects of a water-soluble polymer (PVP) on the SL intensity generated in aqueous solutions. This polymer differs from the solutes previously studied in that its surface activity and volatility are both negligible, physical properties that will prevent polymer molecules from evaporating into the bubbles and from concentrating at the bubble surface. The study is of relevance to the optimization of sonochemical processes, such as in the degradation of organic contaminants in wastewater. It also provides new insight into the phenomenon of bubble coalescence, of more general interest with respect to bubble–bubble interactions that occur in a number of diverse systems.

## EXPERIMENTAL DETAILS

**Materials.** All chemicals were used as received unless otherwise noted. Polyvinylpyrrolidone (PVP) samples with three weight average molecular weights ( $M_w$ ) 10 000, 29 000, and 55 000 g/mol were obtained from Aldrich. Propan-1-ol was analytical reagent grade and obtained from BDH. Sodium dodecylsulfate (SDS) was BDH specially pure grade. The water used to prepare all solutions was obtained from a three-stage Milli-Q purification system and had a conductivity of less than  $10^{-6}$  S cm<sup>-1</sup> at 20 °C and a surface tension of 72.0 mN m<sup>-1</sup> at 20 °C.

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**Procedures.** For SL measurements carried out using 20 kHz ultrasound, 100 mL of solution was placed in a cylindrical Pyrex cell of diameter 50 mm that was fitted over a 40 mm cup-horn transducer (Branson B-30 Cell Disruptor). One hundred milliliters of solution was sufficient to cover the horn completely to a depth of 35 mm. The power absorbed by the insonated solutions (with the generator operated in pulse mode), as measured by calorimetry, was in the range 0.09–0.2 W cm<sup>-2</sup>. The 20 kHz generator was modified in-house to allow it to function in a pulsed or continuous mode. Unless otherwise stated, experiments at 20 kHz were performed in pulsed mode, using 3.5 ms pulses and a duty cycle (on/off) of 1:5. The sonoluminescence intensity was recorded using a Hamamatsu end-on photomultiplier (model no. E849-34), responsive over a wavelength range of 300–650 nm. Signals were displayed on a digital oscilloscope (Tektronix, model no. TDS 360) and stored on a computer.

In a typical experiment, the transducer, reaction vessel, and photomultiplier were placed in a blackened box-enclosure to minimize the background light level. The SL intensities were obtained by first sonicating air-saturated solutions (30 s), and then allowing them to stand undisturbed for about 3 min. This conditioning procedure gave reproducible SL results. Ultrasound pulsing was then commenced again, and when the SL intensity had reached a steady level, the averaging function of the oscilloscope was used to record and average at least 100 pulses.

For 363 kHz SL experiments, 200 mL volumes of solution were placed in a pyrex cell, attached to the stainless steel plate of an Allied Signal transducer (model USW 51-52). The power to the transducer was supplied by an ELAC RF-Generator (model LVG 60). This unit only operated in continuous mode. The experimental procedure was similar to that for pulsed mode operation, but as the 363 kHz generator was unable to operate in a pulsed mode, the solution was conditioned with a 10 s burst of continuous wave US. SL intensity measurements were the average SL intensity measured from several bursts of about 0.5 s duration of continuous wave US. The power absorbed by the insonated solutions (in continuous mode) as measured by calorimetry was around 0.33 W cm<sup>-2</sup>. During experiments, the temperature was monitored using a Jenway electronic thermometer (model 2003, with a precision of 0.1 °C). Generally, the solution temperature did not change by more than 2 °C during the course of an experimental run.

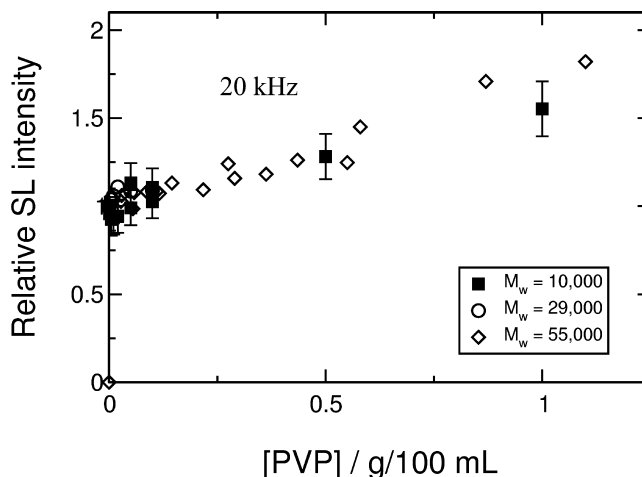
**Coalescence Measurements.** The relative coalescence measurements were conducted with a dilatometer similar to one used in our previous studies.<sup>11,16,17</sup> The dilatometer consisted of a glass cell with an approximate volume of 250 mL and a capillary (internal diameter of 0.9 mm) vertically attached to the top of the glass cell. An ELAC LVG-60 RF generator was used in conjunction with an ELAC Allied Signal plate transducer of diameter 54.5 mm. The ultrasound unit operated at 358 kHz. Each solution was sonicated for 15 s and the overall volume change measured. The temperature change under the conditions used was insignificant. The rate of volume change was 0.9  $\mu$ L/s and linear over at least 80 s.

## RESULTS

The MBSL signals from dilute aqueous solutions of PVP were studied with respect to concentration, average molecular weight ( $M_w$  of 10 000, 29 000, and 55 000 g/mol), and applied ultrasound frequency (20 and 363 kHz). Over the concen-

tration range examined, there was no significant change in the measured bulk viscosity of the aqueous solutions.

The SL intensity from PVP solutions as a function of wt/vol % and molecular weight with pulsed ultrasound at 20 kHz are shown in Figure 1. For each average molecular weight

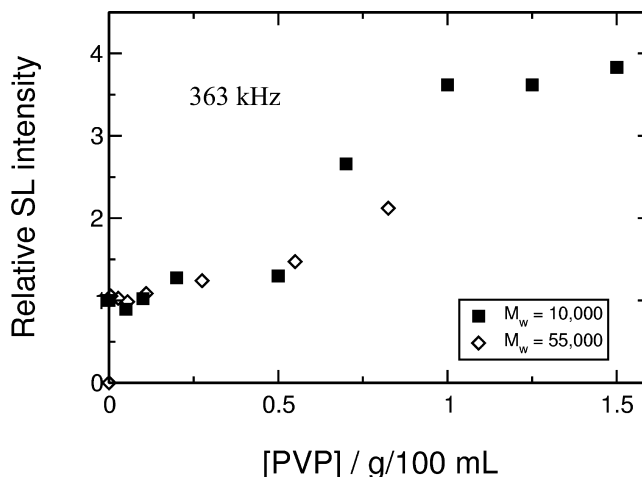


**Figure 1.** Averaged relative SL intensity as a function of PVP concentration ( $M_w$  = 10 000, 29 000, and 55 000 g/mol) using 3.5 ms pulses of 20 kHz ultrasound. SL intensity has been normalized with respect to the average SL intensity for water. A standard error of 10% is shown for  $M_w$  = 10 000 g/mol data.

examined, the SL intensity increased monotonically at a similar rate with respect to polymer mass amount (measured as solute weight with respect to solvent volume (g/100 mL)), rising to around 1.7 times the SL intensity of water at 1 g/100 mL.

The SL signal in the presence of PVP using 363 kHz is shown in Figure 2. As observed with 20 kHz, the rise in SL intensity with PVP percentage at high frequency appears to be independent of the average molecular weight of the polymer. The SL intensity rises to around 3.6 times that of water at a polymer level of 1 g/100 mL.

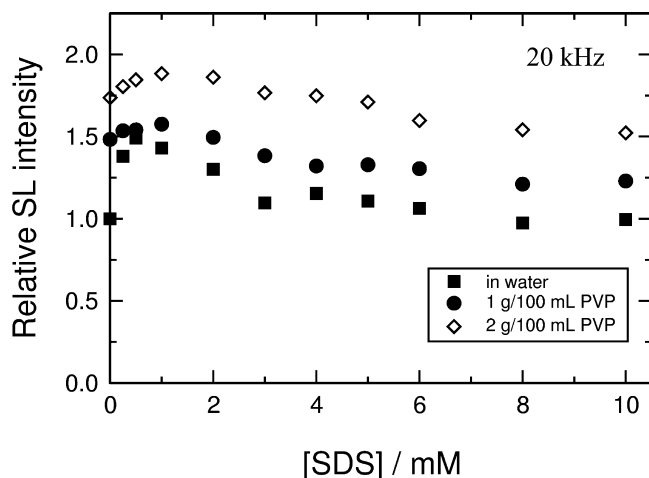
It has previously been shown that SL enhancement in the presence of the anionic surfactant sodium dodecylsulfate (SDS)



**Figure 2.** Averaged relative SL intensity as a function of PVP concentration ( $M_w$  = 10 000 and 55 000 g/mol) using 500 ms of 363 kHz ultrasound. Intensity has been normalized with respect to the average SL intensity for water.

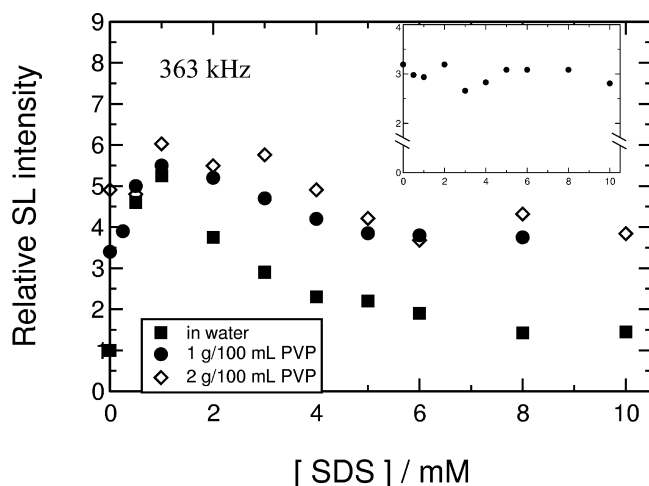
was attributable, in part, to interbubble electrostatic interactions, as demonstrated by screening the effect through the addition of electrolyte.<sup>4,10</sup> In order to see if electrostatic effects were also responsible for the increase in the SL in the polymer solutions, 0.1 M NaCl was added to PVP solutions at both 20 and 363 kHz. The presence of the salt had no influence on the intensity obtained in the PVP solutions. These results are as expected, as the polymer is neither charged nor surface active.

The effect of combining PVP and SDS is shown in Figures 3 (20 kHz) and 4 (363 kHz). In both cases, it can be seen that



**Figure 3.** Averaged relative SL intensity as a function of SDS concentration at two concentrations of PVP ( $M_w = 10\,000$  g/mol). Using 3.5 ms pulses at 20 kHz US and normalizing data to average SL intensity for water.

the same basic features exist in the presence of PVP as with SDS alone. Namely, as the SDS concentration is increased, the intensity of the SL signal rises and a maximum is reached at about 1 mM and then the signal intensity decreases to about

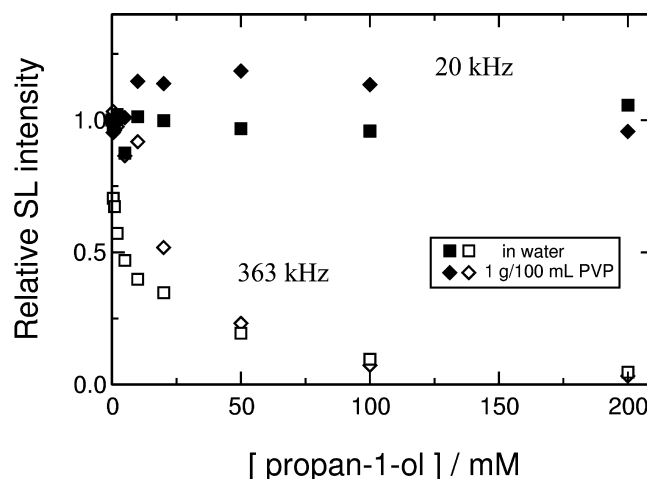


**Figure 4.** Averaged relative SL intensity as a function of SDS concentration at two concentrations of PVP ( $M_w = 10\,000$  g/mol). Using 500 ms pulses of 363 kHz US and normalizing data with respect to the average SL intensity for water. The inset shows averaged relative SL intensity as a function of SDS concentration in a solution of 1 g/100 mL PVP ( $M = 10\,000$  g/mol) and a background of 0.1 M NaCl. Using 500 ms pulses of 363 kHz ultrasound and normalizing data with respect to the average SL intensity for 0.1 M NaCl.

the level without SDS present. The relative effect of SDS on the SL signal is, however, less pronounced the higher the level of PVP present in solution.

When a 0.1 M electrolyte background is used for a system of SDS and 1 g/100 mL PVP (Figure 4, inset), changing the SDS concentration no longer has any effect on the SL intensity, as previously seen for SDS, and SDS with alcohols.<sup>4,7</sup> The resulting intensity corresponds to the SL intensity observed from a 1 g/100 mL sample of PVP in water (Figure 2).

The solid symbols in Figure 5 show the SL intensity behavior at 20 kHz US as a function of propanol concentration, and



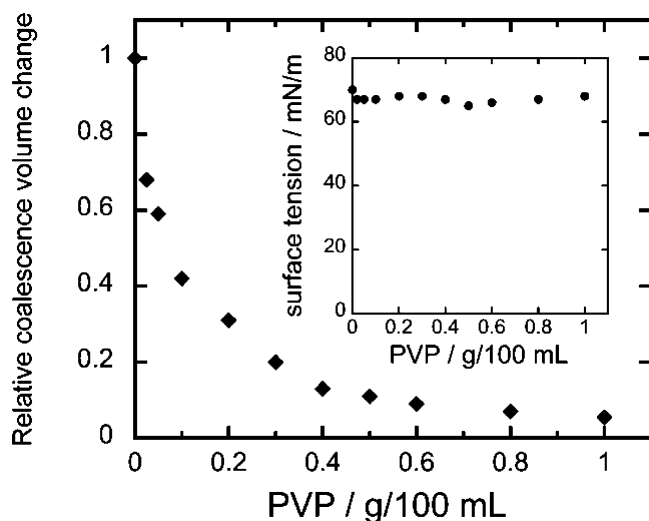
**Figure 5.** Averaged relative SL intensity as a function of propanol concentration in water and in 1 g/100 mL PVP ( $M_w = 10\,000$  g/mol) solutions, using 3.5 ms pulses of 20 kHz US (solid symbols) and 500 ms pulse of 363 kHz US (open symbols). The SL intensities for propanol in water have been normalized to the SL intensity of pure water, whereas the SL intensities for solutions in PVP have been normalized to the SL intensity for a solution of 1 g/100 mL PVP.

compares the relative SL intensity of propanol in 1.0 g/100 mL solutions of PVP ( $M_w = 10\,000$  g/mol) to solutions of propanol in water. It can be seen in both cases that the SL intensity is unaffected by the presence of propanol. At the higher frequency of 363 kHz, however, it can be seen that the relative SL intensity falls away with propanol concentration (Figure 5). The scaling in this figure demonstrates that the extent of this SL decrease is not altered by the presence of polymer.

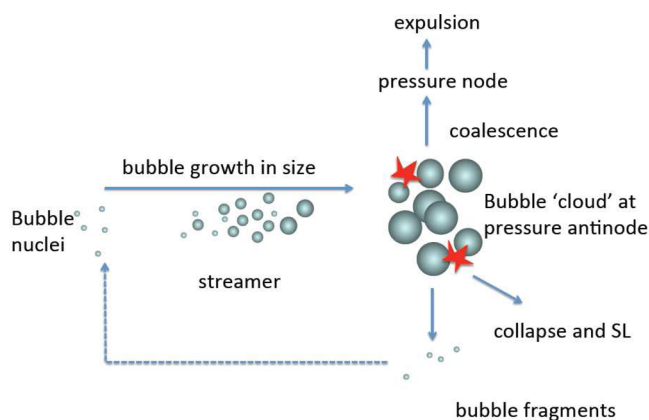
In order to estimate the effect of polymer on the relative degree of bubble coalescence in the system, the net volume change of the solution was measured for a fixed period of sonication. The volume change was linear with time over the time period examined. The results are shown in Figure 6 for PVP ( $M_w = 29\,000$  g/mol) over the wt/vol % range used in the SL experiments.

## DISCUSSION

For a qualitative interpretation of the various solute effects observed, a broad consideration of the behavior of acoustic bubbles in a multibubble system is helpful.<sup>11,18–20</sup> Figure 7 is an illustration of some of the main events that occur when a liquid is exposed to an ultrasound field. Adventitious bubble nuclei in a fluid when acted on by an oscillating pressure field will pulsate and grow in size. In a standing wave field, bubbles smaller than their resonant size are forced to the pressure antinodes in the system. This process creates bubble streamers (primary



**Figure 6.** Relative (to pure water) net volume change of PVP ( $M_w = 29\,000$  g/mol) aqueous solutions sonicated for 15 s as a function of PVP content. (In pure water, the absolute volume change was  $0.9\ \mu\text{L/s}$ .) The inset shows the surface tensions (using the pendant drop method with a DataPhysics OCA tensiometer) of PVP ( $M_w = 29\,000$  g/mol) solutions at  $24 \pm 1\ ^\circ\text{C}$ .



**Figure 7.** Schematic representation of the generation of bubble “clouds” in a liquid by an acoustic field and subsequent processes associated with the bubbles in these clusters. See text for details.

Bjerknes forces), culminating in bubble “clouds” at the antinodes in the system. Bubbles smaller than their resonance size will also oscillate in phase with the sound field and if close enough together will set up hydrodynamic flow conditions (secondary Bjerknes forces) that draw them together. In the high bubble-volume-fraction regions of the bubble clouds, and also possibly in the streamers, bubble–bubble coalescence will occur. Bubbles larger than their resonance size will oscillate out of phase with the sound field and be driven to pressure nodes in the system, or fragment if large enough, or be expelled from solution due to their buoyancy. Bubbles that undergo large radial excursions in a sound field can collapse inertially, producing localized “hot spots” from which SL is emitted and also where sonochemistry is initiated. These bubbles are mainly located in the bubble clouds at the pressure antinodes. Also, the bubble clouds effectively reflect acoustic waves and the outer bubbles of these clusters are an impedance to sound propagation through the bubble field.<sup>19</sup> The consequence of this is that only a relatively small proportion of bubbles exposed

to an acoustic field are of a size that undergoes violent collapse (active bubbles) and hence initiate sonochemical events.

Previous MBSL studies have attributed SL intensity increases to either increases in the number of active bubbles undergoing collapse (as suggested in the case of SDS solutions<sup>4,15</sup>) or solute affected changes to bubble dynamics (with respect to an increase in the radial expansion of a bubble or the symmetry of collapse) that may increase the maximum temperature reached inside a bubble during collapse. The results of Figure 5, however, would rule against the latter of these two possibilities. The data shown in Figure 5 demonstrate that the quenching effect of the alcohol is the same in the presence and absence of PVP. At high frequencies, alcohols evaporate into oscillating bubbles and build up hydrophobic combustion products over many expansion–collapse cycles.<sup>5,12</sup> As a result, the bubble temperature is reduced and the SL intensity diminished. At lower frequencies, the bubble population is dominated by transient cavities that undergo only a few oscillations<sup>8,9,14</sup> and therefore do not build up combustion products and SL quenching does not occur. The close similarity in the way that propanol affects the SL in solutions with and without PVP present is strong evidence that bubble dynamics are not seriously affected by the PVP in solution.

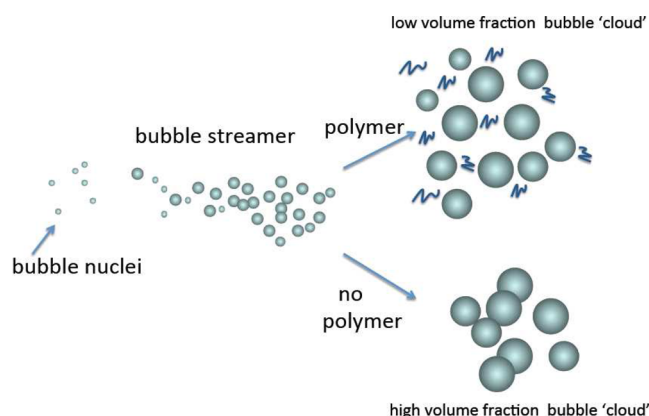
On examining the SL intensity trends obtained on combining SDS with PVP (Figures 3 and 4), it can be seen that as the level of PVP is increased the base SL intensity increases relative to the SL intensity in water alone, whereas the maximum SL intensity reached for all three systems stays almost the same. This maximum intensity also occurs around about the same SDS concentrations irrespective of whether or not polymer is present. If the effects from polymer and surfactant on the sonoluminescing bubbles were independent of each other, then it would be expected that the resulting SL intensity is a superposition of the intensities seen independently for each of PVP and SDS at their respective concentrations. This indicates that the effects of SDS and PVP on the SL intensity are not independent and suggests that both SDS and PVP act on the same population of bubbles.

For bubble systems in the presence of SDS alone, it has been concluded that interbubble electrostatic interactions, due to adsorbed dodecylsulfate ions onto the bubbles in solution, hinder bubble coalescence processes as well as creating spatially more open bubble clouds. It has been suggested that this latter condition reduces the acoustic impedance experienced by bubbles within such clouds and increases the relative number of active bubbles.<sup>4</sup> However, interbubble electrostatic interactions cannot be responsible for the resultant increase in SL in PVP solutions because adding a background level of electrolyte (0.1 M NaCl) to the PVP system does not change the SL intensity significantly from the same system in the absence of salt (Figure 4).

In systems containing PVP and SDS, it is known that complexes between the polymer and surfactant form. Over the range of SDS concentrations used in the present study, these complexes range from simple surfactant polymer complexes to polymer chains possessing hemimicelle structures spaced along the polymer backbone.<sup>21,22</sup> It is difficult to assess the influence such structures have on the bubbles. However, the observation that the SL intensities at high SDS concentrations (10 mM) in PVP solutions, in Figures 3 and 4, are about the same as the intensities in the absence of SDS would suggest that the complexes do not impart any additional effects on the behavior of the bubble clouds in the polymer systems.



A model that allows a rationalization of the way the soluble polymer may act to affect bubbles in an acoustic field is shown in Figure 8. The physical presence of polymer molecules in the



**Figure 8.** Diagrammatic representation of the generation of low volume-fraction bubble “clouds” in the presence of soluble polymer (not to scale) during sonication. Close approach of bubbles to each other and polymer molecules during and post streaming may lead to a different spatial structure of the bubbles in the clouds than in the absence of polymer.

liquid film between colliding bubbles can act to hinder bubble–bubble coalescence. A number of studies examining the forces of interaction between deformable interfaces (drops, bubbles) in aqueous solutions have identified the existence of solute induced structural forces during the collision process.<sup>23,24</sup> These structural forces arise because nanoscale solutes, e.g., polyelectrolytes, surfactant micelles, and nanoparticles, can interact with each other during film thinning, thereby inhibiting the rate of coalescence. As a result, bubbles are less likely to approach as close to one another as they would in the absence of PVP (and SDS), and the resulting bubble cluster is likely to be a relatively more open bubble cloud (analogous to the bubble cloud produced in the presence of low concentrations of SDS), thereby increasing the number of active bubbles.

On the basis of the model depicted in Figure 8, the trends observed in Figures 1 and 2 can be interpreted. Figures 1 and 2 indicate that a steady, and almost linear, increase in SL intensity with increasing polymer content (g/100 mL) is a basic feature of solutions containing the water-soluble polymers, over the solute level and weight average molecular weight range studied. As the trend is linear in wt/vol %, irrespective of the polymer molecular weight, it indicates that the effect is molar concentration dependent. That is, for a given mass of polymer, the higher molecular weight of the PVP, the greater the effect on the SL increase and that, in turn, implies that the effect is polymer size dependent.

Some appreciation of this can be gained by considering the polymer size and the interpolymer spacing for different polymer  $M_w$  and amounts. These are listed in Table 1. The polymer size has been calculated assuming that each polymer molecule is randomly coiled (with the added restriction that bond angles are 109.5°); the spacing between the two ends of the polymer molecule is given by the root mean squared ( $R_{rms}$ ) distance

$$R_{rms} = (2N)^{1/2}l \quad (1)$$

where  $N$  is the number of monomer units in the polymer and  $l$  ( $=2.5$  Å) is the average length of a monomer unit.<sup>25</sup>

**Table 1.** Polymer Coil Size ( $R_{rms}$ ) and Separation Dimensions (Center-to-Center) of PVP for Selected Solution Levels and  $M_w$

$M_w$ (g/mol)	number ( $N$ ) of monomer units	$R_{rms}$ (Å)	separation spacing 0.5 g/100 mL (Å)	separation spacing 1.0 g/100 mL (Å)
10 000	90	33	150	118
29 000	261	57	213	169
55 000	495	79	264	209

The values in Table 1 reveal that the average spacing between polymer molecules is comparable to the polymer size at concentrations where significant changes in SL are observed. It can also be seen that for a particular amount of polymer in solution the spacing between polymer coils increases as the polymer  $M_w$  increases. For example, at 1 g/100 mL, the spacings between polymer coils (center-to-center separation,  $R_{rms}$ ) are 85, 112, and 130 Å for  $M_w$  of 10 000, 29 000, and 55 000 g/mol, respectively. This quantitatively reinforces the observation that polymer size is an important controlling factor in the effect of SL enhancement and not just concentration.

Finally, the reduction in rate of bubble coalescence (Figure 6) is strong support of the proposal that water-soluble polymers hinder bubble coalescence, thereby raising the number of active bubbles in the system. Previous work with polyethylene oxide ( $M_w = 100\,000$  g/mol), where high-speed photographic images of cavitation bubbles were recorded, showed quite diffuse bubble cloud images compared with bubble clouds in pure water,<sup>26</sup> further evidence that water-soluble polymers act to keep bubbles apart.

## CONCLUSIONS

The enhancement in intensity of SL with increasing concentration of the water-soluble polymer PVP, at both high and low ultrasound frequencies, can be attributed to the polymer hindering the process of bubble–bubble coalescence. It is suggested that the presence of polymer in the film between colliding bubbles provides a structural disjoining pressure and this acts to retard coalescence, yielding more active bubbles and/or lower volume fraction bubble clusters. Acoustic bubbles have a relatively short lifetime ( $<1$  ms); nevertheless, the polymer effect observed here can be expected to operate in any multibubble swarms. From a sonochemistry point of view, it can be taken that increases in yield of a particular cavitation bubble process in aqueous solution may be enhanced on the addition of a water-soluble polymer to the system.

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### Notes

The authors declare no competing financial interest.

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