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Foam Counter-current Chromatography: New Foam Separation Technique with Flow-Through Coil Planet Centrifuge

Yoichiro Ito[a]; Robert L. Bowman[a]
[a] LABORATORY OF TECHNICAL DEVELOPMENT, NATIONAL HEART AND LUNG INSTITUTE, BETHESDA, MARYLAND

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Foam Countercurrent Chromatography: New Foam Separation Technique with Flow-Through Coil Planet Centrifuge

YOICHIRO ITO and ROBERT L. BOWMAN

LABORATORY OF TECHNICAL DEVELOPMENT
NATIONAL HEART AND LUNG INSTITUTE
BETHESDA, MARYLAND 20014

Abstract

Under a rotating centrifugal force field, foams are created within a long fine coiled tube to carry foam active materials through the solvent trapped in each turn of the coil. Rhodamine B introduced at 1 ppm was almost entirely recovered at 620 ppm. High-speed chromatographic separation is possible with a small amount of sample.

Foam separation techniques (*1–3*) have been increasingly used for separations of a variety of substances ranging from small ions to microscopic and macroscopic particles. Despite their great potential in versatility and selectivity, the method still remains relatively unpopular because of the unavailability of an efficient apparatus. This paper introduces a new foam separation technique which may open a broad application to scientific research. In this method, foams are passed through a long coiled tube against a countercurrenting solvent by means of the flow-through coil planet centrifuge reported earlier (*4*, *5*).

The principle of the method is demonstrated by a simple model which consists of a coiled tube rotating about its horizontal axis. Liquid and gas

confined in the coiled tube soon reach a dynamic equilibrium state where each turn of the coil is occupied by a nearly equal amount of each phase starting from one end of the coil, called "the head," and any excess of either phase occupies the other end of the coil, called "the tail," as in the case of two-phase liquid systems. In general this dynamic equilibrium state has an interesting feature in that a third phase heavier than the gas phase and lighter than the liquid phase is forced to move toward the tail end of the coil. Thus, when the liquid contains surfactant, foams created by rotation of the coil undergo a countercurrent flow toward the tail carrying any solute or particles having an affinity to the foam. This countercurrent process can be further enhanced if the gas is constantly supplied through the head to the tail end of the coil. In order to achieve an efficient separation with a long fine coiled tube, it is necessary to apply a centrifugal force which prevents a plug flow under a high countercurrent flow and also quickly eliminates excess liquid from the foams.

The flow-through coil planet centrifuge was used to provide a rotating centrifugal force field with respect to the coiled tube. Since the apparatus avoids the use of rotating seals, a desired number of flow tubes can be used to move the liquid or gas phase in and out through the rotating column against a pressure of several hundred psi.

The elution schemes are described—one for continuous removal and/or concentration of a minute amount of substance present in a large quantity of liquid and the other for a chromatographic separation of a relatively small amount of sample. The first scheme is shown in Fig. 1.

The separation column is prepared from a 2.5-mm i.d., 30 m long Teflon tubing coiled onto six plastic pipes with 1.3 cm o.d., making approximately 1000 turns. The column has four flow tubes, the liquid collection line and the gas feed line on the head side, and the liquid feed line and the foam collection line on the tail side as illustrated.

After the apparatus reaches the desired speed, the solvent containing a surfactant is pumped through the liquid feed line while nitrogen gas is fed through the gas feed line, the gas flow rate being monitored at the outlet of the foam collection line. When the foams begin to elute from the foam collection line, the needle valve on the liquid collection line is opened to elute the liquid phase at the desired rate, while the solvent is switched to the sample solution. The foam flow rate through the foam collection line is equal to the difference between the liquid feed rate and collecting rate and can be adjusted by the needle valve or any resistance placed after the valve. A gas trap chamber on the liquid collection line automatically

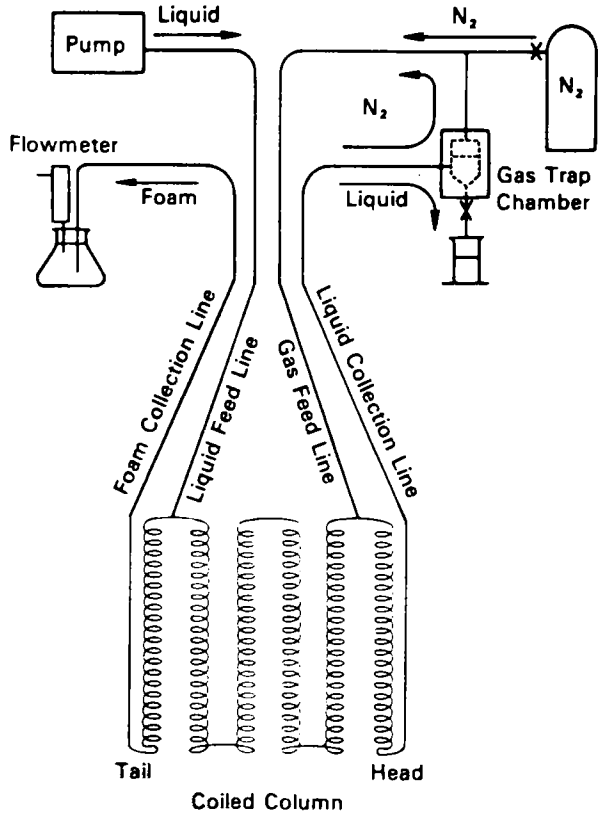

FIG. 1. Diagram of elution scheme for removal and enrichment of foam active material. The flow-through coil planet centrifuge carries six helical column units, connected head to tail in series, equipped with four flow tubes. The solvent fed through the liquid feed line elutes through the liquid collection line while N_2 introduced through the gas feed line escapes through the foam collection line as foams carrying any foam active material. Thus the sample solution introduced through the liquid feed line is continuously eluted through two lines, the stripped solution from the liquid collection line and the enriched foams from the foam collection line.

returns the gas phase to the gas feed line,* thus facilitating the liquid collection. Consequently, the sample solution is continuously separated into two streams, the stripped solution from the liquid collection line and the enriched foams from the foam collection line. This technique is suitable for continuous removal of a minute amount of substance from a large quantity of liquid.

When the objective is enrichment of the foam active substance, the technique is modified in such a way that the substance is first retained in the column, washed, and eluted in a concentrated state. This can be done with the same elution system described above. In this case the liquid collection rate is adjusted equal to or slightly below the liquid feed rate so that the foam is not eluted through the foam collection line and the active material is retained in the column. After the sample feed is completed, the washing solution is introduced through the liquid feed line to wash the column contents until the eluate through the liquid collection line becomes clean. Then the needle valve on the liquid collection line is closed and the

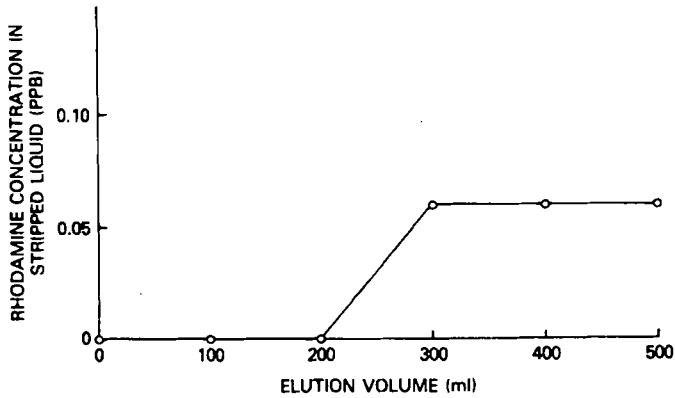

FIG. 2A. Stripping power of the method: Sample solution containing 1 ppm rhodamine B and 100 ppm sodium lauryl sulfate as a collector was eluted through the column without sampling the foams so that the dye was retained within the column. The dye stripping power is estimated by sampling the eluate at 100 ml interval for fluorescence analysis. The fluorescence activity was not detectable until the 300-ml sampling period when the activity rose to 0.06 ppb which continued up to the end of the experiment.

*The dynamic equilibrium state mentioned earlier creates a pressure gradient linearly increasing from the tail to the head end of the coil. Thus, connection between two portions of the coil immediately results in circulation of the contents through the loop.

liquid feed rate is slowed down to efficiently collect the enriched foams through the foam collection line.

Efficiency of the above technique was examined on removal and enrichment of rhodamine B using sodium lauryl sulfate as a collector. Five hundred milliliters of sample solution, containing 1 ppm rhodamine B, 100 ppm sodium lauryl sulfate, and 0.01 *M* NaCl, were fed at a flow rate of 120 ml/hr while the apparatus was operated at 360 rpm with a 30-cm revolutional radius. The pressure gauge at the nitrogen tank was set at 200 psi, which gave a flow rate of 4 l/min at the outlet of the foam collection line. Under these conditions the liquid volume held in the column was about 27 ml.

The eluate through the liquid collection line was sampled at 100 ml intervals for fluorescence analysis, the result being shown in Fig. 2A. No fluorescent activity was detected until the 300-ml sampling period when the activity rose to 0.00006 ppm, which continued steadily up to the end

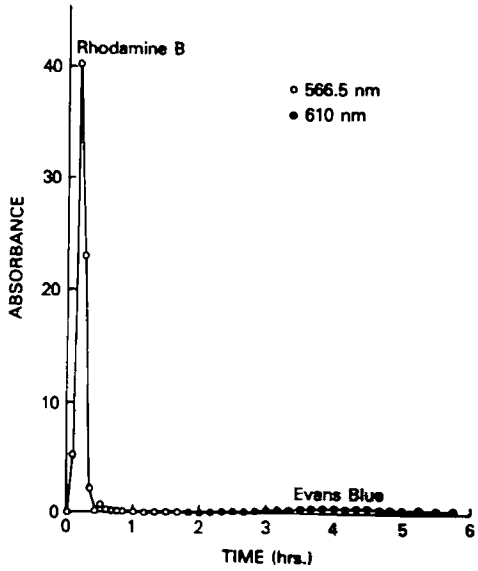

FIG. 2B. Foam chromatogram of rhodamine B and Evans blue. Sample solution containing each dye at 250 ppm (sample size, 0.5 ml) was eluted through the column with a solvent containing 100 ppm sodium lauryl sulfate as a rhodamine B collector. At a feed rate of 6 ml/hr, rhodamine B was eluted within 20 min with a sharp peak while Evans blue appeared in 3 hr with a broad peak.

of the elution. Then the retained dye was eluted through the foam collection line at 2.4 ml/hr. The dye was almost entirely recovered in less than 1 ml at a concentration of 620 ppm.

The second elution scheme for chromatographic separation uses two flow lines, the gas/liquid feed line at the head and the foam collection line at the tail. The column is first equilibrated with the gas/liquid phase at a predetermined revolutional speed, and the sample solution is introduced through the feed line. Then the column is fed with a mixture of gas and liquid phases at a given ratio through the feed line while the foams are fractionated through the foam collection line. In order to achieve an efficient separation, a high gas flow rate and a slow liquid feed rate should be applied.

Capability of the method was demonstrated on separation of rhodamine B and Evans blue using sodium lauryl sulfate as a collector of rhodamine B. A sample size of 0.5 ml containing each component at 250 ppm was eluted at 6 ml/hr of liquid flow and 3 l/min of gas flow at the outlet of the foam collection line under 270 rpm. Figure 2B shows a foam chromatogram where rhodamine B was eluted in 20 min with a sharp peak while Evans blue began to appear in 3 hr with a broad peak which continued up to the end of the experiment.

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