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Facilitated Transport of Zinc Chloride through Hollow Fiber Supported Liquid Membrane. Part 3. Module Operation

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Abstract

The third paper of this series of three papers on the facilitated transport of zinc chloride through a hollow fiber supported liquid membrane of tri-*n*-octylamine diluted in *n*-dodecane with 2-ethylhexyl alcohol deals with module operation. The Type A module, in which 300 polyethylene fibers were simply bundled, revealed that only a fraction of the total surface was effective. The Type B module, with a sheet of glass fiber cloth as a spacer and a cross-flow arrangement of the outside strip solution, showed the same performance as with single fiber operation. The continuous impregnation discussed for a single fiber operation in the second paper was demonstrated to be effective by a Type C module.

INTRODUCTION

For a hollow fiber supported liquid membrane (HFSLM) operation to be utilized as an industrial operation, it is vital to use a module with a large number of fibers in order to obtain a practical transport area per unit equipment volume. It has been reported that the effective surface area in a module with simply bundled fibers is much less than the whole surface area of the fibers used (*1*). This is probably caused by the bridging of neighboring fibers by the organic phase exuded to the outside and the

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increase in the film resistance by an insufficient flow of the outside solution at the outer surface of the fibers. A number of considerations to avoid this problem have been reported, such as the use of a spacer between fibers (2), a weaving arrangement of the fibers (3), and a cross-flow arrangement of the outside solution (3).

In this final paper of a series of three papers, the effects of these considerations on module operation are examined for the facilitated transport of zinc chloride through HFSLM of tri-*n*-octylamine diluted in *n*-dodecane with 2-ethylhexyl alcohol. Module operation with continuous impregnation is also examined.

EXPERIMENTAL

The system examined is the same as previously (4, 5). The liquid membrane phase impregnated into the micropores of polyethylene hollow fibers (EHF-270T, supplied by Mitsubishi Rayon Co.) is *n*-dodecane with 6 vol% 2-ethylhexyl alcohol (EHA) which contains tri-*n*-octylamine (R_3N) as the carrier of zinc chloride. The feed solution is a 1.0 kmol/m^3 aqueous hydrochloric acid solution with zinc chloride ($\sim 2.5 \times 10^{-4} \text{ kmol/m}^3$) and the strip solution is a 0.1 kmol/m^3 aqueous hydrochloric acid solution.

The experimental set-up is shown in Fig. 1. It is basically the same as that used for a single fiber operation (4). The feed solution flows by the head pressure of a constant head tank (6) through a flowmeter (7) and degassing equipment (8) to the lower end of the module. The degassing equipment is a bundle of polyethylene hollow fibers in which the feed solution runs through the inside and the outside is evacuated. The level in tank (6) is maintained constant by the overflow arrangement from the supply tank (5). The feed solution passes through the inside of fibers and the exit flow is sampled from time to time to measure the flow rate, the concentration of zinc chloride, and the acidity. The strip solution, maintained at 298 K in constant temperature bath (12), is continuously circulated on the outside of the fibers in the module. The organic phase from tank (13) is either impregnated into the micropores of the fiber wall by running it through the inside of the fiber for a short period of time instead of the feed solution or is continuously impregnated by supplying it to the lower compartment (3) together with the vacuum at the upper compartment (4).

The three types of modules shown in Fig. 2, of ~ 300 fibers of 21 cm effective length, were constructed. In the Type A module the fibers were

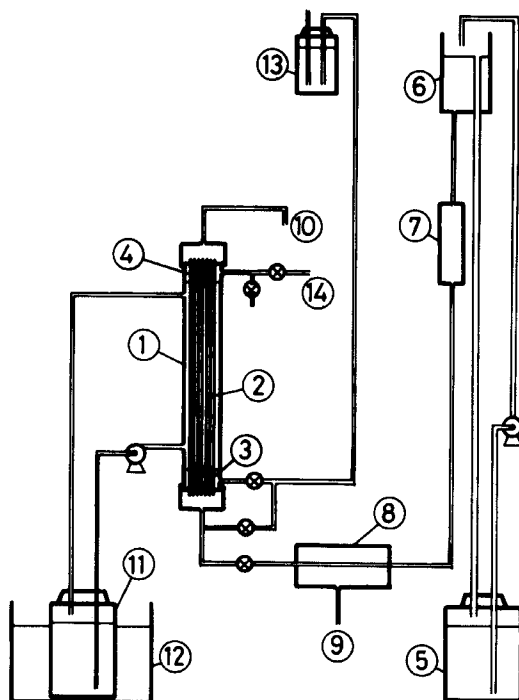


FIG. 1. Experimental set-up for module operation. ① Module. ② Hollow fibers. ③ Compartment for continuous impregnation. ④ Compartment for vacuum. ⑤ Feed supply tank. ⑥ Constant head tank of feed solution. ⑦ Flowmeter. ⑧ Degassing equipment. ⑨ Vacuum. ⑩ Sample of exit raffinate solution. ⑪ Strip solution tank. ⑫ Constant temperature bath. ⑬ Organic phase tank. ⑭ Vacuum.

simply bundled. The strip solution flows on the outside of the fiber bundle in a countercurrent flow configuration. Fibers in the Type B module are separated by bundling them with a sheet of glass fiber cloth (Asahi Fiber Glass Co., MS-100-1030-24NH-00FS). The strip solution flows out of holes of the central tube (40 holes of 0.5 mm diameter) and is made to pass the fiber bundle in a perpendicular manner.

The Type C module has two compartments on both sides to enable continuous feeding of the organic phase to the micropores of the fiber wall. The organic phase is continuously fed to the left compartment by the additional head pressure of the organic phase and the compartment on the right-hand side is evacuated. Other arrangements are similar to the Type B module.

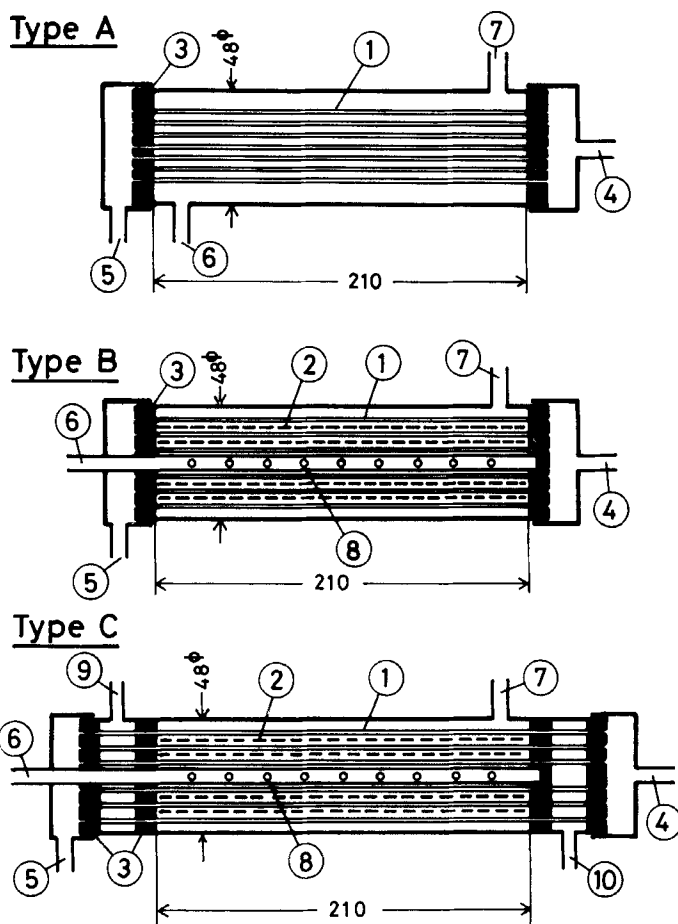


FIG. 2. Module construction. ① Hollow fibers. ② Glass fiber cloth. ③ Adhesive resin. ④ Feed solution inlet. ⑤ Feed solution outlet. ⑥ Strip solution inlet. ⑦ Strip solution outlet. ⑧ Holes. ⑨ Compartment for continuous impregnation of organic phase. ⑩ Compartment for exit flow of organic phase.

The modules, made of methyl methacrylate resin and potting reagents of the epoxy type (LST607 R-1 and H-1) and of the urethane type (LST 607 R-2 and H-2), kindly supplied by Japan Ciba-Geigy, Hyogo, Japan, were used to glue and seal the fibers. The actual modules constructed are shown in Photos 1, 2, and 3 of Fig. 3.

RESULTS AND DISCUSSIONS

Module Arrangement of Types A and B

Modules of Types A and B were compared for the facilitated transport of zinc chloride. Figure 4 shows the initial overall resistances for the two types of modules. Both aqueous phases used were presaturated with the organic phase. The initial overall resistance for the Type A module is approximately five times that for a single fiber operation (broken line), which indicates that only 20% of the whole surface area of the fibers is effective.

The initial overall resistance for the Type B module, however, is only slightly larger than the one for a single fiber operation, which indicates that the use of a spacer and the cross-flow arrangement of the strip solution is effective. After 1 h, the overall resistance starts to increase. This is due to the partial plugging of the fiber bores, because the flow rate of the feed solution started to decrease after 1 h.

Continuous Impregnation of Organic Phase

Figure 5 shows the time course change of the flux of zinc chloride with a Type C module. The aqueous phases without presaturation with the organic phase were used for all experiments using this type of module. The decrease of the feed rate was observed, as shown by the filled squares in the figure. No particles blocking the hollow fibers were observed when the module was disassembled after the experimental run, and this decrease of the flow rate is therefore thought to be caused by either the small gas bubbles which appeared or the existence of the organic phase in the small bores of the fibers. The zinc chloride flux shown in Fig. 5 by open circles was calculated by assuming the number of "alive" fibers using the data on the decrease of the flow rate. The flux level agreed with that of a single fiber (5) and is stable for 5 d. It is

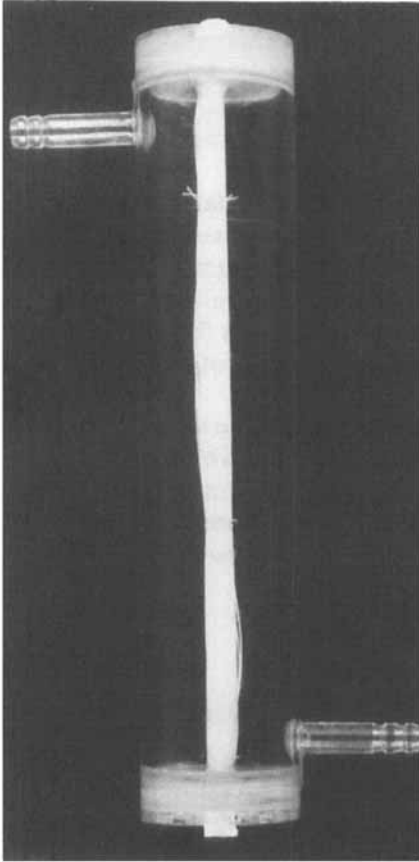


Photo 1

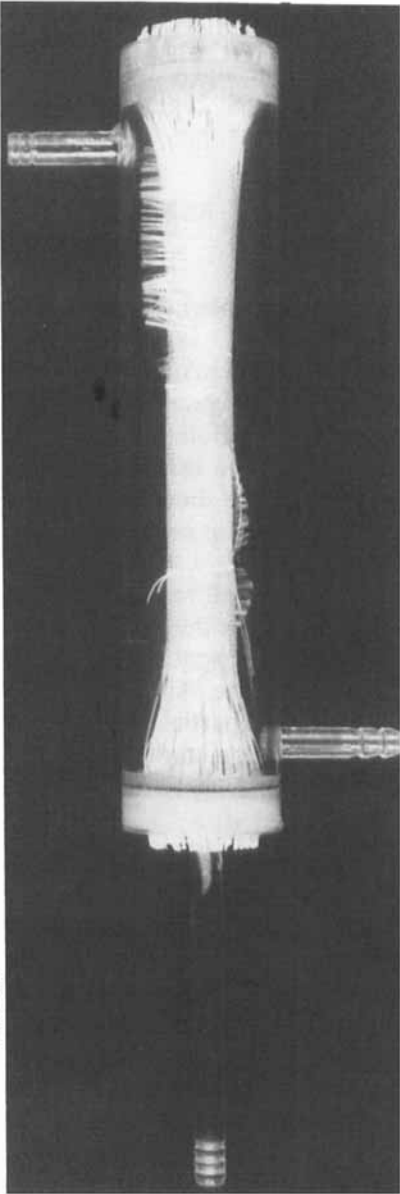


Photo 2

FIG. 3. Photo 1: Type A module of 300 polyethylene fibers. Photo 2: Type B module of 300 polyethylene fibers. Photo 3: Type C module of 300 polyethylene fibers. Photo 4: Type C module of 60 polytetrafluoroethylene fibers.

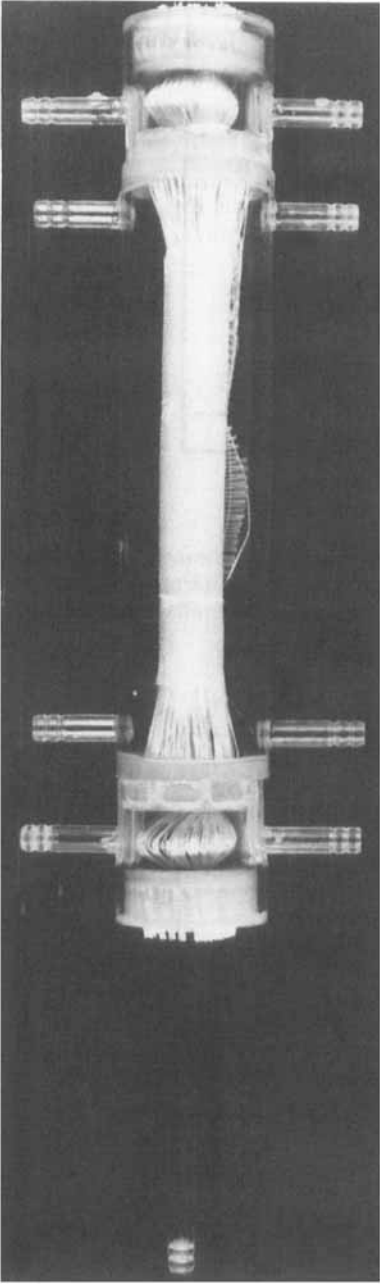


Photo 3

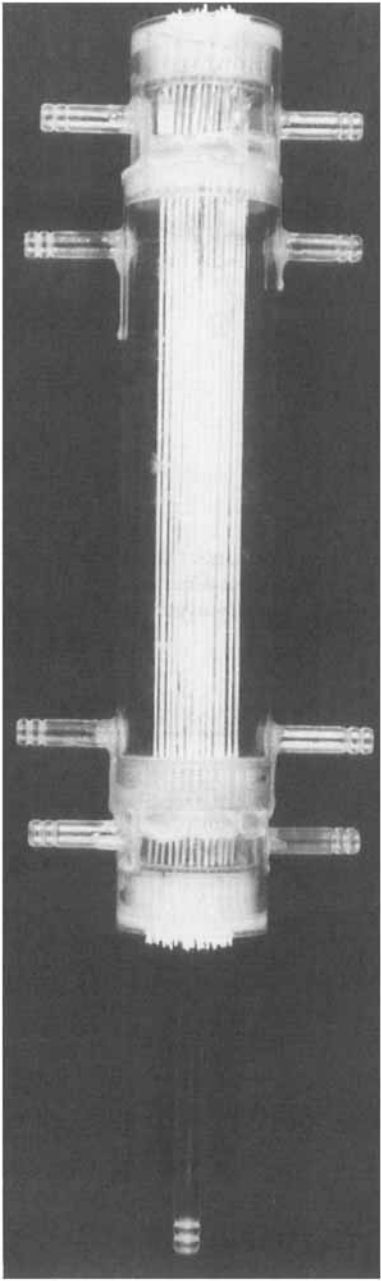


Photo 4

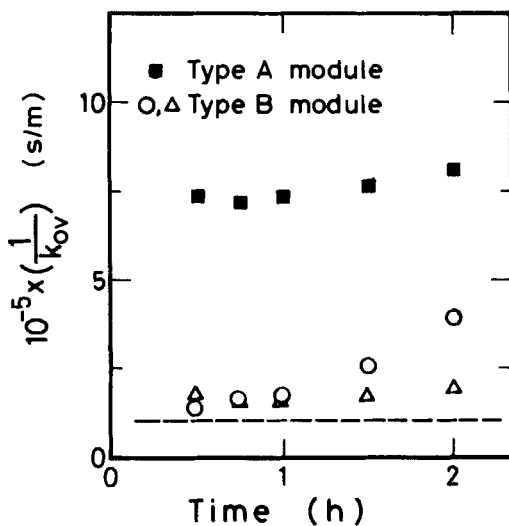


FIG. 4. Overall resistance ($1/k_{ov}$) in the permeation of zinc chloride through HFSLM of *n*-dodecane with $0.1 \text{ kmol/m}^3 \text{ R}_3\text{N}$ and 6 vol% EHA at 298 K. $C_{H,1} = 1.0 \text{ kmol/m}^3$, $C_{H,2} = 0.1 \text{ kmol/m}^3$. Both aqueous phases were presaturated with the organic phase. Broken line: single fiber operation (4).

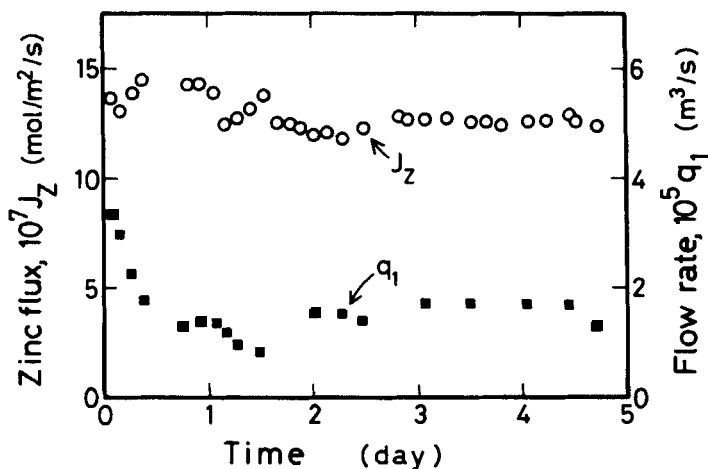


FIG. 5. Zinc chloride flux, J_Z , and the feed solution flow rate, q_1 , through HFSLM of *n*-dodecane with $0.1 \text{ kmol/m}^3 \text{ R}_3\text{N}$ and 6 vol% EHA at 298 K with Type C polyethylene module. $C_{H,1} = 1.0 \text{ kmol/m}^3$, $C_{H,2} = 0.1 \text{ kmol/m}^3$. Aqueous phases were not presaturated with the organic phase.

believed that the module operation with continuous impregnation of the organic phase was successfully demonstrated.

The inside diameter of the polyethylene hollow fibers used in the present work seems to be too small for a practical industrial operation. The inside bore size of the fibers used in recent work reported in the literature is 1–2 mm in diameter (6–11). Because fibers with a larger bore size were not at hand, a module of polytetrafluoroethylene fibers (TB-12, supplied by Sumitomo Electric Ind.), with an inside diameter of 0.8 mm, a wall thickness of 210 μm , an average pore size of 0.7 μm , and a porosity of 0.7, was constructed and examined. The construction of this module of 60 fibers, shown in Photo 4 of Fig. 3, made use of the glass fiber cloth spacer unnecessary.

The result with this module is shown in Fig. 6. The filled triangles represent a one-time impregnation of the organic phase before the experimental run. Because the aqueous phase used was not presaturated with the organic phase, the original high flux dropped rapidly. The open circles are for the case of continuous impregnation of the organic phase. The flux decreased somewhat initially, but stayed constant afterwards,

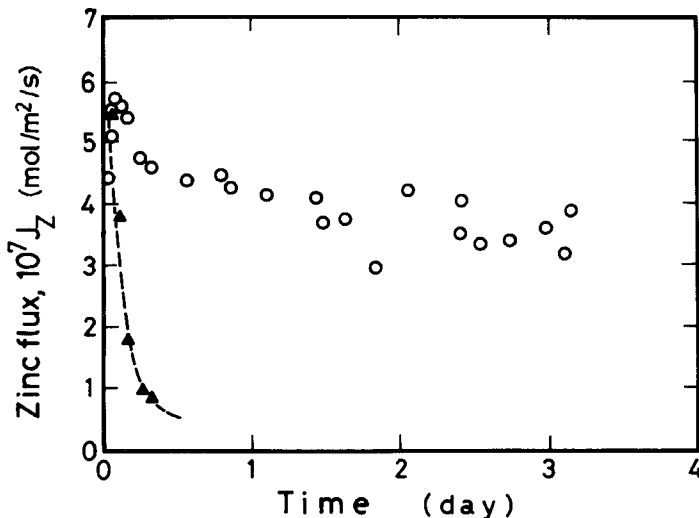


FIG. 6. Zinc chloride flux, J_Z , through HFSLM of *n*-dodecane with 0.1 kmol/m^3 R_3N and 6 vol% EHA at 298 K with Type C polytetrafluoroethylene module. $C_{\text{H},1} = 1.0 \text{ kmol/m}^3$, $C_{\text{H},2} = 0.1 \text{ kmol/m}^3$. Aqueous phases were not presaturated with the organic phase. (\blacktriangle) Impregnation of organic phase once before the experimental run. (\circ) Continuous impregnation.

even without presaturation of the aqueous phases with the organic phase. The overall resistance at this level of flux agrees with the one calculated with a tortuosity factor of 2.0, which is a reasonable value although it has not been verified with a single fiber experiment. The flow rate of the feed solution was constant for the entire experimental run of over 3 d.

CONCLUSIONS

Module operation for the facilitated transport of zinc chloride through HFSLM of tri-*n*-octylamine diluted in *n*-dodecane with 2-ethylhexyl alcohol was examined. With the Type A module, in which 300 polyethylene hollow fibers were simply bundled and the outside strip solution was in a countercurrent flow arrangement, the effective surface area was only ~20%. The use of a glass cloth spacer and a cross-flow arrangement of the outside strip solution in the Type B module succeeded in increasing the effective surface area drastically.

The continuous impregnation of the organic phase, which was proven to be effective earlier in a single fiber operation, was also demonstrated to be effective in Type C modules of polyethylene and of polytetrafluoroethylene fibers. Even without presaturation of the aqueous phases with the organic phase used, the flux was stable for days.

Acknowledgments

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