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P. G. Chaphalkar^a; K. T. Valsaraj^a; D. Roy^a

^a DEPARTMENTS OF CIVIL AND CHEMICAL ENGINEERING, LOUISIANA STATE UNIVERSITY, Baton Rouge, Louisiana

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Flotation Using Microgas Dispersions for the Removal of Pentachlorophenol from Aqueous Solutions

P. G. CHAPHALKAR, K. T. VALSARAJ,* and D. ROY

DEPARTMENTS OF CIVIL AND CHEMICAL ENGINEERING

LOUISIANA STATE UNIVERSITY

BATON ROUGE, LOUISIANA 70803

ABSTRACT

Microgas dispersions called colloidal gas aaphrons (CGAs) were generated using cationic, anionic, and nonionic surfactants, and were used in an adsorptive bubble flotation process in a semibatch mode to remove pentachlorophenol (PCP) from the aqueous phase. The aqueous solution was maintained at pH values by using buffers. CGAs generated with Tergitol, which is a nonionic surfactant, were found to be the most efficient for the removal of PCP; the efficiency remained nearly independent of pH. In the case of an anionic surfactant, sodium dodecylbenzene sulfonate (DDBS), the efficiency of removal improved from 15 to 36% with a change in pH from 10.1 to 3.0. For a cationic surfactant, hexadecyltrimethylammonium bromide (HTAB), the removal at pH 10.1 was 81%, which decreased to 68.1% at pH 3.0. The charges on the encapsulating film of CGAs may explain the higher percentage of adsorption of PCP on the CGAs generated using HTAB as compared to CGAs generated using DDBS. For all the surfactants, an increase in concentration improved the removal efficiency. These results were compared with the removal efficiencies using conventional flotation techniques used by other researchers. Solvent sublation appears to be effective in the removal of PCP, but even in the presence of a surfactant it required 300% more air volume per volume of liquid when compared with CGA flotation.

INTRODUCTION

Pentachlorophenol (PCP) is a priority pollutant that has been used extensively in agricultural and industrial applications. PCP contamination

* To whom correspondence should be addressed.

of water and sediments is well documented in the literature. In a study conducted in Michigan, 156 of the 223 industrial and municipal sewage sludges were found to have PCP concentrations ranging from 0.2 to 8490 mg/kg (1). A USEPA report (2) indicates that acute and chronic toxicity to freshwater aquatic life due to PCP occurs at concentrations as low as 55 and 3.2 $\mu\text{g}/\text{L}$, respectively. For saltwater aquatic life, the corresponding values are 53 and 34 $\mu\text{g}/\text{L}$. The recommended level in drinking water is 1.01 mg/L, but the undesirable taste and odor threshold in water is 30 $\mu\text{g}/\text{L}$.

In this study the effectiveness of a modified flotation method that uses microbubble dispersions to remove PCP from an aqueous stream was studied and compared with the conventional flotation process. Diffused aeration has long been utilized to remove volatile and surface-active organics from wastewaters. However, for surface-inactive compounds called "colligends," a surface-active material or surfactant needs to be added to the solution to form a complex with the compound, making it hydrophobic, so that it is adsorbed at the air bubble surface in a flotation process. This process is called foam flotation (3). The adsorbed compound at the interface then rises to the surface with the bubble and is concentrated at the surface in the foam formed atop the aqueous phase, from where it can be skimmed off. This method is especially effective for the separation of materials at low concentrations.

The microgas dispersion utilized in this study is called colloidal gas aphrons (CGAs). This name was proposed by Sebba (4). These bubbles display different properties than the conventional gas cavities in aqueous solutions (air bubbles) because of the nature of its encapsulating film. The most striking feature of a CGA suspension is its small bubble size, which yields a very large specific surface area per unit volume. CGA suspension entraps around 66% air by volume but is stable enough to be pumped from the point of generation to the point of application. CGAs therefore have been shown to be useful in a variety of environmental applications. They have been used in the microflotation of colloids and in the removal of heavy metal ions from wastewater using precipitate flotation (5-7). Roy et al. (8) recently demonstrated an application of CGA flotation in separating synthetic dyes from an industrial waste.

In this study CGAs were generated using cationic, anionic, and nonionic surfactants in concentrations below and at their critical micellar concentration (cmc) values. CGAs thus generated were used in an adsorptive bubble flotation process to remove PCP from a buffer solution at three different pH values. These results were compared with removal efficiencies using conventional flotation techniques.

EXPERIMENTAL

Chemicals

A pH 10.1 buffer was prepared by the addition of 11 mL of 1 N NaOH to a 0.025 M NaHCO₃ solution, and a pH 3.0 buffer was prepared by adding 4 mL of 1 N H₂SO₄ to a 0.005 M NaH₂PO₄ solution. pH 7.4 solution was prepared from a 10% stock solution of 0.087 M KH₂PO₄ and 0.304 M Na₂HPO₄. PCP stock solution was prepared by dissolving 0.8 g in 100 mL methanol. To achieve an 8 mg/L concentration of PCP in the aqueous solutions, 1 mL stock was diluted to a liter.

CGAs were generated using Tergitol 15-S-12 (Union Carbide), sodium dodecylbenzene sulfonate (Sigma), and hexadecyltrimethylammonium bromide (Eastman Kodak). Tergitol is a nonionic surfactant that has an average chain length of 15 carbon atoms with a cmc value of 0.15 mM (MW = 738). Sodium dodecylbenzene sulfonate (DDBS) is a salt of dodecylbenzene sulfonic acid with a chain of 12 carbon atoms. It is an anionic surfactant with a cmc value of 1.5 mM (MW = 348.5). Hexadecyltrimethylammonium bromide (HTAB) is a cationic surfactant with a chain of 16 carbon atoms and has a cmc value of 0.9 mM (MW = 364.6).

Production of CGAs

Based on methods suggested by Sebba (9), a unit was fabricated in our laboratory for the production of CGAs. Briefly, it consists of a 0.5 hp motor fitted on top of a cylindrical container that held approximately 3 L liquid. A flat disk, 50 mm in diameter, is mounted at the end of a shaft. The level of the surfactant solution is initially adjusted to approximately 15 mm above the disk, which can rotate up to 8000 rpm. Two flat baffles are fixed to the lid of the container. The disk, when rotated at very high speeds, creates strong waves on the liquid surface. They strike the baffles and entrain air in a form of a microbubble dispersion or colloidal gas aphrons (CGA) upon reentering the solution. The size distribution of CGAs was determined by using a particle size analyzer (Microtrac 7995-10, Leeds and Northrup). A detailed description of the CGA apparatus and size distribution can be found in our previous work (10).

Experimental Setup

A glass column, 100 cm in height and with an internal diameter of 8 cm, was used for the flotation experiments. The height of liquid in the column was maintained at 60 cm, which gave a total liquid volume of 2.8 L. Two stopcocks were positioned at 15 and 45 cm from the bottom to

allow samples to be collected from different heights. A pulse of 50 mL CGA suspension was pumped from the bottom of the column with a Masterflex positive displacement pump (Cole Parmer Inc.) at a constant flow rate of 35 mL/min. This was followed by a period of 10 minutes during which no CGAs were introduced, thus allowing the bubbles to rise to the top. Samples were collected from the bottom and top sections, but since the difference in the concentrations was found to be insignificant, only the concentrations of samples collected from the top part are reported.

Analysis

Analysis of pentachlorophenol was performed using a Hewlett-Packard 1050 liquid chromatograph equipped with a multiple wavelength detector. A reverse phase Envirosep-PP (125 × 3.2 mm) column by Phenomenex was used for chromatography. The wavelength for detection was set at 213 nm with a 6-nm bandwidth, and the reference wavelength was set at 400 nm with an 80-nm bandwidth. The mobile phase flow was set at 0.5 mL/min and consisted of 90% methanol and a 10% solution of 0.025 M NaH₂PO₄, which was adjusted to pH 3 by the addition of 1 N H₂SO₄.

RESULTS AND DISCUSSION

Structure of a CGA

The fundamental properties of CGAs were studied and discussed by Sebba (11). An air bubble in pure water can be treated as a cavity enclosed by water. When surfactant molecules are present in water, any cavity introduced in water creates a water-gas interface where surfactants are adsorbed. In the case of colloidal gas aphrons, Sebba hypothesized that the encapsulating soap film has an inner as well as an outer surface, and that these surfaces have surfactant monolayers adsorbed on them. In a separation processes that utilizes CGAs, solubilization of the organics occurs in these layers. The encapsulation of a microbubble in a double layer of surfactant molecules, as observed with CGAs, retards its coalescence, increasing its stability. "Stability" of microbubble dispersion can be defined as the length in time over which the number of bubbles and their size distribution remain constant. CGA dispersions, if left undisturbed, will eventually cream and be converted to ordinary foam, leaving a clear aqueous layer below.

Results of Particle Size Analysis of CGAs

The detailed results of particle size analysis can be found in our previous publication (10). In most cases, CGAs were found to have diameters be-

tween 30 and 300 μm , rarely falling below 30 μm . The mean diameter (MD), which is calculated from the mean volume of the suspension, was found to be characteristic of the surfactant and affected slightly by its concentration. The volume of sample material in the system is reported by the instrument as a dimensionless parameter V_s . The quantity V_s was therefore used as a surrogate parameter to represent both the ability of the CGA suspension to entrap air over time and its stability in the system. The data presented in Table 1 lists MD, standard deviation, and initial V_s of a CGA suspension. From Table 1 it is apparent that CGAs generated from nonionic Tergitol have a larger air content than those generated from ionic DDBS or HTAB.

The data also indicate that the variation in the range of mean diameters of the CGAs and the initial air entrapment in the dispersion was specific to the surfactant. The concentration of surfactant in the solutions used to generate CGAs was found to influence the characteristics of the microbubble dispersions. In general, the increase in concentration of ionic surfactants was found to reduce the mean diameter and increase the V_s of suspension for all three surfactants (10).

Ion Flotation

Addition of surfactant to water beyond a certain value changes the physical characteristics of the solution. The concentration at which these changes occur is called the critical micellar concentration (cmc). Physically, aggregation of long chain surfactants into large units, called micelles, begins to occur at that concentration, and they behave as colloids (12). The colloids thus formed are capable of solubilizing otherwise insol-

TABLE 1
Initial Size Characterization of CGAs Produced with Different Surfactants^a

Surfactant	Concentration (mg/L)	V_s	Particle size analyzer results	
			MD (μ)	Standard deviation
Tergitol	50	0.022	63.0	29.0
Tergitol	100	0.1163	68.6	32.0
HTAB	200	0.0102	148.1	78.6
HTAB	328	0.0183	105.9	57.2
DDBS	200	0.0072	131.9	77.3
DDBS	500	0.0134	119.2	57.9

^a V_s = initial volume of CGA suspension in the particle counter cell (mL). MD = mean diameter.

ble organic molecules from the suspension by incorporating them into the micellar structure. At surfactant concentrations below the cmc, data available in the literature show essentially no enhancement in organic solute solubilization for relatively soluble organics. However, in the case of water-insoluble organic solutes, a significant increase in its solubility has been reported even in the presence of surfactant concentrations below its cmc (13). The relationship of moles of solubilizate with moles of surfactant is linear over a wide range of concentrations above the cmc. Although micelles may solubilize organics, unlike monomers they have no tendency to adsorb at the air–water interface.

In an ion flotation process, gas cavities are introduced in an aqueous solution that contains a dissolved surface-active agent. These molecules are adsorbed at the gas–water interface with their hydrophilic end oriented toward water, which enables them to form an ion pair with oppositely charged species in the solution. This attraction can be purely columbic or a chemical bond may form between the two. The solute-laden bubble is then buoyed to the surface where it will concentrate the ions in the froth. However, once the surfactant concentration reaches cmc levels, separation efficiency reduces dramatically because micelles have no tendency to adsorb at the air–water interface and therefore do not float with the bubble. Efficiency of removal drops because micelles solubilize the organics and retain them in the aqueous solution. Therefore, in an ion flotation process it is important that the surfactant should be at a concentration below cmc.

In the case of separation with CGAs, the mechanism of flotation is somewhat different. The process may involve either solubilization of the solute within the film surrounding a CGA or adsorption on the film surface, or a combination of both. The adsorption and/or solubilization by a CGA occurs because of the unique structure of CGA as described above. Figure 1 illustrates the solubilization process schematically in the presence of micelles and CGAs. When the concentration of surfactant in the aqueous solution is below its cmc, solute molecules will be distributed between CGAs and the monomers. At concentrations above cmc, surfactant molecules will form micelles, but because the CGAs are in high concentration, the solute will distribute mostly between CGAs and monomers. Theoretically, it is therefore possible to achieve separation by using CGA dispersions even at concentrations above cmc.

Effects of Surfactant Type on the Removal of PCP

Pentachlorophenol is a weak hydrophobic acid with a pK_a of 5.25. At $pH < 2.7$, only about 0.1% of PCP remains in ionic form, while at $pH >$

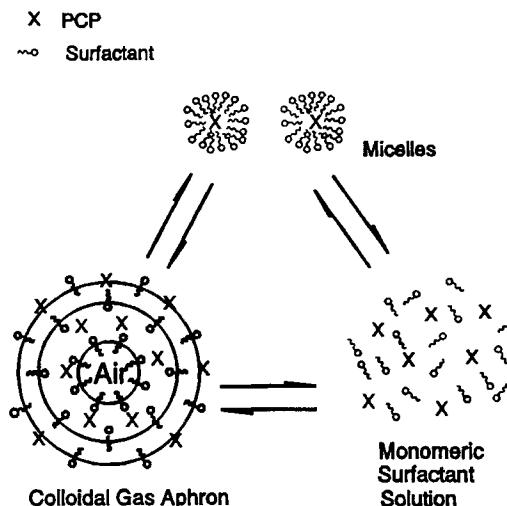


FIG. 1 Schematic of the solubilization process in the presence of CGAs and micelles.

10 nearly all of it will be in the dissociated form. Its sodium salt is highly water soluble, but in its pure form its solubility depends on pH and is reported to be 14 mg/L under acidic conditions. The initial concentration of PCP in these experiments was 8 mg/L, which ensured that all of the PCP was in the dissolved form.

The charge on the encapsulating film was thought to affect the removal efficiencies of PCP from the solution. To study this effect, CGAs generated from the three types of surfactants were used in the separation process. Figure 2 shows the percentage removal at pH 10.1 using CGAs produced at the cmc concentrations. The reason for the higher removal by Tergitol may be its small bubble diameter in the suspension and higher air entrainment (Table 1) even when it is mixed in a solution. A smaller bubble size would not only yield a higher surface area but the bubbles would also tend to rise more slowly to the surface, thereby increasing the contact time. A higher surface area coupled with a greater contact time would result in better removal rates of PCP. The large difference in the removal efficiencies of HTAB and DDBS, however, cannot be attributed solely to the above factors. The difference in their mean diameter and V_s is not large enough to justify the disparities in the removal efficiencies. The charges on the encapsulating film can explain the higher percentage of adsorption of PCP on the CGAs generated by the HTAB as compared to CGAs generated using DDBS. At pH 10.1, most of the PCP will be

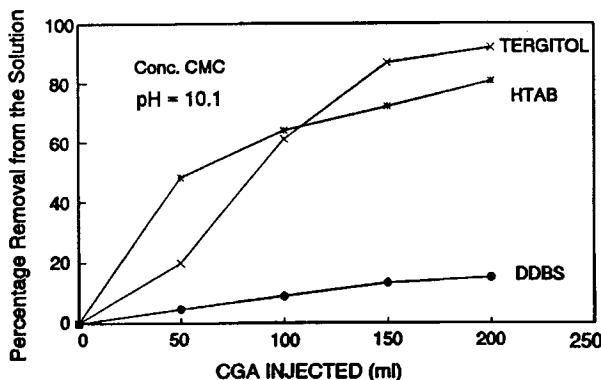


FIG. 2 Removal of PCP using CGAs formed from different surfactants under alkaline conditions.

present in the ionic form, and ion pairing with HTAB molecules in the CGA film will improve its removal efficiency. Repulsive forces between DDBS and the phenolate ion would hinder the adsorption process, yielding poor recovery.

Effect of pH on the Removal of PCP

Aqueous solutions were maintained at pH 3.0, 7.4, and 10.1 by using buffers to study the effect of ionization of PCP on the removal efficiencies. Figure 3 shows the effect of pH on the removal of PCP from aqueous solution under these conditions. It can be seen that the effect is more pronounced for ionic surfactants. In the case of Tergitol, which is a non-ionic surfactant, the removal is nearly independent of pH. In the case of anionic DDBS, the removal percentage improved from 15.1 to 35.6%, when the pH was changed from 10.1 to 3.0. The net negative surface charges on CGAs generated with the anionic surfactant may reduce the formation of phenolate-surfactant complexes, thus producing negligible removal under basic conditions when only phenolate ions exist in the aqueous solution. However, the hydrophobic nature of PCP under acidic conditions will enable adsorption of PCP on the CGA film, yielding higher removal. This conjecture appears to be supported by the removal efficiencies of CGAs generated from the cationic surfactant HTAB. The removal at pH 10.1 is 80.8% while at pH 3.0 it drops to 68.1%.

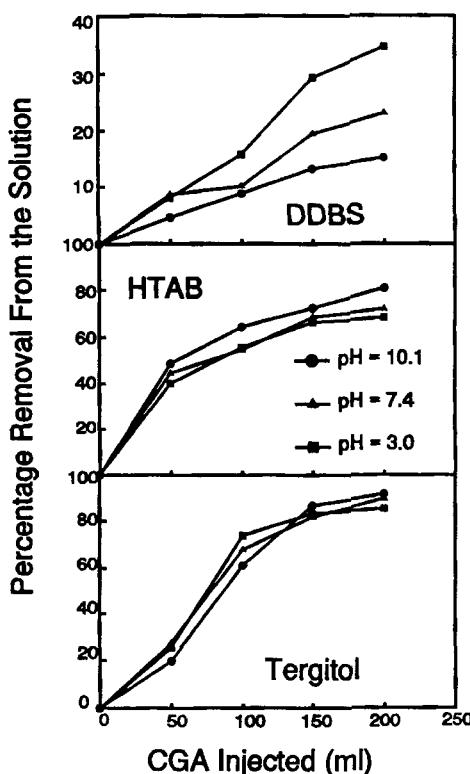


FIG. 3 Comparison of surfactant type in the removal of PCP using CGAs formed at their cmc concentration.

Figure 4 compares the removal of PCP by CGAs generated from all three surfactants at pH 3.0 at their cmc concentration. Although under acidic conditions virtually all the PCP is in the undissociated form, the removal efficiency using Tergitol is higher, again because of the smaller size and far higher air entrapment for CGAs produced using Tergitol as compared to ionic surfactants. However, the difference in the efficiencies of HTAB and DDBS cannot be ascribed to these factors. As Table 1 shows, initial MD and V_s values are nearly comparable for both surfactants, so the difference in efficiencies can be attributed only to differences in their ionic nature. It is most likely that because of its positive charges, HTAB forms complexes with PCP and is able to remove it more efficiently.

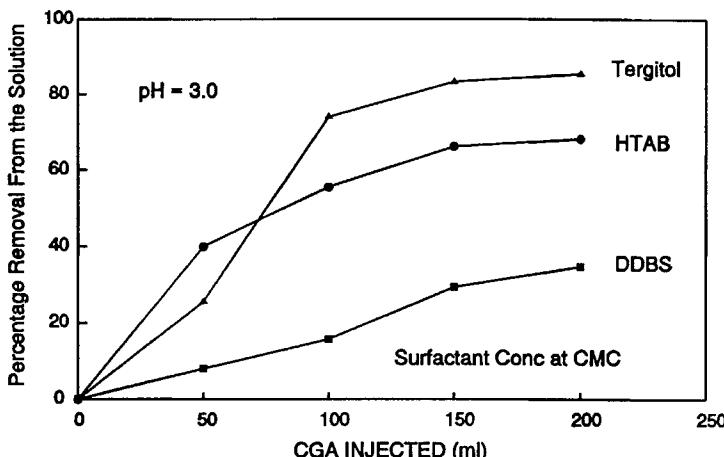


FIG. 4 Removal of PCP using CGAs formed from different surfactants under acidic conditions.

Effect of Concentration of Surfactant on the Removal of PCP

The removal efficiencies for all three surfactants at various concentrations are shown in Figs. 5 through 7. As expected, for all surfactants an increase in their concentrations improves the removal efficiency. The removal of PCP from the solution decreased with each pulse of CGA, except in the case of Tergitol at the cmc concentration.

For HTAB (Fig. 5), the removal increased about 10% with an increase in its concentration from 200 to 328 mg/L (cmc) at all pH values. As mentioned above, for both concentrations of HTAB the removal is higher under alkaline conditions. Figure 6 shows the removal of PCP by CGAs produced with DDBS at different concentrations. At 200 mg/L DDBS, removal was negligible at all pH values. Apparently the CGAs generated from this anionic surfactant at concentrations below its cmc failed to adsorb PCP even at pH 3.0, when PCP in the solution exists in its hydrophobic undissociated form. However, at the cmc concentration the removal was a modest 34.6% and seemed to increase with each pulse, so it is conceivable that removal can be further increased with additional pulses of CGAs. Similar results were obtained for CGAs generated with Tergitol (Fig. 7). The removal in the first pulse was the same for both concentrations of Tergitol. However, at the cmc the incremental removal by the

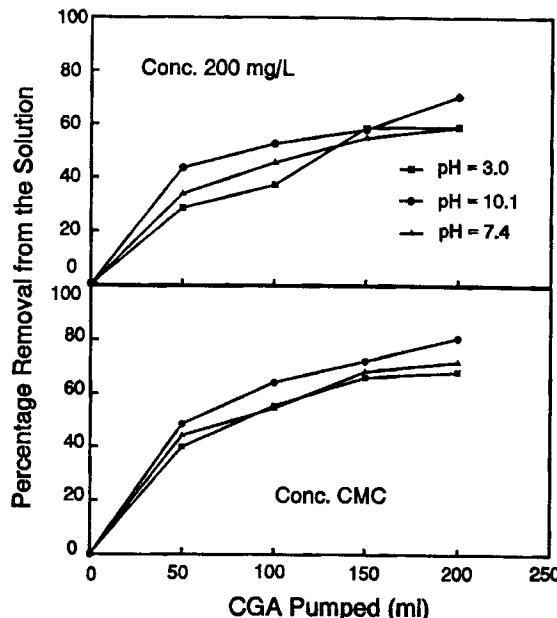


FIG. 5 Removal of PCP using CGAs formed using a cationic surfactant, HTAB.

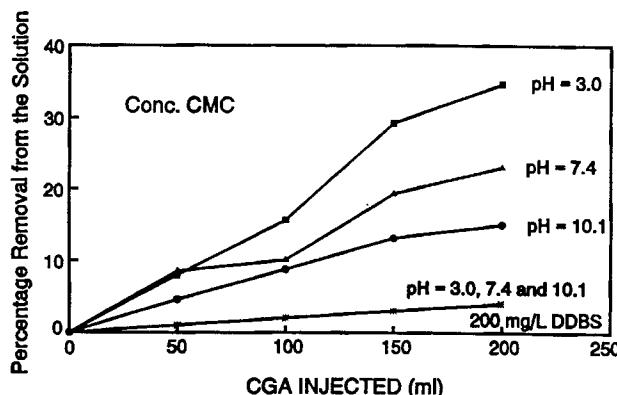


FIG. 6 Removal of PCP using CGAs formed using an anionic surfactant, DDBS.

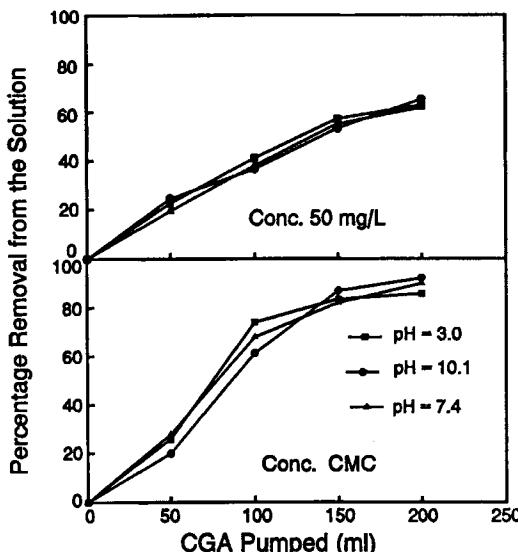


FIG. 7 Removal of PCP using CGAs formed using a nonionic surfactant, Tergitol.

second pulse is better than that of the first pulse. The incremental removal is seen to reduce again in the third and fourth pulses.

The increase in removal at the micellar concentrations of the surfactants above their cmc indicates that the adsorption of PCP must be occurring on the film of CGA in the aqueous phase much more than being solubilized in a micelle. Since micelles do not float, the removal in an ion flotation process is reported to drop precipitously when the solution reaches micellar concentration. That does not seem to be the case when using CGAs because PCP concentration in the encapsulating film may be far in excess of that within the micelle (Fig. 1).

Comparison of CGA Suspension for Flotation with Conventional Techniques

There are other conventional flotation techniques that utilize the surface-active nature of compounds to separate them at the air–bubble interface from the aqueous phase. The simplest form of this technique is air stripping. In this technique a porous stone or a glass frit is used to introduce air bubbles at the bottom of a column. The hydrophobic organics adsorb on the rising bubbles, effecting their removal. This method is extremely inefficient in practice for nonvolatile organics because of the lack

of adsorptive capacity of the bubbles and the redistribution of solute back into the aqueous phase at the surface when the bubble bursts. Solvent sublation, which was first introduced by Sebba (14), is another technique that overcomes this drawback. In this technique a layer of organic solvent is maintained atop the aqueous phase to prevent redistribution. In the case of CGA flotation, the chances of redistribution of solute back to the aqueous phase are reduced because the gas dispersion, after rising to the surface, forms an ordinary foam and the solute remains adsorbed in that matrix.

Table 2 presents data for a comparison of flotation by CGA and conventional techniques as carried out by Valsaraj and Thibodeux (15). The initial concentration of PCP in the aqueous phase was nearly the same in both cases. To compare the efficiency of removal under different conditions, the ratio of volume of air injected to the volume of aqueous solution in the column (V_G/V_L) was used. Since both experiments were carried out in a semibatch mode but for different liquid volumes, this ratio would give us an absolute volume of air per liquid volume required for a given degree of removal of PCP. The volume of air, V_G , in the case of CGA was calculated from the observation that nearly 66% of a CGA suspension volume is comprised of air.

TABLE 2
Comparison of Semibatch Flotation of PCP by Different Techniques*

Method of flotation†	Efficiency <i>E</i>	<i>V_L</i> (mL)	<i>V_G/V_L</i>	pH
Air stripping ^a	0.05	1450	19.1	3.0
Solvent sublation ^b	0.80	1450	17.4	3.0
Solvent sublation ^c	0.98	800	0.45	3.0
Solvent sublation with HTAB ^d	0.98	800	0.15	8.9
CGA (HTAB) ^e	0.68	2800	0.05	3.0
CGA (HTAB) ^e	0.85	2800	0.05	10.1
CGA (Tergitol) ^f	0.81	2800	0.05	3.0
CGA (Tergitol) ^f	0.92	2800	0.05	10.1

* V_L = total volume of aqueous phase in the column (mL). V_G = total volume of air injected into the column to achieve the indicated removal efficiency, E (mL). Q_a = air flow rate, mL/s.

† Other conditions of the experiments are as follows:

^a Q_a = 1.1 mL/s; no solvent on top; no surfactant added.

^b Q_a = 3.0 mL/s; solvent, mineral oil (15 mL); no surfactant added.

^c Q_a = 3.0 mL/s; solvent, decyl alcohol (30 mL); no surfactant added.

^d Q_a = 3.0 mL/s; solvent, decyl alcohol (15 mL); HTAB 5×10^{-5} M.

^e CGA flow rate = 0.583 mL/s, HTAB 2.1×10^{-5} M.

^f CGA flow rate = 0.583 mL/s, Tergitol 3.5 $\times 10^{-6}$ M.

The removal efficiency of conventional air stripping was far less than that of solvent sublation. Extrapolating the results, we should expect that V_G/V_L for 95% removal of PCP by air stripping will be far too large. In the case of solvent sublation, a layer of mineral oil on top of the aqueous phase improves the efficiency as well as reducing V_G/V_L , but a layer of decyl alcohol does even better. For CGA flotation, V_G/V_L is very small. Without the presence of a surfactant in solvent sublation, there is a difference of an order of magnitude in air volume per volume of liquid when compared to CGA flotation. When solvent sublation was carried out in the presence of HTAB under basic conditions (pH 8.9), it required 300% more air volume per volume of liquid and more than twice the amount of surfactant when compared with CGA flotation under similar conditions (pH 10.1). CGA flotation is more effective because of its small bubble size and its ability to solubilize as well as adsorb the solute. It is therefore conceivable that CGA flotation can be as effective as solvent sublation and still remain economical because it uses a far lower volume of air per liquid volume in flotation.

CONCLUSIONS

Microgas dispersions were generated using cationic, anionic, and non-ionic surfactants and were used in an adsorptive bubble flotation process to remove PCP from an aqueous phase. The aqueous solution was maintained at three different pH values by buffering the solutions. The charge on the encapsulating film was found to affect the removal efficiencies of PCP from the solution. CGA generated with Tergitol, which is a nonionic surfactant, was found to be most efficient in the removal of solute, and the efficiency remained nearly independent of pH. In the case of anionic DDBS, the removal percentage improved from 15 to 35% when the pH was changed from 10.1 to 3.0. Apparently the CGAs generated from this anionic surfactant failed to adsorb PCP at concentrations below its cmc even at pH 3.0, when PCP in the solution exists in its hydrophobic undissociated form. The reason for the higher removal by Tergitol may be its small bubble diameter in the suspension and higher air entrapment even when it is mixed in a solution. The large difference in the removal efficiencies between HTAB and DDBS, however, can be attributed solely to the charges on the encapsulating film. The ionic nature of adsorption of PCP is revealed even in its undissociated form because CGAs generated from HTAB were more efficient in the alkaline range than in the acidic range. An increase in concentration of the surfactants improved the removal efficiency. The increase in removal at micellar concentrations of the surfactants above their cmc indicates that in the aqueous phase the adsorption

of PCP must be occurring on the film of CGA much more than being solubilized in a micelle.

These results were compared with the removal efficiencies of conventional flotation techniques used by other researchers. The removal efficiency of air stripping was found to be far less than that of solvent sublation or of CGA flotation. Solvent sublation appears to be most effective for the removal of PCP, but even in the presence of HTAB it required 300% more air volume per volume of liquid when compared with CGA flotation.

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