

more dense than the ethane-rich liquid over the entire three-phase region.

DATA ANALYSIS

Isothermal vapor and liquid composition data for the carbon monoxide-propane system were fitted to empirical equations in the form of power series in pressure. The mathematical form of the vapor and liquid polynomials, as well as the least-squares constants, are presented by Trust (7).

The average standard deviation of the experimental liquid phase data from the curve was 0.28 mole % carbon monoxide. For the vapor phase the average standard deviation of the experimental data from the curves was 0.40 mole % propane.

The experimental data for the carbon monoxide-ethane system were not fitted to equations.

ACKNOWLEDGMENT

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NOTATION

- P = pressure, lb./sq.in.abs.
 T = temperature, °C.
 X = liquid phase composition, mole %
 Y = vapor phase composition, mole %

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Precipitate Flotation of Chromium

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A three-stage process of reduction of acid chromate (HCrO_4^-), with NaHSO_3 followed by precipitation of Cr(III) with NaOH followed by flotation with an anionic surfactant, provides 97% removal of chromium from aqueous suspensions containing 48.3 mg./liter Cr (0.929 mM). The required dosage of surfactant (sodium laurylsulfate) is 0.093 mole SLS/mole Cr, compared to a dosage of 1.1 mole cationic surfactant/mole Cr, used to ion float soluble acid chromate. The precipitate flotation process is pH dependent: below pH 6.3, soluble chromium species become appreciable and the flotation results agree with calculated soluble chromium concentrations; above pH 9.7, the charge of the precipitate is reversed, as indicated by surface potential measurements. The optimum pH range for flotation is 7.0 to 8.8. The effect of initial surfactant concentration on the flotation efficiency is established. Small quantities of calcium enhance the flotation of Cr, while higher concentrations, 0.5 mM and greater, decrease flotation efficiency. For suspensions with a doubled chromium concentration (96.6 mg./liter), the optimum pH range for flotation is lowered and narrowed to 6.3 to 6.5, indicating modifications in particle surface characteristics validated by measurements of surface potential and surfactant adsorption. Eighty-seven percent flotation is achieved at a 0.093 mole/mole ratio.

The precipitate flotation technique has received attention from a number of investigators, beginning with the work of Skrylev and Mokrushin (1) and that of Baarson and Ray (2). A number of studies have been carried out (3 to 6), including an excellent group of papers by Pinfold et al. (7 to 12) and another by Rubin et al. (13 to 15).

Grievess and Bhattacharyya (16) have investigated the separation of complexed cyanide by precipitate flotation. The present study involves the precipitate flotation of chromium from aqueous suspension as chromic hydroxide. Grievess et al. (17 to 20) have investigated the ion flotation technique for the removal of chromium (as soluble acid

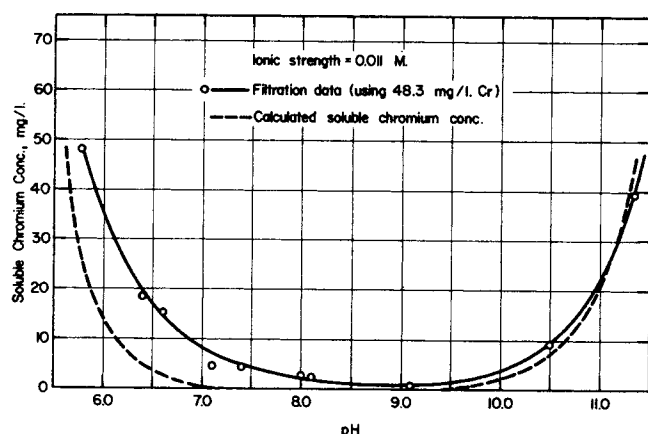


Fig. 1. Total soluble chromium concentrations calculated from equilibrium constants and determined from filtration experiments.

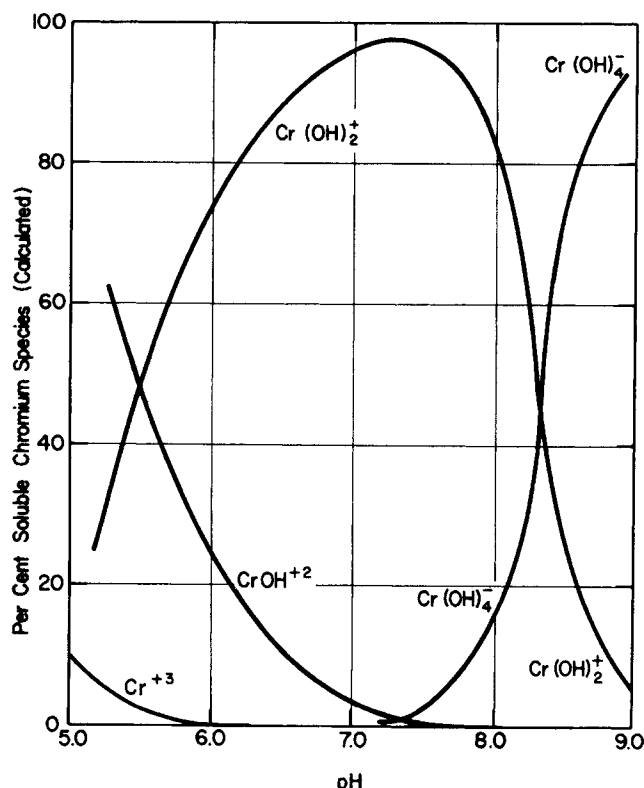


Fig. 2. Distribution of soluble chromium species (calculated) as a function of pH.

chromate) from aqueous solution. Ion flotation requires stoichiometric or greater concentrations of surfactant, whereas precipitate flotation may require a quantity of surfactant only 5 to 20% of the stoichiometric (16).

In any precipitate-solution system, the initial charge of the precipitate plays an important role in the adsorption of a surfactant on the particles. The precipitate acquires the charge either by desorption of one of the ionic species of the solid or by the adsorption of ions from solution onto the surface of the crystal. The constituent ions of the precipitate present in solution are preferentially adsorbed over other ions; because OH^- is a constituent ion, pH has a most pronounced effect on the charge of hydroxide precipitates. The surfactant, added as a collector-frother in a flotation process, plays three roles: the adsorption of the surfactant on the surfaces of the particles makes the pre-

cipitate suitable for gas bubble attachment; interaction between surfactant adsorbed on the particles and "free" surfactant adsorbed at the gas-liquid interfaces associated with the bubbles produces bubble attachment of the particles; and "free" surfactant acts as a frother producing a stable foam, which may be further stabilized by the presence of particulates.

EXPERIMENTAL PROCEDURE

Initial solutions of chromium (VI), containing 48.3 mg./liter (0.929 mM) or 96.6 mg./liter as Cr, were prepared by dissolving known amounts of $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ in distilled water. For some experiments varying quantities of CaCl_2 or NaCl were added to the solutions. The pH of the solutions was then adjusted to between 2.5 and 3.0 with 1.0 N HCl. Sodium bisulfite in the molar ratio of $\text{HSO}_3^-/\text{HCrO}_4^-$ of 2:1 was used for complete reduction of Cr(VI) to Cr(III). After the addition of NaHSO_3 the solution was mixed for 15 min. to ensure 100% reduction. The precipitation of Cr(III) to chromic hydroxide was accomplished by adding 1.0 N NaOH; the pH was adjusted within the range 6.0 to 12.3, and the suspension was allowed to mix for 5 min. The anionic surfactant, sodium laurylsulfate (SLS), also called sodium dodecylsulfate or dodecyl sodium sulfate, was then added and was mixed with the suspension for an additional time period of 5 min. The initial SLS concentration was varied from 5.0 (0.017 mM) to 60.0 mg./liter. In a few experiments ethylhexadecyldimethylammonium bromide (EHDA-Br), a cationic surfactant, was used instead of SLS.

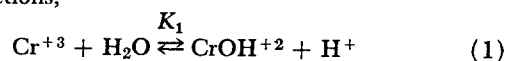
Two liters of the suspension were then placed in the foam separation column. The apparatus used has been described previously (21, 22). The foam separation column was made of Pyrex and was 82 cm. high and 9.7 cm. diam. Air was saturated with water, metered with a calibrated rotameter, and dispersed through twin, sintered glass diffusers of 50 μ porosity at a rate of 1,300 ml./min. (at 1 atm. and 25°C.). The suspension was foamed for 25 min., with continuous foam removal from a port located 7.0 cm. above the initial suspension level. Temperature was maintained at about 25°C. After each experiment was terminated, the volume of the residual suspension was measured, and the concentration of chromium was determined by atomic absorption spectrophotometry. All chromium concentrations were measured as mg./liter Cr. The residual concentration of SLS was determined by reacting the SLS with a known amount (in excess) of a cationic surfactant (EHDA-Br) and then by determining the unreacted EHDA-Br by a two-phase titration technique, using sodium tetraphenylboron (23).

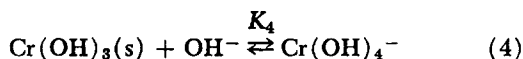
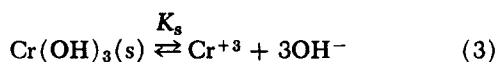
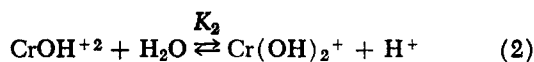
An estimate of precipitate particle size (before the addition of surfactant) was made by observing the particles under a microscope (Bausch and Lomb with zoom lens, 920 \times). The particles were ellipsoidal in shape with an average dimension in the range 15 to 20 μ . Since the major part of the flotation experiments was conducted at a chromium concentration of 48.3 mg./liter, microscopic observations were limited to this concentration only, with the suspension pH adjusted to 8.0. The number of particles present per unit volume of the suspension was measured by using a hemacytometer of 0.1 mm. depth and the microscope. The average number of particles per liter was computed to be 8.8×10^9 . The hemacytometer determination should have been reasonably accurate because 10-hr. settling tests yielded a supernatant with only 0.6 mg./liter Cr, indicating the absence of an appreciable number of small particles, the size of which would be beyond the range of the instrument.

RESULTS AND DISCUSSION

Effect of pH

The Precipitation of Chromium as Hydroxide. The variation of the concentration of soluble chromium species with pH can be calculated by studying the following hydrolysis reactions,





Bronsted and King (24) found that the first hydrolysis constant K_1 varied with ionic strength. The other constants undoubtedly are also functions of ionic strength but no information is available. The formation of the negatively charged Cr(OH)_4^- species occurs only at high pH values. At any pH,

$$\begin{aligned} &\text{Total soluble chromium (molar)} \\ &= [\text{Cr}^{+3}] + [\text{CrOH}^{+2}] + [\text{Cr(OH)}_2^+] + [\text{Cr(OH)}_4^-] \end{aligned} \quad (5)$$

From the equilibrium constant expressions for Equations (1) to (4),

$$\begin{aligned} &\text{Total soluble chromium (molar)} \\ &= K_s' [\text{H}^+]^3 + K_s' K_1 [\text{H}^+]^2 + K_s' K_1 K_2 [\text{H}^+] + \frac{K_4'}{[\text{H}^+]} \end{aligned} \quad (6)$$

$$K_s' = K_s(10^{+42})$$

$$K_4' = (K_4)(10^{-14})$$

Constant	log K	Reference
K_1	$-4.0 - 2.0\sqrt{\mu} + 4.5\mu$	24
K_2	-5.55	25
K_s	-30.0	26
K_4	-0.4	27

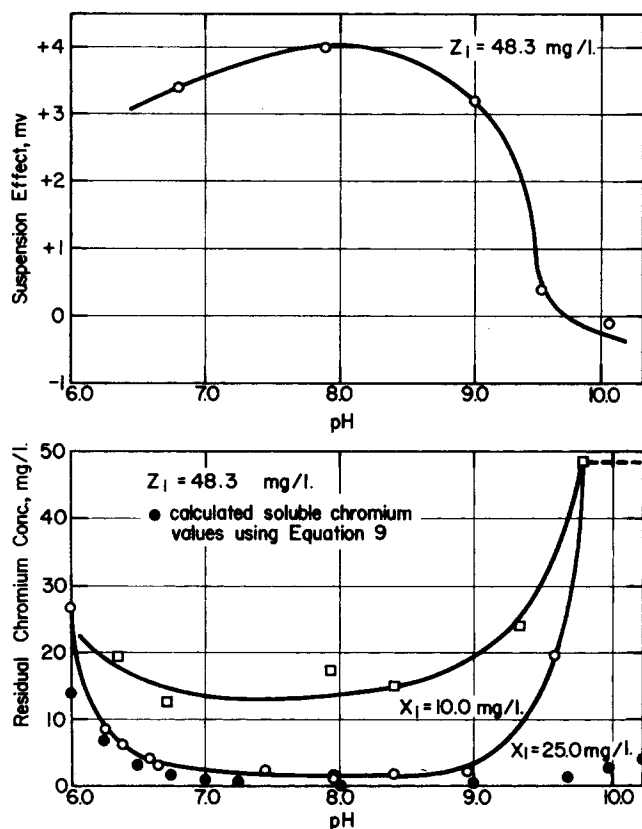


Fig. 3. Flotation results and surface potential measurements as functions of pH.

μ = ionic strength, molar

Below a pH of about 10.0, the last term in Equation (6) is insignificant.

Equation (6) was programmed and the values of the concentrations of total soluble chromium were evaluated at different pH's and ionic strengths. In Figure 1 the dotted curve shows the variation of the soluble chromium concentration (mg./liter) with pH at an ionic strength of 0.001 M. The concentration of soluble Cr(OH)_4^- becomes significant above pH 10.0. Figure 1 also shows the results of filtration experiments. These data were obtained by adjusting the pH of a 48.3 mg./liter Cr(III) suspension with 1.0 N NaOH and filtering the suspension through 0.45 μ Millipore filter paper. The filtrate was analyzed for chromium. Below pH 8.0 the soluble chromium concentrations obtained by filtration experiments are considerably higher than the calculated values. This may have been produced due to incomplete removal of all the particles by the filter paper.

Figure 2 shows the distribution of soluble chromium species (calculated) as a function of pH. The percentage of each of the species at a certain pH is computed by evaluating each term in Equation (6) and then by dividing the concentration of each species by the concentration of total soluble chromium at that pH. It should be emphasized that between pH 7.0 and 9.8, the concentration of total soluble chromium is less than 1.0 mg./liter.

The Flotation of Chromium. An initial series of flotation experiments (using SLS as the surfactant) was carried out in an effort to establish the optimum pH for the flotation of chromium, and also to determine the effect of particle characteristics (as modified by pH) on flotation results. All the results are presented in terms of residual chromium (including both total soluble and precipitated chromium) concentrations. Figure 3 (lower) shows the effect of pH on the residual chromium concentration Z_r with $Z_i = 48.3$ mg./liter and two values of X_i , the initial surfactant concentration. The use of 25.0 mg./liter surfactant gave almost 97% removal of total chromium in the optimum pH range of 7.0 to 8.8. Soluble chromium concentrations, computed from Equation (6), are also shown for comparison. Below a pH of about 7.0, as the soluble chromium concentration increased (precipitated chromium decreased), Z_r also increased; this can be explained by the fact that the removal of the soluble species Cr(OH)_2^+ and/or CrOH^{+2} (see Figure 2) would require stoichiometric concentrations of SLS (far in excess of 25 mg./liter). At pH 6.0, the incomplete flotation of the precipitate was the result of the enhanced reaction of the Cr(OH)_2^+ and CrOH^{+2} ions with the free surfactant, which was evidenced by the approach of V_r , the residual suspension volume, to 2.0 liters, the initial solution volume: at pH 6.6, $V_r = 1.59$, at pH 6.4, $V_r = 1.85$, and at pH 6.0, $V_r = 2.0$. In the last experiment, only a scum was formed atop the liquid suspension with very little froth, indicating that little surfactant was left unassociated with Cr species and was free to act as a frother.

Figure 3 shows a sharp increase in the residual chromium concentration above a pH of about 9.4. One value of $Z_r = 48.3$ mg./liter (no flotation) at $X_i = 25.0$ mg./liter and pH = 10.4 is not shown. Between pH 9.0 and 10.0 the flotation values are well above the calculated soluble chromium values, indicating inefficient or no flotation of the precipitate. This was possible if the precipitate had the same charge as the surfactant. This was the case, as can be seen from the results of the determination of the surface charge of the precipitate which are presented

in the top part of Figure 3, indicating the relation between suspension effect [potential difference (mv.)] and pH. The suspension effect was determined by the technique of Honig and Hengst (28). The positive values indicate positive charge on the precipitate. Reversal of charge occurred at about pH 9.7. Parks (29) reported charge reversal between pH 8.2 and 9.3. At and above pH 9.7, all of the soluble chromium species were in the form of $\text{Cr}(\text{OH})_4^-$. These negatively charged species, along with the excess OH^- ions, imparted a negative charge to the precipitate by exchanging with ions present in the crystal. Flotation of a negatively charged precipitate with a surfactant of the same charge is not feasible, particularly if coordination between the ionic species present in the precipitate and the surfactant is not significant.

A set of flotation experiments (with variable pH) was carried out using a cationic surfactant, EHDA-Br (28.0 mg./liter = 0.074 mM), with results given in Figure 4. Above pH 10.0, where the precipitate was negatively charged, partial flotation of the precipitate was observed. Complete flotation could be made possible by using higher EHDA-Br concentrations. The incomplete flotation was partially due to the competition of soluble $\text{Cr}(\text{OH})_4^-$, requiring stoichiometric concentrations of EHDA cations.

The concentration of the surfactant in the residual suspensions was monitored throughout the study. For all experiments no noticeable change in X_r with pH was observed. With SLS at $X_i = 25.0$ (pH 6.2 to 10.4), X_r ranged between 4.6 to 5.5 mg./liter and at $X_i = 10.0$ (pH 6.7 to 7.8), X_r ranged between 1.8 to 2.6 mg./liter. For the experiments with EHDA-Br the average value of X_r was 0.5 mg./liter.

Effect of Initial Surfactant Concentration

A second series of experiments was performed to study the effect of X_i (sodium laurylsulfate) on Z_r at two different pH values. The results of these experiments are presented in the bottom part of Figure 5. Variation in surfactant adsorption on the particulates with surfactant concentration was also determined for three of these experiments. It was found that equilibrium was approached within 5 min. of contact of surfactant with the suspension. The suspension was then filtered through 0.45 μ Millipore filter paper and the filtrate was analyzed for surfactant concentration. In Figure 5 (upper) the amount of SLS adsorbed (in milligrams) per unit amount of Cr in the precipitate is related to the equilibrium SLS concentration. For example, at $X_i = 25.0$ mg./liter, 3 mg./liter SLS was adsorbed on the precipitated particles and 22 mg./liter was in the form of free surfactant.

Effect of the Addition of Calcium Ion and of Sodium Ion

A third series of experiments was carried out to determine the effect of Ca^{+2} on the flotation of chromium. Calcium (in the form of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$) was added to the solutions before reduction with bisulfite. All experiments were performed at pH 8.0 and at two different initial SLS concentrations ($X_i = 10.0$ and $X_i = 25.0$ mg./liter). The selection of pH 8.0 was based on the fact that precipitation of Cr is complete at this pH and the increase in ionic strength should not change the concentration of precipitated chromium. Figure 6 (left side) presents the effect of added Ca^{+2} on the removal of chromium. Z_r passes through a minimum around 1.0 meq./liter Ca^{+2} for both

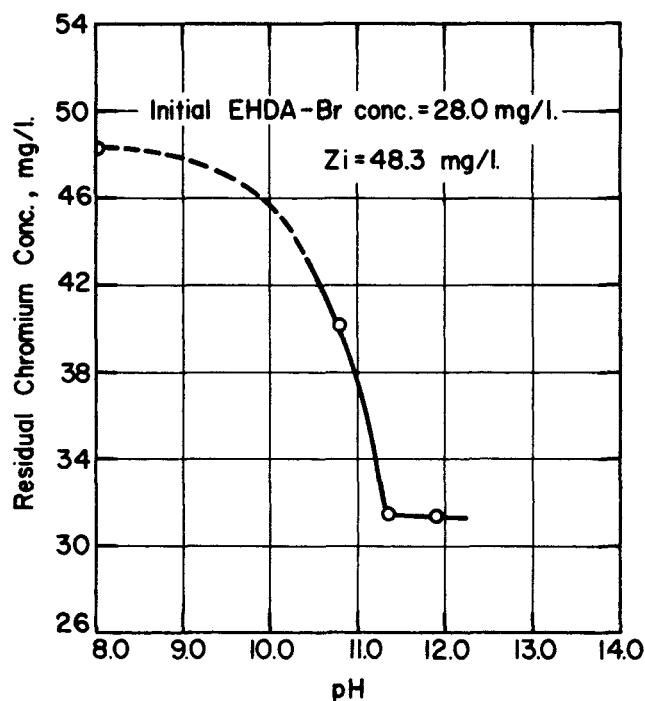


Fig. 4. Flotation results with a cationic surfactant as a function of pH.

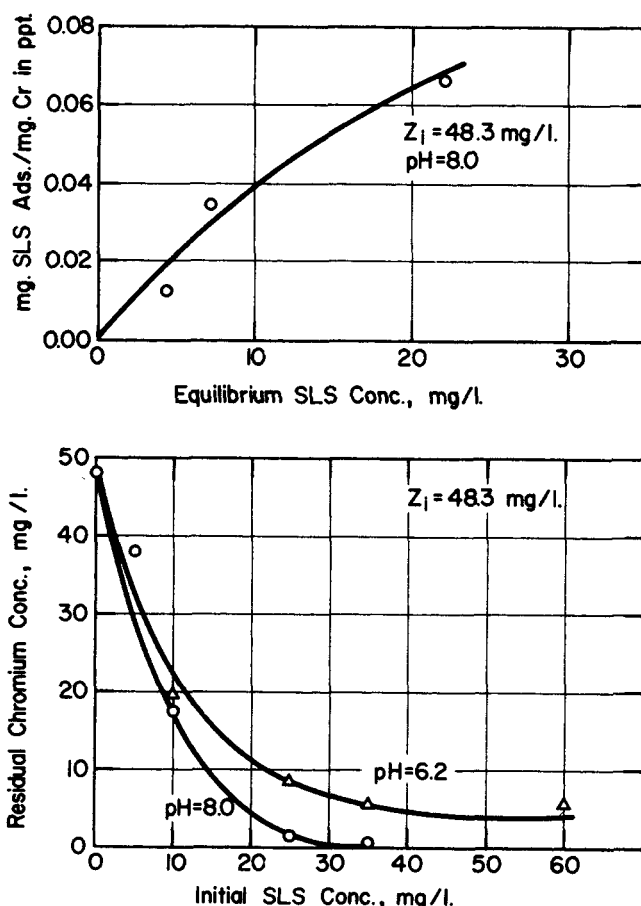


Fig. 5. Flotation results and adsorption measurements as functions of surfactant concentration.

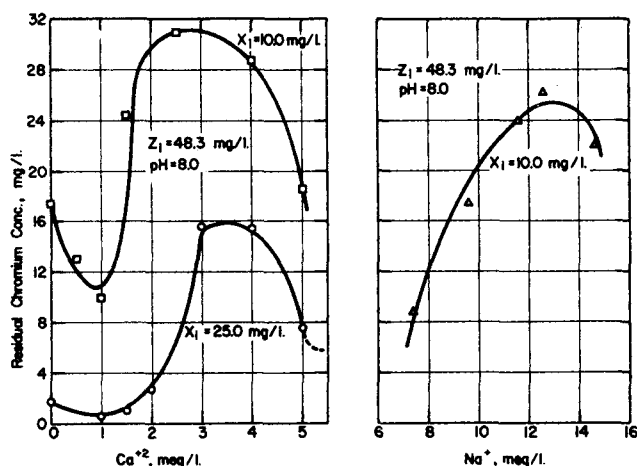


Fig. 6. Flotation results as functions of calcium and sodium concentrations.

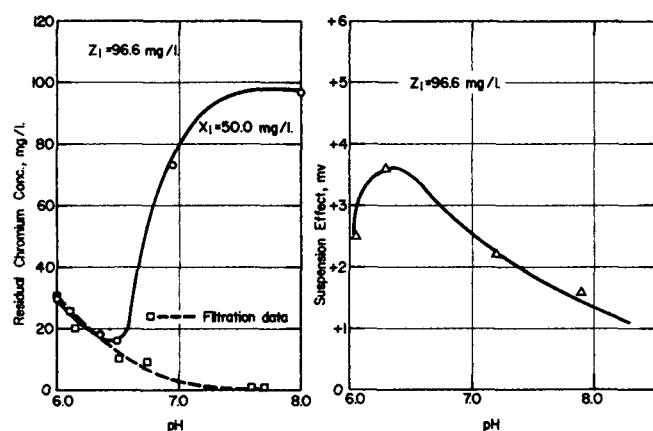


Fig. 7. Flotation results and surface potential measurements as functions of pH.

values of X_i , and the effect is more pronounced in the case of 10 mg./liter SLS. The minima were probably produced by modification by Ca^{+2} of the state of aggregation of the chromic hydroxide and consequently the surface area charge; and/or modification of the orientation of the adsorbed surfactant anions by extension of the hydrocarbon chains from the surface in a close-packed array with Ca^{+2} (30, 31). It is not likely that the increased ionic strength lowered the critical micelle concentration of the surfactant to a sufficient extent to produce micellization (32).

With an increase of Ca^{+2} from 1.0 to about 3.0 meq./liter the flotation of the precipitate decreased. Reduced flotation produced by increases in concentration of ions with like charge to the precipitate have been observed previously and possible causes have been detailed (12, 16). It should also be mentioned that over the entire range of added Ca^{+2} ions, flotation of about 0.15 to 0.2 meq./liter Ca^{+2} was observed.

Another series of experiments was conducted to determine the effect of Na^{+} (added as NaCl) on flotation characteristics. Figure 6 (right side) shows the results of these experiments performed at $X_i = 10.0$ mg./liter and at pH 8.0. A higher Na^{+} concentration was required than Ca^{+2} concentration to have an appreciable effect on Z_r . The maxima in the curves of Figure 6 (right and left) may have been produced by further modifications of surface area charge.

Effect of Initial Chromium Concentration

A final series of flotation experiments was performed with initial solutions containing 96.6 mg./liter Cr. The effects of pH and of initial SLS concentration were determined. The effect of pH was surprisingly different than that observed in the case of 48.3 mg./liter. Figure 7 (left) shows the effect of pH on the residual chromium concentration. The optimum pH was found to be between 6.4 and 6.5. Figure 7 also shows the values of soluble chromium obtained from filtration experiments. The filtration results were slightly lower than those obtained with 48.3 mg./liter Cr. This was caused by the increased ionic strength ($\mu = 0.016$ M) and can be predicted from Equation (6). It should be stressed that an increase in the initial chromium concentration also entailed an increase in ionic strength, principally due to SO_4^{-2} , produced from the reduction of Cr(VI) by NaHSO_3 .

Between pH 6.0 and 6.5, complete flotation of the precipitate occurred. To explain the behavior above pH 6.5, variations of suspension effect with pH were again determined. The measured values are presented in Figure 7 (right). The precipitate surfaces had the maximum positive charge between pH 6.2 and 6.5. Above this pH the potential differences dropped, indicating reduction of charge on the precipitate. The reduction of the charge on the precipitate, particularly above pH 7.0 (above pH 7.0 practically all of the Cr was in the precipitate form), may have been caused by the adsorption of excess SO_4^{-2} (33, 34). As the Cr concentration was doubled (48.3 \rightarrow 96.6 mg./liter), the SO_4^{-2} concentration was also doubled. It is also obvious that above pH 7.0 essentially no flotation occurred, and yet the suspension effect remained positive (although decreasingly). The zero flotation could have been produced by excessive quantities of free, nonadsorbed surfactant saturating the gas bubbles and preventing the attachment of virtually any of the particulates. This theory was further evidenced by a sharp increase in foam volume (decrease in V_r) when the pH was increased from 6.5 to 7.0 and above.

The effect of pH on the flotation of chromium can also

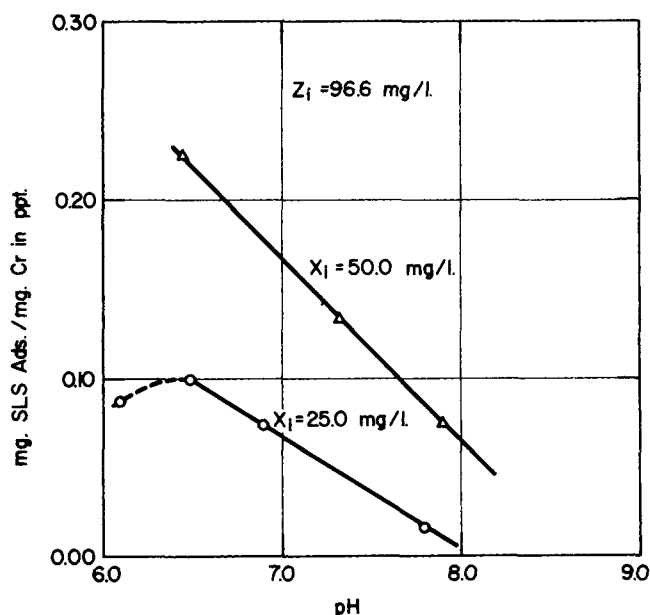


Fig. 8. Adsorption measurements as a function of pH.

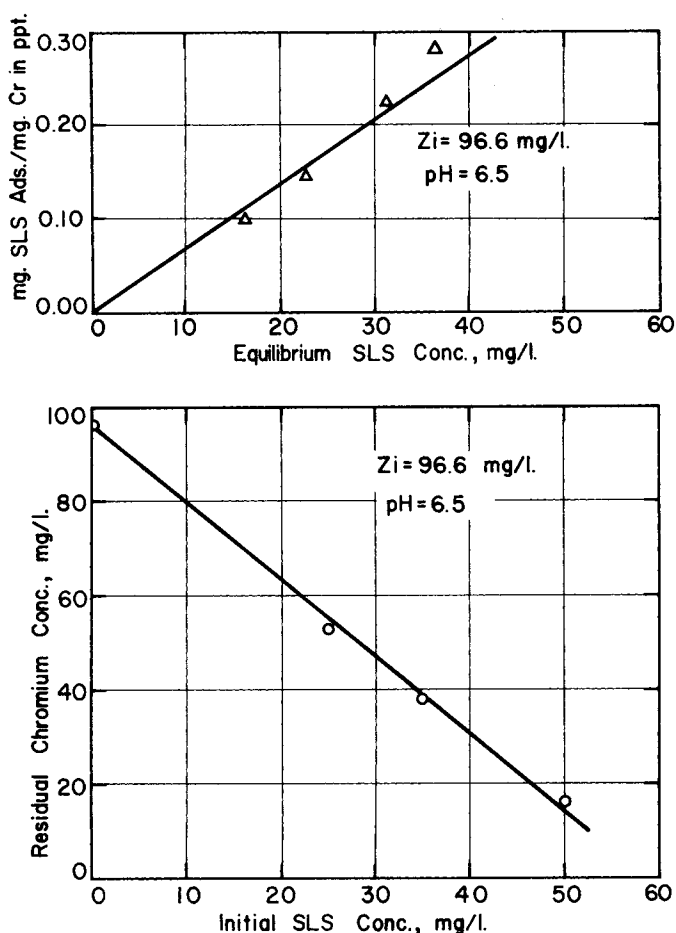


Fig. 9. Flotation results and adsorption measurements as functions of surfactant concentration.

be explained on the basis of adsorption of SLS on the precipitate. The results of the adsorption experiments are shown in Figure 8 as a linearly decreasing function of pH for two different values of X_i , all for $Z_i = 96.6$ mg./liter.

The effect on the flotation of varying the initial SLS concentration was determined at a fixed pH of 6.5, as shown in Figure 9 (bottom). Between 0 and 50 mg./liter SLS, Z_r decreased linearly with X_i , indicating increased removal of the precipitate. An initial SLS concentration of 50.0 mg./liter gave 98% removal of the precipitate. Figure 9 (top) shows the linear increase of adsorption of SLS on the precipitate with an increase in the equilibrium SLS concentration. This relation may be contrasted with Figure 5 (top) indicating much less adsorption per unit weight of precipitated Cr for the suspensions with the lower initial chromium concentration.

Bubble Size

The average bubble size during the course of an experiment was determined using a photographic technique, as described previously (16). From a measurement of 200 bubbles, the average bubble diameter was between 600 to 700 μ .

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NOTATION

- V_i = volume of initial suspension at beginning of each experiment, 2.0 liters
 V_r = volume of residual suspension at termination of each experiment, liter
 X_i = surfactant concentration in initial suspension, mg./liter
 X_r = surfactant concentration in residual (left after flotation) suspension, mg./liter
 Z_i = chromium concentration in initial suspension, mg./liter
 Z_r = chromium concentration in residual (left after flotation) suspension, mg./liter
 μ = ionic strength, M

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