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Extraction of L-Lysine from Its Dilute Aqueous Solutions by Rotating Film Pertraction

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ABSTRACT

Removal and concentration of L-lysine in a three liquid phase pertraction system was studied using a laboratory three stage rotating disk pertractor. A 5% (vol) solution of Di-2-ethyl hexyl phosphoric acid (D2EHPA) in normal paraffins was used as an intermediate membrane liquid. The amino acid was extracted from its dilute aqueous solution (feed F) and concentrated in the stripping liquid R—a 1*N* solution of HCl in water. D2EHPA dissolved in the membrane liquid played the role of a ion exchange carrier. It was found that the most important parameters controlling the efficiency of this continuous pertraction process are the speed of disk rotation and the feed solution flowrate. The counter-current flow arrangement in the apparatus and the high flowrate ratio chosen provided nearly complete removal of solute from the feed as well as a concentrated solution of L-lysine.

Index Entries: L-lysine; liquid membranes; pertraction; Di-2-ethyl hexyl phosphoric acid.

INTRODUCTION

In the last 30–35 yr, amino acid production based on microbiological processes increased considerably. Among the amino acids produced, L-lysine is the most important one. Because of the product inhibition

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effect upon biomass growth (*1*), lysine concentration in the fermentation broth is kept low, thus decreasing the efficiency of product removal. However, the residual concentration of the amino acid is too low to justify the cost for its complete recovery from the exhausted liquor by solvent extraction or ion exchange methods (*2*). On the other hand, its concentration is high enough to cause significant contamination, creating a favorable environment for the growth of various microorganisms (*3*). Recently, regarding the amino acid recovery and concentration, several authors paid attention to the liquid membrane (liquid pertraction) processes (*4–7*). In addition to the main advantages of the membrane techniques, namely the absence of phase transition and low energy consumption that made them extremely suitable for treatment of thermosensitive substances, the use of liquid as a membrane allows higher selectivity and fluxes to be obtained. Essentially, the liquid membrane separation combines in space and time two separation operations, extraction and stripping (*8*), that provides better recovery and concentration of the amino acids.

The objective of the present paper is to study L-lysine recovery from its dilute aqueous solutions and the feasibility for its concentration by means of a RF (Rotating Film)-pertractor, a liquid membrane apparatus, successfully used previously in other separational studies (*9–10*).

EXPERIMENTAL

Description of the RF-Pertractor

Figure 1 shows a diagram of the laboratory pertractor design used. The apparatus comprises a horizontal cylinder (1) made of organic glass, divided in three sections or mass transfer stages (2). Each stage is divided in two compartments of 200 mm in diameter and 20 mm width (5) intended for the donor phase (F) and the acceptor phase (R), respectively. Disks of 195 mm in diameter and 5 mm thick rotate in each compartment (3). All disks are mounted on a common shaft, driven by electric motor (4). The donor (F) and the acceptor (R) flow from one stage to the next in countercurrent, while the membrane liquid S, being common for both compartments, does not leave the stage. The disks, are immersed up to 1/3 of their diameter in the aqueous solutions F or R, respectively, as shown in the figure. Rotating, their hydrophilic surfaces (2/3 of total disk areas) are covered by mobile, continuously renewed aqueous films, exposed to be in contact with the surrounding, well-agitated organic membrane phase S.

Reagents and Analytical Methods

The initial donor solution contained 10 mol/m^3 (0.15 wt%) L-lysine (pure, >99%, Merck, Darmstadt, Germany) dissolved in distilled water. A 1*N* hydrochloric acid (p.a., TIC-Varna Co., Varna, Bulgaria) solution in

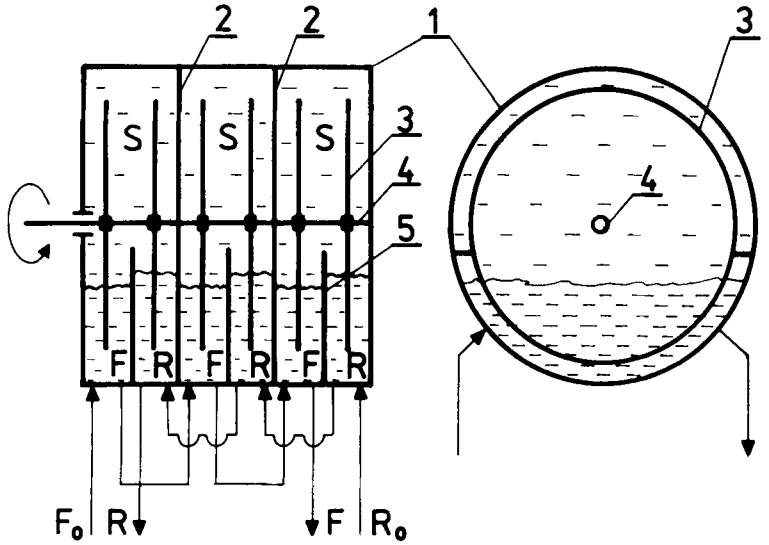

Fig. 1. Scheme of a multistage RF-pertractor. (1) Pertractor body; (2) Stage separators; (3) Rotating discs; (4) Rotating shaft; (5) Separating segments between donor (F) and acceptor (R) phases; (S) membrane liquid.

distilled water was used as acceptor phase. The membrane phase consisted of 220 mol/m^3 di-2-ethyl-hexyl-phosphoric acid (D2EHPA, >70%, Fluka, Buchs, Switzerland) in *n*-paraffines, fractions C_{11}-C_{13} (Neftochim Co., Bourgas, Bulgaria). The exact composition of the paraffinic solvent is given elsewhere (*11*). The carrier concentration was chosen based on previous detailed studies (*7*).

In all runs the temperature was 25°C and the disk rotation speed 20 rpm, corresponding to a disk peripheral velocity of 0.092 m/s. Higher rates of rotation were avoided, because although they lead to improved mass transfer, the risk of aqueous film rupture and drop formation from the disk peripheries became too high.

Lysine concentrations in both aqueous phases were measured by colorimetry, based on the color ninhydrin reaction (*12*) by means of a spectrometer (Specol 11, Carl-Zeiss, Jena, Germany) at wavelength $\lambda = 570$ nm.

RESULTS AND DISCUSSION

As already mentioned, the aim of this study was to assess the feasibility for continuous recovery and concentration of lysine by a RF-pertractor. A detailed description of the mechanism of interaction between L-lysine and the carrier D2EHPA, as well as details on the transfer mechanism that is of cation exchange type, were already given in our earlier publication (*7*). The latter also contains the equilibrium data for the studied system at various pH-values.

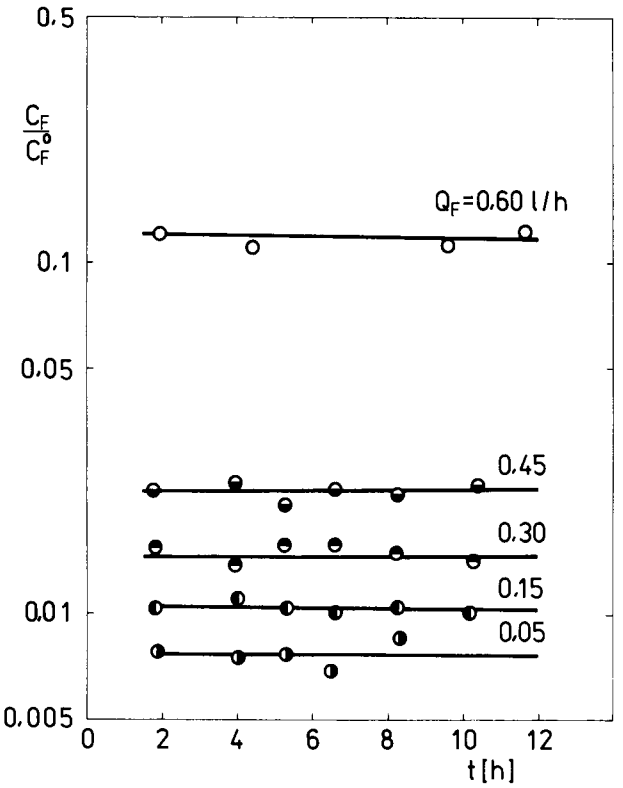

Fig. 2. Recovery vs time at various donor solution flowrates.

In the present study, the ratio between the flowrate of the donor and acceptor solutions was kept constant at $Q_F:Q_R = 10$. Twelve hour runs at various donor liquid flowrates, between 0.05 L/h and 0.6 L/h, were performed. The results are shown in Fig. 2. As seen from the figure, the rate of lysine recovery from the donor solution remained constant and varied within the limits of 86 and 99.3%, depending on the flowrate or the mean residence time of the treated solution, F in the apparatus. The relationship "recovery vs residence time" in solution F, as illustrated in Fig. 3, shows that under the condition chosen, a 90% recovery is attained in about 1 h mean residence time, while it takes more than 5 h to obtain a 99% recovery.

In order to study the effect of lysine concentration in the acceptor solution, R, upon the recovery efficiency, a continuous run was carried out by recirculation of the acceptor solution R in a closed loop system within the appropriate compartments. In this 60 h run, the flow rate of the treated solution F was 0.15 L/h and its initial concentration -10 mol/m^3. Concentrations obtained in both aqueous solutions F and R, are given in Fig. 4. As seen from the figure, a lysine content of 40 mol/m^3 in the acceptor solution did not influence the level of recovery that was 99% in this case. The results obtained show that the solute concentration in

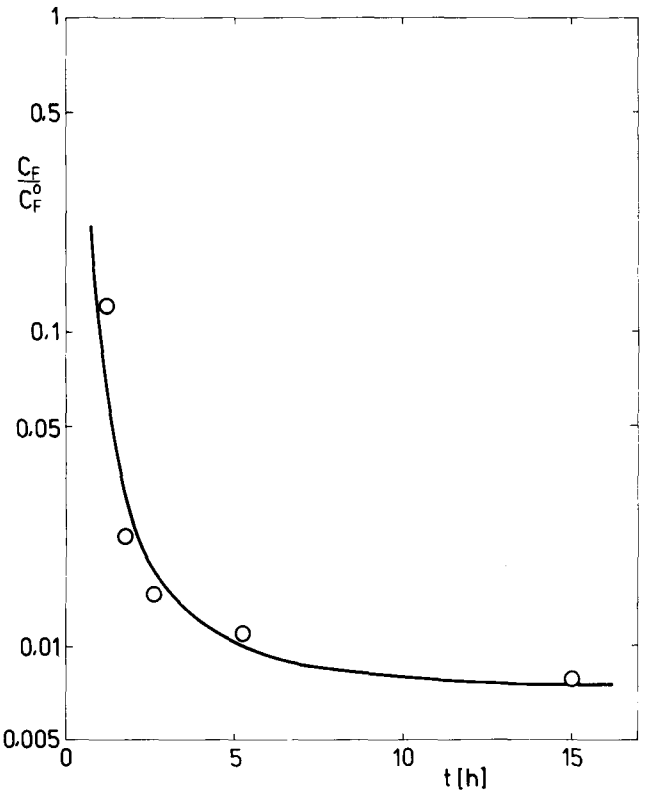

Fig. 3. Recovery vs mean residence time of treating donor liquid.

the acceptor solution can exceed several times its initial concentration in the donor liquid, as illustrated by the same figure. It should be mentioned that in this long-lasting initial moment, increased continuously without reaching its final equilibrium value. This is the reason for the incomplete solute mass balance when aqueous phase flowrates and concentrations are taken into account, only. This result confirms the possibility of recovering lysine from its dilute aqueous solutions with its subsecond concentration.

The aim of this study was to assess initial feasibility of the RF-liquid membrane technique for extraction of L-lysine. Hence, no attempt was made to optimize process parameters. Further detailed studies and economical analysis would be necessary to reduce such an approach to practical use.

CONCLUSION

The results obtained show that the RF-pertraction technique can be used successfully to recover and concentrate L-lysine from its dilute aqueous solutions. The process is continuous and ensures efficient recovery of the amino acid. It was established that the recovery efficiency depends

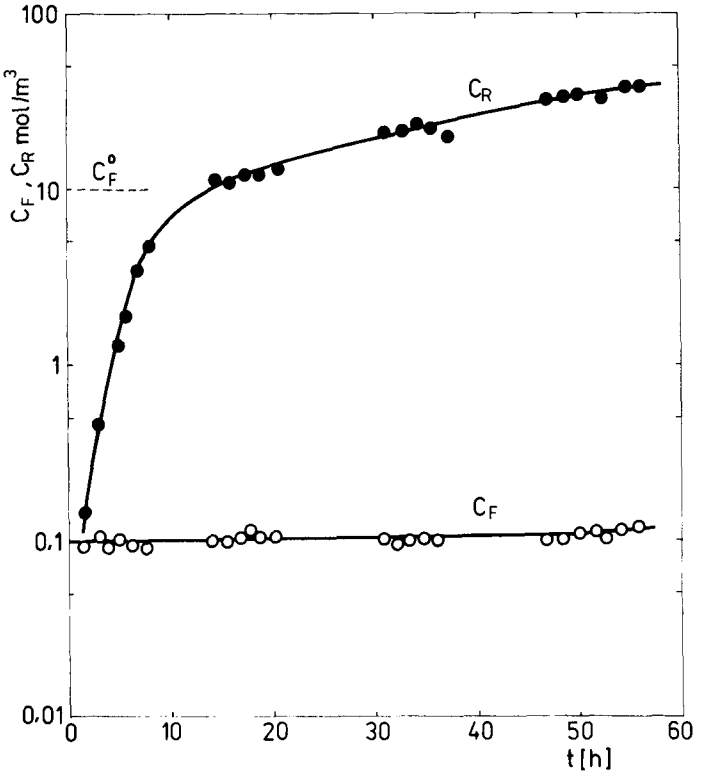

Fig. 4. Influence of L-lysine concentration in the acceptor solution C_R on final amino acid concentration in the donor liquid C_F.

mainly on the mean residence time of the treated donor solution in the apparatus, as defined by the feed flowrate.

The method permits use of a relatively dilute solution of D2EHPA in normal paraffins as membrane liquid. Furthermore, a counter-current operation mode is easily applied for this liquid membrane separation technique.

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