

OPERATING A FOAM FRACTIONATING COLUMN IN SIMPLE MODE

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This paper discusses the correct way of operating a foam fractionating column strictly in simple mode. The system chosen was zinc and sodium lauryl sulphate. It was experimentally noticed that if the surface excess was measured at random operating conditions the column was producing inconsistent data, violating the conditions of simple mode operation. After thorough examination it was determined that the height of foam-liquid interface, air flow rate and bubble diameter must be mutually adjusted in order to bring the column close to its simple-mode operation. The results were compared with those of previous investigators, who used the similar apparatus, and it was concluded that the experimental method illustrated in this paper furnished more reliable data of simple-mode operation.

Introduction

Foam fractionation is applicable to removal of objectionable surfactants and/or trace metal ions from dilute effluents, and can well be used as a tool for studying surface phenomena.¹⁾ It is an important adsorptive bubble separation method in which dissolved solutes are selectively adsorbed on the surface of gas bubbles that rise through a liquid pool. A non-surface active substance can be made surface active by deliberately adding a suitable surfactant,

called a collector.¹³⁾ The degree of adsorption of a particular component at the bubble surface is generally expressed by the surface excess,^{11a)} defined as the number of moles of the solute in a portion of the solution per unit interfacial area in excess of the number of moles of the same solute in a portion of the interior which has exactly the same volume.⁷⁾ The separation that can be accomplished is a direct function of the amount of foam generated per unit interfacial area and the concentration of the foamate.

There are two modes of foam fractionation, as shown in Fig. 1:

- (i) Simple mode (batchwise or continuous) and

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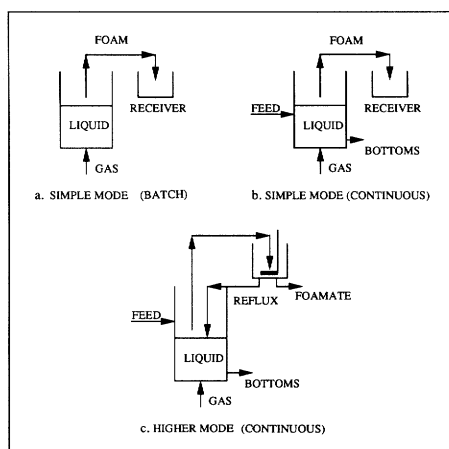


Fig. 1. Types of foam fractionation

(ii) Higher mode with enriching and/or stripping.

"Simple mode unit" means one theoretical (perfect) stage, which requires careful operation for avoiding bubble coalescence in the rising foam. Such coalescence unwillingly releases adsorbed solute back down through the rising foam, causing internal reflux—thereby establishing an erroneous separation beyond that of a single stage.^{11b)} Moreover, there should be no concentration gradient in the liquid pool.

Only a few publications are available on the operation/use of a foam fractionation unit in simple mode. A spherical vessel-type apparatus with flat neck was proposed by Walling *et al.*¹⁸⁾ and Krieg *et al.*⁹⁾ A vertical-type column with recycled foamate in laboratory scale, was originally proposed by Brunner and Lemlich¹⁾ as a one-theoretical stage continuous separator. Haas⁵⁾ and Brunner and Stephan²⁾ then employed the same type of column in pilot plant size. Later on, Syed¹⁷⁾ published some work using a vertical continuous-type column without giving evidence of simple mode. Lee and Maa¹⁰⁾ recently published data on continuous-type simple mode operation, revealing however no attempts to minimize bubble coalescence. Most recently Yu *et al.*²⁰⁾ published results obtained from a vertical column apparatus—which still does not explain how a foam fractionating column can be operated strictly in simple mode.

A batchwise simple mode unit is preferable, especially when perfect single-stage separation is obligatory. It also avoids concentration gradients in the liquid pool. A tapered column representing a simple mode apparatus was repeatedly used by Dick and Talbot,⁴⁾ St. Eloi,¹⁶⁾ Huang and Talbot⁶⁾ and Siy and Talbot,¹⁴⁾ who directly obtained results on the separation of metals from dilute aqueous solutions. In these papers, no evidence of simple-mode operation was presented nor did they discuss any reliable operating method.

It was therefore of utmost interest to critically investigate the correct operating method of the

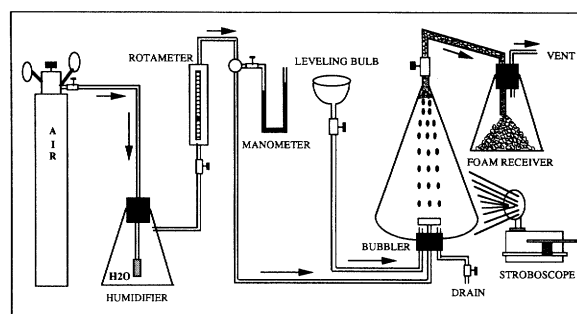


Fig. 2. Simple-mode apparatus for foam fractionation

simple-mode apparatus in order that the data generated from such an experiment would be meaningful. For this purpose it was decided to measure surface excess in a wide range of operating conditions with a major object to recommend proper operating procedure that would minimize bubble coalescence.

1. Experimental

1.1 Description of experimental apparatus

The apparatus, shown in Fig. 2, consisted of a tapered column ($3.0 \times 10^{-3} \text{ m}^3$) made of Pyrex. The advantages of a tapered column are⁷⁾ that (a) it gives support to the rising foam; (b) since the surface area is minimum at the foam-liquid interface, it is easier to collapse the foam; and (c) it essentially assists in minimizing the bubble coalescence.

The major component of the tapered column was a bubbler inserted into the rubber stopper. The bubbler was designed with five glass capillaries ($6.0 \times 10^{-3} \text{ m}$ long and $1.78 \times 10^{-4} \text{ m}$ internal diameter) imbedded in a Teflon chamber to produce bubbles of uniform size. A stroboscope was placed alongside, focussing the array of bubbles to measure the bubble generation rate. The column was painted light blue on the back in order to clearly observe and count bubbles. A level control bulb ($0.3 \times 10^{-3} \text{ m}^3$) was mounted by the side of the column and its tail extended into the column through the rubber stopper. The main purpose of this bulb was to control the height of the foam-liquid interface by moving it upwards and/or downwards. The rubber stopper was also equipped with a drain. The top end of the column was fitted to a vertical foam delivery tube ($5.0 \times 10^{-2} \text{ m}$ height of neck and $9.0 \times 10^{-3} \text{ m}$ internal diameter) which sloped downwards into a receiver in order to avoid recycling of solute. A vent was mounted on the top of the foam receiver to release air. An oil-free compressed air cylinder, a saturator with fritted sparger immersed in distilled water, a calibrated rotameter and a mercury manometer were interconnected as an assembly to pass air into the bubbler.

1.2 Experimental procedure

Deionized distilled water, zinc standard solution

and sodium lauryl sulfate were mainly used in preparing any feed solution. The pH of the feed solution was adjusted to the desired value by adding HNO_3 , NaOH or both. The flowrate of air, from a regulated pressure of 143 kPa, was brought to the desired value by simultaneously manipulating two needle valves of the rotameter and manometer.

The tapered column was filled with feed solution by dismantling and reassembling the top portion. The level control bulb was also filled to the required mark. When the level of the foam-liquid interface was perfectly constant, a foam receiver of known weight was placed in position and the timer started. Then the bubble generation rate was determined using the electronic stroboscope. Foam was collected into the receiver over a measured time period of 600 s. The receiver was then weighed and stoppered. The foam was allowed to freeze, followed by thawing. The foamate solution was analysed for the concentration of zinc by means of an atomic absorption spectrophotometer.

2. Experimental Results and Discussion

2.1 Preliminary comments

The system chosen for the simple mode foam fractionation was a dilute solution containing 0.15 mol/m^3 zinc as the metal ion, and 1.73 mol/m^3 sodium lauryl sulphate (NaLS) as the collector. The most stable foam was obtained with the chosen collector concentration. The chosen zinc concentration was found to be optimum in a separate study.⁸⁾ However, it should be borne in mind that this paper is not dealing with the chemistry involved in the foam fractionation of metals but is exploring the correct way of operating a simple-mode unit.

After a keen literature survey and extensive experimentation it became evident that simple-mode operation is very sensitive to height of foam-liquid interface, flowrate of the gas that generates bubbles and bubble diameter. The following discussion therefore concerns their influence. **Table 1** shows the range of several parameters encountered during experimentation.

2.2 Height of foam-liquid interface

Refer to **Fig. 3**, which shows the results of the first experiment, conducted exactly as explained in the experimental procedure but by filling the column with feed solution up to 0.15 m above the bubbler. The same experiment was repeated by filling the column up to 0.20 m, 0.23 m, and so on. It is evident in **Fig. 3** that the concentration of zinc in foamate increased exponentially as the height of foam-liquid interface above the bubbler was decreased. In other words, the foamate became enriched in zinc more intensely as the foam height above the liquid was decreased. As $H_{f/l}$ was decreased from 0.32 to 0.15 m, the foamate's zinc

Table 1. Range of parameters

S.No	Parameter	Units	Range
1	C_f	$[\text{mol/m}^3]$	0.15 to 4.43
2	$d \times 10^3$	$[\text{m}]$	2.56 to 3.76
3	$H_{f/l}$	$[\text{m}]$	0.15 to 0.36
4	$\Delta H_{f/l}$	$[\text{m}]$	-1.25 to +0.5
5	n	$[-]$	2 to 5
6	N	$[\text{s}^{-1}]$	63 to 184
7	pH	$[-]$	1.8 to 7.6
8	$Q \times 10^6$	$[\text{m}^3/\text{s}]$	0.33 to 3.4
9	$S \times 10^3$	$[\text{m}^3/\text{s}]$	2.5 to 5.5
10	$V_f \times 10^8$	$[\text{m}^3/\text{s}]$	0.61 to 7.1
11	$(V_f/S) \times 10^6$	$[\text{m}]$	6.4 to 13.0
12	$\Gamma \times 10^6$	$[\text{mol/m}^2]$	0.32 to 1.46

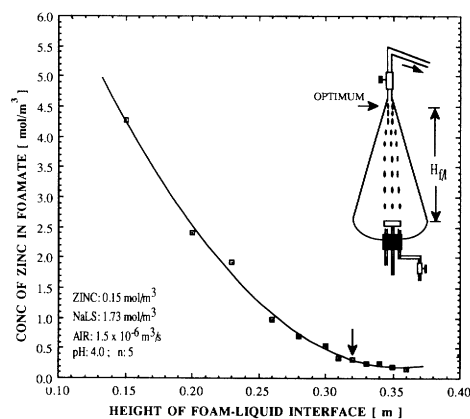


Fig. 3. Effect of $H_{f/l}$ on foamate concentration

content increased by at least 15 times.

The column was designed particularly to carry out batchwise foam fractionation in simple mode. The most important condition that can never be violated is to say “no bubble coalescence in simple mode.” But in this case the bubble coalescence deliberately created in the rising foam by lowering the $H_{f/l}$ released adsorbed solute that ran down, due to gravity, through the rising foam as an internal reflux, enriching the foam beyond that of a single-stage separation. Moreover, solute molecules migrated to the air-liquid interfaces associated with the bubbles and the liquid passed downward among bubbles—causing the foam to be continuously enriched in solute. The higher enrichment was the consequence of internal bubble gas diffusion and the rupture of the film between bubbles.^{11c)} Even though such enrichment is essential under higher-mode operation, it is undesirable in simple mode foam fractionation.

At each datum point of the plot in **Fig. 3**, the concentration of foamate was tested for uniformity and the results are shown in **Fig. 4a** and **4b**. It was obvious that if the foam was being enriched with drainage and coalescence, the concentration would not be uniform with time. This fact was experimentally verified by measuring the concentration against time

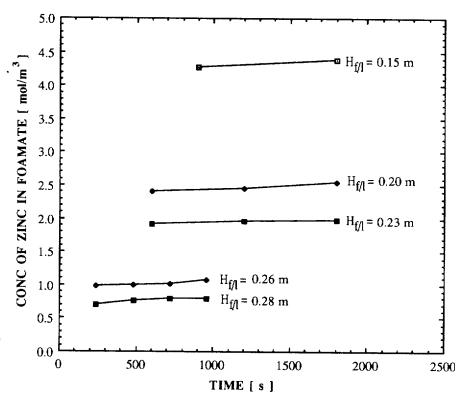


Fig. 4a. Variation of foamate concentration with time

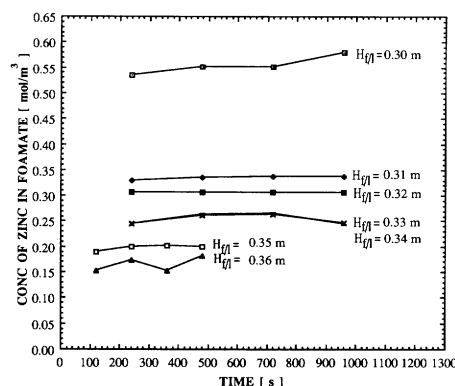


Fig. 4b. Variation of foamate concentration with time

at several heights. For a given $H_{f/l}$, the experiment was conducted exactly as explained in the experimental procedure but by collecting the foam samples continuously into several receivers at equal intervals of time. It was noticed that, for heights between 0.15 and 0.32 m (higher foam heights), the concentration of zinc in foamate increased with time, indicating the continuous growth of coalescence and drainage. For heights between 0.33 and 0.36 m (lower foam heights), the foam was delivered wet due to liquid entrainment diminishing the concentration to an unreasonable value. Moreover, the concentration determined at equal intervals of time was found to be non-uniform. The lower concentrations also implicated that single-equilibrium stage separation was not being accomplished perfectly. Therefore, the heights between 0.33 and 0.36 m in the column were disregarded because the separation efficiency was poor. The present attempt was to choose the proper level which would achieve a maximum concentration without drainage or coalescence. This was possible only at a height of 32 cm—which was reasonably chosen as the optimum height.

2.3 Surface excess

The surface excess (Γ) was determined from the mass balance over a single equilibrium stage^{1,7)}

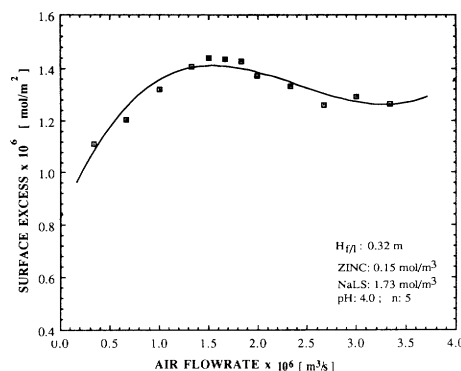


Fig. 5. Effect of air flowrate on zinc surface excess (usual operation)

$$V_f C_f = S\Gamma + V_f C_b \quad (1)$$

or

$$\Gamma = (V_f/S)(C_f - C_b) \quad (2)$$

The surface generation rate (S) was determined with the knowledge of the air flowrate and bubble generation rate.⁸⁾

The above equation was applied under the following three assumptions: (a) the surface excess was considered at the surface of the bubble only; (b) the adsorption occurred within the liquid phase of the column with sufficient contact time to reach the equilibrium; and (c) bubbles were all spherical and uniform, with negligible breakage of bubbles.

The first assumption is obvious. The second assumption was verified by using Eq. (3)¹⁹⁾:

$$\frac{d\Gamma}{dt} = \left[\sqrt{\frac{D^*}{\pi}} \right] \left[\frac{C_b}{\sqrt{t}} \right] \quad (3)$$

Dick⁴⁾ used Eq. (3) and determined the time required to form a complete monolayer of surfactant as 0.013 s. In the current experiment the residence time of bubbles within the bulk was around 0.5 s—which obviously means there was sufficient time to form a complete monolayer of NaLS. The third assumption was convenient in determining the bubble generation rate. However, proper care was taken in designing the bubbler to produce uniform spherical bubbles.

2.4 Air flowrate

Figure 5 shows the effect of air flowrate on zinc surface excess as was determined experimentally. It is observed from the plot that the measured zinc surface excess reached maximum at an air flowrate of $1.5 \times 10^{-6} \text{ m}^3/\text{s}$. This flowrate was regarded as optimum because the same flowrate was used to minimize bubble coalescence while determining the optimum height in the previous section. For flowrates other than optimum, the measured surface excess decreased gradually. It was also observed that the foamate concentration at lower flowrates increased

more than that at the optimum flowrate, and at higher flowrates it decreased. For a perfect single equilibrium stage, the measured surface excess calculated from the surface concentration that is in equilibrium with bulk liquid concentration should not be affected just by the change in physical entrainment of the bubbles.

Equation (2) suggests that the measured surface excess depends on the (V_f/S) ratio and C_f (C_b is almost the same as feed concentration). In the current experimental data, the (V_f/S) ratio—which represents the volume of foam per unit interfacial area—increased with air flowrate as shown separately in Fig. 6. C_f increased at lower flowrates because of the high residence time of bubbles in the rising foam, and C_f decreased at higher flowrates because too much liquid entered the foam and the interfacial turbulence caused re-entry of the adsorbed ions into the solution.¹²⁾ A similar type of experience was earned when $H_{f/l}$ was increased above 0.32 m in the previous section. The data revealed bubble coalescence and drainage at lower flowrates, and poor performance at higher flowrates—violating the conditions of simple mode. The measured surface excess obtained at the optimum air flowrate was considered as the true result, and that obtained at any other flowrate was treated as a false result.

Serious attempts were then made to correct any false surface excess by adjusting the operating condition such as $H_{f/l}$ so that a constant (V_f/S) ratio would be accomplished at all flowrates. For this purpose the optimal (V_f/S) ratio was computed as 9.4×10^{-6} m at the corresponding optimum air flowrate of 1.5×10^3 m³/s. The foam-liquid interface for lower air flowrates was moved upwards so that the (V_f/S) ratio increased and the concentration of the foamate decreased, and for higher air flowrates it was moved downwards so that the (V_f/S) ratio decreased and the concentration of the foamate increased. This approach was successfully implemented for other flowrates, and the corrected results are shown in Fig. 7.

The change of height of foam-liquid interface required in achieving the constant (V_f/S) ratio is plotted against air flowrate in Fig. 8—which gave the following linear regression equation:

$$\Delta H_{f/l} = 1.21 \times 10^{-2} - 7942.8Q \quad (4)$$

Eq. (4) predicts approximately the change of height of foam-liquid interface for a given air flowrate. It would be useful for any other column of the same type when the initial optimum height is estimated at an air flowrate of 1.5×10^{-6} m³/s.

2.5 Bubble diameter

Figure 9, in which the measured surface excess is plotted against pH, shows the influence of bubble diameter. The bubbler was designed with 5, 4, 3 and

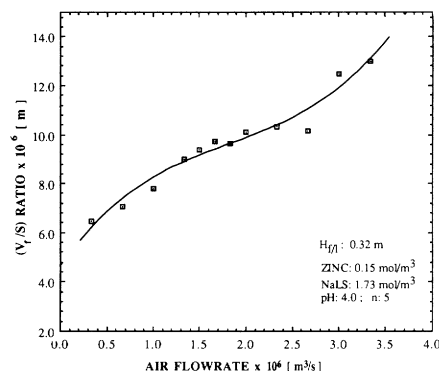


Fig. 6. Effect of air flowrate on (V_f/S) ratio

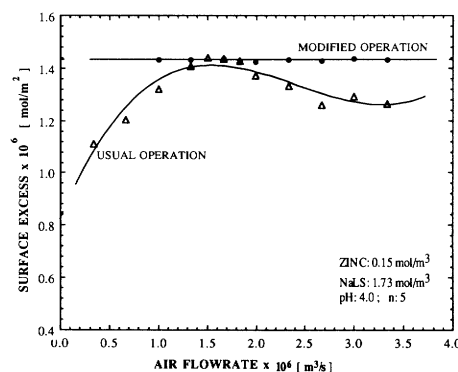


Fig. 7. Effect of air flowrate on zinc surface excess (modified operation)

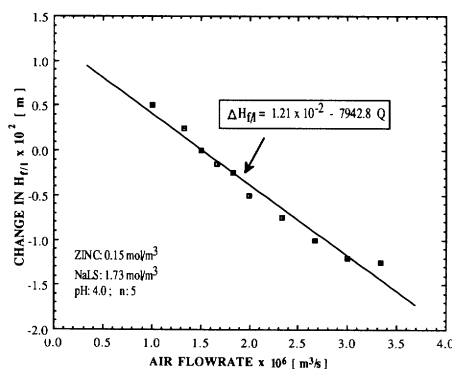


Fig. 8. Change in $H_{f/l}$ versus air flowrate

2 capillaries respectively to generate bubbles of various sizes. Surface excess was measured under these four different situations. It was noticed that when the bubble size was minimum ($n = 5$), the interfacial area was maximized, providing maximum surface excess. The surface excess decreased gradually with increase in bubble diameter. It was also realized from the results that this change in surface excess was due apparently to a change in (V_f/S) ratio. This change in (V_f/S) ratio with bubble diameter is fundamentally correct as far as the soap bubbles are concerned (the more the interfacial area available, the greater will be the surface excess and vice versa⁷⁾). The measured surface

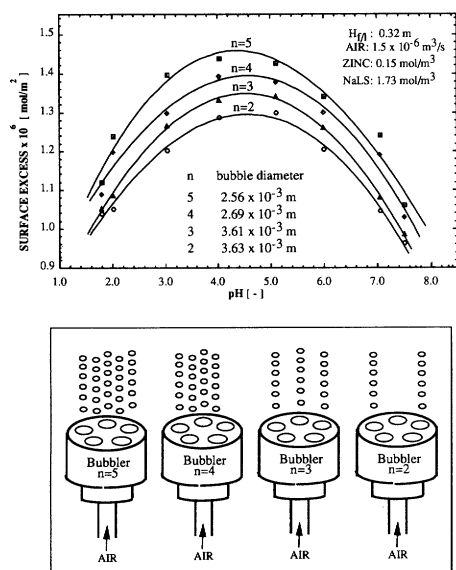


Fig. 9. Effect of bubble diameter on zinc surface excess (usual operation)

excess in any particular simple-mode apparatus, however, should not be affected by the physical entrainment of bubbles. It was therefore concluded that the curves obtained for $n=4$, 3 and 2 represented false surface excess.

Serious attempts were then made to correct the false surface excess to true value by simultaneously adjusting the air flowrate and height of foam-liquid interface so that a constant (V_f/S) ratio would be accomplished. The required change in $H_{f/l}$ was computed from Eq. (4). The corrected data with adjusted air flowrates are shown in Fig. 10. For instance, for $n=4$, when the air flowrate and height of foam-liquid interface were adjusted, the bubble diameter and bubble generation rate increased slightly. As a result, the surface generation rate and foam generation rate increased adequately so that a constant (V_f/S) ratio of $9.4 \times 10^{-6} \text{ m}$ was obtained. In this attempt the change in $H_{f/l}$ was not significant. A similar approach was successfully implemented for other cases ($n=3$ and 2).

2.6 Importance of the (V_f/S) ratio:

The (V_f/S) ratio is the amount of foam collected per unit interfacial area. For a particular unit (simple-mode apparatus), the amount of foam varied according to the entrainment of the bubbles, which could coalesce and produce inconsistent results. No conscious effort was made by previous investigators to determine an optimal (V_f/S) ratio nor did they keep this ratio constant.

The (V_f/S) ratios were computed from the data of other investigators, who used similar apparatus, and are shown in Tables 2, 3, 4 and 5. These results implied that pH had some effect on the (V_f/S) ratio, without a definite trend. It also seemed that the foam

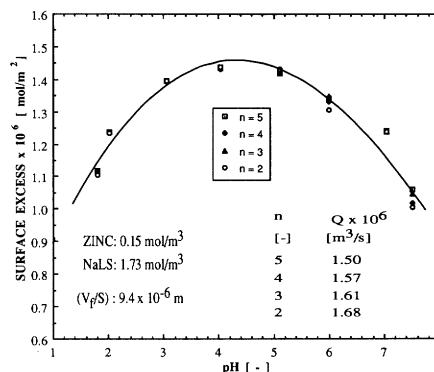


Fig. 10. Effect of bubble diameter on zinc surface excess (modified operation)

Table 2. Present results (System: Zinc-NaLS)

S.No	pH [—]	$Q \times 10^6$ [m ³ /s]	$V_f \times 10^8$ [m ³ /s]	$S \times 10^4$ [m ² /s]	$(V_f/S) \times 10^6$ [m]
1	1.80	1.5	3.29	35.10	9.37
2	2.02	1.5	3.29	35.09	9.38
3	3.05	1.5	3.31	35.19	9.43
4	4.02	1.5	3.27	35.10	9.32
5	5.10	1.5	3.26	35.10	9.29
6	6.00	1.5	3.32	35.09	9.46
7	7.05	1.5	3.32	35.10	9.46
8	7.51	1.5	3.29	35.10	9.37

Table 3. Results of Dick and Talbot (System: Copper-NaLS)

S.No	pH [—]	$Q \times 10^6$ [m ³ /s]	$V_f \times 10^8$ [m ³ /s]	$S \times 10^4$ [m ² /s]	$(V_f/S) \times 10^6$ [m]
1	1.09	1.07	2.03	32.82	6.19
2	1.75	1.07	2.32	33.15	7.00
3	2.88	1.07	2.37	32.80	7.23
4	3.20	1.07	2.45	32.34	7.58
5	4.39	1.07	2.45	32.68	7.50
6	4.58	1.07	2.30	32.98	6.97
7	5.43	1.07	2.18	33.62	6.48
8	6.00	1.07	3.88	31.70	12.24

Table 4. Results of St. Eloi (System: Zinc-NaDBS-NaCl)

S.No	pH [—]	$Q \times 10^6$ [m ³ /s]	$V_f \times 10^8$ [m ³ /s]	$S \times 10^4$ [m ² /s]	$(V_f/S) \times 10^6$ [m]
1	1.60	1.78	1.40	42.93	3.26
2	1.86	1.26	1.33	33.21	4.00
3	2.00	2.03	1.57	45.66	3.44
4	2.38	1.78	1.78	42.89	4.15
5	2.54	1.26	1.28	33.16	3.86
6	2.46	1.26	1.60	33.74	4.74
7	3.10	1.78	1.42	42.92	3.31
8	3.48	1.26	1.72	33.34	5.16
9	3.85	1.26	1.45	33.11	4.38
10	4.05	1.78	1.47	43.02	3.42
11	5.00	1.78	1.42	43.53	3.26
12	5.15	1.26	1.70	33.12	5.13
13	6.12	1.26	1.90	33.56	5.60
14	6.13	1.78	1.43	43.28	3.30
15	7.05	1.78	1.52	43.10	3.53
16	7.49	1.78	1.43	45.56	3.14

Table 5. Results of Siy and Talbot (System: Zinc-NaDBS)

S.No	pH [—]	$Q \times 10^6$ [m ³ /s]	$V_f \times 10^8$ [m ³ /s]	$S \times 10^4$ [m ² /s]	$(V_f/S) \times 10^6$ [m]
1	2.04	1.63	4.65	39.97	11.63
2	2.49	1.63	5.58	39.75	14.04
3	2.59	1.63	6.05	39.44	15.34
4	2.76	1.63	5.95	39.48	15.07
5	2.86	1.63	5.56	39.39	14.12
6	2.90	1.63	5.60	39.55	14.16
7	3.22	1.63	5.88	39.49	14.89
8	3.42	1.63	3.73	38.36	9.72
9	3.79	1.63	3.85	39.21	9.82
10	4.12	1.63	4.58	39.32	11.65
11	4.50	1.63	3.67	38.77	9.47
12	5.18	1.63	4.56	40.02	11.39
13	5.78	1.63	3.52	39.02	9.02
14	5.84	1.63	3.63	40.50	8.96
15	6.57	1.63	3.82	38.47	9.93
16	7.02	1.63	4.68	40.00	11.70

generation rate at constant air flowrate did fluctuate significantly. In the results of Dick and Talbot^{3,4)} the (V_f/S) ratio varied from 6.22×10^{-6} to 7.60×10^{-6} m at the same air flowrate. In St. Eloi's results¹⁶⁾ the (V_f/S) ratio, which varied from 3.14×10^{-6} to 5.64×10^{-6} m, seemed very low at specified high flow rates. In the results of Siy and Talbot^{14,15)} the (V_f/S) ratio varied from 8.99×10^{-6} to 15.35×10^{-6} m. This was due to the fact that the surface generation rate and foam generation rate fluctuated for the same air flowrate. However, the survey revealed that previous investigators never attempted to maintain a constant (V_f/S) ratio nor did they verify simple-mode operation. In fact, the optimal (V_f/S) ratio is the key parameter that would conveniently verify whether or not the column operates meaningfully in simple mode.

Conclusion

A simple-mode foam fractionating column was thoroughly examined and the optimal operating procedure illustrated. For the particular unit employed, a height of 0.32m above the bubbler was proved to be the only optimum. This optimum height was very sensitive to air flowrate, and its deviation can be correlated as:

$$\Delta H_{f/l} = 1.21 \times 10^{-2} - 7942.8Q$$

The measured surface excess represented a false value, being disturbed from simple mode, whenever the bubble size was inappropriate. This discrepancy was overcome by simply maintaining an optimal (V_f/S) ratio of 9.39×10^{-6} m. Ignorance of the (V_f/S) ratio might generate a false surface excess and henceforth a false degree of adsorption.

It was finally concluded that a careful tuning of height of the foam-liquid interface, air flowrate and bubble diameter towards minimizing bubble coa-

lescence is the correct way of operating a foam fractionating column in simple mode.

Recommendation

A simple-mode pilot plant unit should be installed to further correlate the height of foam-liquid interface as a function of the air flowrate, geometry of the column and geometry of bubbles.

Nomenclature

C_f	= conc of metal in foamate	[mol/m ³]
C_b	= conc of metal in bulk liquid	[mol/m ³]
D^*	= diffusion coefficient	[m ² /s]
d	= bubble diameter	[m]
$H_{f/l}$	= height of foam-liquid interface	[m]
$\Delta H_{f/l}$	= change in $H_{f/l}$	[m]
N	= number of bubbles per sec per capillary	[s ⁻¹]
n	= number of capillaries	[—]
pH	= negative log of hydrogen ion conc	[—]
Q	= air flowrate	[m ³ /s]
S	= surface generation rate	[m ² /s]
t	= time	[s]
V_f	= foam generation rate	[m ³ /s]
Γ	= zinc surface excess	[mol/m ²]
NaCl	= sodium chloride	
NaDBS	= sodium dodecyl benzene sulfonate	
NaLS	= sodium lauryl sulphate	

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