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# Surface interactions between nickel ions and sodium dodecyl sulfate

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## ABSTRACT

In this research the interaction of nickel ions with the surfactant sodium dodecyl sulfate at the liquid-air interface was studied. Surface tension measurements were performed on pure sodium dodecyl sulfate solutions, with and without the addition of nickel ions. Results of these measurements showed that the addition of nickel ions lowered the equilibrium surface tension values of pure sodium dodecyl sulfate solutions and shifted the critical micelle concentration to lower concentrations.

The foam fractionation technique was used to determine if the nickel ions could be removed from surfactant solutions. It was found that the removal of nickel ions was influenced by pH and SDS concentrations.

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## INTRODUCTION

Ion-flotation, a technique that removes ions from water, involves bubbling air through water containing surfactant, which serves to form a foam and extend the liquid-air interface. There is an attraction between the surfactant and the ion being removed, the attraction being greater for polyvalent ions (1). As the foam drains and the bubbles break, the surfactant and ion remains. This technique has been suggested for the removal of ions from waste liquor before they are released into and pollute natural waterways. Because nickel is present in many plating solutions, and constitutes such a problem, it was chosen as the ion in question in this thesis. The objective of this research was to study the interaction of nickel ions with the surfactant sodium dodecyl sulfate (SDS) in order to correlate surface parameters such as surface tension lowering and micelle formation to ion-flotation conditions.

The surface activity of a molecule is caused by the characteristic structure of the molecule consisting of a long hydrophobic and a short hydrophilic group. The hydrocarbon portion of the surfactant is the hydrophobic group and the polar portion is the hydrophilic group. Strong hydrophilizing action requires that the group in question be capable of building a dense hydrated envelope around itself.

For ionic surfactants, there are several factors that control the adsorption at liquid-air interfaces. The van der Waals or cohesive forces between the hydrocarbon chains, the repulsive forces between ionized portions of the molecules, and the attractive forces between the ionized portions and the water dipoles, as well as those between

water dipoles. The surfactant molecules orient themselves in the system so that the interaction energy between molecules is at a maximum. Hence, the hydrophobic group will point away from the water and the hydrophilic points toward the water. The increase of surface containing non-polar molecules in the surface requires a smaller force than the surface containing polar molecules. By creating a less polar surface, the surface tension will be lowered.

The equation that best describes the relationship between surface tension and adsorption at the liquid-air interface is the Gibbs adsorption isotherm. For the two-component system where surface adsorption of the solvent is assumed to be zero this equation can be expressed as

$$d\gamma = -RT \Gamma d \ln A_2 \quad (1)$$

where

$\gamma$  = surface tension

R = gas constant

T = absolute temperature

$\Gamma$  = surface excess of solute at  
the liquid-air interface

$A_2$  = activity of solute

For ideal solutions, the mole fraction  $X_2 = A_2$  so that equation 1 reduces to

$$d\gamma = -RT \Gamma d \ln X_2 \quad (2)$$

If the solution is dilute,  $X_2$  becomes proportional to the concentration  $C_2$  and the equation 2 becomes

$$d\gamma = - RT \Gamma d \ln C_2 \quad (3)$$



For ideal surfactant systems below the critical micelle concentration; a plot of experimental surface tension values versus the natural logarithm of the concentrations will yield a straight line with slope equal to  $-F RT$ . When the slope is negative, positive adsorption is implied and the surface excess of solute is positive. When the slope is positive, negative adsorption is implied and there is a surface deficiency of solute at the liquid-air interface. At the critical micelle concentration (CMC) and for surfactant concentrations above the CMC, the surface tension becomes nearly constant. For concentrations at and above the CMC, the surfactant molecules or ions aggregate to form micelles, thought to be spherical. The hydrocarbon portion of the surfactant points to the inside of the micelle and the hydrophilic portion toward the outside. The counter ions, such as sodium ions, remain unaggregated.

## LITERATURE REVIEW

It is generally accepted that surface tension values for surfactant solutions below the CMC fit the Gibbs equation with good agreement, but when impurities are present its applicability has been questioned. It was shown by Hutchinson (2) that when surface-active impurities are present, the system cannot be treated as a two-component system. Brady (3) concluded that the presence of surface-active impurities causes a minimum in the surface tension concentration curve which can be removed by foam fractionation; this minimum arises from the adsorption of a small amount of these impurities below the CMC of the pure surfactant. The rise in surface tension beyond the minimum was attributed to solubilization or incorporation of the impurity within the micelles of the surfactant molecules. Robinson (4) suggests that the existence of a minimum is due to metal ions initially present in the water. Reichenberg (5) postulated that the minimum is due to salts of polyvalent ions present in water. Harrold (6) gave experimental evidence that the presence of excess salt removes the minimum, while Clayfield and Matthew (7) showed that the addition of a divalent salt produces the minimum.

According to Burcik (8) the addition of an added electrolyte increases the rate of dynamic surface tension lowering, but it had little effect on the equilibrium surface tension. For anionic surfactants, the rate of surface tension lowering depends largely on the added cation while for cationic surfactants, the rate depends on the added anions (9). For the surfactant, sodium dodecyl

sulfate, it was found that the rate was independent of pH. (10)

Research of Klevens (11) relates the decrease in the CMC of potassium laurate solutions to equivalent additions of added electrolyte. Goddard and co-workers (12) report that CMC decreases are related to the decreasing size of the hydrated ion in question.

## EXPERIMENTAL

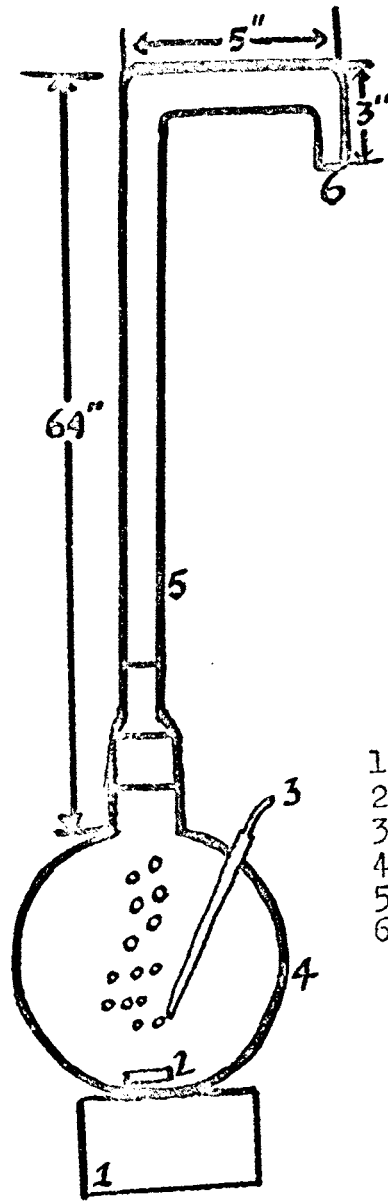
### Preparation of Distilled Water

Distilled water was obtained by two methods. In method I, double distilled water was prepared by distilling nuclear reactor water in alkali permanganate solution followed by one redistillation. In method II, distilled water was obtained by distilling tap water in a Corning AGIA Still and redistilling it once from an alkali permanganate solution. The alkali permanganate solution was prepared by using 0.6 gram sodium hydroxide and 0.5 gram of potassium permanganate per liter of water. The surface tension value for the distilled water was 72.3 dynes/cm for method I, and 72.1 dynes/cm for method II. These values agree reasonably well with literature values for distilled water. Most of the distilled water used in the research for this thesis was obtained from using method II.

### Purification of Sodium Dodecyl Sulfate

The sodium dodecyl sulfate used was procured from the Eastman Kodak Company and was purified by the foam fractionation technique. The technique used was similar to the method described by Elworthy and Mysels (13). Figure 1 shows the foam fractionation apparatus that was used.

For the purification of SDS solutions, the following procedure was used. Weighed amounts of SDS were dissolved in sufficient amounts of distilled water to give a SDS concentration just below the CMC. Nitrogen was bubbled into the flask and up through the solution to produce foam in the liquid-air interface which was



- 1. Magnetic stirrer
- 2. Stirring bar
- 3. Nitrogen entrance
- 4. 3-liter distilling flask
- 5. Column
- 6. Foam exit

FIGURE 1 - Foam fractionation apparatus

passed up the column and removed at the top of the column. Surface active impurities such as dodecyl alcohol in SDS adsorb at the liquid-air interface and can be removed by foaming. The solutions were then foamed until samples taken for surface tension measurements showed no drop in surface tension with respect to time. This was an indication that the surfactant was pure.

#### Surface Tension Measurements

All surface tension measurements were made by the Wilhelmy Slide Method. This method was chosen because it is relatively free of correction factors. A laboratory chainomatic balance was modified by removing one of the pans and replacing it with a hanger to hold a platinum slide. A platinum slide, 0.875 by 1.225 by 0.001 inches and weighing 2.364 grams was used. In an actual experiment, the slide was hung vertically above the surface of the liquid. The sample, 100 milliliters, was placed in a surface tension dish and was raised by means of a lift until the liquid came in contact with the slide. Weights were added until the slide was detached from the surface. To correct for buoyancy effects, the sample was lowered by means of the lift so as to keep the balance arm at zero. The lift consisted of a set of gears which were operated by turning a handle. One complete turn of the handle moved the surface tension dish up 0.1 inches. Water from a constant temperature bath regulated at  $25 \pm 0.05^{\circ}\text{C}$  was circulated through the surface tension dish in order to keep the liquid at constant temperature.

Surface tensions were calculated according to the relationship:

$$\Delta W = W_2 - W_1 = \gamma p \quad (4)$$

where

$W_2$  = weight to detach slide from surface

$W_1$  = weight of dry slide

$\gamma$  = surface tension in dynes/cm

$p$  = perimeter of plate in cm.

Since the perimeter is constant and was found to be 6.249 cm, then

$$\gamma = \frac{\Delta W}{p} \quad (5)$$

On conversion to mass units

$$\gamma = 156.9 \Delta W \quad (6)$$

The plate was cleaned in chromic acid and flushed with distilled water and dried with an air dryer before each measurement. The surface tension dish was cleaned in chromic acid before each series of tests. It was flushed with distilled water until the pH of the washings were the same as that of the distilled water.

#### Preparation of Samples for Surface Tension Measurements

Sodium dodecyl sulfate solutions below the CMC were prepared by diluting the foamed solutions. For concentrations above the CMC, the foamed solution was stored in a freezer to precipitate the surfactant. This precipitate was dissolved in water to make a concentrated surfactant solution and the concentrated solution diluted to give the desired concentrations. To determine concentrations of the surfactant solutions, refractive index differences were determined using the Brice-Phoenix Differential Refractometer. Concentrations were obtained by referring to figure 2 that shows the relationship between surfactant concentrations and the refractive index differences.

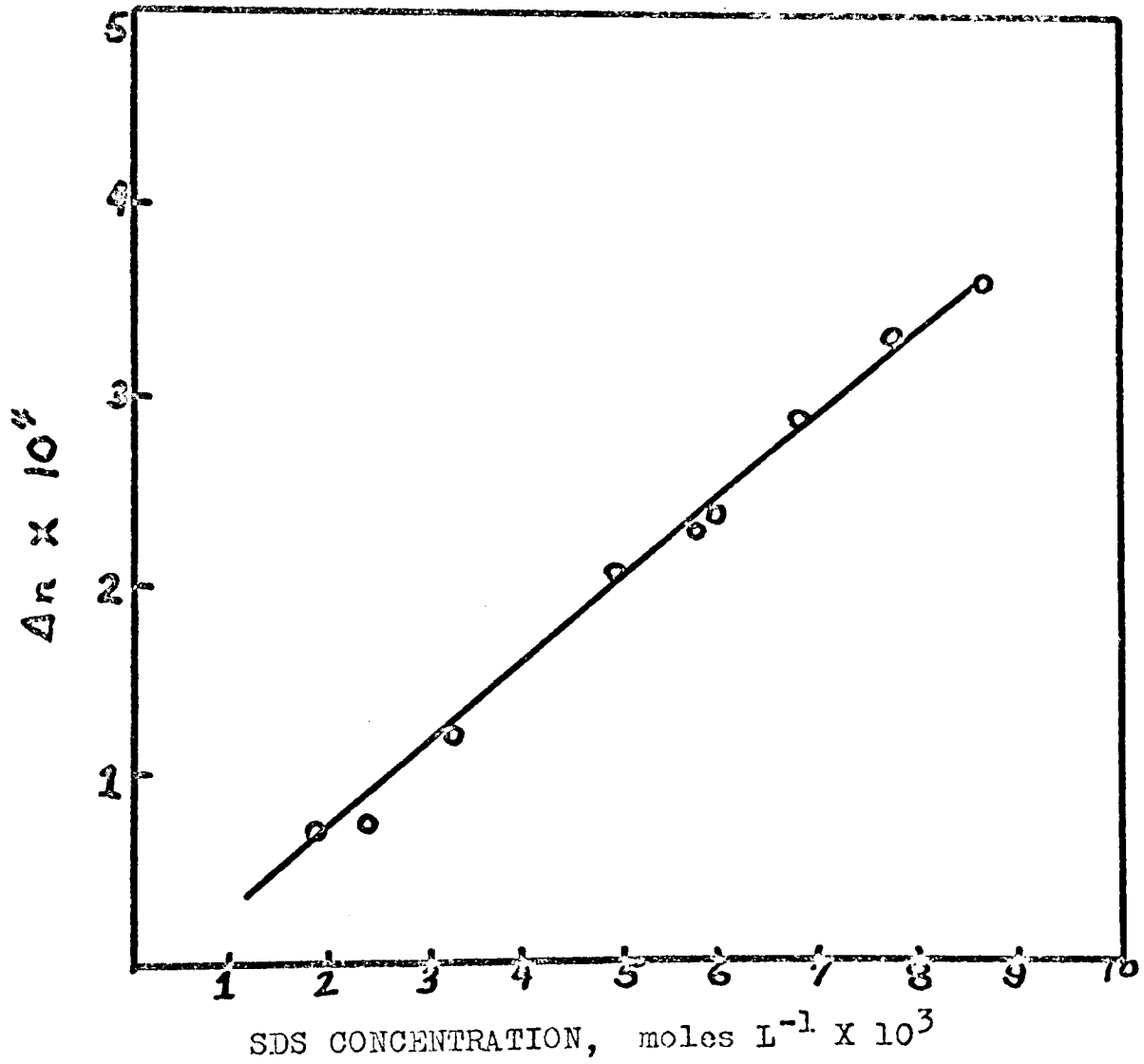


FIGURE 2 - Refractive index differences ( $\Delta n$ ) versus sodium dodecyl sulfate concentration



Figure 2 was obtained by the following procedure. A purified concentrated SDS solution was prepared from precipitated SDS. The concentration of this solution, 0.0144 M, was determined by evaporating 25 milliliters of this solution to dryness in an oven at 40°C, and weighing to determine the grams of SDS per milliliter. Aliquots were taken from the 0.0144 M SDS solution and diluted with distilled water to prepare solutions of various concentrations. Refractive index differences were determined on these solutions by using the relationship

$$\Delta n = k \Delta d \quad (7)$$

where

$\Delta n$  = refractive index difference between  
a solution and its solvent

$k$  = calibration constant for the  
selected wavelength

$\Delta d$  = total slit image displacement  
of the instrument.

The cell constant was determined using potassium chloride solutions of known refractive index differences; the cell constant was found to be  $9.68 \times 10^{-4}$ .

Surfactant solutions containing nickel ions were prepared in the following way. Concentrated solutions of known weights of nickel nitrate were prepared and aliquots were transferred into 100 milliliter volumetric flasks containing the pure surfactant. According to the data of Purohit and Sogani (14) divalent nickel ion will begin to hydrolyze to the hydroxyl complex at pH 6.9. It was therefore assumed that nickel ion is divalent and has 6 molecules of water attached to it below the pH of 7.0.

### Flotation Experiments

The foam fractionation technique was used to remove nickel ions from SDS solutions. Pure SDS solutions were prepared in five different flotation cells at different concentrations; all below the CMC of pure SDS. The flotation cells were those designed by Oko (15). The cells, 1,000 milliliter round bottom flasks, were a slight modification of the apparatus shown in figure 1. Nitrogen was bubbled through distilled water and the flowrate measured with flowmeters before entering the flotation cell through a capillary tube. Water was circulated around the cell in a jacket at 25°C and the solutions were stirred with magnetic stirrers. During a flotation test, the foam was raised up a column and was caught in 1,000 milliliter flasks. One pellet of sodium hydroxide was added to the collection flask to collapse the foam. For determining the nickel content, the residue was dissolved in 50 milliliters of water containing 5 milliliters of dilute sulfuric acid. The Perkin Elmer 303 atomic absorption spectrophotometer was used to determine the absorption of ultra-violet radiation at 2320 Å in these solutions. The nickel was determined from a standard curve of concentrations of nickel versus absorption of radiation.

## RESULTS AND DISCUSSION

Surface Tension Measurements

Figure 3 shows surface tension values plotted versus the logarithm of the SDS concentration in distilled water at pH 5.8. It is noted that the plot is not linear below the CMC. The CMC is located in the region of  $8.2 \times 10^{-3}$  M. Rehfield (16) reports a CMC value for SDS to be  $8.1 \pm .1 \times 10^{-3}$  M by surface tension measurements using the drop-volume technique and showed that the surface tension logarithm plots fit a polynomial of the second degree. The plot of the data in figure 3 resembles those of Rehfield, although the surface tension values are slightly lower.

Cook and Talbot (17) observed a pH effect on the surface tension values of SDS solutions below pH 7.0. Since the data in figure 3 were for below pH 6.0, surface tension values were obtained in SDS solutions in the region of pH 7.0. Sodium hydroxide solutions were used to adjust the pH. It was found that there was very little effect from the pH in this region upon the surface tension values. To show that there is no salt effect due to the addition of the sodium hydroxide, measurements were also obtained for SDS solutions containing an equivalent amount of sodium nitrate.

Surface tension measurements were usually completed in 15 minutes. In this time the surface tension became constant. For SDS solutions containing divalent nickel ions, surface tension measurements were completed in the same period of time, however, nickel was added to volumetric flasks two hours before the measurements were taken.

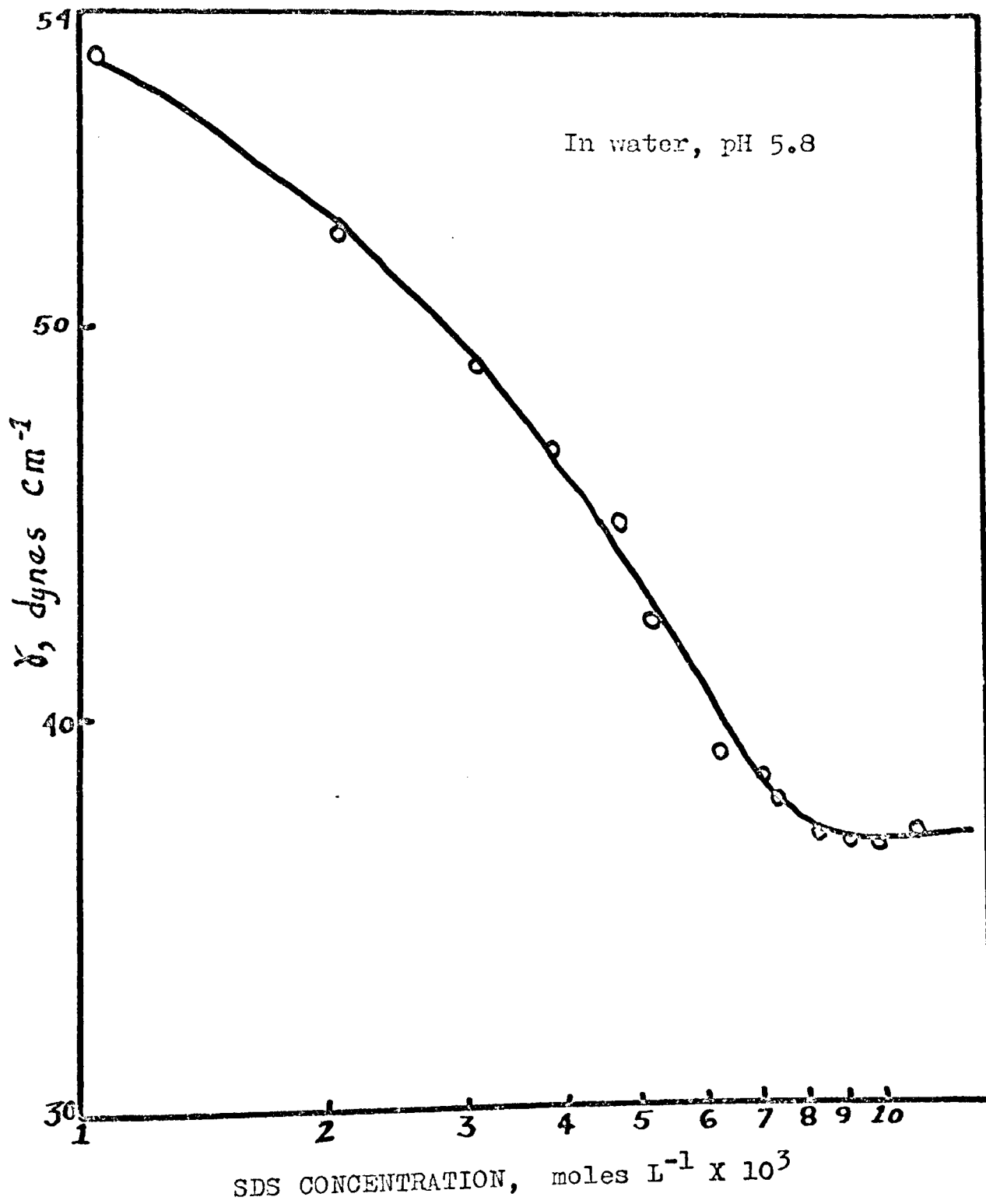


FIGURE 3 - Surface tension values versus the logarithm of the SDS concentration

The results of surface tension measurements of SDS solutions containing nickel ions are shown in figures 4 and 5. Figure 4 shows that the addition of the nickel causes a large decrease of surface tension for the  $3.75 \times 10^{-3}$  M SDS, and a much smaller decrease for the  $6.3 \times 10^{-3}$  M SDS. At both concentrations a linear plateau is approached.

Figure 5 shows the effect on the surface tension values due to varying the SDS concentration with a constant concentration of nickel ions of  $1.075 \times 10^{-4}$  M. The curves shown are for solutions of surfactant in water at pH 5.8, in  $1.53 \times 10^{-6}$  M sodium hydroxide at pH 7.0, and in  $1.53 \times 10^{-6}$  M sodium nitrate at pH 5.7. All three curves show a broad minimum near  $5.0 \times 10^{-3}$  M. This shows that the position of the minimum does not change between pH 5.8 or 7.0; and does not change in the presence of small amounts of added salt. All three curves are similar and give the same position of the minimum. If this value for the broad minimum is taken as the CMC, it is more than the CMC reported for nickel dodecyl sulfate which was  $1.24 \times 10^{-3}$  M. (18)

The fact that the addition of nickel ions lowers the surface tensions indicates that there is some interaction at the liquid-air interface. Generally the addition of soluble inorganic salts will raise the surface tension. A replacement of monovalent sodium ions with divalent nickel ions should increase the electrostatic attraction between the dodecyl sulfate ions and counter-ions, and a lower surface tension would result. The rise in the curve at high concentrations in figure 5 is an indication that some

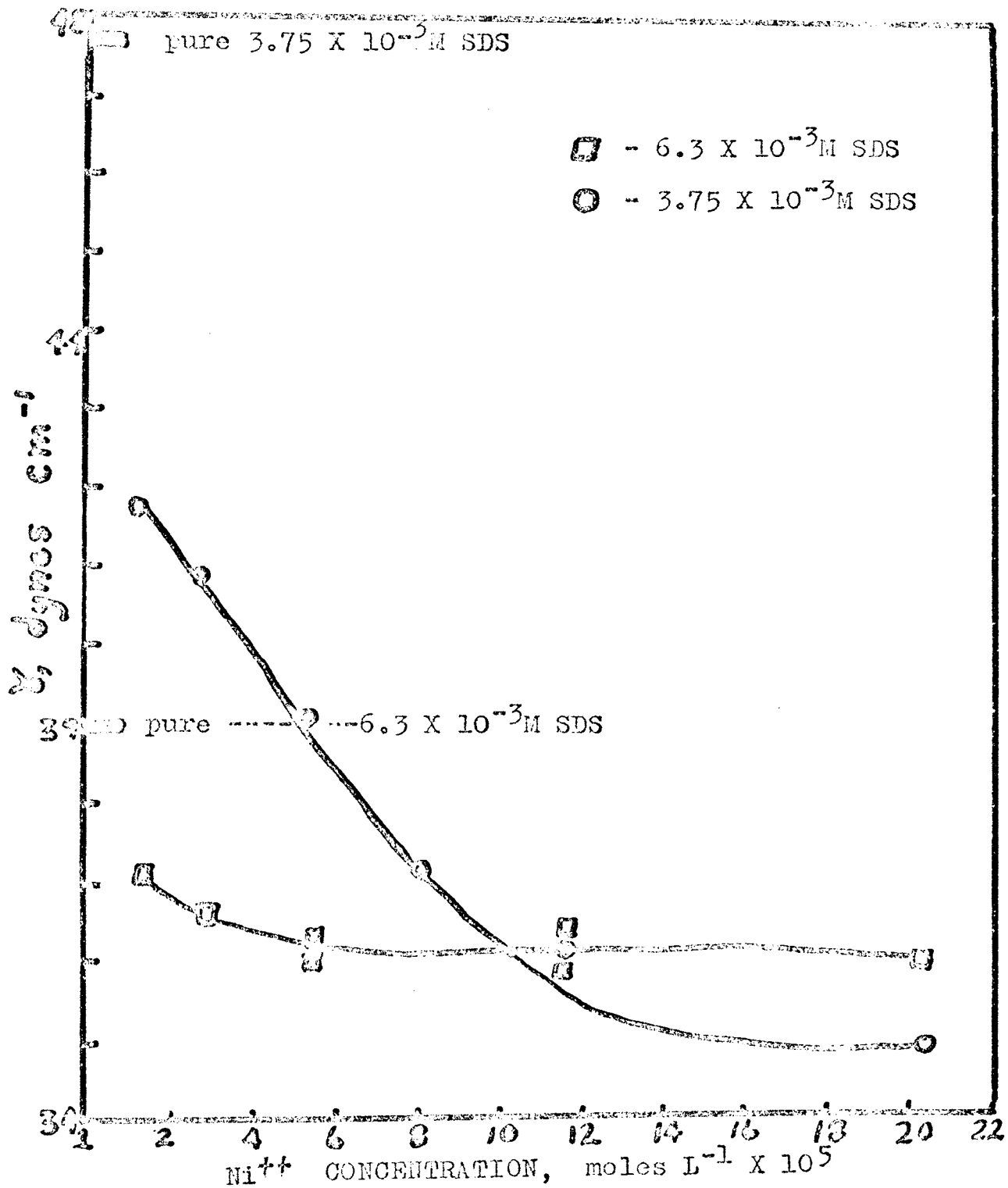


FIGURE 4 - Effect of Nickel ion concentrations on surface tension values of SDS solutions

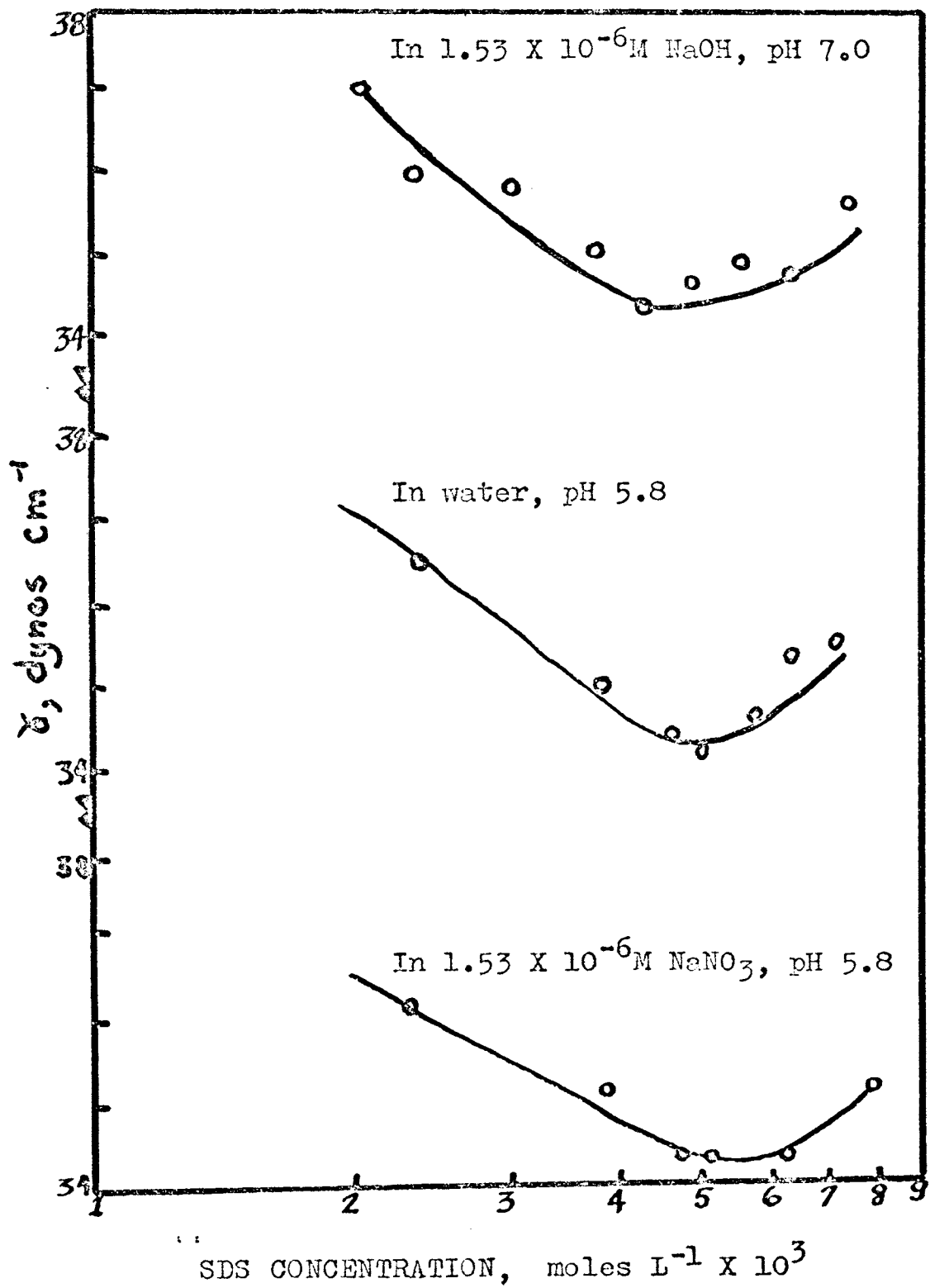


FIGURE 5 - Surface tension values as a function of SDS concentration,  $1.075 \times 10^{-4} \text{M}$  Nickel ions added

solubilization is occurring in the micelles. Corkill and Goodman (19) reported that interactions of sodium dodecyl sulfate micelles with calcium ions are complex and mixed micelles are formed.



### Flotation Results

The primary concern of the flotation studies was to determine if nickel ions could be removed by the foam fractionation technique, i.e. to determine if ions adsorb at the liquid-air interface in the presence of anionic surfactants. Such factors as pH, added salts or electrolytes, and the effect of nickel concentration were considered. Nickel ions were added to SDS solutions at various concentrations below the CMC of pure SDS in the flotation cells. A flotation time of four hours was used. It was found that it took this amount of time to remove enough nickel to get sufficient sensitivity for the atomic absorption analysis; otherwise, a small volume of water had to be used to dissolve the foam residue and questionable results were obtained. Corrections were made to obtain the same flow rate, about 16 ml/min. for each cell. The calibration curves for the flowmeters showed nearly a linear relationship between the volume of wet nitrogen and the flowmeter readings in the particular region that was investigated.

Figure 6 shows the removal of nickel as a function of the SDS concentration at three nickel ion concentrations,  $5.35 \times 10^{-5}$  M,  $10.75 \times 10^{-5}$  M, and  $25.4 \times 10^{-5}$  M. The amount of nickel removed was determined by relating the amount of nickel in a 50 milliliter volume of the dissolved foam residue to the volume of the flotation solution.

The results shown in figure 6 are believed to show no significant effect of nickel ion concentration on flotation, rather it is presented to show the effect of SDS concentration. It is seen that there is a decrease in the removal of nickel ions at about  $5.0 \times$

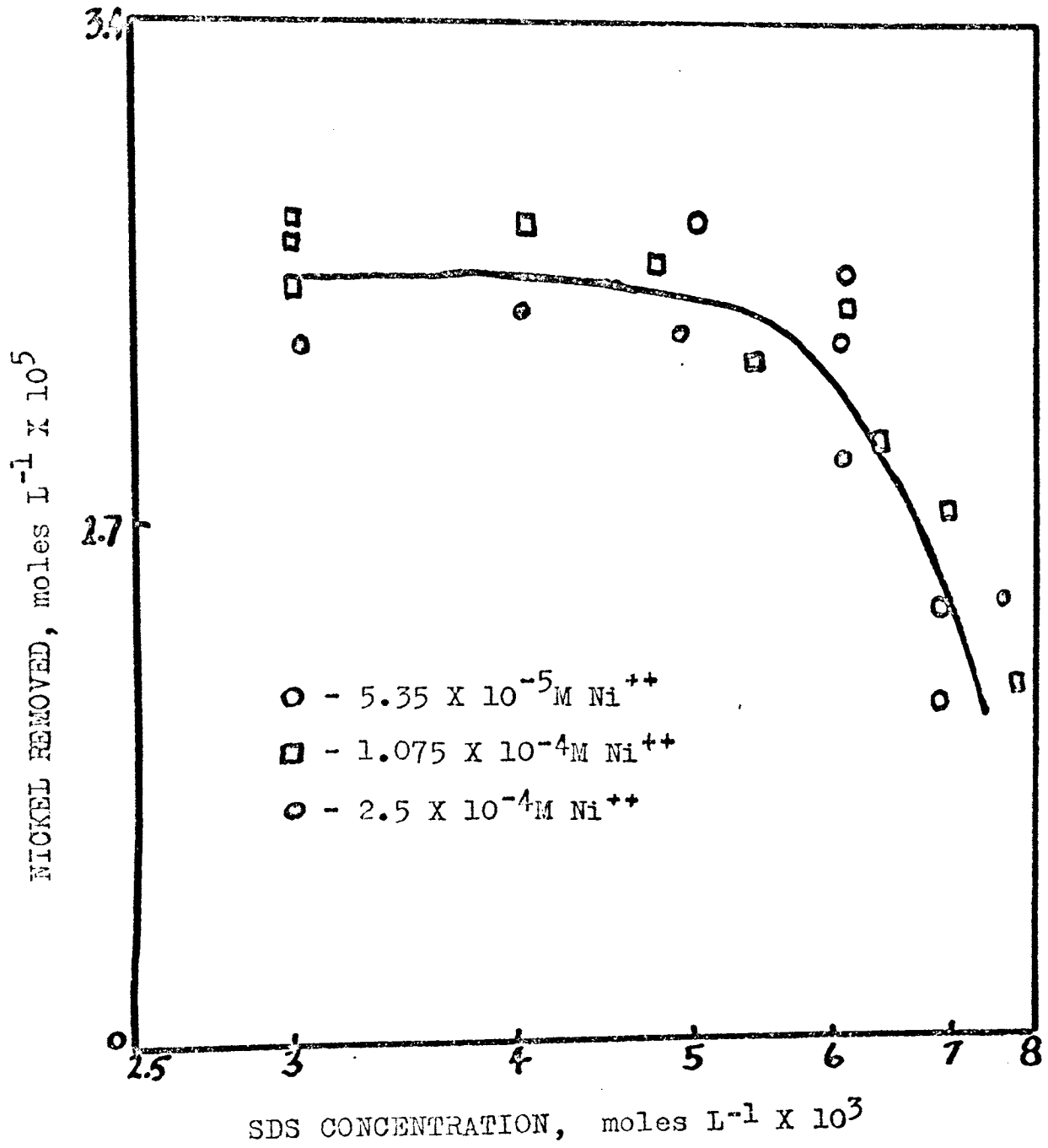


FIGURE 6 - Nickel removal as a function of the SDS concentration

$10^{-3}$  M of SDS. This value is very near the CMC determined from the surface tension measurements, so it seems logical to say that the formation of micelles decreased flotation efficiency. This can be explained by the adsorption or solubilization of nickel species in the micelles. Not all of the nickel ions were removed; however, in a commercial process the foam rates could be adjusted to get sufficient removal. Rubin and co-workers (20) have shown that in the ion-flotation technique, an increase in flow rate increases the rate of recovery.

Figure 7 shows the effect of pH and added salt on the removal of nickel ions. At pH 2.1, the removal is decreased at SDS concentrations less than  $7 \times 10^{-3}$  M SDS; compared to pH 5.5, when no salt was added. At pH 2.5, the removal is a little greater than at pH 2.1. The decrease in the removal of nickel ions could be attributed to some adsorption of hydrogen ions at the interface instead of nickel ions. Bujake and Goddard (21) gave evidence of the preferential adsorption of hydrogen ions when the hydrogen ion concentration approaches the sodium ion concentration.

Surface tension values were determined on the flotation solutions at pH 2.1 after nickel removal, and also on pure SDS solution containing  $1.075 \times 10^{-4}$  M  $\text{Ni}^{++}$  ions at pH 2.2. These surface tension values are plotted in figure 8; also shown are surface tension values at pH 5.8. The plots in figure 8 show that the surface tension values of the solutions at pH 2.1 are higher. No explanation is given for the higher surface tensions at the lower pH. A minimum is observed near the  $2.7 \times 10^{-3}$  M concentration and is

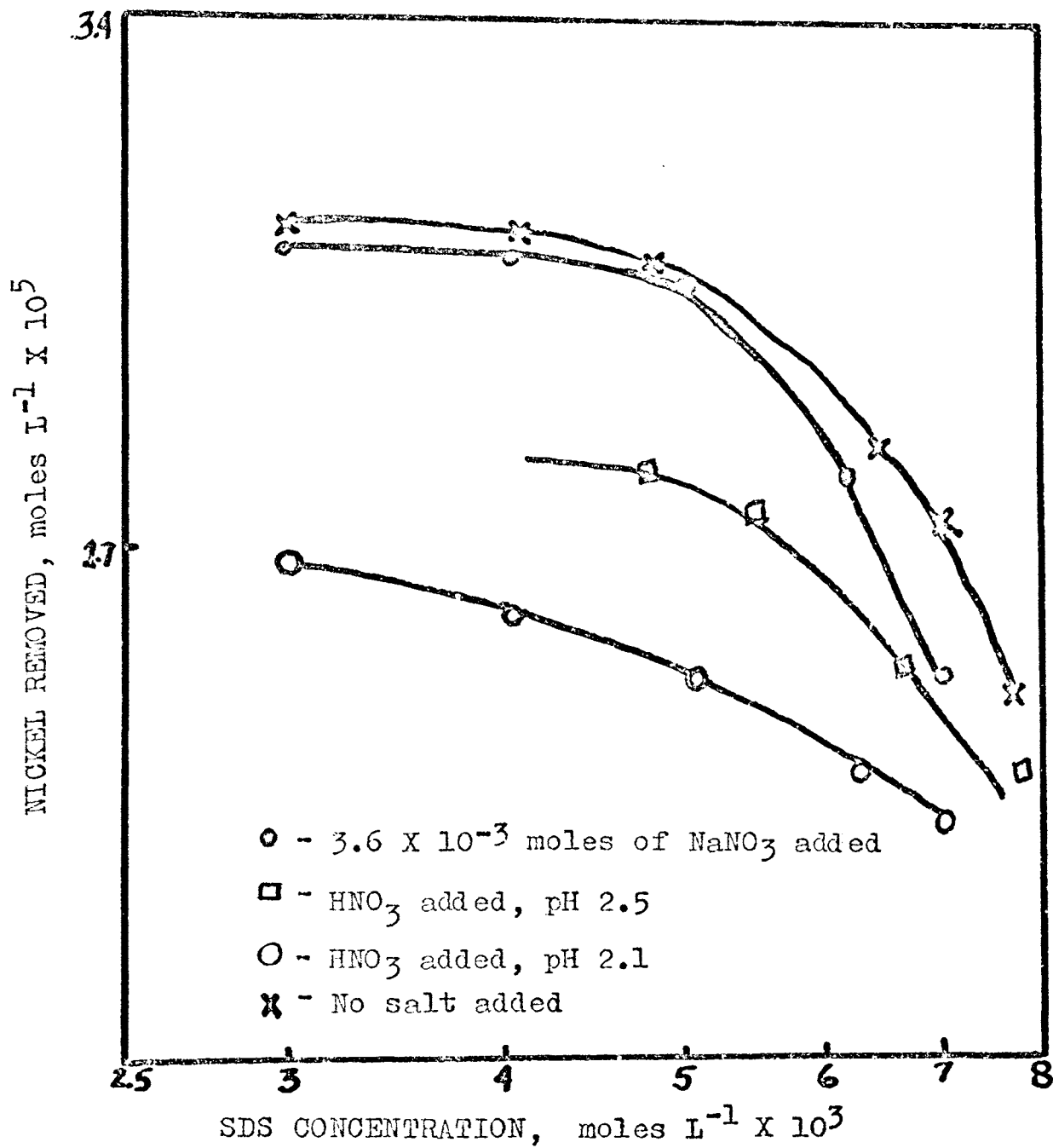


FIGURE 7 - Effect of pH and added salt on the removal of nickel ions,  $1.075 \times 10^{-4} \text{ M Ni}^{++}$  in SDS solutions

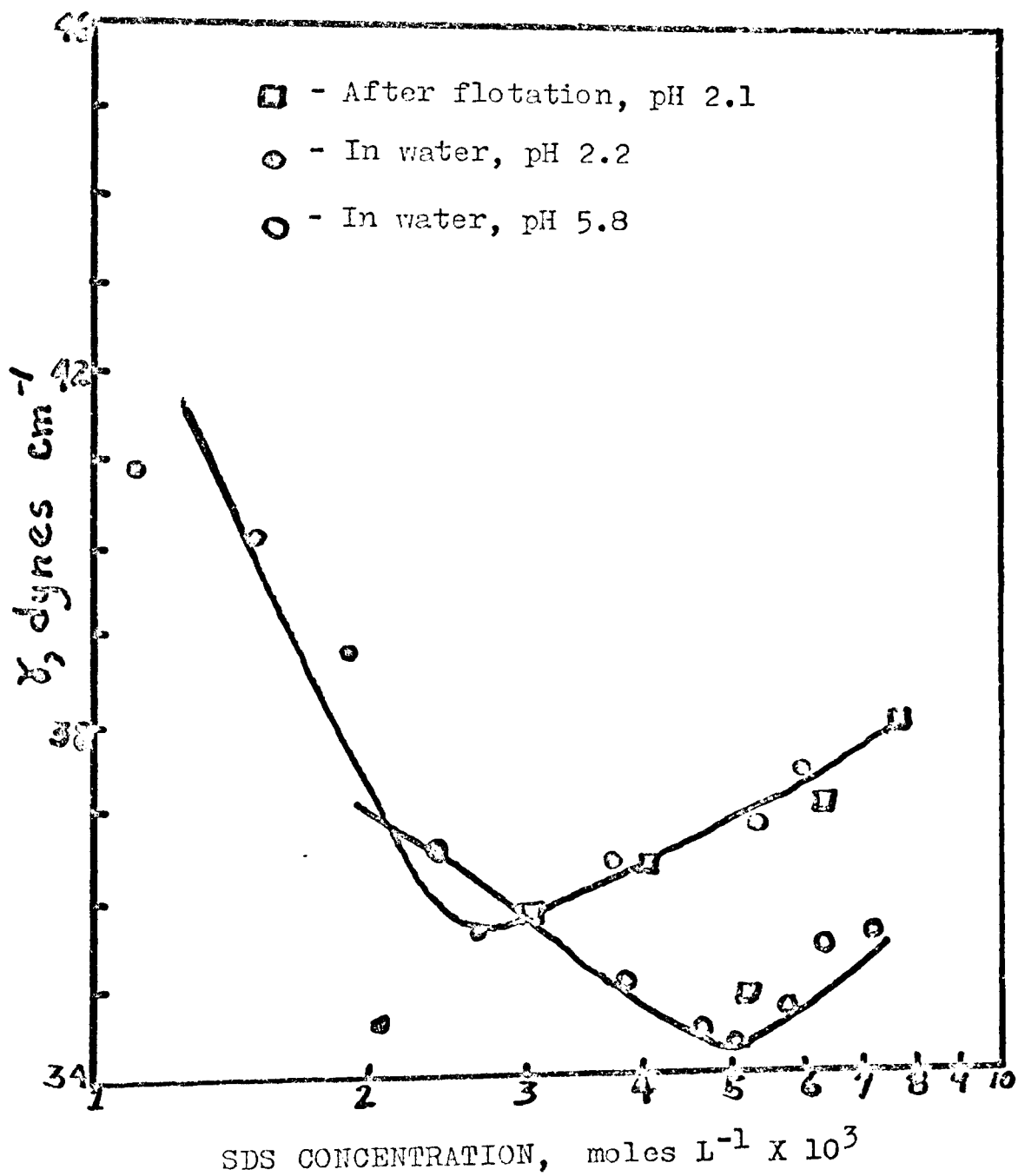


FIGURE 8 - Comparison of surface tension values of SDS solution at pH 2.1 and 2.2 to pH 5.8

considered to be the CMC.

The curve in figure 7 shows the removal of nickel for added sodium nitrate, which is equivalent in concentration to the added nitric acid at pH 2.5. This shows that there is very little difference in the nickel removal with added salt compared to the curve in figure 7 with no added salt. Therefore, the decrease in the nickel removal is not due to added salt and low pH does have an effect on the removal of nickel ions. This decrease can possibly be attributed to micelle formation.

Finally, the question arises as to whether foam stability really affects the nickel removal. According to Burcik, (8) foam stability is related to (a) low equilibrium surface tension, (b) low viscosity, (c) low rate of foam production and (d) high rate of surface tension lowering. Thus, the foam should be most stable at the CMC where the lowered surface tension was found. Since the surface tension values in figure 5 increase beyond the minimum, it is not certain whether the decrease in flotation efficiency can be related to a decrease in foam stability.

## CONCLUSIONS

The addition of nickel ions to pure SDS solutions lowers the equilibrium surface tension values and shifts the CMC to lower concentrations indicating increasing surface activity. For constant nickel ion concentrations, a broad minimum is produced in the curve of surface tension versus SDS concentration. As the concentration of the nickel ion increases, at constant SDS concentrations, the equilibrium surface tension values assume constant values.

Foam fractionation of SDS solutions containing nickel ions removes these ions at a slow rate. This rate decreases when micelles are formed in the solution. Flotation efficiency is reduced in extremely acid solutions (pH 2.5) although, the additions of salts have little effect at equivalent electrolyte concentrations.

These results indicate that the optimum flotation conditions for the removal of the divalent nickel ion  $\text{Ni}(\text{H}_2\text{O})_6^{++}$  is at low hydrogen ion concentration and at a SDS concentration where micelles are not formed.

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## VITA

Ralph Edward Coleman was born on April 12, 1933, in Slater, Missouri. He obtained his primary education in the rural Saline County Schools and his secondary education at Slater High School. He received a Bachelor of Science degree in Chemistry and Mathematics from Central Missouri State College in 1955. He served in the U. S. Army from 1955 to 1958.

He did graduate work at the University of Missouri at Columbia from 1959 to 1960. In late 1960 he was employed by the Absorbent Cotton Company in Valley Park, Missouri as a chemist. The following year he was employed with the Bureau of Mines in Rolla and remained there until he entered graduate school at the University of Missouri - Rolla in September, 1967.

On August 5, 1961, the author was married to Carolyn Goetting. One son, Royce Lamont, was born on December 31, 1965.

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