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Surfactant Recovery from Water Using Foam Fractionation: Effect of Temperature and Added Salt

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Surfactant Recovery from Water Using Foam Fractionation: Effect of Temperature and Added Salt

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ABSTRACT

The purpose of this study was to investigate the use of foam fractionation to recover surfactant present at low concentrations in aqueous streams. A simple continuous mode foam fractionation was used, and three surfactants were chosen for this study: sodium dodecyl sulfate, cetylpyridinium chloride, and sodium *n*-hexadecyl diphenyloxide disulfonate. In a previous study the effects of surfactant concentration, air flow rate, liquid- and vapor-phase heights, and sparger type were investigated for these surfactants. Here, the effects of temperature and added salt are studied. It is found that the foam flow rate and enrichment ratio increase whereas the foam wetness and the rate of surfactant recovery decrease with increasing temperature. Increasing

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the concentration of added salt decreases the CMC of the surfactants. The foam flow rate, foam wetness, and the rate of surfactant recovery increase, while the enrichment ratio decreases with increasing concentration of salt.

INTRODUCTION

Surfactants appear in the effluent water from a number of industries such as textile, paper and pulp, food processing, and detergent manufacturing. As environmental regulations tighten, there is specifically a concern about reducing the surfactant concentration in the effluent streams. Surfactant-based separation processes are of increasing interest in the removal of pollutants from wastewater and groundwater (1). Generally, in these processes the surfactants will be added to remove the pollutants from effluent streams. Sometimes, these treated effluent streams contain a low surfactant concentration. In addition to satisfying environmental regulations, the value of the surfactant being emitted sometimes make recovery operations economical. The surfactant concentration in these effluent streams is often around or below the critical micelle concentration (CMC) (2).

Micellar-enhanced ultrafiltration (MEUF) is one surfactant-based separation which shows great promise (2). The surfactant aggregate called micelles either solubilizes or bind target pollutants and is then ultrafiltered from solution. However, surfactant not in micelles (monomer) leaks into the permeate stream coming through the membrane at a concentration around the CMC. Generally, the surfactant must be recovered from the stream for an economical separation (3). Some methods which have been considered to solve this problem include the use of a surfactant with a very low CMC so leakage is tolerable, use of a polymeric surfactant which should not pass into the permeate at all, and foam fractionation to remove and recover the surfactant from the permeate.

In many studies, foam fractionation has been extensively studied for the purpose of removing pollutants such as heavy metals from water by adding surfactant on purpose. In the current study the purpose is to recover surfactant itself by using foam fractionation. In previous work (4) the effects of air flow rate, foam height, liquid height, surfactant feed concentration, and sparger porosity were studied. In this work the effect of salt concentration and the temperature of the system are investigated. This research involves the systematic study of these variables on the efficiency of a foam fractionation operation in a pilot-scale fractionator for several different surfactants.

BACKGROUND

The foam separation process is an adsorptive bubble separation technique that selectively separates surface-active compounds from a solution due to

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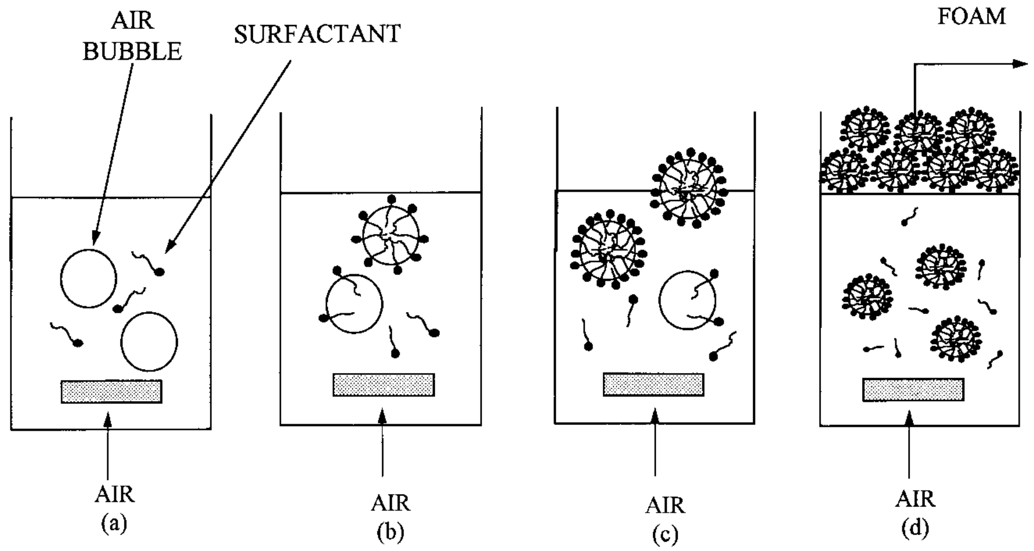

FIG. 1 Schematic of foam fractionation.

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adsorption of surfactant at the interface between the liquid and gas (5). This process is especially effective for separation of materials at low concentration. Surface-inactive compounds (colligens) can be removed from solution if an appropriate surface-active material (surfactant) is added to the system (6). The foam separation process can be divided into two types, foam fractionation and froth flotation. Foam fractionation separates dissolved material, while froth flotation separates insoluble material (7).

In foam fractionation, air is sparged to produce bubbles which rise to the top of a liquid column producing foam as illustrated in Fig. 1. As the bubbles travel through the liquid phase, surfactant adsorbs at the air–liquid interface. When the air bubbles emerging from solution form a cell in the foam honeycomb, the thin liquid film in the foam (lamellae) is stabilized by the adsorbed surfactant (8). Drainage of liquid in the lamellae occurs due to gravity, and eventually the foam breaks or collapses (9). The collapsed foamate solution that is collected from the top of column has a higher concentration of the surfactant than the initial solution. There are two modes of foam fractionation: simple mode (batchwise or continuous) and higher mode with enriching and/or stripping, as shown in Fig. 2 (10). In this work a simple continuous mode of operation is used to recover the surfactant from water.

The foam fractionation process has been widely studied for the purpose of removing colligends such as organic molecules (11), cations (12–14), and anions (15, 16) from the aqueous phase by using a surfactant as the collector. Many publications are available on different modes of operation of a foam fractionation unit such as batch mode (10, 17, 18), simple continuous mode

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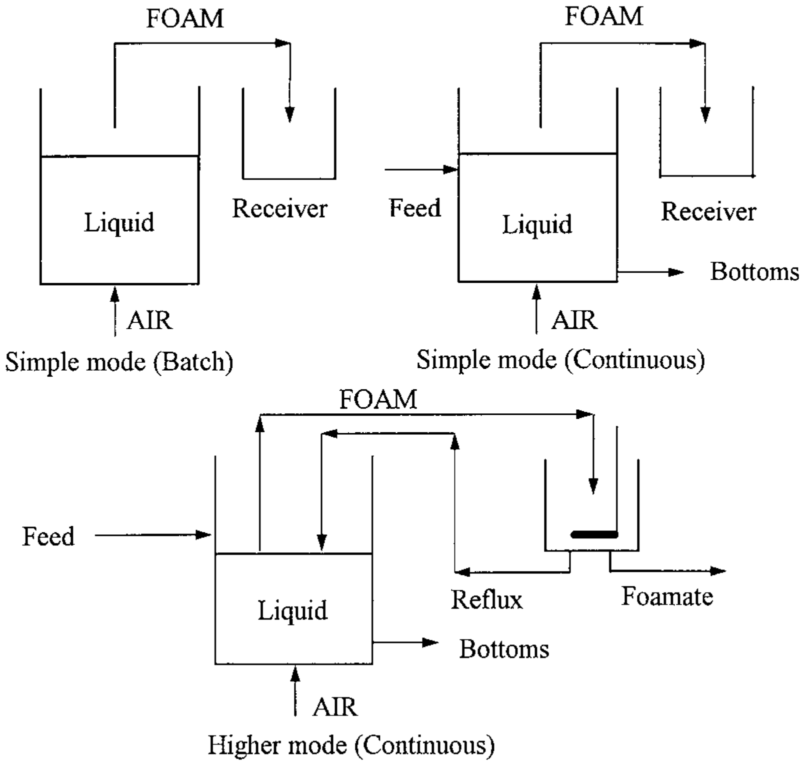

FIG. 2 Types of foam fractionation.

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(19), stripping mode (15), enriching mode (20, 21), and combined mode (22). The effects of such key variables as temperature, feed rate, pH, gas flow rate, feed concentration, foam height, and bubble diameter on the operation of both batch and continuous fractionation have been studied. There are few studies addressing surfactant recovery by foam fractionation as is the purpose of this work.

EXPERIMENTAL

Materials

Three different surfactants were chosen for study. Sodium dodecyl sulfate (SDS) was supplied from Kao Industrial R&D with a purity of 96.28%. Cetylpyridinium chloride (CPC) from Pfaltz & Bauer Inc. was 99.9% pure. Sodium *n*-hexadecyl diphenyloxide disulfonate (DADS) was supplied from Dow Chemical (trade name Dowfax-8390) in a 36.5 wt% surfactant in water form. Sodium chloride (NaCl) was supplied from Ajax Chemical with a purity of 99.9%. All of the above materials were used without further purification. Distilled water with a conductivity of 2 μmoh/cm was used in all experi-

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TABLE 1
Information on Manufacturer-Supplied Surfactant Properties

Surfactant	Formula weight	Quality	Source
Sodium dodecyl sulfate (SDS)	288.38	96.28% SDS 1.12% volatile matter 0.39% *n*-hexane 2.207% SO_4	Kao Industrial (Emal 10 P) Lot 611
Cetylpyridinium chloride (CPC)	358.01	>99.9% pure	Pfaltz & Bauer
Sodium *n*-hexadecyl diphenyloxide disulfonate (DADS)	640	36.5 wt% active 10% NaCl 0.51% Na_2SO_4	Dow Chemical
Sodium chloride (NaCl)	58.44	99.9% pure	Ajax Chemical

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ments. Manufacturer-supplied information about the chemicals used is shown in Table 1.

Equipment

A schematic flow diagram of the foam fractionation apparatus used in this study is shown in Fig. 3. The jacketed cylindrical fractionator column consisted of two 100-cm long acrylic columns with diameters of 12 and 15 cm diameter, and a thickness of 3 mm. The feed stream tube, drainage stream tube, and cooling water tube were made of acrylic with 1.5 cm O.D. and 15 cm length. For the foam outlet tube, an acrylic tube with 2.5 cm O.D. and 15 cm length was used.

Methods

The simple continuous mode of foam fractionation system was used in this study, as shown in Fig. 3. The surfactant feed solution was continuously pumped through a flowmeter at a flow rate of 100 mL/min (10 L/min·m^2) by a diaphragm pump before entering the column at a position 40 cm from the bottom of the column. The liquid level in the column was controlled at a position 45 cm from the bottom of the column by adjustment of the bottom stream withdrawal rate. Compressed air was allowed to pass through the air flowmeter at a flow rate of 200 mL/min and was introduced to the column through a sintered-glass diffuser. Sintered-glass No. 2 was used to produce bubble sizes of 100–160 μm. Foamate at the top of the solution was collected at a position 45 cm from the liquid surface in a beaker for a measured time period. The foam was frozen, thawed, and then weighed to get the collapsed

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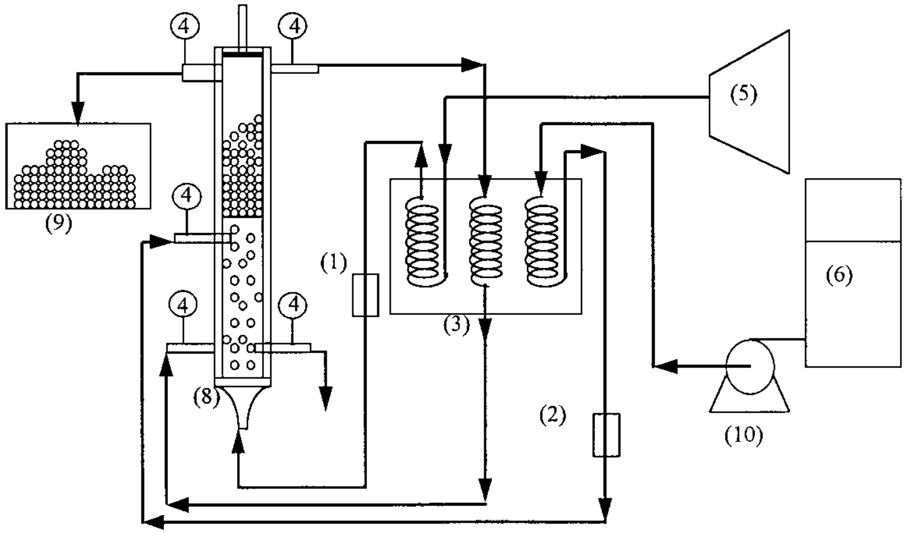

Equipment:

(1) Flow meter
(2) Air flow meter
(3) Circulated heating & cooling bath
(4) Thermocouple
(5) Air compressor
(6) Feed storage tank
(7) Foam fractionation column
(8) Sintered-glass diffuser
(9) Foam storage collector
(10)Diaphragm pump

FIG. 3 Schematic diagram of foam fractionation experimental system.

foamate volume. The column operating temperature was held at the desired value by using a circulated cooling–heating bath and the water jacket around the column. The temperature at any point around this column was measured continuously by a scanning thermocouple thermometer.

The foam fractionation was studied under steady-state conditions. The base condition was 200 mL/min (20 L/min·m^2) air flow rate, 100 mL/min (10 L/min·m^2) liquid feed flow rate, 20°C, 45 cm foam height, and 45 cm liquid height. The surfactant concentration in the feed solution was kept at 80% of the CMC of each surfactant (the CMC at 20°C) when the effects of temperature were studied. In studies of the effects of added salt, the surfactant concentration was held at 10% of the CMC (with no added salt) for SDS and CPC, and 20% of the CMC (in the absence of added salt) for DADS to avoid micelle formation. Each experiment was carried out for a minimum of 3 hours. Steady-state was insured when all measured parameters were invariant with time.

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Three parameters, volumetric foam production rate (L/min·m^2), foam wetness (grams of foam solution/L of foam), and the surfactant concentration (g/L) in the collapsed foam solution were measured. The concentrations of CPC and DADS were determined by UV visible spectroscopy at wavelengths of 260 and 237 nm, respectively, while SDS concentration was measured by a high performance liquid chromatography (HPLC) or a total organic carbon (TOC) analyzer.

The CMC of each surfactant was calculated as the concentration where the specific surface tension versus the surfactant concentration showed an abrupt change in slope (9).

RESULTS AND DISCUSSION

In this study there are four parameters used to illustrate the efficiency of the foam fractionation. Results involving rates are on a unit cross-sectional area basis for use in designing units. The enrichment ratio is defined as the ratio of the concentration in the collapsed foam solution to that in the feed solution (C_f/C_i). Foam wetness (W) is defined as the grams of collapsed foam solution per liter of foam. Volumetric foam production rate (V) is defined as the rate of foam formation (liter of foam/min·m^2). Rate of surfactant recovery (R) is defined as the amount of surfactant that is removed per unit time in the foam (g/min) and calculated by

$$R = WVC_fM_w$$

where C_f = concentration of the surfactant in the collapsed foam solution (M) and M_w = molecular weight of that surfactant (g/mol).

Effect of Salinity

Table 2 gives the results for the foam fractionation experiments at 20°C in which added NaCl concentration varies. The CMC of each surfactant de-

TABLE 2
Experimental Results: Effect of Added Salt on Foam Fractionation

[NaCl] (mol/L)	Enrichment ratio, C_f/C_i			Foam wetness (g/L)			Volumetric foam production rate (L/min·m^2)			Rate of surfactant recovery (g/h·m^2)		
	CPC	DADS	SDS	CPC	DADS	SDS	CPC	DADS	SDS	CPC	DADS	SDS
0.000	58.85	97.96	69.54	0.14	0.11	1.02	19.15	12.78	21.93	5.06	0.48	25.57
0.025	50.93	56.49	59.24	0.17	0.32	1.23	20.53	15.94	22.41	5.84	0.98	28.04
0.050	47.68	37.26	44.11	0.23	0.47	1.54	21.21	18.71	22.92	7.43	1.13	28.43
0.100	41.37	29.49	36.15	0.28	0.83	2.00	22.49	20.16	23.58	8.69	1.76	31.56

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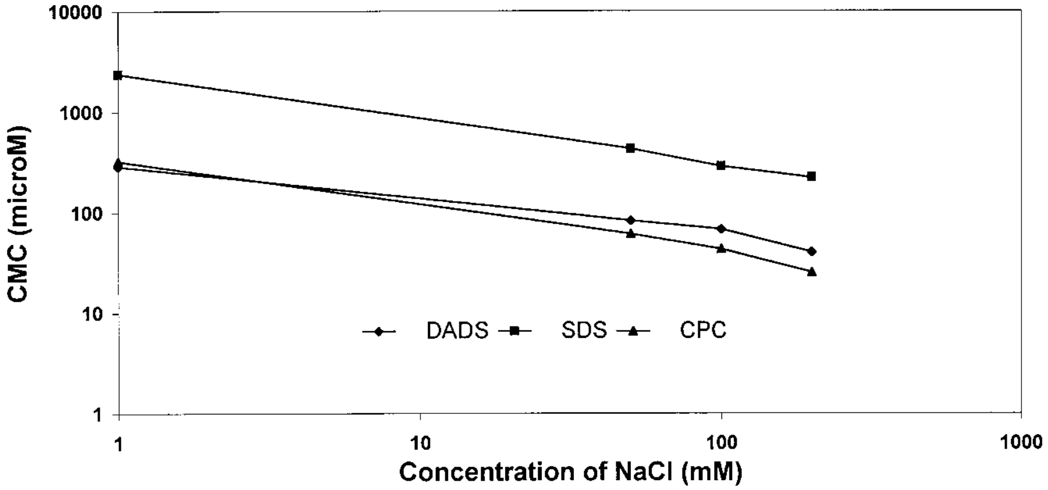

FIG. 4 Effect of NaCl concentration on CMC of surfactants.

creases with increasing concentration of salt, as shown in Fig. 4. This CMC depression effect is due to the compression of the electrical double layer around the micelle which causes a reduction in repulsion between the head groups of surfactant at the micelle surface (9, 23, 24). Therefore, micelles form at a lower surfactant concentration as micelle formation is synergized. To avoid the formation of micelles in the system when salt was added, the initial concentration of CPC, SDS, and DADS were kept at 10, 10, and 20% of the CMC value in the absence of salt, respectively.

The effect of added salt on foam fractionation parameters is shown in Figs. 5–8. The volumetric foam production rate increases with increasing concentration of added salt, as seen in Fig. 5. Increasing the salt concentration tends to increase the adsorbed surfactant at the air–water interface of the thin liquid film (lamellae) comprising the foam (Fig. 9) because the surfactant monolayer is more efficiently packed and the repulsion between two monolayers on each side of the liquid film decreases as electrolyte concentration increases. As a result, the liquid in the lamellae is more structured and surface viscosity is higher, leading to a decreased rate of film drainage (7, 9, 23, 24). The foam wetness increases as added salt concentration increases, as shown in Fig. 6, presumably due to the decreased rate of film drainage and the increased amount of water contained in the film lamellae. The enrichment ratio decreases with increasing concentration of salt, as shown in Fig. 7, presumably also due to this decreased rate of drainage. Figure 8 shows that increasing the concentration of salt increases the rate of surfactant recovery. The increased volumetric foam production rate and higher foam wetness more than compensates for the lower concentration of surfactant in the collapsed foam as salinity is increased.

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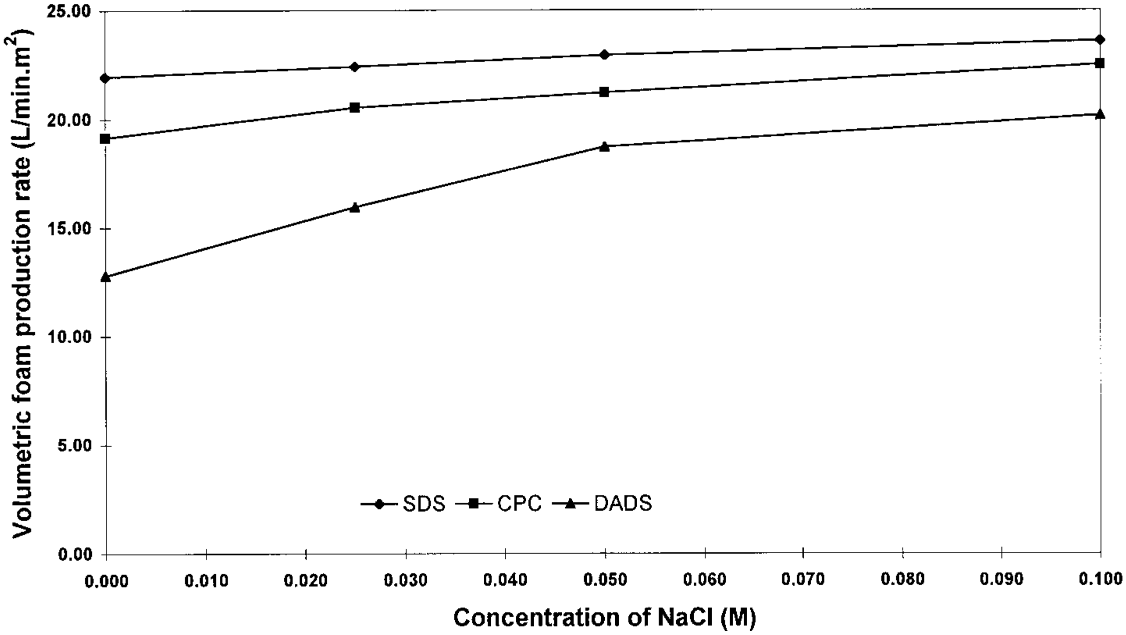

FIG. 5 Effect of NaCl concentration on volumetric foam production rate.

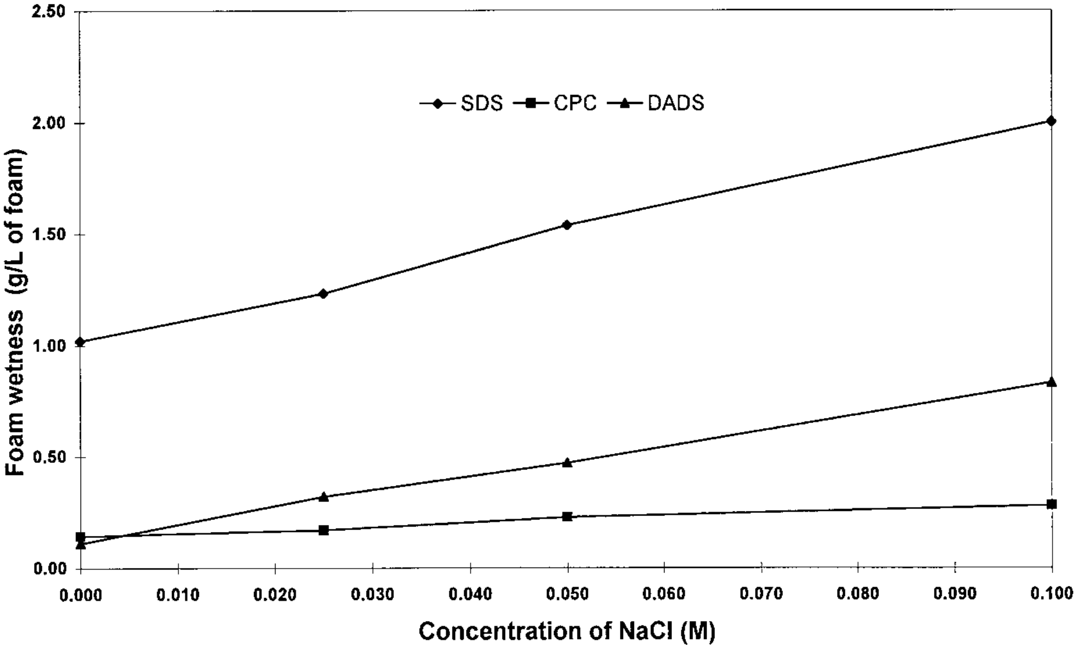

FIG. 6 Effect of NaCl concentration on foam wetness.

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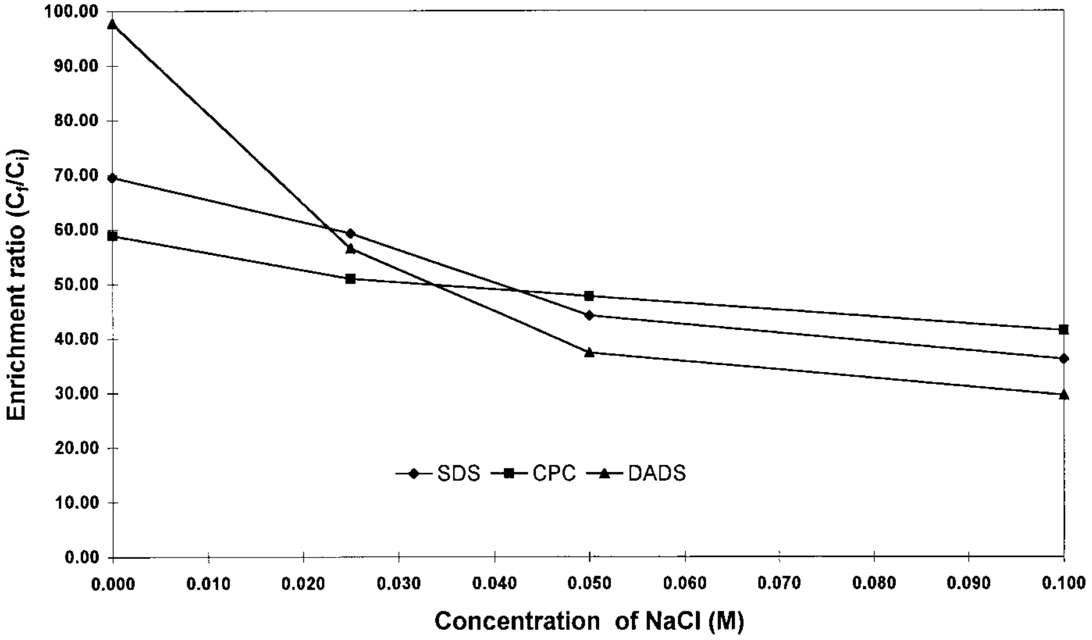

FIG. 7 Effect of NaCl concentration on enrichment ratio.

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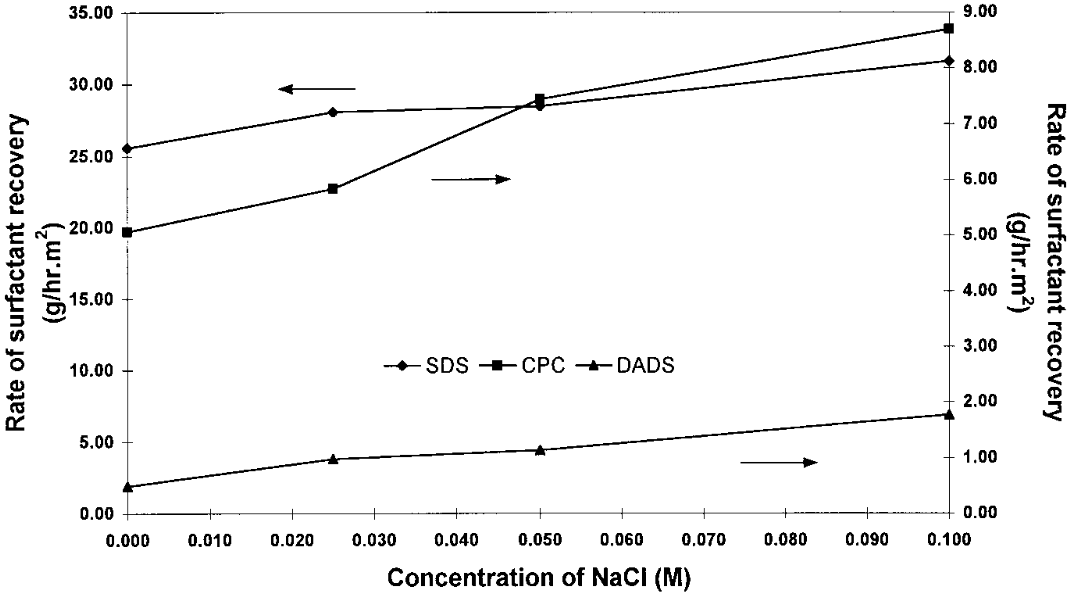

FIG. 8 Effect of NaCl concentration on rate of surfactant recovery.

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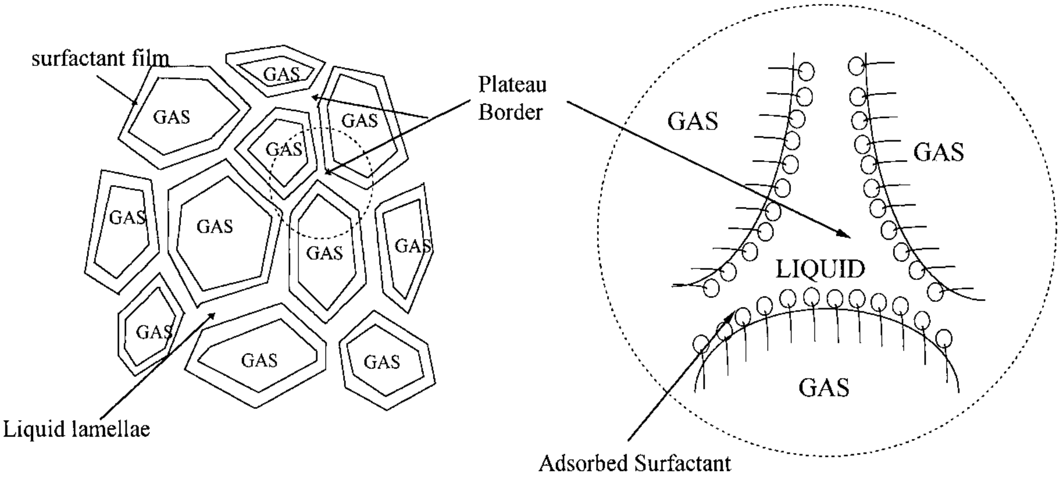

FIG. 9 Structure of foam.

Effect of Temperature

Table 3 shows the effect of temperature on foam fractionation parameters with no added salt. Figure 10 shows that the volumetric foam production rate increases with increasing temperature. This may be due to the reduction in the surface tension of the thin liquid film lamellae with increasing temperature, which can increase foam formation (8, 25). Increasing the temperature decreases the foam wetness, as shown in Fig. 11, perhaps because drainage rates increase as viscosity (bulk and surface) decreases and evaporation of lamellae water increases with increasing temperature, resulting in decreasing water content in the thin liquid film. This could also explain the increase in enrichment ratio when the temperature is increased, as seen in Fig. 12. As temperature increases, foam formation is generally enhanced, but foam stabil-

TABLE 3
Experimental Results: Effect of Temperature on Foam Fractionation

Temperature (°C)	Enrichment ratio, C_f/C_i			Foam wetness (g/L)			Volumetric foam production rate ($\mathrm{L/min{\cdot}m^2}$)			Rate of surfactant recovery ($\mathrm{g/h{\cdot}m^2}$)		
	CPC	DADS	SDS	CPC	DADS	SDS	CPC	DADS	SDS	CPC	DADS	SDS
10	13.72	30.05	2.32	3.52	3.71	6.28	19.91	15.54	20.06	15.03	26.69	42.92
20	16.14	49.44	2.87	3.11	2.43	6.14	20.25	16.78	20.34	16.37	25.23	38.31
30	80.84	58.12	5.26	0.59	1.73	2.16	21.44	19.15	22.74	16.35	26.78	27.99
35	96.14	57.58	6.77	0.43	1.73	1.58	22.06	20.11	23.75	14.18	25.72	25.57

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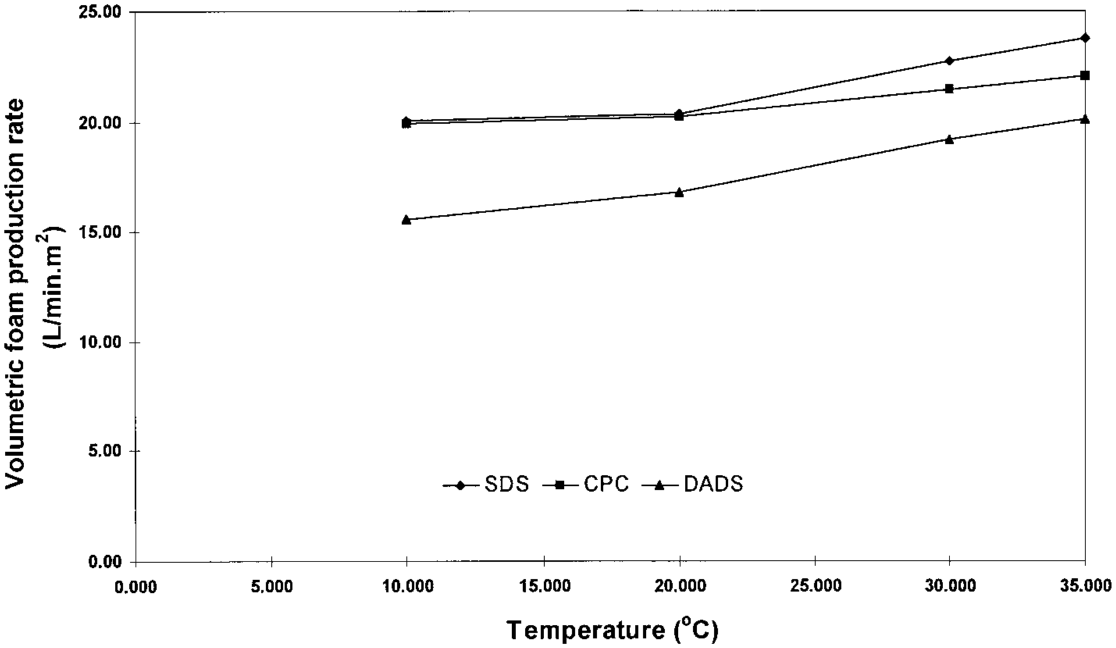

FIG. 10 Effect of temperature on volumetric foam production rate.

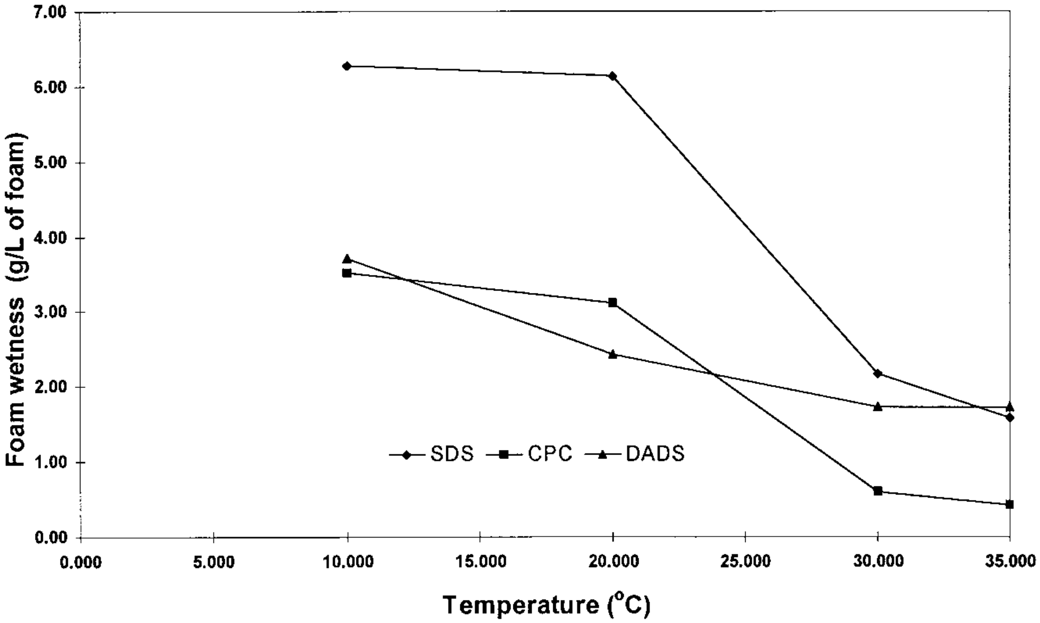

FIG. 11 Effect of temperature on foam wetness.

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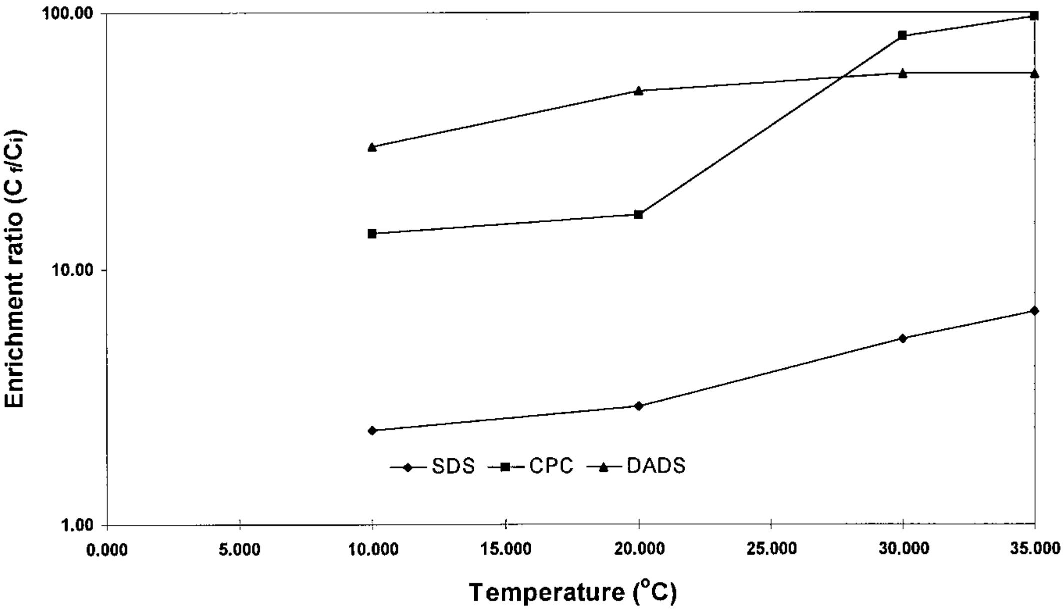

FIG. 12 Effect of temperature on enrichment ratio.

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ity (once formed) is generally decreased. These opposing effects make prediction of temperature effects on foam fractionation difficult. For SDS, the rate of surfactant recovery decreases with increasing temperature, as shown in Fig. 13, because the decrease in the collapsed foam flow rate is much higher than the increase in the surfactant concentration in the collapsed foam as the temperature increases. These two counteracting effects appropriately cancel for CPC and DADS so that the rate of surfactant recovery is almost independent of the temperature for these surfactants.

The effect of temperature on foam wetness, enrichment ratio, and rate of surfactant recovery are in reasonable agreement with Grieves and coworkers (26, 27), whereas the effect of temperature on volumetric foam production rate contrasts with that found in those works, probably because this effect is very system-dependent. Another possible explanation is that Grieves and Wood (26) studied higher temperatures than those used here.

Since different surfactant concentrations were used in the experiments to avoid micelle formation and because surfactant concentration can have a large effect on foam fractionation (4), it is difficult to generalize about the effect of surfactant structure. However, the removal of SDS seems to be more temperature-dependent than that of CPC or DADS, possibly because the SDS CMC is so much higher (Fig. 5), so that at a surfactant concentration which is a defined fraction of the CMC, the surfactant monomer concentration is higher for SDS.

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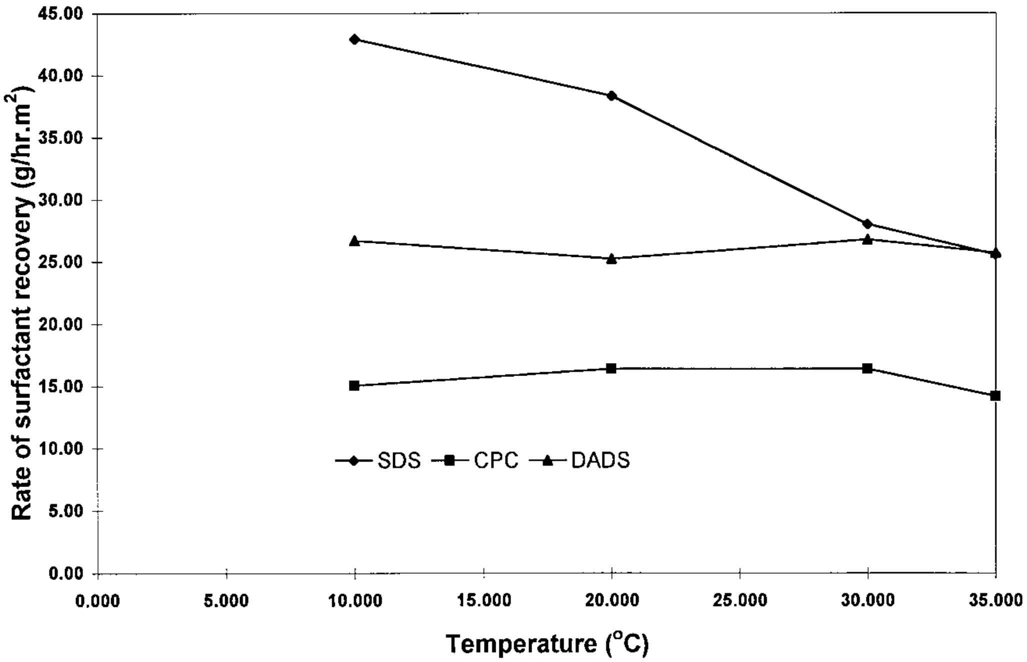

FIG. 13 Effect of temperature on rate of surfactant recovery.

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CONCLUSION

As temperature increases, the enrichment ratio increases for all three surfactants (approaching 100 in one case) while rate of surfactant recovery stays approximately constant for CPC and DADS and mildly decreases for SDS. Therefore, increasing temperature has a generally positive impact on foam fractionation as a more concentrated foam liquid is recovered overhead.

As salinity is increased, the foam wetness increases as the enrichment ratio decreases, so generally a higher volume of a less concentrated solution is foamed overhead with a slightly increased surfactant recovery rate. Therefore, whether increased salinity is beneficial or not depends on the goals of the separation.

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