Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Wungrattanasopon, Pukpong , Scamehorn, John F. , Chavedej, Sumaeth , Saiwan, Chintana and Harwell, Jeffrey H.(1996) 'Use of Foam Flotation to Remove *tert*-Butylphenol from Water', Separation Science and Technology, 31: 11, 1523 — 1540

To link to this Article: DOI: 10.1080/01496399608001411

URL: http://dx.doi.org/10.1080/01496399608001411

SEPARATION SCIENCE AND TECHNOLOGY, **31**(11), pp. 1523–1540, 1996

Use of Foam Flotation to Remove *tert*-Butylphenol from Water

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ABSTRACT

The surfactants cetyl pyridinium chloride (CPC) and sodium dodecyl sulfate (SDS) were used in a study of an adsorptive bubble flotation process in batch mode to remove *tert*-butylphenol (TBP) from water. The TBP removal is maximized when the surfactant concentration is around the critical micelle concentration (CMC). Since micelles form above the CMC, this indicate that the higher the surfactant monomer concentration, the better the removal, but the micelles compete with the air/water interface for the TBP, resulting in micelles reducing removal efficiency. The addition of NaCl to the feed solution results in a significant reduction of the ability of CPC to remove TBP, while it improves the ability of SDS to remove TBP.

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INTRODUCTION

Phenol and its derivatives have long been problem toxic pollutants. They are widely used as photographic developers and in several processes such as fiber glass manufacturing and wood distillation. Because of their widespread use, they are often found in contaminated wastewater, groundwater, and soil (1).

The use of foam flotation to remove organic solutes from water is illustrated in Fig. 1. A surfactant (called a collector) is introduced into the water, and gas is introduced into the system through a sparger which generates bubbles. A surfactant tends to strongly adsorb at the air/water interface with the hydrophobic or tail groups (e.g., hydrocarbon tail) in the air and the hydrophilic or head (water soluble) groups in the water. Even at low surfactant concentrations, a close-packed monolayer is formed at the bubble surface as shown in Fig. 1. The environment in the hydrophobic region formed by the surfactant tail groups is favorable for organic solutes which tend to co-adsorb at the bubble surface. Polar regions of the solute molecule can interact with the surfactant head group, affecting solute adsorption.

Foam flotation has proven to be extremely effective at removing contaminants from wastewater streams (2–10). Many variables were considered to have a significant effect upon removal efficiency, such as the height of the foam–liquid interface, the air flow rate, the bubble diameter, and the feed concentration (11–13). However, the effect of added electrolyte has received little attention. In this study, batch foam flotation experi-

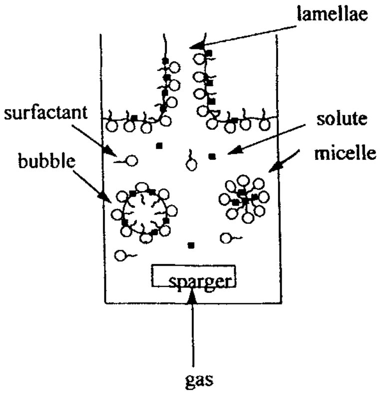

FIG. 1 Schematic of the foam flotation process.

ments were performed to investigate removal of *tert*-butylphenol from water using an anionic or a cationic surfactant and varying amounts of added NaCl.

EXPERIMENTAL

Materials

tert-Butylphenol (TBP) from Aldrich Chemical Company had a purity of 99%. Sodium chloride (NaCl), analytical purity grade, was obtained from Aldrich Chemical Company. Sodium dodecyl sulfate (SDS) was commercial grade with a purity of 90% and was obtained from Henkel Company. Cetyl pyridinium chloride (CPC), with a purity higher than 99%, was obtained from Zealand Chemical. Double distilled water was used in all of the experiments.

Methods

A schematic diagram of the foam flotation apparatus used in this study is shown in Fig. 2. An acrylic cylindrical column, 100 cm in height and with an internal diameter of 5.4 cm, was used as the flotation column. One liter of premixed liquid (containing water, TBP, surfactant, and NaCl) was placed in the column. A sample port was located 10 cm. from the bottom of the column. Filtered air was introduced into the bottom of the column through a sintered glass disk, pore size number 3. Samples of the liquid phase were taken at 30 and 45 minutes after the bubbling started for the SDS system, and at 30 and 60 minutes for the CPC system. Foam

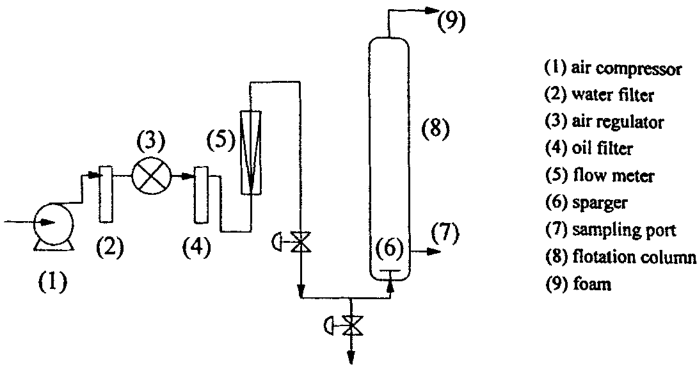

FIG. 2 Schematic diagram of the experimental apparatus.

from the overheads was collected at 40 and 50 minutes. The foam was broken for analysis by freezing the foam samples for 20 minutes and then allowed them to melt at ambient temperature for 20 minutes. Concentrations were analyzed by HPLC using a Nova Pak, C18 reverse phase column. A UV detector was used for TBP and CPC, while a refractive index detector was used for SDS.

An initial TBP concentration of 50 ppm was used in every experiment. Three initial concentrations of NaCl (3.2, 10, and 32 mM) were chosen to study the effect of salinity on removal efficiency. The initial concentration of SDS was varied from 0.1 to 10 times its critical micelle concentration (CMC). The initial concentration of CPC was varied from 0.1 to 2 times its CMC.

RESULTS AND DISCUSSION

The effect of NaCl concentration on the CMC of each surfactant is shown in Fig. 3. The logarithm of the CMC of each surfactant decreases with increasing logarithm of total counterion (e.g., Na^+ for SDS) concentration for ionic surfactants (14). The counterion is contributed from the surfactant and from added salt. The counterion reduces the head group repulsion of surfactant at the micelle surface, making micelle formation more favorable, reducing the CMC.

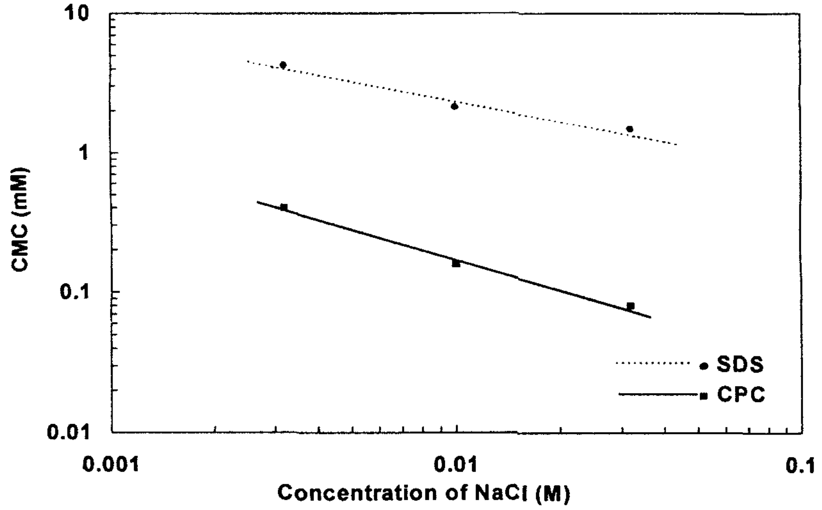

FIG. 3 Effect of NaCl concentration on the CMC of surfactants.

The effect of surfactant concentration and NaCl concentration on the removal efficiency of TBP is shown in Fig. 4 using CPC as the collector and in Fig. 5 using SDS as the collector. The average concentration was calculated by adding the initial and final concentration and dividing by two. The percentage of TBP removed shows a maximum around the CMC with SDS as the collector. The percentage of TBP removed shows a maximum at a concentration a little lower than the CMC with CPC as the collector. With added NaCl, this TBP removal does not exhibit a clear maximum with increasing CPC concentration, but after reaching a plateau in the region of the CMC, could increase with further increases in average CPC concentration.

The percentage of TBP removed plotted as a function of NaCl concentration at a constant average surfactant concentration for both CPC and SDS is shown in Fig. 6. The run time in Fig. 6 is different than that used in Figs. 4 and 5 to better illustrate the effect of salt concentration under the same conditions for CPC and SDS. Decreasing the ionic strength of solution decreases the electrical potential or charge density at the micelle surface due to electrical diffuse double layer compression (15). The reduction in the CMC with added electrolyte due to this effect has already been discussed. The same effect will stabilize the surfactant monolayer adsorbed at the air/water interface—a specified adsorption density will be attained at a lower surfactant concentration. However, a monolayer is generally nearly completely formed at equilibrium at surfactant concen-

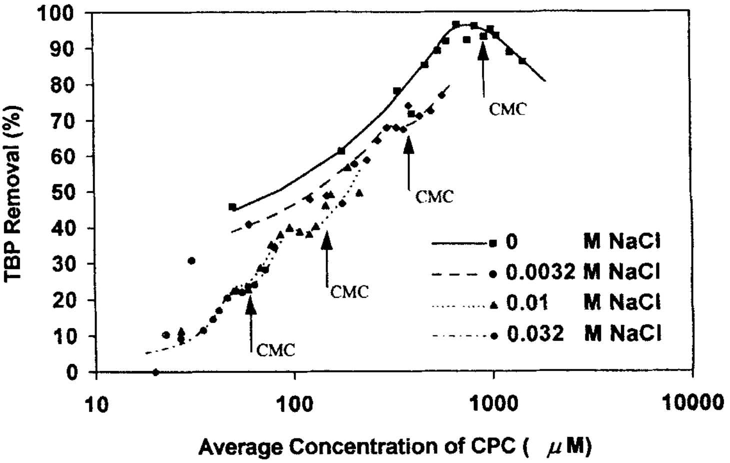

FIG. 4 Effect of average CPC concentration on TBP removal with added NaCl. Run time: 60 minutes.

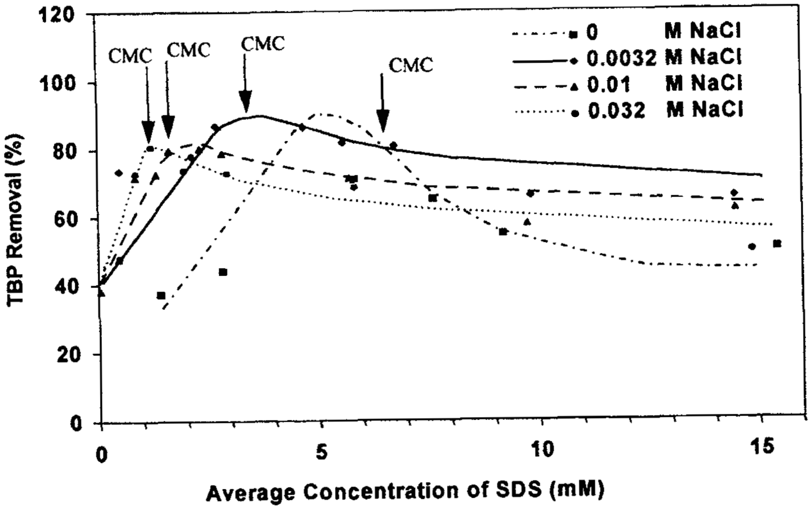

FIG. 5 Effect of average SDS concentration on TBP removal with added NaCl. Run time: 45 minutes.

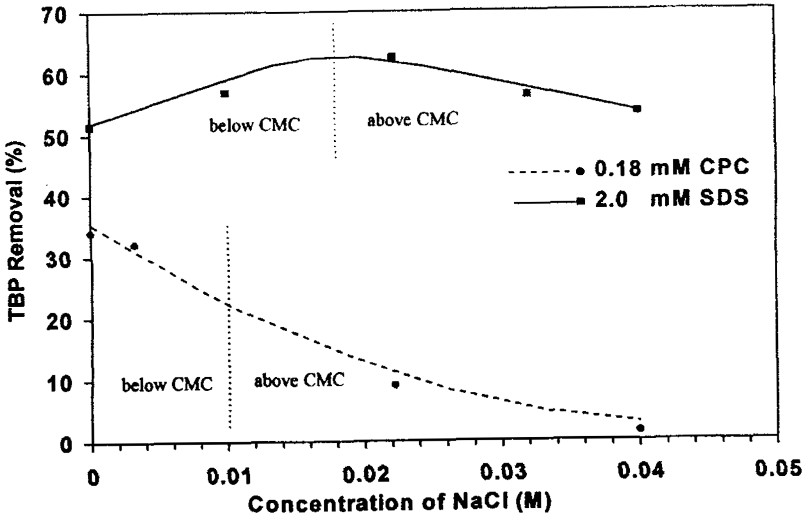

FIG. 6 Effect of NaCl concentration on TBP removal with SDS and CPC collector. Run time: 30 minutes.

trations above about 10% of the CMC, i.e., the conditions used in this work. Steric effects can cause the TBP hydroxyl groups to have less space to insert themselves between head groups in either the micelle or adsorbed monolayer as these head groups come closer together. Therefore, below the CMC, this effect in the monolayer causes a decrease in TBP removal when CPC is the collector.

Alcohols solubilize better in micelles composed of cationic surfactants than anionic surfactants due to ion–dipole interaction between the surfactant head groups and alcohol hydroxyl groups (16). Increasing ionic strength of the solution would be expected to decrease this ion–dipole interaction. If this effect also applied to the air/water interfacial monolayer, it could help explain why the TBP removal efficiency decreases with increasing electrolyte concentration more for the CPC than for SDS.

Foam stability of ionic surfactants generally reaches a maximum at a certain concentration of added electrolyte (17). This could explain the maximum in removal efficiency observed in Fig. 6 for SDS. Of course, in addition to organic pollutants, simultaneous removal of cationic heavy metals (e.g., copper) can be achieved with anionic surfactants, while multivalent anionic pollutants (e.g., chromate) can be removed by cationic surfactants, which may dictate surfactant type selection. Since the added

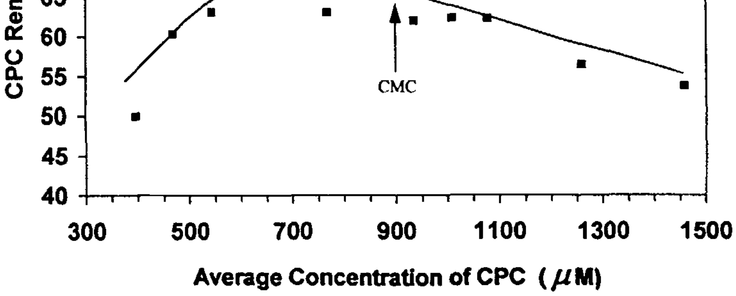

FIG. 7 Effect of average CPC concentration on CPC removal with no added NaCl. Run time: 60 minutes.

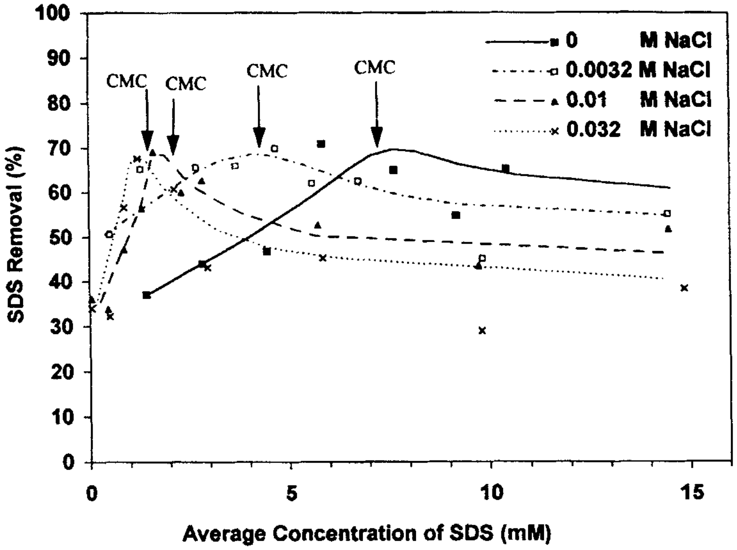

FIG. 8 Effect of average SDS concentration on SDS removal. Run time: 45 minutes.

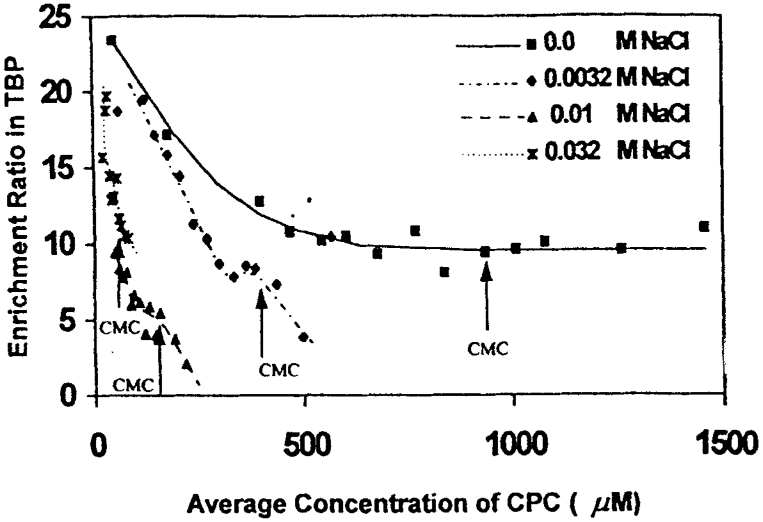

FIG. 9 Enrichment ratio of TBP in the foam with CPC collector. Run time: 45 minutes.

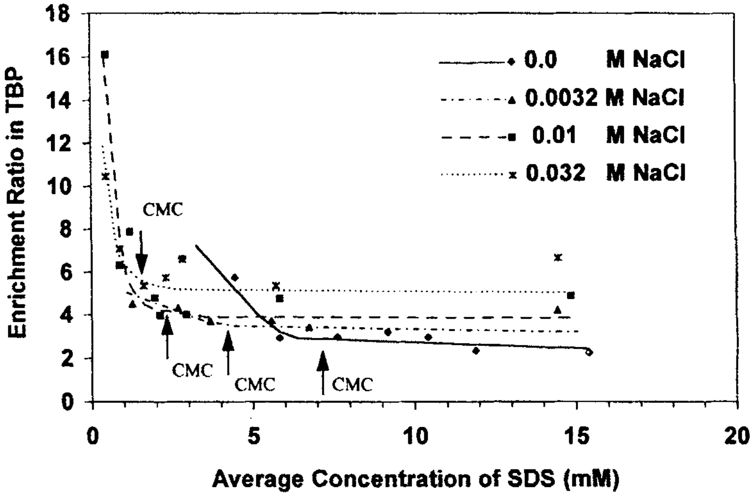

FIG. 10 Enrichment ratio of TBP in the foam with SDS collector. Run time: 45 minutes.

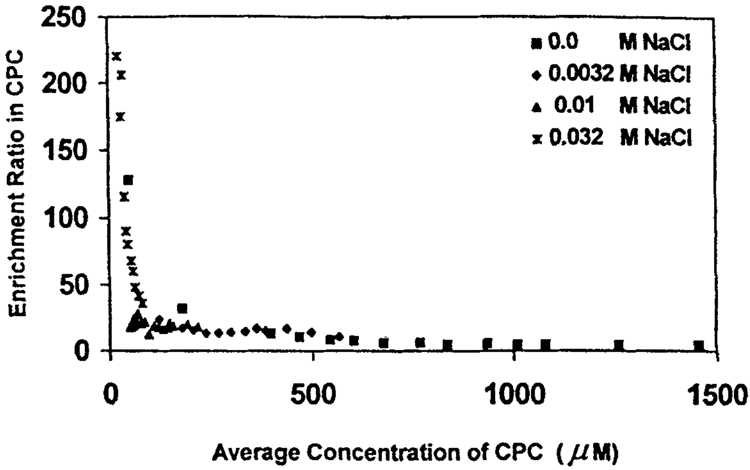

FIG. 11 Enrichment ratio of CPC in the foam. Run time: 45 minutes.

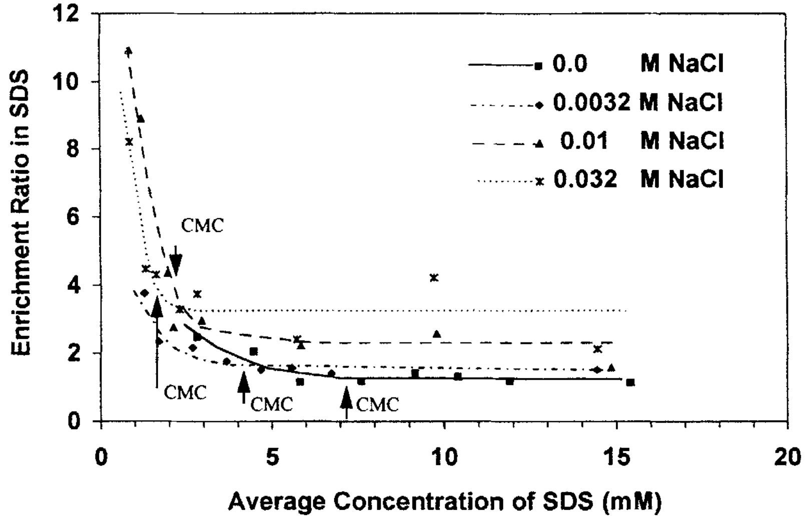

FIG. 12 Enrichment ratio of SDS in the foam. Run time: 45 minutes.

electrolyte reduces the CMC and the optimum removal occurs around the CMC for SDS as illustrated in Fig. 5, added electrolyte can reduce the surfactant concentration required to attain maximum removal efficiency for SDS, although this is not true for CPC.

The effect of average surfactant concentration on the removal efficiency of TBP is shown in Figs. 4 and 5 and for the surfactant in Figs. 7 and 8

TABLE 1
Experimental Results Using

	Time (min)	Initial CPC						
		64.36	233.01	370.81	423.38	587.54	722.32	801.09
Solution TBP	0	45.63	43.10	38.99	33.35	41.08	40.92	40.97
concentration	30	34.08	29.12	25.25	24.75	23.07	22.07	20.71
(ppm)	60	25.36	17.72	10.00	11.29	7.96	6.06	4.92
Solution CPC	0	64.36	233.01	370.81	423.38	587.54	722.32	801.09
concentration	30	27.72	153.97	299.92	370.79	442.73	525.50	595.75
(μM)	60	9.34	85.84	130.00	252.75	305.15	366.82	393.18
Liquid volume	0	1.00	1.00	1.00	1.00	1.00	1.00	1.00
(L)	30	0.97	0.93	0.86	0.84	0.82	0.80	0.79
	60	0.98	0.94	0.85	0.76	0.76	0.73	0.68

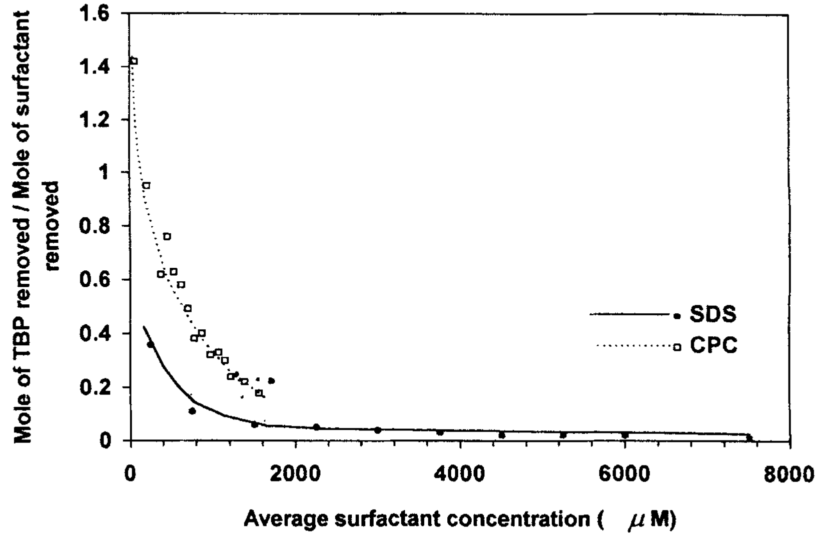

FIG. 13 Comparison of performance of CPC and SDS on TBP removal with no NaCl. Run time: 30 minutes.

for CPC and SDS, respectively. The maximum removal of both surfactants occurred at approximately the CMC in the absence of added salt. This result is in agreement with the those obtained by Peng and Di (18) who found that the maximum arsenic removal occurred at the CMC with SDS as the collector. This may be because higher surfactant concentrations cause a higher surface excess of surfactant at the air–water interface,

CPC without NaCl Addition

concentration (μM)							
861.38	944.92	1073.10	1157.80	1226.20	1298.20	1417.40	1678.70
39.27	38.99	41.91	42.34	40.10	43.27	41.87	45.61
19.69	21.28	19.97	22.85	22.74	23.57	25.89	27.86
2.33	4.78	2.93	4.63	3.21	4.75	7.55	9.85
861.38	944.92	1073.10	1157.80	1226.20	1298.20	1417.40	1678.70
661.04	747.39	865.05	968.07	1042.40	1088.80	1247.70	1419.70
451.81	544.35	591.08	698.78	757.05	804.69	986.90	1204.20
1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
0.76	0.78	0.75	0.78	0.77	0.78	0.78	0.79
0.62	0.64	0.57	0.63	0.61	0.61	0.62	0.64

TABLE 2
Experimental Results Using

	Time (min)	Initial CPC						
		49.64	97.16	133.29	201.16	230.60	291.37	319.69
Solution TBP concentration (ppm)	0	47.01	47.56	41.55	42.40	41.26	43.10	41.73
	30	36.01	35.78	34.07	32.04	32.60	27.41	27.75
	60	50.85	29.87	23.49	23.23	23.47	19.35	18.72
Solution CPC concentration (μM)	0	49.64	97.16	133.29	201.16	230.60	291.37	319.69
	30	0.00	45.32	92.05	119.05	143.42	152.77	183.97
	60	0.00	0.00	43.60	56.25	75.22	89.60	113.02
Liquid volume (L)	0	1.00	1.00	1.00	1.00	1.00	1.00	1.00
	30	0.77	0.95	0.95	0.94	0.93	0.91	0.90
	60	1.00	0.94	0.92	0.93	0.94	0.93	0.92

leading to a greater surface density of co-adsorbed TBP molecules. In contrast, at concentrations of surfactant above the CMC, TBP molecules prefer to solubilize in micelles than co-adsorb at bubble surfaces. The reason that the optimum removal is not exactly at the CMC may be because an average concentration is used here, not a point concentration. Since the final solution surfactant concentration might be as low as 20% of the initial concentration, the arbitrary nature of using an arithmetic average may lead to some small differences compared to results at a single concentration.

TABLE 3
Experimental Results Using

	Time (min)	Initial CPC						
		12.49	22.82	49.18	57.93	79.64	95.43	113.88
Solution TBP concentration (ppm)	0	46.11	49.98	48.11	48.68	46.03	48.39	46.82
	30	48.18	39.71	40.30	40.06	40.06	37.88	37.83
	60	45.65	44.65	39.08	39.58	34.59	33.09	30.74
Solution CPC concentration (μM)	0	12.49	22.82	49.18	57.93	79.64	95.43	113.88
	30	11.04	11.84	11.54	36.84	21.97	27.37	40.55
	60	0.00	0.00	9.87	10.79	10.87	10.63	10.48
Liquid volume (L)	0	1.00	1.00	1.00	1.00	1.00	1.00	1.00
	30	1.00	0.98	0.95	0.95	0.96	0.96	0.96
	60	0.99	0.99	0.96	0.95	0.95	0.95	0.94

Experimental Results Using CPC with 3.2 mM NaCl

concentration (μM)							
352.95	392.38	447.60	462.80	515.87	554.34	632.93	747.75
40.99	42.23	43.96	42.75	45.86	45.42	45.62	49.91
28.66	28.17	26.75	31.24	29.57	28.64	27.34	30.68
17.08	16.95	16.17	16.15	14.09	15.52	15.22	14.47
352.95	392.38	447.60	462.80	515.87	554.34	632.93	747.75
232.54	258.99	285.69	338.24	370.79	400.38	444.43	561.99
135.32	155.12	184.98	201.47	207.31	271.15	314.36	371.54
1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
0.89	0.88	0.88	0.88	0.87	0.88	0.86	0.85
0.91	0.89	0.87	0.86	0.85	0.85	0.82	0.80

The effect of the average concentration of SDS and CPC on the enrichment ratio (concentration in collapsed foam/initial concentration in the liquid pool) of TBP and surfactant is shown in Figs. 9–12. The TBP enrichment ratio indicates the ability of the process to concentrate the solute, which is the purpose of the operation. The previously discussed effects on the fraction of solute removed in a given time period influence such parameters as residence time required and optimum surfactant concentration. The enrichment ratio indicates whether the separation is substantial when it is done. The enrichment ratio for TBP is above 10 using CPC,

CPC with 10.0 mM NaCl

concentration (μM)							
126.88	137.25	151.02	156.72	201.49	213.15	250.54	276.23
46.10	43.80	43.55	40.99	40.58	45.10	43.76	42.77
37.40	36.80	35.95	33.58	31.72	32.24	33.24	31.53
30.15	28.64	28.61	25.86	23.13	24.47	20.11	22.98
126.88	137.25	151.02	156.72	201.49	213.15	250.54	276.23
48.63	71.86	83.95	83.10	81.55	106.79	160.04	176.80
11.61	17.31	25.33	26.53	39.72	40.95	73.92	89.64
1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
0.95	0.96	0.96	0.96	0.96	0.95	0.96	0.95
0.92	0.94	0.94	0.95	0.95	0.94	0.94	0.94

TABLE 4
Experimental Results Using

	Time (min)	Initial CPC					
		11.46	39.83	71.30	62.37	65.24	81.41
Solution TBP	0	47.89	49.02	48.87	48.33	47.99	48.65
concentration	30	49.17	44.59	43.03	42.14	41.56	41.48
(ppm)	60	48.55	43.59	44.08	33.37	42.63	41.94
Solution CPC	0	11.46	39.83	71.30	62.37	65.24	81.41
concentration	30	6.98	6.19	7.60	7.66	7.07	10.62
(μM)	60	5.52	5.35	5.93	5.60	5.75	6.29
Liquid volume	0	1.00	1.00	1.00	1.00	1.00	1.00
(L)	30	1.00	1.00	1.00	0.99	0.98	0.98
	60	1.00	1.00	1.00	1.00	1.00	0.99

but only above 2 using SDS, both in the absence of NaCl. Added NaCl substantially reduces the enrichment ratio using CPC, but has a minor effect using SDS. The enrichment ratio of the CPC itself is substantially higher than that of the SDS, consistent with the TBP enrichment ratio trends. Both the TBP and surfactant are more concentrated in the collapsed foam using CPC than when SDS is used, even in the presence of

TABLE 5
Experimental Results Using SDS Without NaCl Addition

	Time (min)	Initial SDS concentration (mM)									
		2.00	1.59	2.95	4.55	6.02	7.50	9.00	11.09	13.24	16.12
Solution TBP	0	50.55	47.49	48.33	49.42	48.31	50.00	48.46	50.14	51.34	47.63
concentration	30	32.65	29.71	30.58	21.54	29.05	32.04	35.65	36.91	38.31	42.00
(ppm)	45	28.15	21.34	20.61	17.82	16.14	16.29	26.92	20.03	27.04	33.08
Solution SDS	0	2.00	1.59	2.95	4.55	6.02	7.50	9.00	11.09	13.24	16.12
concentration	30	0.00	1.42	2.87	4.93	6.53	8.92	9.22	10.82	12.17	14.94
(μM)	45	0.00	1.33	2.64	3.87	5.65	7.73	9.36	10.34	11.83	15.83
Liquid volume (L)	0	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
	30	0.69	0.79	0.73	0.73	0.56	0.57	0.63	0.61	0.55	0.57
	45	0.93	0.75	0.63	0.63	0.31	0.34	0.44	0.37	0.31	0.35

CPC with 32.0 mM NaCl							
concentration (μM)							
98.44	101.11	115.19	135.82	139.00	151.14	174.99	208.74
47.34	47.40	47.82	47.21	46.64	46.46	45.63	47.18
40.97	40.70	40.34	40.41	39.94	39.08	38.43	38.12
39.86	38.57	38.42	38.08	36.95	36.79	34.34	32.74
98.44	101.11	115.19	136.82	139.00	151.14	174.99	208.74
6.71	13.83	27.24	22.39	23.40	36.82	49.09	65.31
4.22	5.08	7.03	6.62	6.82	7.77	8.49	9.12
1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
0.97	0.96	0.97	0.97	0.96	0.97	0.96	0.96
0.99	0.98	0.97	0.97	0.97	0.96	0.95	0.95

added NaCl. The enrichment ratio of both TBP and surfactant decreases with increasing average concentration of surfactant. This may be due to the foam wetness, as higher surfactant concentration result in foams of higher wetness (as shown by the increasing volume reduction with increasing surfactant concentration in Tables 1–8) and, therefore, lower enrichment ratios. A possible reason for this is because higher surfactant concen-

TABLE 6

Experimental Results Using SDS with 3.2 mM NaCl

	Time (min)	Initial SDS concentration (mM)									
		0.50	1.50	2.00	3.00	4.00	5.00	6.00	7.00	10.00	15.00
Solution TBP concentration (ppm)	0	47.89	48.91	47.86	48.36	48.32	47.84	45.46	45.31	49.59	46.98
	30	30.79	24.60	24.65	29.05	28.11	29.72	31.33	32.23	37.18	40.75
	45	24.78	13.67	11.07	16.96	15.92	16.96	20.25	21.46	30.83	35.17
Solution SDS concentration (mM)	0	0.51	1.55	1.92	3.26	3.95	5.58	5.56	7.03	9.63	14.23
	30	0.38	1.35	1.61	3.04	3.96	4.67	5.85	6.44	9.64	13.93
	45	0.49	1.05	1.42	2.37	3.34	4.35	5.16	6.50	9.65	13.91
Liquid volume (L)	0	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
	30	0.65	0.65	0.58	0.65	0.60	0.59	0.61	0.61	0.72	0.61
	45	0.51	0.51	0.40	0.47	0.40	0.39	0.41	0.41	0.55	0.46

TABLE 7
Experimental Results Using SDS with 10.0 mM NaCl

	Time (min.)	Initial SDS concentration (mM)									
		0.10	0.50	1.00	1.50	2.00	2.50	3.00	6.00	10.00	15.00
Solution TBP concentration (ppm)	0	50.00	48.94	49.57	50.46	48.66	48.77	47.95	49.02	51.29	50.48
	30	40.15	35.07	29.19	32.96	32.79	31.34	31.11	36.11	41.76	43.21
	45	38.07	28.81	23.17	23.37	20.70	22.03	22.68	29.44	37.09	38.59
Solution SDS concentration (mM)	0	0.26	0.56	0.82	1.54	1.95	2.33	3.17	5.51	9.80	14.45
	30	0.20	0.48	0.85	1.08	1.97	2.36	2.81	5.64	9.62	14.39
	45	0.00	0.42	0.71	1.14	1.25	2.11	2.63	5.46	9.46	13.92
Liquid volume (L)	0	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
	30	0.86	0.88	0.70	0.72	0.64	0.61	0.70	0.64	0.71	0.66
	45	0.81	0.89	0.60	0.59	0.48	0.44	0.45	0.48	0.59	0.50

trations may cause a more viscous surface film of foam and decrease the foam drainage. At concentrations above the CMC, a liquid–crystalline phase can be formed in the lamellae, reducing the foam drainage (19).

The relative performance of CPC and SDS is shown in Fig. 13 by the ratio of the moles of TBP removed to the moles of surfactant removed plotted against the average concentration of surfactant, at run times of 30 minutes, with no added NaCl. The CPC exhibits a better performance than SDS, perhaps due to the charge on the head group or the longer alkyl chain length of CPC. Moreover, CPC produces a dryer foam than SDS as shown by the greater volume reduction in the SDS system than in the

TABLE 8
Experimental Results Using SDS with 32.0 mM NaCl

	Time (min.)	Initial SDS concentration (mM)									
		0.10	0.50	1.00	1.50	2.00	2.50	3.00	6.00	10.00	15.00
Solution TBP concentration (ppm)	0	49.11	48.66	47.84	48.37	48.56	48.26	47.35	50.77	49.87	49.31
	30	41.57	31.93	29.08	28.20	30.21	30.37	32.60	36.49	41.76	42.33
	45	36.64	27.26	19.93	17.55	21.75	20.70	22.72	27.21	37.36	39.08
Solution SDS concentration (mM)	0	0.10	0.65	1.11	1.57	1.99	2.32	2.88	6.12	10.01	15.23
	30	0.00	0.53	0.86	1.31	1.82	2.03	2.59	5.80	9.86	14.63
	45	0.00	0.47	0.73	0.95	1.93	1.77	2.89	5.70	9.60	14.70
Liquid volume (L)	0	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
	30	0.89	1.00	0.73	0.67	0.70	0.67	0.70	0.71	0.82	0.75
	45	0.78	0.93	0.65	0.54	0.59	0.52	0.57	0.59	0.74	0.64

CPC system in Tables 1–8. In additions, CPC yields higher enrichment ratios for both TBP and surfactant. However, removal efficiencies for TBP using CPC decrease dramatically as NaCl is added.

CONCLUSIONS

Cetyl pyridinium chloride (CPC) and sodium dodecyl sulfate (SDS) were used as surfactants to remove *tert*-butylphenol (TBP) from water in a flotation process. Over 90% of the solute can be removed using either surfactant with a residence time of 1 hour or less. An enrichment ratio of more than 20 is observed, and the ratio of moles solute removed/moles surfactant removed can be greater than unity. The addition of NaCl affects surfactant monolayer formation and micelle formation, and it enhances the removal ability of SDS while reducing the removal ability of CPC. Micelle formation was found to reduce the removal efficiency because TBP prefers to solubilize in the micelle rather than co-adsorb at the bubble surface, resulting in a reduction in the percentage of TBP removal at surfactant concentrations above the CMC. The foam is more concentrated in both TBP and surfactant using CPC instead of SDS.

ACKNOWLEDGMENTS

Financial support for this work was provided by National Science Foundation Grant CBT 8814147, an Applied Research Grant from the Oklahoma Center for the Advancement of Science and Technology, the Center for Waste Reduction Technologies of the American Institute of Chemical Engineers, Agreement No. N12-N10, the TAPPI Foundation, the National Research Council of Thailand, the Petroleum Authority of Thailand, and U.S. AID (University Development Linkages Project). In addition, support was received from the industrial sponsors of the Institute for Applied Surfactant Research including Akzo Nobel, Amway, Amoco, Aqualon, Calgon-Vestal, Colgate-Palmolive, Dow, Dowelanco, DuPont, Henkel, ICI, Kerr-McGee, Reckitt and Colman, Lubrizol, Phillips Petroleum, Pilot Chemical, Shell, Sun, and Witco. Dr. Scamehorn holds the Asahi Glass Chair and Dr. Harwell holds the Conoco-DuPont Professorship in chemical engineering at the University of Oklahoma. Surfactant was provided by Henkel Co. and Zealand Chemical Co.

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Received by editor September 25, 1995