Foam Fractionation with Reflux

Abstract. A surface-active aqueous solution was foamed up a glass column, removed, collapsed, and returned as downcoming liquid to drain countercurrently against the rising foam. This deliberate refluxing action greatly increased the degree of separation of the surfactant over that obtainable without reflux. The effects of altering the gas rate and the solute concentration were also studied.

There are several common methods for physically concentrating or separating the components of a liquid solution. Examples are distillation and extraction. These methods depend on differences in concentration between different physical phases in contact with one another. Where such differences are small, countercurrently flowing reflux can be used to cascade them and thus increase the over-all separation obtainable.

Now, a less usual method, namely foam fractionation, has been proposed and even used on occasion (*1*). In this method the separation depends on surface activity. One (or more) components of the solution are absorbed at any surface formed, so that bubbling off foam from the solution produces a partial separation of components. Unfortunately, the degree of such separation is usually very modest (*2*). Accordingly, an experimental study (*3*) was begun to examine the feasibility of using reflux to increase analogously the over-all separation obtainable with foam fractionation. Some results of this study are reported here.

The apparatus employed is shown in the abbreviated schematic diagram (Fig. 1). The foam fractionation column was of glass with a diameter of 1 in. and a height of 6 ft. The mixture used was a dilute aqueous solution of the commercial surfactant Aresket-300 (monobutyl diphenyl sodium monosulfonate). The solution was charged to a 2-liter flask at the bottom of the column. Nitrogen, which had first been thoroughly moistened in a preliminary packed absorption column in order to eliminate any spurious evaporative effects, was bubbled through the charge at a controlled and measured rate. Foam rose through the column, out the top, and into a centrifugal foam breaker consisting of a spinning wire basket in a very large funnel. Collapsed foam flowed by gravity from the funnel stem, and a portion was returned to the column some distance down from the top. (Pressure in the column prevented introduction directly at the top.) The remainder of the collapsed foam was drawn off as concentrated "product." However, in order to achieve steady state and avoid the necessity for continually replenishing the charge, the product was simply returned by gravity to the flask at the bottom.

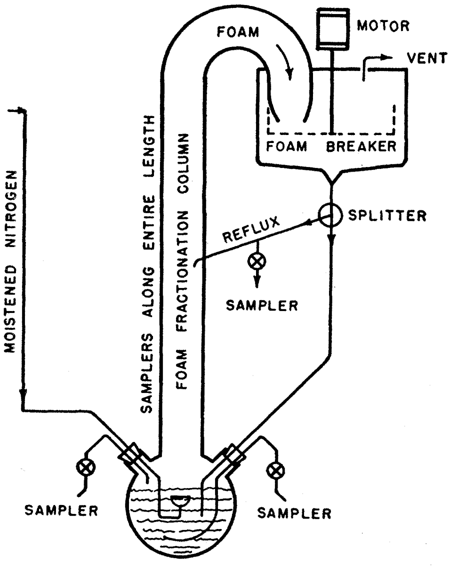

Fig. 1. Foam fractionation apparatus (not to scale).

Samples for analysis were withdrawn repeatedly from the reflux line in order to check the approach to steady state. Some runs were continued for as long as 48 hr. Just before shutdown, samples were drawn from the top, bottom, reflux line, and locations along the column length generally at 5-in. intervals.

Much of the work was carried out in a factorial experiment. The effects of reflux ratio, solute concentration, gas rate, and column level were studied. At high reflux ratios the concentration increased considerably up the column. In fact, for total reflux an overhead concentration in excess of 10 times the charge concentration was obtained, as shown in Fig. 2. However, without external reflux the increase in concentration was far less. In other words, the downcoming reflux greatly enriched the rising foam so that the column acted as a rectifier (enricher). Thus the utilization of reflux can greatly increase the ability of a foam fractionation device to concentrate a constituent of a solution.

Decreasing the gas rate increased the degree of separation. This is attributed in part to decreased channeling (which was readily visible) and longer residence time at the lower rate, which make for better over-all contact between rising bubbles and downcoming liquid. A larger residence time also permits somewhat more foam drainage and collapse in the column (which were also visible), thus promoting some degree of self-reflux. Also in line with these several reasons, in some auxiliary work an increase in separation resulted from an increase in column diameter.

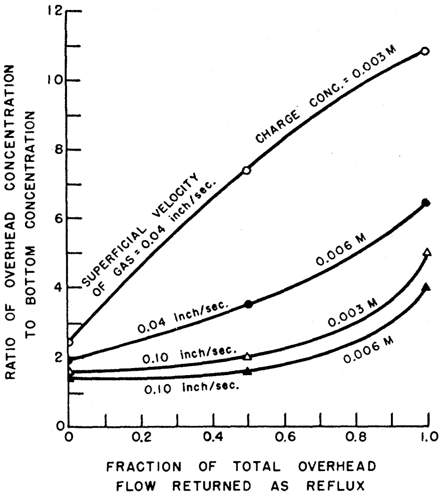

Fig. 2. Performance of a foam fractionation column, showing the beneficial effect of reflux. The effects of different solute concentrations in the charge and different gas rates are also shown.

The degree of separation increased somewhat with a decrease in charge concentration. This follows from the relatively stronger tendency toward surface adsorption that generally exists at lower concentrations. However, it is worth noting at this point that direct quantitative estimation of such adsorption by the Gibbs equation (*4*) cannot be reliably made for foams, even without reflux. The Gibbs equation applies to conditions of true equilibrium, which of course do not exist at the mobile surface of a foam bubble.

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References and Notes

1. L. Shedlovsky, *Ann. N.Y. Acad. Sci* **49**, 279 (1948).
2. For an interesting exception, see R. W. Schnepf, E. L. Gaden, Jr., E. Y. Mirocznik, and E. Schonfeld, *Chem. Eng. progr.* **55**, No. 5, 42 (1959).
3. This work is supported by a U.S. Public Health Service grant (RG-5870). We also thank E. L. Gaden, Jr., of Columbia University, for his suggestions concerning the foam breaker and the choice of Aresket-300.
4. S. Glasstone, *Textbook of Physical Chemistry* (Van Nostrand, New York, ed. 2, 1946), p. 1206.

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