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Recovery of Dodecylbenzene Sulfonate from Adsorbing Colloid Flotation Foamates

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Recovery of Dodecylbenzene Sulfonate from Adsorbing Colloid Flotation Foamates

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Abstract

Dodecyl benzene sulfonate, used in the foam separation of $Fe(OH)_3$ floc, can be recovered from the collapsed foamate by treating the foamate with various salts (such as NaOH, Na_2CO_3, or Na_2SO_4), thereby displacing the surfactant from the floc. Both NaOH and Na_2CO_3 are very effective in surfactant recovery; Na_2SO_4 is much less effective.

INTRODUCTION

The removal of trace levels of toxic metals has become a problem of current interest in the area of advanced wastewater treatment. Toxic metals are not efficiently removed by biological treatment or by conventional drinking water treatment techniques. They not only interfere with the efficient operation of secondary (biological) treatment plants, but their presence in biological sludge interferes with the disposal of this material by some methods, especially land application. The extent of the problem has been discussed in detail in several publications (*1–5*).

Foam flotation methods have shown considerable promise for the removal of a variety of pollutants from wastewaters. A number of reviews on foam separation are available (*6–10*). These techniques are based on the fact that surface-active material tends to concentrate at the gas–liquid interface. On bubbling air through the solution, one adsorbs the surface-active material at the surface of the bubble, and separates it from the solution in the foam. The

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substance to be removed, if not surface active, can be made surface active through union with or adsorption of a surface-active material. For instance, adsorbing colloid flotation involves the addition of a coagulant (alum or ferric chloride) to produce a floc. The dissolved metal is adsorbed onto the floc particle and/or coprecipitated with it. A surfactant is then added, adsorbs onto a floc particle and renders it hydrophobic, and the floc (with adsorbed metal) is removed by air flotation.

When dealing with dilute waste, foam flotation techniques appear to possess some distinct advantages: low residual metal concentrations, rapid operation, low space requirements (important where land costs are high), flexibility of application to various metals at various scales, production of small volumes of sludge highly enriched with the contaminant, and moderate cost. The chemical costs and capital costs of wastewater treatment by adsorbing colloid flotation have been recently estimated and compared with those of lime precipitation (*11–13*). For instance, estimated costs (capital cost plus chemical costs) for lead removal from dilute wastewater by adsorbing colloid flotation were 75¢/1000 gal, while the corresponding costs by lime precipitation were $1.47/1000 gal. Economics appear to favor adsorbing colloid flotation by a substantial margin (*12, 13*).

Recycling of surfactant, the most expensive chemical used in the process on a cost-per-pound basis, would greatly reduce the chemical cost of foam separation treatment. Surfactant recovery would not only markedly improve the economics of the process, it would also reduce the likelihood of contamination of surface and ground water with surfactant if the sludge is deposited in a landfill. Recent reports have shown that the recovery of sodium lauryl sulfate from the collapsed foamate is feasible (*13–15*). Wilson et al. examined the displacement of surfactants by salts from the floc–water interface by statistical mechanical methods (*15*).

A surfactant which has been widely used for foam separation, other than sodium lauryl sulfate (*13–16*), is sodium dodecylbenzene sulfonate (*17, 18*). Here we report the recovery of sodium dodecylbenzene sulfonate (DBS) from a collapsed foamate containing ferric hydroxide floc by treatment of the foamate with various salts. It was found that hydroxide ion produced by bases (such as NaOH, Na_2CO_3) is extremely effective in surfactant displacement. Sulfate ion, despite its double negative charge, is far less effective in surfactant recovery than is hydroxyl ion.

EXPERIMENTAL

The apparatus used for foam flotation was a simple batch-type column previously described (*19*). A Pyrex glass column 90 cm in length with an inside diameter of 3.5 cm was used for the flotation. The bottom of the column was closed with a rubber stopper with holes for a gas sparger and a stopcock to take samples and to drain the column. The gas sparger was a commercially available gas dispersion tube. A lipped side arm near the top of the column served as a foam outlet.

House air was passed through glass wool to remove particulates, and through distilled water for controlled rehumidification, then through the gas dispersion tube at the bottom of the column. The air flow rate was adjusted with a needle valve. Laboratory grade DBS was used as the collector and frother without further purification. Fisher reagent-grade ferric nitrate was used for floc preparation. A stock solution of 1000 ppm Fe(III) was prepared weekly and kept refrigerated.

The procedure used for the DBS analysis is the methylene blue method for the analysis of anionic surfactant as outlined in *Standard Methods* (*20*). The method consists of forming a methylene blue–surfactant complex in a buffered aqueous solution; the complex is then extracted quantitatively with chloroform. The extract is diluted with chloroform to a final volume of 100 mL, and analyzed colorimetrically. In this work colorimetric readings were determined using a Bausch and Lomb Spectronic 20 Spectrophotometer. A calibration curve was prepared from DBS solutions of known concentrations.

Procedure for Surfactant Recovery

The pH of 400 mL of solution containing 50 ppm Fe(III) was adjusted to the value 6.0, 25 ppm DBS was added, and final adjustment of the pH to 6.0 was made. The solution together with the floc was poured into the flotation column. It was very difficult to collect the floc quantitatively if some of the floc sticks on the wall of the glass column; this occurs if low air flow rates are used. Therefore a high air flow rate (about 250 mL/min) was used at the beginning of the runs; the air flow rate was adjusted to a lower value (about 100 mL/min) as soon as the top of foam reached the outlet of the column to minimize the amount of water carried out by the foam. The air flow rate was readjusted to a high value at the end of the run to remove the surfactant from the solution as completely as possible. Some of the floc still adhered to the top of the column; the floc could be easily collected in a beaker by rinsing the top of the column with distilled water. The volume of the collapsed foamate and washings was about 40 mL. The collapsed foamate and the floc were treated with solutions of NaOH, Na_2CO_3, or Na_2SO_4, and the solution was diluted to 100 mL. The solution was agitated with a magnetic stirrer for 20 min. The floc was then removed by filtration. The filtrate was diluted to 200 mL. The concentrations of DBS in the filtrate and in the effluent were measured by the methylene blue extraction method described above.

RESULTS AND DISCUSSIONS

The optimum pH for foam separation of ferric hydroxide with DBS is about 6.0. At pH 5, a higher concentration of surfactant is needed to maintain a stable foam. Separation efficiency can be easily observed visually by the removal of the yellow to brown colored floc from the solution into the foam. No floc remained in the solution after aeration at pH 6 for a few minutes. All runs for the flotation of ferric hydroxide were performed at pH 6.0. The surfactant was recovered from the foamate. DBS concentration in the liquid in the column at the end of a run was about 1.5 ppm for all runs.

An initial series of experiments was carried out by adjusting the pH of the collapsed foamate and floc with NaOH solution to determine the effect of pH on surfactant recovery. The results are presented in Table 1. Approximately 95% of the total surfactant was utilized for foam production; the remainder was left behind in the solution in the column (the effluent).

Essentially 100% of the surfactant in the foamate was recovered by adjusting the pH of the foamate to 12. The total surfactant (surfactant in the foamate and that in the effluent) recovered was 1.018 g, which compares with the theoretical value of 1.000 g with a 2% error. 95% of the total surfactant was recovered from the foamate for the run at pH 12, and 86% of the total surfactant was recovered from the foamate for the runs at pH 9 or 10. Only 30% of the total surfactant was recovered from the foamate when the pH of the foamate was left at approximately 6.0. It is evident that most of the surfactant was adsorbed on the floc in this case.

Another series of experiments was performed by treating the floc and the foamate with various amounts of Na_2CO_3. 93% of the total surfactant was

TABLE 1
Recovery of DBS by Treating the Foamate with NaOH

pH	DBS in effluent (mg)	DBS in filtrate (mg)	DBS recovered (%)	Total DBS found (%)
6	0.560	2.99	30	36
9	0.504	8.64	86	91
10	0.555	8.60	86	92
11	0.480	9.70	97	102

TABLE 2
Recovery of DBS by Treating the Foamate with Na_2CO_3

Na_2CO_3 (m*M*)	pH	DBS in effluent (mg)	DBS in filtrate (mg)	DBS recovered (%)	Total DBS found (%)
0.5	9.6	0.504	9.30	93	98
2.5	10.4	0.525	9.10	91	96

recovered from the foamate when the foamate was made 0.5 m*M* in Na_2CO_3 (shown in Table 2). The pH of the solution was found to be 9.6. The recovery of DBS was improved somewhat by treating the foamate with Na_2CO_3 rather than with NaOH to the same pH (9.0 to 10.0); this is probably due to the displacement of the surfactant from the surface of the floc by carbonate ion in addition to hydroxyl ion.

The result of surfactant recovery by treating the foamate and the floc with Na_2SO_4 is shown in Table 3. Less than 60% of the total surfactant was recovered when the foamate was treated with Na_2SO_4 solution (with the concentration of Na_2SO_4 as high as 0.01 *M*). The doubly charged sulfate ion is not as effective as hydroxyl ion for surfactant recovery. The high efficiency in surfactant recovery from the foamate and the floc by treating with base may be due to specific chemisorption of hydroxide ion on the ferric hydroxide, on which it is a potential-determining ion. The surface of the floc is positively charged at pH 6 and negatively charged at pH 9 or higher. The anionic surfactant can much less easily remain adsorbed on the surface of the floc in the basic solution due to coulombic repulsion.

All the surfactants recovered were found to be reusable for foam flotation with qualitatively the same efficiency as that of the freshly prepared surfactant.

TABLE 3
Recovery of DBS by Treating the Foamate with Na_2SO_4

Na_2SO_4 (m*M*)	pH	DBS in effluent (mg)	DBS in filtrate (mg)	DBS recovered (%)	Total DBS found (%)
1	6.5	0.525	4.60	46	51
5	6.7	0.525	5.90	59	64
10	6.7	0.555	5.30	53	58

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