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Włodzimierz Zwierzykowski<sup>a</sup>; Krystyna B. Mędrzycka<sup>a</sup>

<sup>a</sup> Department of Fat Chemistry, Technology Institute of Organic and Food Chemistry and Technology Gdańsk Technical University, Gdańsk, Poland

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## Multistage Removal of Detergents from Aqueous Solutions by Foaming

WŁODZIMIERZ ZWIERZYKOWSKI and  
KRYSTYNA B. MĘDRZYCKA

DEPARTMENT OF FAT CHEMISTRY AND TECHNOLOGY  
INSTITUTE OF ORGANIC AND FOOD CHEMISTRY AND TECHNOLOGY  
GDAŃSK TECHNICAL UNIVERSITY  
GDAŃSK, POLAND

### Abstract

The anionic (sodium dodecylbenzenesulfonate, Nansa HS-80) and nonionic (oxyethylated nonylphenol, Rokaphenol NX-8) detergents were foamed from their solutions of different initial concentrations. The experimental results were used for the construction of diagrams which were found to be helpful for establishing the number of foaming processes in a multistage system. In another series, solutions of high detergent concentration were foamed in a multistage system. It was found that only the first processes of the multistage experiments gave good agreement with the results found in the diagrams. An explanation of the phenomenon is suggested.

### INTRODUCTION

The amount of a detergent removed by foaming is a function of the adsorption equilibrium state at the phase boundaries of a system and a number of the process parameters. An increase in detergent removal from a solution can be attained by changing these parameters, e.g., by an increase in the gas flow rate (1-5), by a decrease in the flow rate of the feeding solution to a column (6), and by feeding a column to the foam layer instead of to the liquid layer (3, 6, 7). Inorganic salts dissolved in a solution of detergents change the equilibrium state at the phase boundary, thus improving the decontamination factor (2, 3, 5, 7, 8).

In order to obtain a raffinate product of a still lower detergent concentration, multistage foaming may be used. The product of the first stage of foaming is subjected repeatedly to further refining to obtain a solution of the desired concentration in the final process. In the previous paper (7) we presented a simple method for the graphical calculation of the number of foaming processes in the multistage system required to obtain the desired detergent removal from the solution. The same method has been applied in this paper. Diagrams for the calculation of the number of steps needed for the removal of anionic and nonionic detergents from their aqueous solutions were constructed, and then multistage foaming was carried out with greater solution amounts subjecting the same solution to several consecutive processes.

## EXPERIMENTAL

The foaming processes were carried out in a continuously working glass column (3, 5, 7). The column height ( $h_c$ ) was 78 cm; the liquid height ( $h_l$ ) was 24 cm; the height of feeding the column with a solution ( $h_f$ ) was 23 cm; the gas flow rate ( $V_g$ ) was 10 or 15 l/hr; and the flow rate of a solution introduced into the column ( $V_o$ ) was 2.24 l hr.

## DIAGRAM CONSTRUCTION

Foaming was carried out with aqueous solutions of sodium dodecylbenzenesulfonate (Nansa HS-80) and oxyethylated nonylphenol (Rokaphenol NX-8) in concentration ranges from 40 mg/l to about 320 mg/l. A new solution of the required concentration was prepared for each foaming process. Detergent concentrations were determined in feeding solution ( $C_o$ ), raffinate ( $C_r$ ), and in the solution obtained from the collapsed foam ( $C_f$ ). The concentrations were obtained spectrophotometrically by measuring the absorption at 223 nm. The decontamination factor ( $C_o/C_r$ ) and quantity of water removed with foam ( $V_f/V_o \times 100\%$ ) were calculated for each process.

The results obtained are used in Figs. 1, 2, and 3 to represent the relationship between the detergent decontamination factor and its concentration in the initial solution or in the raffinate.

It has been proved that the lower the detergent concentration in the solution subjected to foaming, the higher is the decontamination factor of this detergent (5, 7). It follows from Figs. 1, 2, and 3 that this holds true only for a concentration level below which the decontamination factor

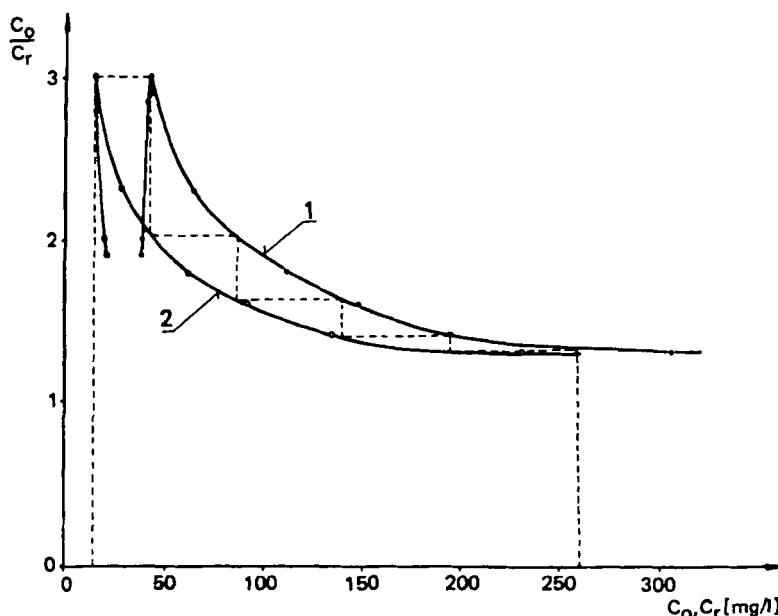


FIG. 1. Decontamination factor dependence for Rokaphenol on its concentration in feed and raffinate. Gas flow rate  $V_g = 10$  l/hr; (1)  $C_o/C_r = f(C_o)$ ; (2)  $C_o/C_r = f(C_r)$ .

decreases with the detergent concentration decrease. Thus the maximum value of the decontamination factor is obtained for the defined concentration. In the case of Rokaphenol, the highest decontamination factor is obtained for solution concentrations of about 40 mg/l (at  $V_g = 10$  l/hr) and about 80 mg/l (at  $V_g = 15$  l/hr), while for Nansa it equals about 70 mg/l.

The relationship observed may be explained by the considerable drop of foam stability during the lowering of the detergent concentration in a solution below a critical value. The worst foam stability is related to an increase of foam leak in the column, and consequently to an increase of the detergent concentration in the raffinate (lower decontamination factor). Because the increase of gas flow lowers the foam stability, lowering of the decontamination factor at  $V_g = 15$  l/hr occurs at higher detergent concentrations than at 10 l/hr (Figs. 1 and 2).

An increase of the foam leak is very difficult to observe because at these concentrations the foam has not collapsed while in a column. Only at

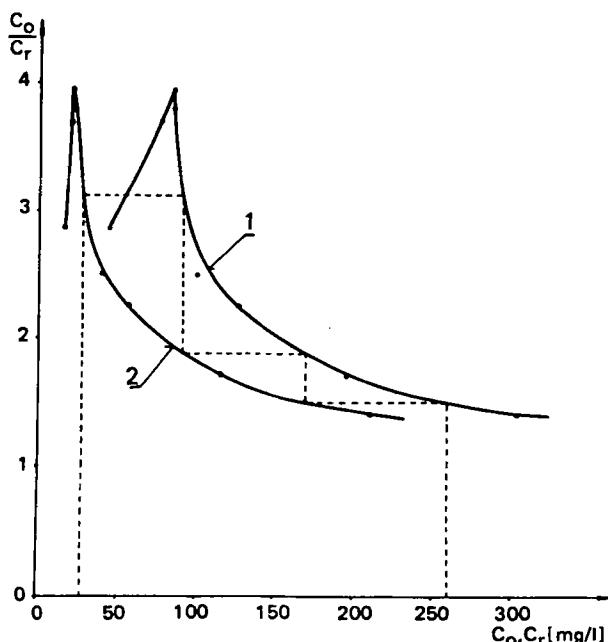


FIG. 2. Decontamination factor dependence for Rokaphenol on its concentration in feed and raffinate. Gas flow rate  $V_g = 15 \text{ l/hr}$ ; (1)  $C_o/C_r = f(C_o)$ ; (2)  $C_o/C_r = f(C_r)$ .

very low detergent concentrations does the foam collapse in the column, but such experiments have not been taken into account. The quantity of water removed with the foam is measured with a large error. Therefore it is possible to observe the increase of water quantity removed with the foam (Fig. 4). Only for Nansa was the quantity of water removed with the foam considerably lowered by a reduction of the concentration of the initial solution below 80 mg/l.

The method for the determination of the number of processes in the multistage removal of detergents is represented graphically by dotted lines on Figs. 1, 2, and 3. Thus, in order to obtain the raffinate containing 14 mg/l from the solution of Rokaphenol of initial concentration 260 mg/l, it is necessary to carry out five processes under conditions previously defined and at a gas flow rate equal to 10 l/hr (Fig. 1). At a flow rate of 15 l/hr a detergent concentration in the raffinate equal to about 26 mg/l is attained after only three foaming processes (Fig. 2). Thus, at higher

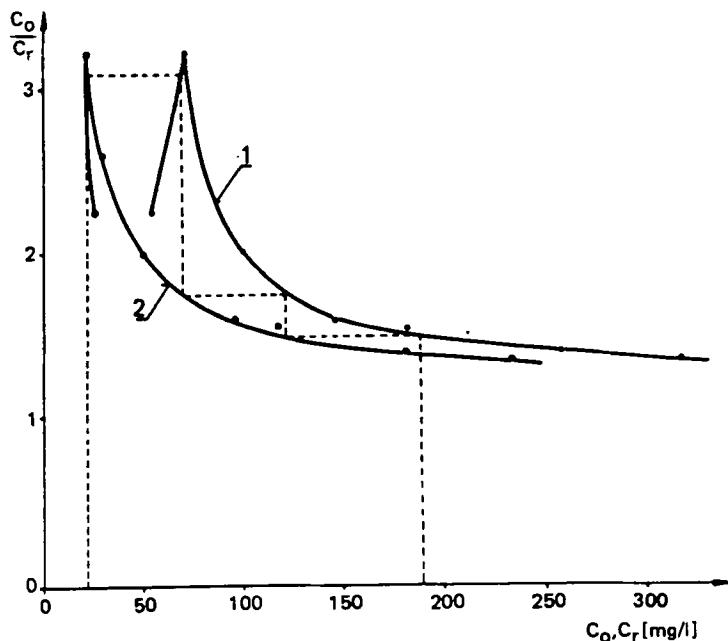


FIG. 3. Decontamination factor dependence for Nansa on its concentration in feed and raffinate. Gas flow rate  $V_g = 15 \text{ l/hr}$ ; (1)  $C_o/C_r = f(C_o)$ ; (2)  $C_o/C_r = f(C_r)$ .

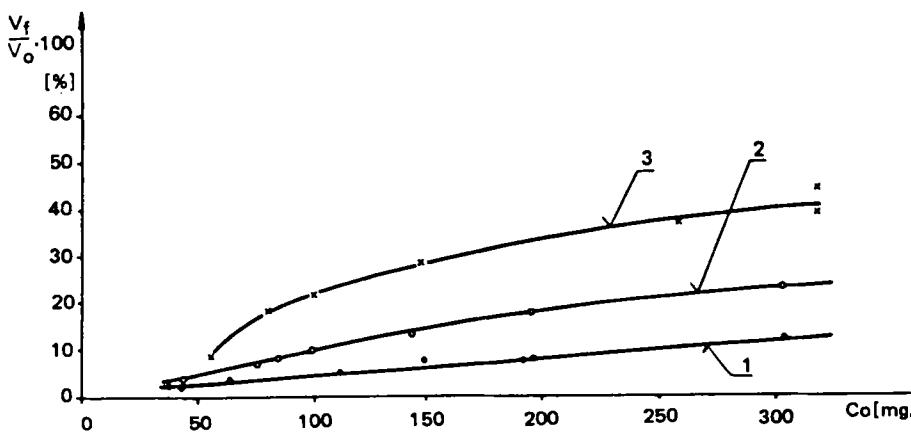


FIG. 4. The dependence of water removal with foam on the detergent concentration in the solution. (1) Rokaphenol,  $V_g = 10 \text{ l hr}$ ; (2) Rokaphenol,  $V_g = 15 \text{ l hr}$ ; (3) Nansa,  $V_g = 15 \text{ l hr}$ .

gas flow rates, the number of columns in the multistage process can be reduced. However, the increase of  $V_g$  increases the quantity of water removed with foam (Fig. 4). It also follows from Fig. 4 that the higher the initial concentration of the solution subjected to foaming, the more humid is the foam. Thus, in order to improve the effectiveness of the multistage process, it is advisable to perform it at different gas flow rates. Solutions of higher concentrations have to be foamed at lower gas flow rates, e.g., 10 l/hr, and then the next stage can be carried at a higher  $V_g$ , e.g., 15 l hr (Fig. 5).

The multistage foaming process was carried out in such a way that the solutions made by dissolving the detergents in distilled water were used only in the first stage. In the next stages the raffinates of the previous processes of foaming were the feeding solutions. This procedure was applied for two Rokaphenol solutions and for two Nansa solutions containing 0.01 mole/l sodium chloride. The results obtained are compiled in

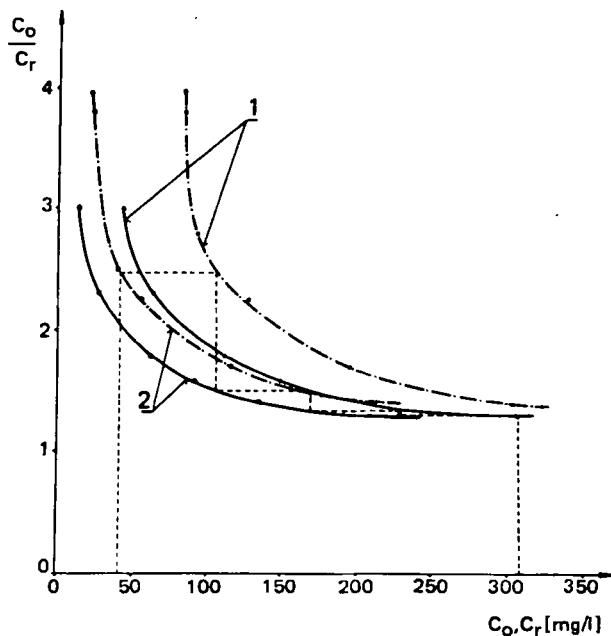


FIG. 5. The determination of the foaming processes number for Rokaphenol in multistage systems. Gas flow rate:  $V_g = 10 \text{ l/hr}$  (—);  $V_g = 15 \text{ l/hr}$  (---).  
 (1)  $C_o/C_r = f(C_o)$ ; (2)  $C_o/C_r = f(C_r)$ .

Tables 1 and 2. The foaming of Rokaphenol solutions has been carried out at  $V_g = 10 \text{ l/hr}$  and that for Nansa solutions at  $V_g = 15 \text{ l hr}$ .

The foaming of Rokaphenol solutions was also carried out at gas flow rates of 10 and 15 l/hr. The results are compiled in Table 3.

Tables 1, 2, and 3 also contain the values of the detergent concentrations in raffinates and the decontamination factors found from the corresponding diagrams of Figs. 1, 2, and 3. These values should agree with those obtained in the multistage processes. It follows from the tables that only the first two stages of the multistage foaming process give results in good agreement with the diagrams. In each sequential process the raffinates obtained contain more of a detergent and the decontamination factors are lower than appears from the diagrams. This fact was observed for both

TABLE 1  
Multistage Foaming of Rokaphenol NX-8. Gas Flow Rate:  $V_g = 10 \text{ l hr}$

| Series | Value determined     | Stage of foaming |      |      |      | Results found from Fig. 1 |      |      |      |
|--------|----------------------|------------------|------|------|------|---------------------------|------|------|------|
|        |                      | I                | II   | III  | IV   | I                         | II   | III  | IV   |
| I      | $C_o \text{ (mg/l)}$ | 193              | 140  | 90   | 45   | 193                       | 137  | 82   | 40   |
|        | $C_r \text{ (mg/l)}$ | 140              | 90   | 45   | 23   | 137                       | 82   | 40   | 14   |
|        | $C_o/C_r$            | 1.40             | 1.56 | 2.00 | 1.95 | 1.41                      | 1.66 | 2.05 | 2.85 |
| II     | $C_o \text{ (mg/l)}$ | 206              | 145  | 87   | 46   | 206                       | 147  | 90   | 45   |
|        | $C_r \text{ (mg/l)}$ | 145              | 87   | 46   | 24   | 147                       | 90   | 45   | 15.5 |
|        | $C_o/C_r$            | 1.42             | 1.65 | 1.89 | 1.89 | 1.40                      | 1.63 | 2.00 | 2.90 |

TABLE 2  
Multistage Foaming of Nansa HS-80. Gas Flow Rate:  $V_g = 15 \text{ l hr}$ . Content of Sodium Chloride in the Solutions: 0.01 mole/l

| Series | Value determined     | Stage of foaming |      |      | Results found from Fig. 3 |      |      |
|--------|----------------------|------------------|------|------|---------------------------|------|------|
|        |                      | I                | II   | III  | I                         | II   | III  |
| I      | $C_o \text{ (mg/l)}$ | 186              | 123  | 79   | 186                       | 122  | 70   |
|        | $C_r \text{ (mg/l)}$ | 123              | 79   | 47   | 122                       | 70   | 23   |
|        | $C_o/C_r$            | 1.51             | 1.56 | 1.67 | 1.52                      | 1.75 | 3.10 |
| II     | $C_o \text{ (mg/l)}$ | 190              | 128  | 85   | 190                       | 123  | 70   |
|        | $C_r \text{ (mg/l)}$ | 128              | 85   | 52   | 123                       | 70   | 23   |
|        | $C_o/C_r$            | 1.48             | 1.51 | 1.64 | 1.50                      | 1.76 | 3.10 |

TABLE 3  
Multistage Foaming of Rokaphenol NX-8. Gas Flow Rates:  $V_g = 10$  or  $15 \text{ l/hr}$

| Series | Value determined | Experimental multistage foaming |      |      | Results found from Figs. 1 and 2 |      |      |
|--------|------------------|---------------------------------|------|------|----------------------------------|------|------|
|        |                  |                                 |      |      | I                                | II   | III  |
|        |                  | I                               | II   | III  | I                                | II   | III  |
| I      | $V_g$ (l/hr)     | 10                              | 10   | 15   | 10                               | 10   | 15   |
|        | $C_o$ (mg/l)     | 211                             | 150  | 100  | 211                              | 154  | 99   |
|        | $C_r$ (mg/l)     | 150                             | 100  | 48   | 154                              | 99   | 37   |
|        | $C_o/C_r$        | 1.40                            | 1.50 | 2.08 | 1.37                             | 1.56 | 2.67 |
| II     | $V_g$ (l/hr)     | 10                              | 10   | 15   | 10                               | 10   | 15   |
|        | $C_o$ (mg/l)     | 208                             | 145  | 90   | 208                              | 150  | 94   |
|        | $C_r$ (mg/l)     | 145                             | 90   | 44   | 150                              | 94   | 32   |
|        | $C_o/C_r$        | 1.43                            | 1.61 | 2.04 | 1.38                             | 1.60 | 2.90 |

anionic and nonionic detergents. Thus, in the foaming process of Rokaphenol from the solution with an initial concentration equal to 193 mg/l, the raffinate containing 23 mg/l was obtained after four consecutive processes while the raffinate concentration read at diagram 1 after four processes was equal to 18 mg/l (Table 1). The foaming process of a solution containing a Nansa concentration of 190 mg/l gives the raffinate with a concentration of 54 mg/l of the detergent after three consecutive processes (Table 2), instead of the 23 mg/l expected on the basis of Fig. 3. The decontamination factor in the last process was 1.74 instead of 3.10. The raffinates after three stages of Nansa foaming could not be subjected to further foaming because of the small quantity of foam being formed. On the other hand, fresh solutions with the same Nansa concentration (about 50 mg/l) formed good foams because they contained a mixture of surface-active substances which were not yet fractionated. For Nansa solutions with concentrations of 50 mg/l, the decontamination factor was equal to 2.1.

## CONCLUSIONS

The poorer foaming of the solutions previously subjected to the process as compared to the fresh solutions of the same concentration can be explained by the removal of dissolved surfactants to a different extent depending on their surface activity. Therefore, the solution already foamed has poorer foaming properties and higher surface tension as can be seen from Fig. 6.

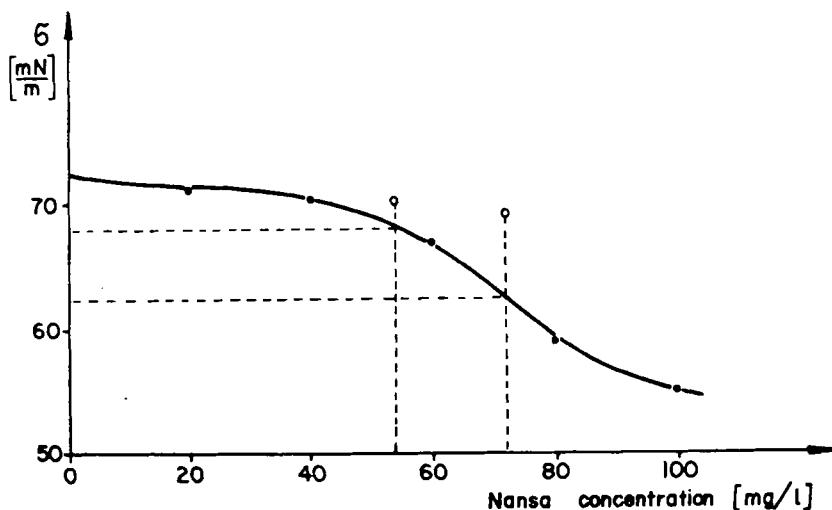


FIG. 6. The dependence of the surface tension of Nansa solutions on their concentration: (●) fresh solutions; (○) solutions foamed 3 times in a multistage system.

Nansa (technical grade) contains, besides sodium dodecylbenzenesulfonate, minor amounts of its homologs and other impurities. It may be assumed that in the case of a series of homologs and other surface-active substances, the more surface-active ones will be foamed first.

The above-mentioned fractionation also takes place during the foaming process of Rokaphenol solutions. It was found earlier (9) that technical Rokaphenol contains about 21 components which vary in their polyoxyethylene chain length. The molecules with shorter polyoxyethylene chains are more hydrophobic and are better foamed. The decontamination factors obtained in the two first stages of the multistage process are in good agreement with those found from the diagram (Table 1). In the next processes the foamed solutions contain molecules with larger polyoxyethylene chains and, consequently, they are poorer foamed (9). Therefore, the final results obtained in the multistage process are less satisfactory than what appears in the diagram.

Almost all of the surfactants of wide application are mixtures of compounds of diverse structure and thus with different surface activities. Therefore, the fact that the diagrams are applicable only to the first stages of the multistage process should be taken into account while designing multistage foaming.

Each consecutive process has values for foaming effectivity which diverge more and more from those calculated on the basis of the diagrams. The agreement of the experimental results with those predicted from the diagrams may be attained only for solutions containing only one component or components of identical surface activity.

## SYMBOLS

|                        |   |
|------------------------|---|
| $h_c$                  | height of the column                            |
| $h_l$                  | height of the liquid phase                      |
| $h_i$                  | height of feeding the column with solution      |
| $C_o$                  | detergent concentration in the feeding solution |
| $C_r$                  | detergent concentration in the raffinate        |
| $C_f$                  | detergent concentration in the collapsed foam   |
| $V_g$                  | gas flow rate                                   |
| $V_o$                  | feeding solutions flow rate                     |
| $C_o/C_r$              | decontamination factor                          |
| $V_f/V_o \times 100\%$ | water removal with the foam                     |

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