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Solvent Sublation and Adsorbing Colloid Flotation of Acid Red

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Abstract

Acid Red 114, an anionic dye, was removed from aqueous solution by solvent sublation of Acid Red–hexadecyltrimethylammonium complex into paraffin oil. 95% of Acid Red was removed from the solution in 5 min by solvent sublation. A stoichiometric amount of surfactant (1 mol surfactant to 1 mol dye) was found to be most effective for Acid Red removal. The effect of neutral salts and ethanol on solvent sublation was studied. Adsorbing colloid flotation of Acid Red with $Fe(OH)_3$ floc and sodium lauryl sulfate was very effective, with 99% of Acid Red removal in 5 min. Adsorbing colloid flotation of Acid Red with $Al(OH)_3$ floc was also effective, with 99% of Acid Red removal in 3 min. The effect of neutral salts on adsorbing colloid flotation was studied.

INTRODUCTION

Solvent sublation, a surface chemical separation method originated by Sebba (*1*), has shown promise for the removal of certain types of organic compounds from aqueous systems. In this procedure a surface-active solute is transported from the aqueous phase to an overlying layer of a nonvolatile organic liquid on the air–water interfaces of bubbles rising through the solvent sublation column. Volatile solutes of low solubility in water may be removed in the interior of the bubble in a similar fashion by air stripping into the organic layer. Although adsorptive bubble separation techniques have been studied extensively, the literature on the subarea of solvent sublation is rather sparse. Lemlich's book on adsorptive bubble separation includes an excellent review on solvent sublation through 1970 by Karger (*2*). Wilson et al. included material on this subject in a more general review (*3*) and two recent books (*4*, *5*).

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Works of particular relevance to the present one include the following studies. Caragay, Karger, and Lee separated methyl orange from rhodamine B with a cationic surfactant at a pH at which the methyl orange was anionic while the rhodamine B was zwitterionic (*6*,*7*). Karger, Pinfold, and Palmer carried out a detailed study of the solvent sublation of a methyl orange–hexadecyltrimethylammonium ion pair (*8*). Womack, Lichter, and Wilson (*9*) reported on the solvent sublation of two dye–surfactant ion complexes, methylene blue–tetradecyl sulfate and methyl orange–hexadecyltrimethylammonium. We recently reported on the solvent sublation of a cationic dye, magenta, with sodium lauryl sulfate (*10*), and an anionic dye, C.I. Direct Red 1, with hexadecyltrimethylammonium ion (*11*).

Other works of particular interest to us are the extensive works of Wilson's group on solvent sublation of various organic pollutants from aqueous systems, such as solvent sublation of alkyl phthalates, volatile chlorinated organics, dichlorobenzenes, nitrophenols, polynuclear aromatics, and chlorinated pesticides (*3–5*, *12–16*). Grieves et al. (*17*) studied the removal of phenol by solvent extraction, solvent sublation, and foam fractionation.

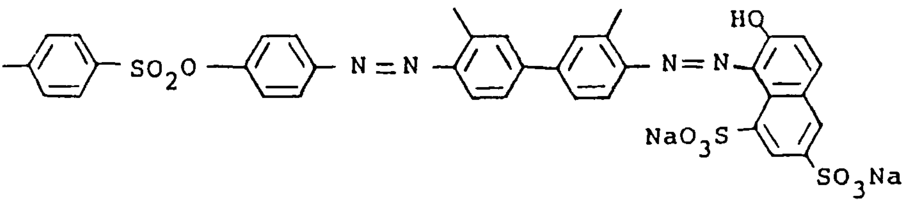
The adsorbing colloid flotation technique has been found to be very effective in removing various inorganic constituents from aqueous solutions. A number of excellent reviews are available (*3*, *5*, *18–20*). This technique involves the addition of a coagulant (alum or ferric chloride) to produce a floc. The dissolved inorganic constituents (metal ions or anion) are adsorbed onto the floc particle and/or coprecipitated with it. A surfactant is then added, adsorbs onto the floc particle, and renders it hydrophobic. On bubbling the air through the solution, the floc is adsorbed on the rising bubble and carried out from the solution by the foam.

Applications of adsorbing colloid flotation for organic constituents removal are rare. We have recently reported that both megenta (a cationic dye) and C.I. Direct Red 1 (an anionic dye) can be effectively removed from aqueous solutions by adsorbing colloid flotation (*10*, *11*).

In this paper we describe some studies we have recently made on the solvent sublation of Acid Red 114 (an anionic dye) from aqueous solution. Hexadecyltrimethylammonium bromide (HTA) was used as the collector, and paraffin oil was used as the top organic layer. It was found that 95% Acid Red is removed from the solution by solvent sublation in 5 min.

It was also attempted to remove Acid Red by adsorbing colloid flotation. Ferric hydroxide and aluminum hydroxide were used as the coprecipitants. Sodium lauryl sulfate (NLS) was used as the collector and frother. The rate of removal was very fast; 97% of Acid Red was removed by adsorbing colloid flotation with 10 ppm of Fe(III) in as little as 3 min.

The structure formula of Acid Red 114 is



EXPERIMENTAL

The adsorbing colloid flotation system used was similar to that described earlier (*10*, *11*, *21*). Figure 1 depicts the apparatus used for the batch separations. A soft glass column 90 cm in length with an inside diameter of 3.5 cm was used for the flotation. There was a side arm with a rubber septum near the bottom to inject the collector. 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.

The solvent sublation system used was similar to that used for adsorbing colloid flotation as described above except that the column was shorter (60 cm in length).

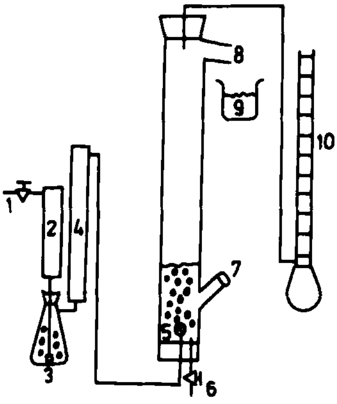


FIG. 1. The apparatus for foam separation. (1) Air needle valve, (2) Ascarite tube for CO_2 removal, (3) humidifier, (4) glass wool column, (5) fritted glass sparger, (6) drain, (7) reagent syringe, (8) foam discharge port, (9) discharged foam, (10) soap film flowmeter.

Compressed air was generated from an air pump. The air flow rate was adjusted with a Hoke needle valve with micrometer control and measured with a soap film flowmeter. The air was purified by passing it through glass wool to remove particulates, through Ascarite to remove carbon dioxide, and through distilled water for controlled rehumidification.

Reagent-grade sodium lauryl sulfate (NLS, Wako Pure Chemical Industry, Japan) and hexadecyltrimethylammonium bromide (HTA, Aldrich, 95%) were used as collectors without further purification. Reagent-grade Acid Red 114 (Everlight Chemical Industry, Taiwan, ROC), ferric nitrate, and aluminum nitrate were used for sample preparation.

For the solvent sublation runs, HTA was added to the sample solution to form the dye–surfactant complex, the pH of the solution was adjusted, the solution was poured into the separation column, 10 mL paraffin oil was added immediately, and the timer was started. Air flow was maintained at 120 mL/min.

For the adsorbing colloid flotation runs, ferric nitrate or aluminum nitrate was added to the sample solution, the pH was adjusted to produce the floc, Acid Red was adsorbed on or coprecipitated with the floc, NLS was added to render the floc hydrophobic, and the solution was then poured into the column for separation. Air flow rate was maintained at 86 mL/min. NLS dose was 50 ppm for the runs.

All runs were made with 250 mL of test solution which contained 25 ppm Acid Red. The experiments were performed at room temperature.

The pH of the solution was measured with a Radiometer pHM83 Autocal pH meter.

The absorption of visible light by the dye solution was measured with a Jasco 505 UV/VIS spectrophotometer. Spectrophotometric experiments revealed a peak shift and change in absorbance for Acid Red in the aqueous phase when HTA was added. The change in absorbance and peak shift depended on the HTA to Acid Red mole ratio (peak maximum at 522.5 nm for dye solution and at 501.0 nm for dye solution with equal mole of HTA). The Acid Red concentration of the solution was estimated by comparing its absorbance at peak maximum to the calibration curves of solutions with the same dye to HTA mole ratio (assuming the dye to HTA mole ratio does not change too much during solvent sublation).

RESULTS AND DISCUSSION

The effect of HTA concentration on the solvent sublation of Acid Red is shown in Table 1. It was found that a 1:1 mole ratio of surfactant to dye gave the fastest rate of separation and the lowest residual dye concentration; 95% of Acid Red was removed in 5 min. At lower surfactant concentration, the rate of separation is slower and the residual dye level

TABLE 1
The Effect of HTA Concentration on Solvent Sublation (percent removal)[a]

HTA/dye (mole ratio)	Time (min)				
	5	10	15	20	25
0.5	<60	<60	<60	<60	—
0.8	68	67	63	—	—
1.0	95	96	96	97	97
1.2	<60	78	80	79	80
2.0	<60	<60	<60	<60	<60

[a] pH = 4.5.

is higher, presumably due to incomplete dye–surfactant complex formation. The complex formed by Acid Red and HTA appears to be a 1:1 mole ratio, in spite of there being two sulfonate group on the dye molecule. This is probably because one of the sulfonate groups cannot react with HTA due to a steric effect. The optimum separation with surfactant dosage at the stoichiometric amount (surfactant to dye with a 1:1 mole ratio) was also observed for the solvent sublation of HTA–Acid Red (*11*) and the solvent sublation of NLS–magenta (*10*). Note that this finding was quite different from the solvent sublation results for methylene blue and methyl orange studied by Wilson et al. (*9*) and Karger et al. (*6–8*), who found that the rate of removal of methylene blue and methyl orange increased with increasing surfactant concentration, which was much in excess of the stoichiometric amount. This contradiction is probably due to the difference in the formation constant of the different surfactant–dye complexes and may also be due to the difference in the dye to surfactant mole ratio of the complexes. A large excess of surfactant dosage resulted in a very poor separation of Acid Red; this is presumably due to the competition for the bubble surface by the excess surfactant ion with the dye–surfactant complex. The excess surfactant also causes the emulsification of the paraffin oil (which was observed during the solvent sublation process with a large excess of surfactant), such that the dye–surfactant complex in the paraffin oil is constantly dispersed back into the solution and the separation efficiency cannot be improved by aeration for a longer time. Note that even at lower surfactant dosage, Acid Red cannot be removed from the solution any further after the first 10 to 15 min of the runs. A similar phenomenon was also observed by Tamamushi and Wilson (*12*) in the solvent sublation of diethylphthalate into paraffin oil. They suggested that some mass transfer of sublate from the paraffin oil back into the water layer is occurring. They proved this assumption by replacing the paraffin oil during a run with a fresh portion of paraffin oil; a very substantial increase in the removal rate was observed. The extremely high rate of separation of the Direct Red–HTA complex by solvent sublation (95% of removal in 5 min) suggests that the aggregates of the Acid Red–HTA complex may exist in the solution such that the removal of the aggregates may be responsible for the high rate of separation. The existence of aggregates of the surfactant–dye complex was also proposed earlier to explain the phenomena of a fast initial removal rate and the subsequent marked decrease in the solvent sublation of magenta–NLS (*10*). The sharp drop of removal rate was also observed on the solvent sublation of Direct Red–HTA complex (*11*).

The effect of pH on separation is shown in Table 2. 95% of Direct Red was removed from the solution by solvent sublation in 5 min at pH 4.0 to 4.5. The rate of separation and separation efficiency decreased significantly if the pH of the solution was either too high or too low, probably due to interference with Acid Red–HTA complex formation by the added acid or base, and may also be due to the influence of the protonation of the acidic group of the dye.

The effects of $NaNO_3$ and Na_2SO_4 on the rate of removal of Acid Red–HTA complex by solvent sublation are shown in Table 3. The separation was not affected by the presence of 0.1 *M* $NaNO_3$, but the separation efficiency decreased significantly when the concentration of $NaNO_3$ was greater than 0.5 *M*. The separation efficiency decreased somewhat for solutions containing 0.05 or 0.10 *M* Na_2SO_4, and the separation became very poor for solutions containing Na_2SO_4 greater than 0.5 *M*. The decrease in separation efficiency is presumably due to the competition for collector between the colligend and the ions of salts (nitrate or sulfate). Wilson et

TABLE 2
The Effect of pH on Solvent Sublation (percent removal)[a]

	Time (min)				
pH	5	10	15	20	25
3.0	87	87	87	87	87
3.5	90	92	92	92	93
4.0	95	96	96	97	97
4.5	95	96	96	97	97
5.0	94	95	96	96	97
5.5	87	88	88	88	88
6.0	85	86	87	88	88
6.5	66	68	64	61	<60
7.0	<60	<60	<60	<60	<60

[a] HTA/dye (mole ratio) = 1.0.

TABLE 3
The Effect of $NaNO_3$ and Na_2SO_4 Concentration on Solvent Sublation (percentage removal)[a]

$NaNO_3$ (*M*)	Na_2SO_4 (*M*)	Time (min) 5	10	15	20
0	0	95	96	96	97
0.1	0	96	97	—	—
0.5	0	<60	<60	<60	<60
1.0	0	<60	<60	<60	<60
0	0.05	90	87	86	—
0	0.10	86	83	79	77
0	0.50	<60	<60	<60	<60
0	1.00	<60	<60	<60	<60

[a] HTA/dye = 1.0, pH = 4.5.

al. (*9*) also observed the inhibiting effect by neutral salt ($NaNO_3$, KCl, and NaH_2PO_4) on the solvent sublation of both methylene blue–tetradecyl sulfate and methyl orange–HTA dye–surfactant complexes.

The effect of added ethanol (representative of a polar organic solute) on the solvent sublation of Acid Red–HTA complex is shown in Table 4. The separation effiency decreased significantly with increasing ethanol concentration, presumably due to an increase of the solubility of the Acid Red–HTA complex by the alcohol in solution. Another possible explanation that the deleterious effect on separation by ethanol is due to interference with Acid Red–HTA complex formation by ethanol, is rejected since neither the absorbance nor absorption peak maximum of the Acid Red–HTA solutions varied with the alcohol content (note that both the absorbance and peak maximum changed with a change in the Acid Red to HTA ratio).

TABLE 4
The Effect of Ethanol Concentration on Solvent Sublation (percent removal)[a]

Ethanol (vol%)	Time (min) 5	10	15	20	25
0.5	89	94	98	—	—
0.8	73	73	75	76	78
1.0	<60	<60	<60	<60	<60
5.0	<60	<60	<60	<60	<60

[a] HTA/dye = 1.0, pH = 4.5.

TABLE 5
The Effect of pH on Adsorbing Colloid Flotation with $Fe(OH)_3$ (percent removal)[a]

Time (min)	pH				
	4.0	4.5	5.0	5.5	6.0
3	97	98	98	97	94
5	98	98	99	98	96

[a] Fe(III) = 10 ppm, NLS = 50 ppm.

Another series of experiments was performed by using adsorbing colloid flotation techniques to remove Acid Red from aqueous solution. The effect of pH on adsorbing colloid flotation of Acid Red with ferric hydroxide floc is shown in Table 5. Sodium lauryl sulfate (NLS) was used as the collector and frother. Iron dosage was 10 ppm. The separation was very effective in the pH range 4.0 to 6.0; over 96% of Acid Red was removed in 5 min. The optimum pH for separation was 5.5, with 98% of Acid Red removal in 3 min and 99% removal in 5 min.

The effects of neutral salts, such as $NaNO_3$ and Na_2SO_4, and Fe(III) dosage on the separation efficiency of adsorbing colloid flotation of Acid Red are shown in Tables 6 and 7. The separation efficiency decreased with increasing ionic strength of the solution, presumably due to a decrease of the surface potential of the floc by the adsorption of anions in the solution, such that the surface potential of the floc was no longer positive enough for significant anionic surfactant adsorption. It was found that less than 60% of Acid Red was removed in 5 min from a solution containing 1.0 *M*

TABLE 6
The Effect of $NaNO_3$ and Fe(III) Concentration on Adsorbing Colloid Flotation with $Fe(OH)_3$ (percent removal)[a]

Fe(III) (ppm)	$NaNO_3$ (*M*)	Time (min)	
		3	5
10	0.5	99	99
10	0.8	75	78
10	1.0	<60	<60
20	0.8	99	99
20	1.0	88	93
30	1.0	90	93
40	1.0	95	97

[a] NLS = 50 ppm, pH = 5.0.

TABLE 7
The Effect of Na_2SO_4 and Fe(III) Concentration on Adsorbing Colloid Flotation with $Fe(OH)_3$ (percent removal)[a]

Fe(III) (ppm)	$NaNO_3$ (*M*)	Time (min) 3	5
10	0.01	82	88
20	0.01	88	94
30	0.01	97	98
30	0.02	97	98
30	0.05	<60	<60
50	0.05	<60	<60
100	0.05	<60	<60

[a] NLS = 50 ppm, pH = 5.0.

$NaNO_3$ by adsorbing colloid flotation with a 10-ppm dose of Fe(III); however, effective separation with 97% removal of Acid Red was achieved with a 40-ppm dose of Fe(III). The reason for the improved separation from a solution of high ionic strength by increasing the dose of Fe(III) can be explained as follows: The adsorption of anionic dye on $Fe(OH)_3$ floc decreases the surface potential of the floc. This may cause the surface potential of the floc to be insufficiently positive for efficient anionic surfactant adsorption. This deleterious effect is reduced by increasing the dose of Fe(III) to decrease the density of dye [mole of dye per unit surface area of $Fe(OH)_3$ floc] on the floc surface. Sulfate ions showed a greater effect on separation than did nitrate ions. The separation of Acid Red by adsorbing colloid flotation was very poor from a solution containing 0.05 *M* Na_2SO_4. Doubly charged anions were found to decrease the separation efficiency of various adsorbing colloid flotation systems to a greater extent than did singly charged anions (*21–25*).

Adsorbing colloid flotation of Acid Red with $Al(OH)_3$ was also studied. The effects of pH and aluminum dosage are shown in Table 8. The separation was also very effective; 99% of Acid Red was removed in 3 min at pH 7.0 to 7.5. The separation improved somewhat and the pH range for an effective separation was wider with a larger dose of aluminum (from 10 to 20 ppm).

The effects of $NaNO_3$, Na_2SO_4, and aluminum dosage on adsorbing colloid flotation of Acid Red with $Al(OH)_3$ are shown in Tables 9 and 10. Effective separations with Acid Red removal greater than 98% were achieved provided that the concentrations of dissolved salts were no greater than 0.5 *M* ($NaNO_3$) or 0.01 *M* (Na_2SO_4). The deleterious effect of in-

TABLE 8
The Effect of pH and Al(III) on Adsorbing Colloid Flotation with $Al(OH)_3$ (percent removal)[a]

Al(III) (ppm)	pH	Time (min) 3	Time (min) 5
10	7.0	87	88
10	7.5	98	98
10	8.0	88	88
20	7.0	99	99
20	7.5	99	99
20	8.0	96	97

[a] NLS = 50 ppm.

creasing ionic strength was not compensated for by increasing the dose of aluminum. A large excess dose of aluminum resulted in poorer separation, presumably due to an increase in the ionic strength of the solution by the added aluminum salt (and the extra base required to adjust the solution pH), and might also be due to the production of a greater amount of floc such that the time needed to remove the floc became longer.

We recently showed that the inhibition effect of neutral salts on adsorbing colloid flotation of various heavy metal ions and an anionic dye (Direct Red) with $Fe(OH)_3$ floc and NLS can be compensated for to quite a large extent with the aid of aluminum ion and zinc ion as the activators (*21*, *22*, *24*, *26*, *27*). We attempted to use this technique on the removal of Acid

TABLE 9
The Effect of $NaNO_3$ and Al(III) on Adsorbing Colloid Flotation with $Al(OH)_3$ (percent removal)[a]

Al(III) (ppm)	$NaNO_3$ (*M*)	Time (min) 3	Time (min) 5
20	0.5	98	99
20	0.8	77	82
30	0.8	79	84
50	0.8	73	79
80	0.8	71	79
40	1.0	69	70
80	1.0	66	70
100	1.0	<60	<60
200	1.0	<60	<60

[a] NLS = 50 ppm, pH = 7.5.

TABLE 10
The Effect of Na_2SO_4 and Al(III) on Adsorbing Colloid Flotation with $Al(OH)_3$ (percent removal)[a]

Al(III) (ppm)	Na_2SO_4 (*M*)	Time (min) 3	Time (min) 5
20	0.01	97	98
20	0.02	85	92
20	0.05	72	82
30	0.02	76	87
30	0.05	63	72
40	0.02	82	87
40	0.05	<60	64
50	0.02	<60	66
50	0.05	<60	65

[a] NLS = 50 ppm, pH = 7.5.

Red from solution with a high sulfate concentration by adsorbing colloid flotation. The results are shown in Table 11. Effective separation with 98% removal of Acid Red was achieved from a solution containing 0.05 *M* Na_2SO_4 by adsorbing colloid flotation with a 30-ppm dose of Fe(III) and a 20-ppm dose of Al(III). Note that the separation of Acid Red from 0.05 *M* Na_2SO_4 solution was very poor if either Fe(III) or Al(III) was used alone. The effect of aluminum ion as the activator is presumably due to the increase of the surface potential of the floc by the adsorption of aluminum(III) species on the $Fe(OH)_3$ floc (or by forming a mixed precipitate), such that a sufficient negatively charged surfactant (NLS) can be adsorbed on the surface of the floc and thus render the surface of the floc hydrophobic. This results in effective separation.

TABLE 11
The Effect of pH and Na_2SO_4 on Adsorbing Colloid Flotation with $Fe(OH)_3$ and Al(III) (percent removal)[a]

pH	Time (min) 3	Time (min) 5
5.0	83	88
5.5	97	98
6.0	74	97
6.5	82	92
7.0	70	79

[a] NLS = 50 ppm, Fe(III) = 30 ppm, Al(III) = 20 ppm, Na_2SO_4 = 0.05 *M*.

Adsorbing colloid flotation of Acid Red with $Fe(OH)_3$ from a solution containing 0.1 *M* Na_2SO_4 was not very effective even if aluminum ion and/or sinc ion were used as the activators. The optimum separation of Acid Red from 0.1 *M* Na_2SO_4 solution by absorbing colloid flotation was a run at pH 5.5 with 30 ppm Fe(III), 20 ppm Al(III), 20 ppm Zn(II), and 50 ppm NLS. 71% of Acid Red was removed in 25 min under such conditions.

CONCLUSION

Acid Red 114, an anionic dye, can be removed effectively from aqueous solution into paraffin oil by solvent sublation of the Acid Red–HTA complex with a cationic surfactant, hexadecyltrimethylammonium (HTA) bromide. 95% of Acid Red was removed from the solution in 5 min by solvent sublation. A stoichiometric amount of surfactant (1 mol surfactant to 1 mol dye) was found to be most effective for Acid Red removal. The removal of the aggregates of Acid Red–HTA complex may be reponsible for the high rate of Acid Red removal by solvent sublation. The separation was not affected by the presence of 0.1 *M* $NaNO_3$, but the separation efficiency decreased significantly when the concentration of $NaNO_3$ was greater than 0.5 *M*. The decrease in separation efficiency is presumably due to the competition for collector between the colligend and the salt ions. Ethanol has a deleterious effect on the solvent sublation of Acid Red–HTA, presumably due to an increase of the solubility of the Acid Red–HTA complex by alcohol.

Adsorbing colloid flotation of Acid Red with $Fe(OH)_3$ floc and NLS (sodium lauryl sulfate) is very effective: 98% of Acid Red removal in 3 min and 99% in 5 min. Effective separation with 97% removal of Acid Red from a solution containing 1.0 *M* $NaNO_3$ was achieved by absorbing colloid flotation with $Fe(OH)_3$. Sulfate ion has a deleterious effect on the separation of Acid Red by adsorbing colloid flotation with $Fe(OH)_3$. This deleterious effect can be somewhat compensated for with the aid of aluminum ion as the activator.

Adsorbing colloid flotation of Acid Red with $Al(OH)_3$ and NLS was also effective, with 99% removal of Acid Red in 3 min. Effective separations of Acid Red by adsorbing colloid flotation with $Al(OH)_3$ were achieved provided that the concentrations of dissolved salts were no greater than 0.5 *M* ($NaNO_3$) or 0.01 *M* (Na_2SO_4).

The deleterious effect of neutral salts on the adsorbing colloid flotation of Acid Red is presumably due to a decrease of the surface potential of the floc by the adsorption of the anions in the solution, such that the surface potential of the floc was no longer positive enough for a significant amount of anionic surfactant adsorption.

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