Separation Science and Technology

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Boyadzhiev, L.(1990) 'Liquid Pertraction or Liquid Membranes—State of the Art', Separation Science and Technology, 25: 3, 187 — 205
To link to this Article: DOI: 10.1080/01496399008050329
URL: http://dx.doi.org/10.1080/01496399008050329

SEPARATION SCIENCE AND TECHNOLOGY, 25(3), pp. 187-205, 1990

REVIEW

Liquid Pertraction or Liquid Membranes—State of the Art

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INTRODUCTION

Liquid pertraction* or liquid membranes appeared as a prospective separation process relatively recently. Due to its obvious advantages over solid membranes and liquid-liquid extraction, liquid pertraction has attracted the attention of many scientists and engineers. At present there are more than 160 research teams around the world exploring this new, emerging separation operation.

The principal idea inherent in liquid pertraction is quite simple—two completely miscible liquids, considered further in this paper as two aqueous solutions, the feed F and the stripping liquor R, are separated, as shown in Fig. 1, by a third, immiscible liquid, the membrane phase S, usually an organic solvent. The specified component or components are extracted from the feed F, due to the favorable thermodynamic conditions set around the first interface F/S, and simultaneously stripped by the stripping phase R because of the new equilibrium conditions at the second interface S/R.

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*The term "pertraction" (from the latin "per-traho") was introduced by Schlosser and Kossaczky in 1975 (*1*). The term "liquid pertraction" is an apt analog of the closest unit operation name "Liquid-liquid extraction" and reflects its multi (three) phase transport mechanism.

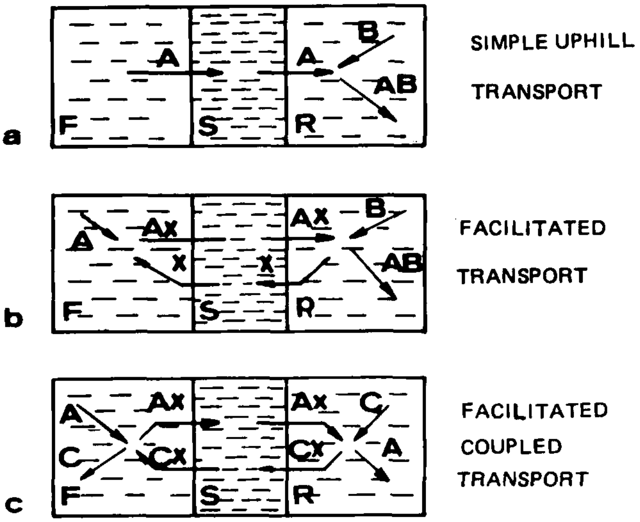

FIG. 1. Pertraction transfer mechanisms of practical interest.

Liquid pertraction is a combination in time and space of two well-known separation operations—extraction and solvent stripping (*2*)—which offers some attractive advantages over classical liquid-liquid extraction. First, liquid pertraction provides a maximum driving force for the transportation of extracted solutes and, therefore, multistage or countercurrent flow diagrams are in principal useless. Second, the organic phase S is merely a short-time mediator and hence its capacity is not a very important characteristic. For that reason an insoluble, harmless, and inert liquid, containing a small amount of very selective, even solid, slightly soluble carriers, can be used as the membrane phase. This special feature reveals new horizons for the development of new, very selective solid "extractants."

TRANSFER MECHANISMS

In general, liquid pertraction is a concentration-driven separation process. Application of a temperature or pressure difference or an electric field considerably diversifies the fields of interest, but these special separation methods are beyond the scope of this paper.

Among the variety of possible transfer mechanisms, controlling the removal and concentration of the transferred component, as shown in Fig. 1, are of practical interest. In the first case (Fig. 1a), known as "simple uphill transport," the solute A is pumped against the apparent concentration gradient because of the irreversible chemical reaction A + B = AB taking place in the stripping solution R. The product AB or the ions existing after its dissociation are insoluble in the membrane liquid S, and therefore there is no backtransport of A across the membrane, but there is an accumulation in the acceptor liquid R. Weak acids or bases such as phenols, amines, or antibiotics are selectively extracted from their dilute solutions and concentrated by this mechanism.

The second mechanism, shown in Fig. 1(b), is called "facilitated transport." In this case a specified carrier X, dissolved in the membrane liquid, plays the role of a shuttle, complexing the solute A around the first interface and delivering it to the stripping phase.

The third mechanism, shown as Fig. 1(c), represents an ion-exchange process. It is sometimes known as "coupled transport." The transfer of the ions of component A from the feed F into the stripping solution R is counterbalanced by an equivalent transport of similar ions C from the R-phase into the F-phase. These are cases for the removal of various metals like copper and zinc from their dilute neutral or slightly acidic solutions by using suitable chelating agents as carriers. The metal ions are replaced in the feed by hydrogen ions from the stripping mineral acid.

PERTRACTION TECHNIQUES

The idea of creating an efficient and commercially acceptable separation method based on this three-liquid-phase combination is not new. The reason that practical achievements are so poor today is due to the lack of a suitable technique to provide a stable system of three liquids with an intensive mass flux across the two opposing large interfaces. However, in the last 15 years several prospective ideas to solve this problem have been proposed, and some of them are undergoing intensive pilot-plant and industrial tests. Three of these techniques will be discussed in this paper, with the accent on the method we developed—liquid film pertraction.

At the end of the 1960s and the beginning of 1970s, N. Li proposed the so-called double emulsion method (DEM) or surfactant liquid membranes (*3*). This technique is currently being studied in many laboratories around the world. The triple liquid system is stabilized in this method by means of emulsifiers which are added to the membrane liquid up to 5% or

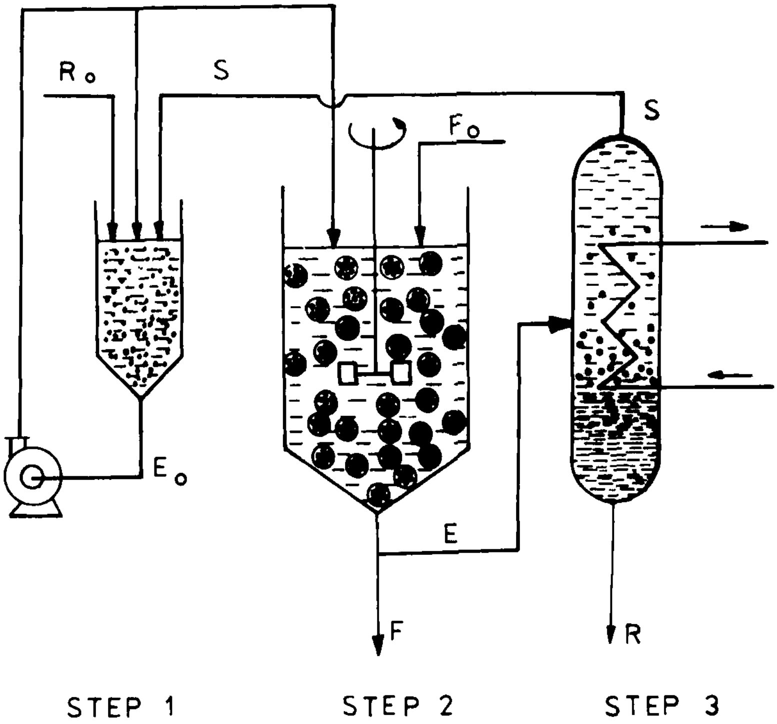

FIG. 2. Flow diagram of the three-step double emulsion method.

more (*4–10*). The double emulsion technique is a three-step procedure. During the first step the stripping solution R is finely dispersed and emulsified into the membrane phase S. After encapulation of the stripping liquid R in the membrane liquid S, the emulsion is dispersed in turn in the feed solution F, as shown in Fig. 2. Due to the large interfacial area between the feed and the solvent (F/S) and to the extremely large interface between the solvent and the strip solution (S/R), solute A is generally transferred into the encapsulated receiving liquid in a few minutes. When this second step is completed, the emulsion is separated from the exhausted raffinate and destroyed. Finally, after this third, emulsion break-up step, a concentrated aqueous product solution R is obtained and the membrane liquid is reused.

Two typical extraction curves are shown in Fig. 3. The first curve represents the change of phenol content in the feed, a phenolic water, and the

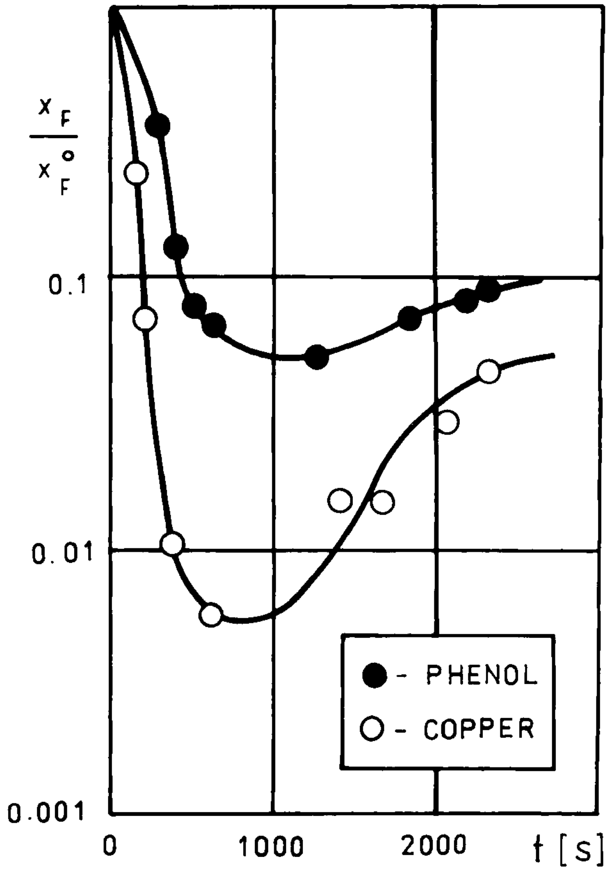

FIG. 3. Solute content vs pertraction time, applying the DE technique. System formulations: (A) Phenol content in the feed, 0.1 g/L; membrane phase, 95% *n*-paraffins + 5% SPAN 80; stripping phase, 0.5% sodium hydroxide. (B) Copper content in the feed, 0.25 g/L; membrane phase, 2% ACORGA P-5100 + 5% SPAN 80 + 93% *n*-paraffins; stripping solution, 3 *N* sulfuric acid.

second one represents the removal of copper from copper-containing wastewater. The corresponding experimental conditions are also given. In both cases, pertraction efficiency shows a maximum due to the superposition of two effects: removal of phenol or copper, respectively, from the feed, controlled by the diffusional processes, and their backtransport due to destruction of the emulsion which contains the concentrated aqueous solutions of the accumulated solutes. The emulsion stability can be improved by modifying the liquid membrane formulation and the emulsion preparation technique, but this causes difficulties in the following emulsion break-up step.

Obviously, this attractive method has several serious drawbacks. In prinicpal it is a batch process, although some modifications for continuous operation have been successfully tested (*11–16*). The emulsion stability depends on the type and the amount of emulsifier used. During the second extraction step there is some emulsion break-up, which deteriorates the process efficiency and selectivity. The emulsifiers added can pollute the outgoing raffinate solutions. Side phenomena like feed or water occlusion, secondary feed emulsification, etc. can reduce the process efficiency to unacceptable levels. In spite of these shortcomings, the double emulsion method is suitable in a number of cases, and it achieved industrial recognition in the People's Republic of China (*9*) and Austria (*17*).

The second technique, known as supported liquid membranes (SLM), has had noticeable success recently. In this technique the open pores of very thin porous polymer sheets or hollow fibers are filled with the membrane liquid S, as shown in Fig. 4. The oleophylic properties of the polymer keep the organic liquid in the pores and prevent both aqueous solutions from direct contact. In order to increase the membrane area,

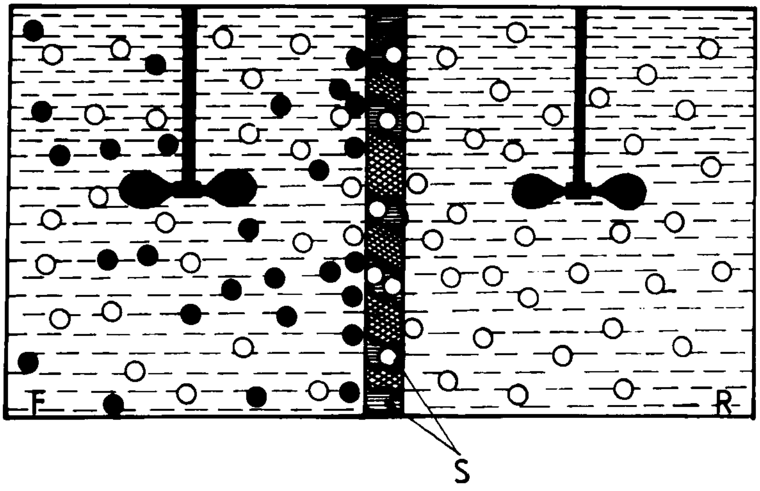

FIG. 4. In the supported liquid membrane technique the open pores of thin porous polymer sheets or hollow fibers are filled with the membrane liquid S.

modules containing large numbers of hollow fibers or long spiral-wound flat sheets are prepared. This method, as confirmed by the increasing number of published studies, practically avoids all the drawbacks of the previous method (*18–26*). However, in spite of the tiny sizes of the hollow fibers or the very thin porous sheets used, the total mass flux across the membrane is quite low. As seen in Fig. 5, the changes of copper concentrations in the feed and stripping solutions take much more time than in the equivalent DE process. Further attempts to reduce the membrane thickness increase the risks of mechanical rupture. Furthermore, the organic liquid filling the pores dissolves over time in both aqueous flows. This means that the membrane must undergo some kind of regenerative treatment. In spite of continuing efforts, membrane lifetime remains the main problem to be solved before there can be industrial application of this pertraction method. Nevertheless, a large number of research laboratories and even commercial companies in the United States, Japan, USSR, and other countries are developing full-scale industrial separation processes based on supported liquid membranes.

An essential common feature of both the described methods is that at least one of the three liquids be stagnant. According to mass transfer

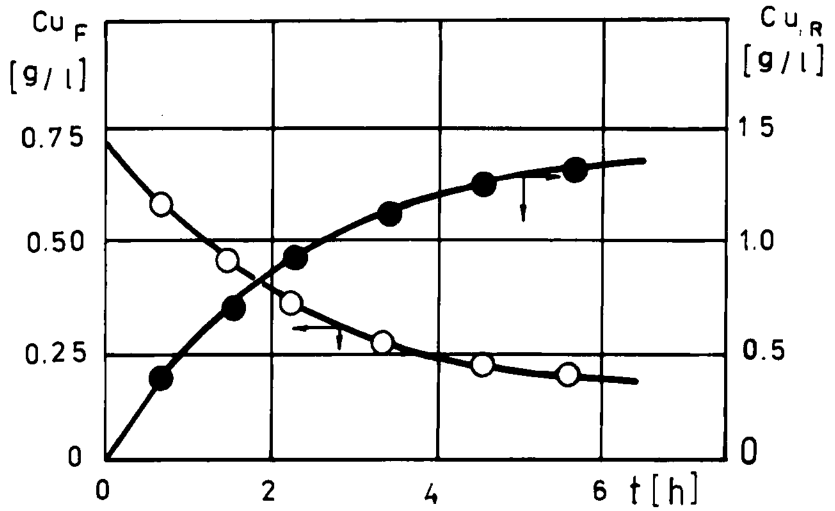

FIG. 5. Evolution of copper concentration in the feed and strip solutions vs time. Supports: 20 μm thick Nuclepore polymer sheet with 15% porosity and 0.5 μm mean pore diameter. Membrane phase: 2% ACORGA P-5100 in *n*-paraffins.

theory, the slowest transfer step in any multistage diffusional process controls the overall transfer rate, so molecular diffusion in the stagnant liquid is an obstacle to mass flux intensification.

In contrast to SLM and DE methods, all three liquids in liquid film pertraction (LFP) (*27*) are in motion, as shown in Fig. 6. The feed and the stripping solutions flow down the vertical solid porous supports, which are arranged in an alternating sequence and spaced at small distances. The latter, as well as the whole pertractor volume, are filled by the intermediate organic phase S, which circulates in a cocurrent or countercurrent mode, forced by an external pump or some other motion generator. Since all three liquids are in motion, the specie transfer is controlled by eddy diffusion, increasing all mass fluxes considerably. Although the real thickness of the vertical liquid "membranes" is in the range of several

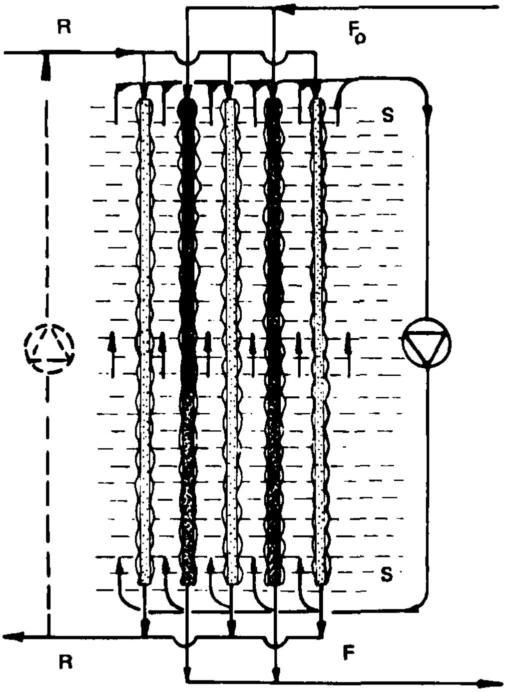

FIG. 6. All liquids in liquid film pertraction are in motion. The feed (F) and the stripping solution (R) flow down the vertical hydrophylic supports, arranged in an alternating sequence.

millimeters, they are (with respect to their mass transfer resistance) equivalent to completely stagnant liquid films only a few microns thick. Typical velocity profiles of the flowing films for feed and strip solutions—down to the porous hydrophylic supports—and for the membrane liquid—upstream between the falling aqueous films—are shown in Fig. 7, together with the tracer response curve registered at the feed outlet, which demostrates the very low level of longitudinal mixing.

Experiments on liquid film pertraction carried out by using a laboratory-type LF-pertractor with a maximum capacity of 100 L/day (with respect to the feed) are presented in Figs. 8 and 9. The apparatus contains seven flat vertical hydrophylic supports, three of them for the feed solution, which expose a 0.12-m^2 active F/S interface. Each support is 4 mm thick, and the distance between them is 3 mm. Figure 8 shows the effect of two process parameters: Mean residence time of the feed in the active pertractor portion, and linear upward velocity of the membrane liquid on zinc removal from zinc-containing wastewater. Normal paraffins (C_{11}-

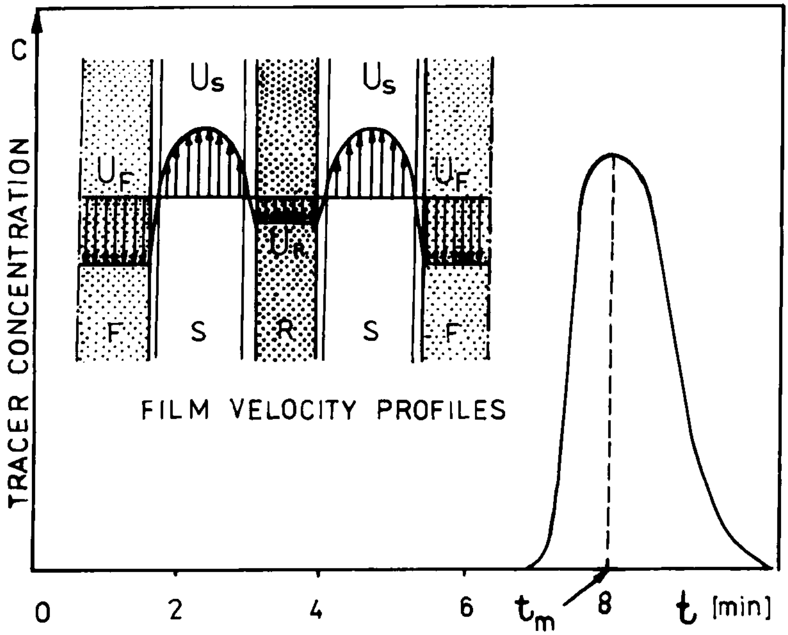

FIG. 7. Velocity profiles of the feed (F), membrane phase (S), and stripping solution (R) in a liquid film pertractor and a typical tracer response curve at the feed outlet.

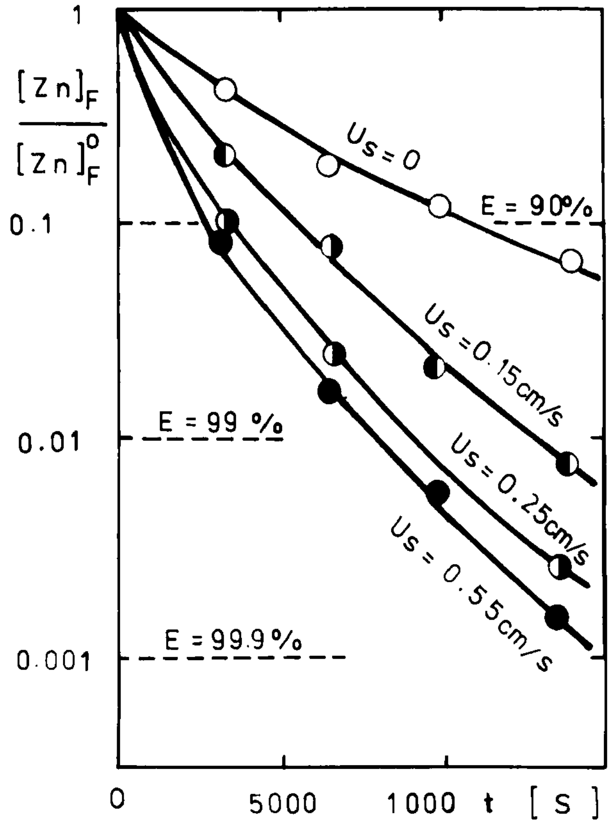

FIG. 8. Dimensionless zinc concentration at the feed outlet vs mean residence time of the feed in the apparatus, varying the linear velocity of membrane liquid (2% D2EHPA in *n*-paraffins).

C_{13}) containing 2 vol% only of di-2-ethylhexylphosphoric acid are used as the membrane phase. The selected flow rate ratio Q(F)/Q(R) = 120 provided up to 25 g/L zinc in the final R product. According to the data presented in the figure, pertraction efficiency, when throughput is the lowest, reaches 99.8% in the case of a flowing membrane liquid and 92% in the case of an inactivated pump. It should be mentioned, however, that according to calculations made by applying Fick's law, the process efficiency must be under 20% for a completely stagnant organic membrane. The adjacent creeping aqueous films obviously disturb the motionless interposed membrane and induce cocurrent displacements to it.

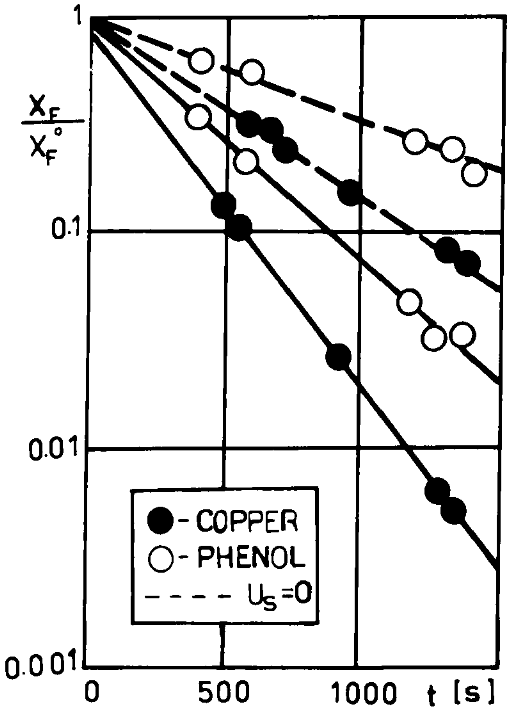

FIG. 9. Removal of phenol and copper by the LP technique. Solute concentrations at the feed outlet vs mean feed residence time for flowing and "stagnant" membrane liquids (*n*-paraffins for phenol removal and 1% ACORGA P-5100 in *n*-paraffins in the case of copper pertraction).

Analogous conclusions can be drawn from data obtained for phenol and copper removal, shown in Fig. 9. Phenol was extracted from phenolic water by using the aforementioned mixture of normal paraffins as the membrane phase and sodium hydroxide solution as the stripping agent (*28*). The two curves correspond to a stagnant and to a flowing intermediate phase. In the case of copper pertraction, the initial feed containing 0.25 g/L copper is stripped down to 1 ppm. This means 99.6% removal efficiency. A 1% solution of a commercial aldoxime ACORGA P-5100 in the same paraffinic oil is the formulation of the organic membrane, and the latter is stripped by a 15% solution of sulfuric acid. When the membrane phase is pseudostagnant, the maximum efficiency reached is 88%. A 200-fold selective accumulation of copper in the strip solution R was achieved. Other metals such as iron, magnesium, calcium, cobalt, and nickel are not cotransported and remain in the raffinate (*29*). These examples show the exceptional possibility for direct treatment of leachates or mine waters containing copper, producing conditioned cathode solutions for copper electrowinning. A general flow sheet of this suggested hydrometallurgical process is shown in Fig. 10.

Figure 11 represents the kinetics of iodine and bromine recovery from brines after their consecutive oxidation with nitrite and chlorine. The membrane liquid is again a paraffinic oil and the stripping liquor is a 40 g/L solution of sodium sulfite. These data confirm once more the good performance of the LF-pertractor tested as well as the strong effect of the feed flow rate. Figure 12 shows the results of the two stability tests carried out for coppepr extraction. In both tests all experimental conditions except membrane hydrodynamics were identical. The efficiencies measured during these continuous, round-the-clock tests for more than 2000 h were identical. The advantages and shortcomings of liquid film pertraction will be discussed, in parallel with the features of the other pertraction techniques, after the following brief survey on the mathematical modeling of pertraction methods.

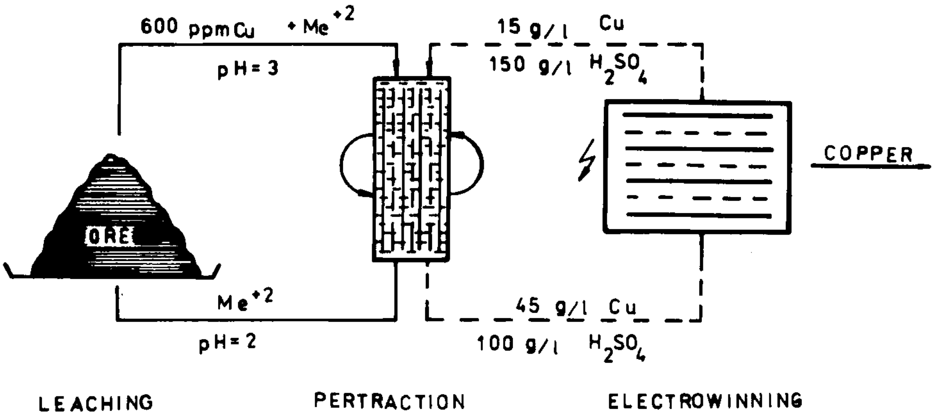

FIG. 10. General flow sheet for cathode copper production. A selective copper enrichment is possible by applying a LFP treatment of the leachates.

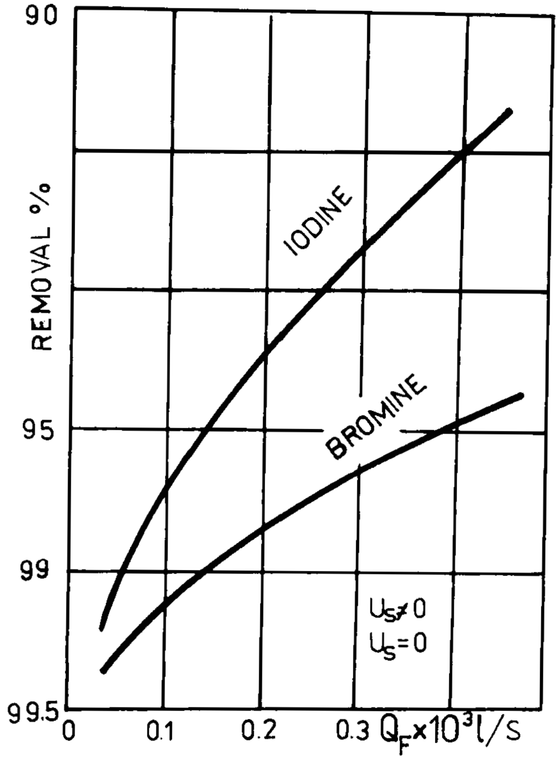

FIG. 11. Removal efficiency vs feed flow rate in the case of consecutive and selective recovery of iodine and bromine from brine by liquid film pertraction. Membrane liquid: *n*-paraffins.

PERTRACTION MODELING

Nowadays, in the era of computerization, a common feature in many research fields is the incredible flood of mathematical models which compensate to some extent for the shortage of good ideas, accurate experimental measurements, and logical conclusions. Liquid pertraction is no exception. Nearly 120 mathematical models or their improved modifications have been published in the last 15 years. Some of the more important, pertinent models will be briefly mentioned, but the sophisticated equations typical of the modern deterministic models will be avoided.

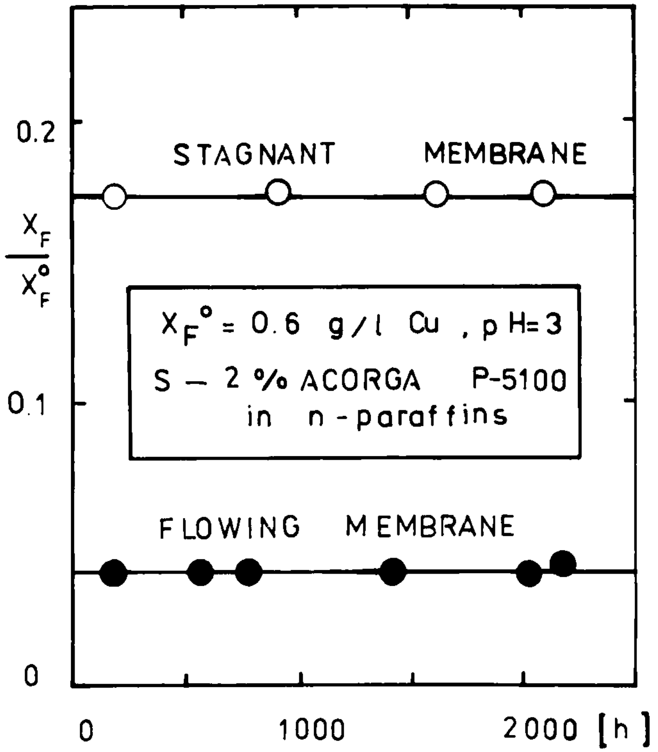

FIG. 12. Pertraction efficiency vs time in prolonged round-the-clock stability test.

There are two principal approaches to modeling liquid pertraction processes (*30*). According to the membrane or differential approach, commonly used in the modeling of separations by polymer membranes, all phenomena taking place in the feed or in the stripping phase, such as diffusion, chemical reactions, etc., are totally ignored. The measured transfer fluxes are supposed to depend on phenomena occurring in the bulk or at the surface of the membrane only. The other, integral approach (*30–32*) considers the three liquid-phase pertraction system to be a closed multiphase system and therefore takes into account the processes and changes in all three liquids.

The flat geometry of supported liquid membranes considerably simplifies the pertraction models. Most frequently, the emphasis is put on reaction mechanisms, since the diffusion, hampered or facilitated, is always assumed to follow Fick's law (*19, 33*). But second-order phenomena are also taken into account in the models based on the integral approach (*34–36*), with the conclusion that the hydrodynamics or reaction rates in the feed usually control the overall separation process (*37, 38*).

Initially, the complicated geometry of double emulsions was oversimplified by representing the organic liquid in the globule as a thin, flat envelope of the stripping phase, assumed to be a large, uniform internal drop. Coefficients of the so-called "effective diffusivity" were calculated, and the thickness of this hypothetical membrane was obtained for steady-state conditions (*39–41*). Later, it was suggested that the stripping phase droplets were dispersed but of equal size and uniformly distributed inside the globule. The transported specie advances gradually, reacting irreversibly with reagent within the droplets. In this way a series of "advancing front" models was initiated and later developed (*44–46*), although elements of this concept had been suggested earlier (*47, 48*). In other advancing front models the location of this front is not fixed and the reaction is assumed to be general in character (*14, 49, 50*).

In the earlier models, only zero-order phenomena were taken into account. Later, in applying the integral approach, secondary, but decisive for process efficiency, phenomena, such as emulsion break-up and equilibrium changes, were incorporated in the models (*30–32, 40, 51*). More recent models are more sophisticated because they assume many possible types of control, nonlinear equilibria, phase intermixing, etc. (*38, 52–54*). It should be noted, however, that in spite of multiple efforts and a high level of model sophistication, solutions based on the multidispersion concept and intraglobular hydrodynamics are still not available.

In models of liquid film pertraction, it was initially assumed that the overall rate of mass transfer can be controlled by any of the diffusion resistances in the three liquid phases, but the mass transfer coefficients were subject to identification procedures (*28*). Later versions of this model evaluated these coefficients by the theory of falling liquid films (*55*). The cases of circulating membrane and chemical reaction control still remain unsolved.

DISCUSSION

By comparing the three described pertraction techniques, it is concluded that they all have their specific advantages and drawbacks. The double emulsion method provides higher transfer rates than the other two techniques, but the negative side phenomena limit its efficiency and selectivity in component separation. Supported liquid membranes and double emulsions can treat solutions containing reasonable amounts of fine solids, while liquid film pertraction demands careful prefiltration of both aqueous solutions. SLM and LFP are in principal one-stage, continuous operations, and they guarantee more reproducible and predictable results than the three-stage, double emulsion method. The most important shortcomings of the SLM technique are the unavoidable losses or washouts of the membrane liquid from the pores of the polymer supports. This necessitates periodic regenerative treatment of the membrane supports or the introduction of special devices for continuous compensation of the lost membrane liquid (*56*). When tiny hollow fibers and long modules are used, pressure losses become very high and the membrane liquid can be pushed out of the pores, followed by intermixing of feed and stripping solutions. In spite of a very large membrane area, overall mass transfer fluxes remain poor because of high tortuosity factors and membrane stagnancy. To achieve useful results and good separation performance, a series of modules of supported liquid membranes must be used.

An attractive feature of the LFP technique is its continuous, round-the-clock operation mode, with no maintenance needed for very long time periods. Large laboratory units have been operated for more than 3000 hours with no sign of efficiency loss or process deterioration. Pertractor design, as in the case of the SLM technique, is simple and without scale-up problems; laboratory and industrial equipment differ only in the number of supports used and their width. The concentrating power is practically unlimited: concentrations of the transferred species in the product solutions up to several hundred times higher than their initial content in the feed can be obtained by simply adjusting the ratio between the feed and strip solution flow rates. Very high concentration enhancement (needed, for example, in the analytical preconcentration practice) is usually obtained by closed loop circulation of the stripping liquid. In this way, extremely dilulte solutions containing less than 1 ppm of any valuable or toxic species can be treated successfully.

The introduction of scrubbing operations in solvent extraction often drastically improves separation selectivity and product quality. LFP is the only pertraction technique among the three considered that allows partial scrubbing of the membrane liquid.

The price of materials used in pertraction techniques can play an important role in which one is chosen. Decreasing prices of porous supports and hollow fibers could be an important future advantage for the LFP and SLM techniques.

CONCLUSIONS

Economic estimations show that liquid pertraction operations are among the cheapest separation techniques because of their simple inventory and low running costs. Energy consumption is very low, and organic liquid losses are often negligible if the membrane formulation is properly chosen.

Potential fields of application for liquid pertraction range from hydrometallurgy to medicine. Biotechnology is a special area of interest because liquid pertraction could be a suitable downstrean operation or an efficient reactor with fixed enzymes. Liquid pertraction is an ideal method for sample preconcentration in analytical chemistry. Redox chemical reactions with simultaneous product separation, with or without an applied electric field, is another interesting possibility.

The increasing interest in liquid pertraction is obvious today. The number of research teams enlisted in the "pertraction club" is increasing, and, as mentioned before, has reached 160.

Today's liquid membranes are more selective and perform better than solid membranes. However, more experimental and economic proof are needed to determine the right place for this challenging new unit operation.

Acknowledgment

This project has been supported financially by the Ministry of Culture, Science and Education under Research Contract 0155.

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Received by editor March 24, 1989