

Chapter 17

Other and Novel Separation Processes

As noted in Part Two, separation processes may be separated into two categories: *phase separation* and *component separation* processes; in the latter, a particular species is separated from a single-phase, multicomponent system. The treatments may fall into one or both of these categories.

Component separation processes remove particular ionic or molecular species, generally without the use of chemicals. Phase separation processes are employed to separate phases, reduce feed volume, and to concentrate the feed into one phase before further treatment and material recovery are performed. Such feed streams, which can include slurries, sludges, and emulsions that contain more than one phase, are the usual candidates for this category.

Many factors need to be considered when selecting a particular type of treatment. These include: the characteristics of the feed stream and the desired characteristics of the output stream, the technical feasibility of the different physical treatments when applied to a particular case, and economic, environmental, and energy considerations.

In this chapter, the less established types of separation treatment methods used today are examined. Part Two of this book provided information, sometimes in significant detail, on the major mass transfer equipment and processes employed by industry. However, there are others involving either component or phase separation, or both, that did not receive any treatment but deserve some consideration. These topics include

- 1 Freeze crystallization
- 2 Ion exchange
- 3 Liquid ion exchange
- 4 Resin adsorption
- 5 Evaporation
- 6 Foam fractionation
- 7 Dissociation extraction

8 Electrophoresis

9 Vibrating screens

A brief review⁽¹⁾ of each of the above is provided in this chapter. Additional details are available in the literature where noted.

FREEZE CRYSTALLIZATION

Freeze crystallization is a phase separation process that is in the process of becoming fully developed or achieving its full potential for significant commercial use. Most of the development has been for use in instant coffee and water desalination. On a laboratory scale, this process has been used in the treatment of feeds containing ammonium nitrate, paper mill bleach solutions, plating liquors, and arsenal redwater. The freeze crystallization process also has potential use in the recovery/removal of 1–10% total dissolved solids (TDS) in aqueous streams.

Contaminated wastewater can be subcooled during freeze crystallization in order to form purified ice crystals. The remaining liquid is more concentrated in soluble organic or inorganic materials. The crystals are subsequently removed, washed, and melted to recover the water. The liquid could be freeze crystallized again to further concentrate the feed and make treatment and/or disposal much easier.

ION EXCHANGE

Ion exchange is generally used for the removal of dilute concentrations of heavy metals and anions from aqueous streams. Though the process is fully developed, it is not commonly used in industry. It has been used in recovering effluents from fertilizer manufacturing, the deionization of water, treating electroplating waste waters, and looks promising for the removal of cyanides and selected heavy metals from streams.

Ion exchange is a two-step process. First, a solid material, the ion exchanger, collects specific ions after coming into contact with the aqueous stream. The exchanger is then exposed to another aqueous solution of a different composition that picks up the ions originally removed by the exchanger. The process is usually accomplished by sending the two aqueous streams through one or more fixed beds of exchangers. The ion-rich product stream may be recovered.

LIQUID ION EXCHANGE

Liquid ion exchange (LIE) serves the same purpose as ion exchange, the removal of heavy metals and anions from aqueous streams. However, LIE may be used for treating a higher concentration of metals and ions. This process is also well-developed

though not widely used in waste treatment; it has promising uses in the removal of cyanide from waste waters and in treating hydroxide slimes produced in electroplating.

Liquid ion exchange uses an organic stream to carry out the transfer of ions from the aqueous stream to a second aqueous stream of different composition. The organic stream is immiscible in both aqueous streams. It removes the ionic or inorganic material from the feed stream and then passes it on to the second aqueous stream. The processing equipment is similar to that of liquid–liquid extraction (see Chapter 12). The ion-rich stream is usually treated further for recovery. The purified aqueous stream usually contains a dilute concentration of organic solvent and may therefore require further treatment.

Capital investment is dependent upon the type of feed stream being treated. As one might suppose, high-volume applications improve the economic aspects of this process.

RESIN ADSORPTION

Resin adsorption is used in the removal of organic solutes from aqueous streams. Solute concentrations may be as high as 8%. It is the preferred method when recovery of the adsorbate is desired since thermal regeneration of carbon destroys the organic material. It is also useful when there is a high concentration of dissolved inorganic salts in the stream.

Synthetic resins may be used to remove hydrophobic or hydrophylic solutes, which may be recovered by chemical means. Resin adsorption is similar to carbon adsorption in that two filter beds are often used—one bed is used for adsorption while the other is being regenerated (see also Chapter 11). The stream flows downward in the system at a rate of 1–10 gal/min · ft² of cross section, and adsorption stops when the bed becomes saturated or the effluent concentration reaches a certain level. Their applications include phenol recovery, fat removal, and color removal (or even change).

Energy costs for this system depend on whether the resin is regenerated or not. If the resin is not regenerated, it must be disposed. Because the high cost of resins results in a high capital cost, regeneration is usually desirable to keep overall costs low.

EVAPORATION

Evaporation is a common process used in both water desalination and the treatment of other feed streams. It may be used to treat a variety of these feeds: liquids, slurries, sludges, organic and inorganic streams, streams containing suspended or dissolved solids, and streams containing nonvolatile dissolved liquids. In other facilities, it has been used in processing radioactive wastes, discharges from paper mills and molasses distilleries, and side streams from trinitrotoluene (TNT) manufacturing.

The process and equipment used in evaporation are similar to that of distillation (see Chapter 9) except that the vapor is not collected and condensed unless organic components are present. The stream usually flows through metal pipes that are heated by low-pressure steam outside the pipe walls. Other modes of operation that

have been used are the solar evaporation from ponds or the heating of open vessels. The process concentrates the original feed stream and reduces its volume. Evaporation is an energy-intensive process and utility and equipment costs may be high.

FOAM FRACTIONATION

By exploiting the difference between the surface and bulk concentrations of surface-active agents, it is possible to affect a separation by forming an air–water foam. If the operation is carried out in a column, it is possible to fractionate the foam phase from the liquid phase continuously. The foam is then collected and collapsed.

Application of this operation to the effluents from industrial plants enables the separation of the detergents present in a discharge. Recycling of the foam fraction to the activated sludge tanks permits further degradation of the detergents. Other refractory contaminants that concentrate in the foam are also removed.⁽²⁾

Extension of foam fractionation to the concentration of other ionic materials requires only the selection of a surfactant that will sequester the particular material. Recovery of the material from the foam fraction usually requires further treatment.

DISSOCIATION EXTRACTION

In solvent extraction, separation of a liquid solution depends on the relative solubility of a component in another liquid that is immiscible with the solvent of the original solution. The separation of such liquid solutions may be accomplished in single-stage batch equipment or, as is more usual, in continuous multi-stage equipment. Separation of the extracted material usually requires removal of the solvent.

The separation of mixtures of organic acids or bases can also be accomplished by distributing them between an aqueous solvent and an organic nonpolar solvent (see Chapter 14 for additional details). This technique has been used for the separation of mixtures of closely related organic acids. Occasionally, acids will not distribute themselves into the two phases; however, if their dissociation constants are different, it is possible to separate them.

The extraction system necessary to accomplish this separation contains the organic acids, a nonpolar organic solvent, and aqueous caustic soda in an amount less than the equivalent of total acid.

If a separation of organic bases is required, a similar system may be used, but an aqueous mineral acid replaces the caustic soda solution. Here, the base with the higher ionization constant is selectively concentrated in the aqueous phase.⁽²⁾

ELECTROPHORESIS

Colloidal systems dispersed in buffered solutions have electrical charges surrounding the particles. These charges consist of a double layer that, in turn, consists of an inner

layer due to the actual charge on the colloid, and an outer one due to the ionic charge from the solution.

If an electrical potential is applied to such a system in a cell, the colloidal particles will migrate toward the electrodes according to their charge. This process is termed electrophoresis. In the electrophoresis of proteins, for example, movement is toward the anode in basic solutions and toward the cathode in acidic solutions. Different proteins show different mobilities and, hence, may be separated into pure components.

Since the electrophoresis of colloid solutions produces density gradients in the electrode cell, spontaneous remixing of the components and the buffered solution may occur because of convection currents. To eliminate these currents, various porous media such as filter paper and starch are used to stabilize the separated components.⁽²⁾

VIBRATING SCREENS

As the name implies, vibrating screens are screens that vibrate. The screen allows for separation of large-sized solid particles from gaseous (usually) or liquid (rarely) streams. The vibration increases the rate of the separation and helps reduce clogging and plugging. Obviously, the key design consideration for this separation process is the size opening of the screen.

Equation (17.1) may be employed to calculate the percentage of open area, S ,

$$S = (1 - ND)(1 - nd) \quad (17.1)$$

where N is number of wires per inch in one direction, n is number of wires per inch, D is the diameter of wires in the perpendicular direction (inches), and d is the diameter of those wires (inches).

To calculate mesh, determine the space width and wire diameter (according to product requirements), then add them together and take the reciprocal. For example, if the space width is 0.048 inch and the wire diameter is 0.035 inch, the mesh is $1/0.083$, or 12.

ILLUSTRATIVE EXAMPLE 17.1

Qualitatively describe design considerations for a ion-exchange system.

SOLUTION: Although ion-exchange systems can be designed with the aid of only a few laboratory tests, a pilot plant operation of substantial duration is generally recommended. Ion exchangers are subject to fouling and loss of capacity in operation, which cannot be detected readily by laboratory tests. Therefore, it is desirable to operate a pilot plant throughout a number of cycles using the commercial plant regeneration procedures and, if at all possible, the actual stream to be treated in the commercial installation. ■

REFERENCES

1. S. DANATOS, "Unusual separations," *Chem. Eng.*, New York City, NY, December 7, 1964.
2. Author unknown, *Chem. Eng.*, **38**, New York City, NY, Sept. 30, 1963.

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