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 Tekić, Miodrag N. , Pekić, Branislav , Vatai, Gyula and Zeković, Zoran(1994) 'Separation of Lanatosides by Membrane-Based Extraction', Separation Science and Technology, 29: 4, 551 — 556
To link to this Article: DOI: 10.1080/01496399408002162
URL: http://dx.doi.org/10.1080/01496399408002162

SEPARATION SCIENCE AND TECHNOLOGY, **29**(4), pp. 551–556, 1994

TECHNICAL NOTE

Separation of Lanatosides by Membrane-Based Extraction

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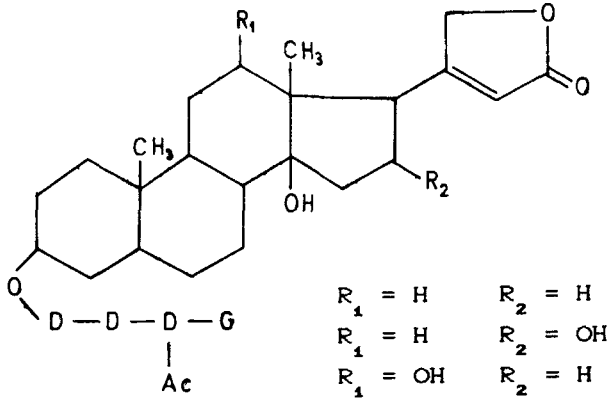
ABSTRACT

Separation of Lanatoside C from a complex mixture of Lanatosides A, B, and C by membrane-based extraction was investigated. The experiments were carried out using a tubular inorganic membrane. The system chloroform–trichloroethylene–methanol–water, characterized by good separation and solubility, was used. The experimental data obtained correlated satisfactorily by using Leveque's equation for the tube-side flow and the Prasad and Sirkar equation for the shell-side flow.

INTRODUCTION

Membrane-aided solvent extraction has been the subject of a number of investigations during the past 10 years (1–3). This dispersion-free solvent extraction technique has several advantages over conventional liquid–liquid extractors. The contactor does not have mechanical moving parts, there is no flooding limitation, and it is possible to handle systems that form emulsions. Also, by using microporous hollow fibers it is possible to provide high mass transfer per unit equipment volume.

Lanatosides are glycosides of complex chemical structure whose crystals are typically isomorphous. A complex mixture of Lanatosides A, B, and C can be extracted from the leaves of *Digitalis lanata Ehrh.* Further, by using liquid–liquid extraction (4), Lanatoside C, which is used as a cardiotonic, can be separated.



The aim of this work is to examine separation of Lanatoside C from complex mixtures of Lanatosides A, B, and C by membrane-based extraction. In the experiments, the system chloroform–trichloroethylene–methanol–water was used. This system shows both good separation and solubility (4, 5). The experiments were carried out with tubular inorganic membranes.

EXPERIMENTAL

On the basis of earlier investigations (4, 5), separation experiments of lanatosides were carried out with a mixture of solvents which has high solubility and selectivity: chloroform–trichloroethylene–methanol–water in a 30:20:30:20 ratio. This system forms two phases: a light phase consisting of water and methanol, and a heavy phase consisting of chloroform, trichloroethylene, and some methanol. The different distribution coefficients of LA, LB, and LC between the light and heavy phases make it possible to separate LC from LA and LB. The physical properties of the investigated system are shown in Table 1.

Because of the aggressivity of the chosen system to polymer membranes, the extraction experiments were carried out with a ceramic tube-type membrane 7/10 mm in diameter, 250 mm long, with pores of 200 nm made by SCT, Cedex, France. For laboratory experiments a laboratory module in a glass tube of 14 mm inner diameter was prepared. The experimental apparatus for extraction was similar to those used by Dahuron and Cussler (1). The flow rates and pressures of the phases were adjusted by vertically positioned storage tanks and needle valves. The flow rates of both feed and extractant were determined by using a measuring cylinder. However, since the viscosity of the heavy phase is three times lower than

TABLE 1
Physical Properties of Investigated System

Component	Distribution coefficient	Phase	Density (kg/m^3)	Viscosity (mPa·s)
Lanatoside A	0.26	Light	942.0	1.766
Lanatoside B	0.64	Heavy	1428.1	0.560
Lanatoside C	5.60			

that of the light one, the pressure was adjusted to be stronger from that side in order to fill the membrane pores with the heavy phase. In this way the membrane resistance, which is proportional to the molecular diffusion rate across the liquid phase in the pores, was decreased. All experiments were carried out at room temperature in a cocurrent flow. The light phase was in the tube and the heavy phase was on the shell side.

The overall mass transfer coefficients in all the experiments were determined by a dynamic method described in the literature (1). On the basis of measured time variations of concentrations from the slope of the model equation for cocurrent flow (1), the overall mass transfer coefficient was calculated:

$$\ln \frac{\Delta C}{\Delta C_0} = -t \left\{ \left[\frac{\frac{1}{V_L} + \frac{1}{mV_T}}{\frac{1}{Q_L} + \frac{1}{mQ_T}} \right] [1 - \exp(-4K_L L/dw_f)(1 + Q_L/mQ_T)] \right\} \tag{1}$$

while the concentration difference on the left-hand side of Eq. (1) is given by

$$\ln \frac{\Delta C}{\Delta C_0} = \ln \left(\frac{C_L(1 + V_L/mV_T) - (C_L^0 V_L/mV_T) - (C_T^0/m)}{C_L^0 - C_T^0/m} \right) \tag{2}$$

In all the experiments, 200 mg of a mixture of Lanatosides A, B, and C (LA 36.445%, LB 14.645%, and LC 41.69%) was dissolved in 100 mL of the light phase. Since the solubility of LA is greater in the heavy phase (distribution coefficient of the heavy/light phase is 1/0.26, i.e., 3.85), its concentration was measured in the light phase. The lanatoside concentrations were determined by HPLC using the external standard method. The column used was a MicroPack MCH-5, and the mobile phase was an acetonitrile–water (50:50 v/v) mixture.

RESULTS AND DISCUSSION

Experimental data (shown in Figs. 1 and 2) can be correlated satisfactorily by using Leveque's equation (6) for the tube-side flow:

$$\frac{kd}{D} = 1.61\left(\frac{d^2w}{LD}\right)^{1/3} \tag{3}$$

and the equation of Prasad and Sirkar (2) for the shell-side flow:

$$\frac{kd_e}{D} = 5.8(d_e/L)(d_e w/\nu)^{0.6}(\nu/D)^{1/3} \tag{4}$$

On the basis of the measured overall mass transfer coefficient, and the calculated values of mass transfer coefficients in the feed (Eq. 3) or the extractant (Eq. 4), the membrane mass transfer coefficient was determined by using Wilson's plot (shown in Fig. 3). The intersection of the straight line representing the experimental data and the vertical axis gives the mass transfer coefficient of the membrane, 0.0144 m/h.

The results presented in this note show that the separation of lanatosides can be carried out efficiently by a membrane-based extraction. By using ceramic membranes in the hollow-fiber form (7), it is possible to provide

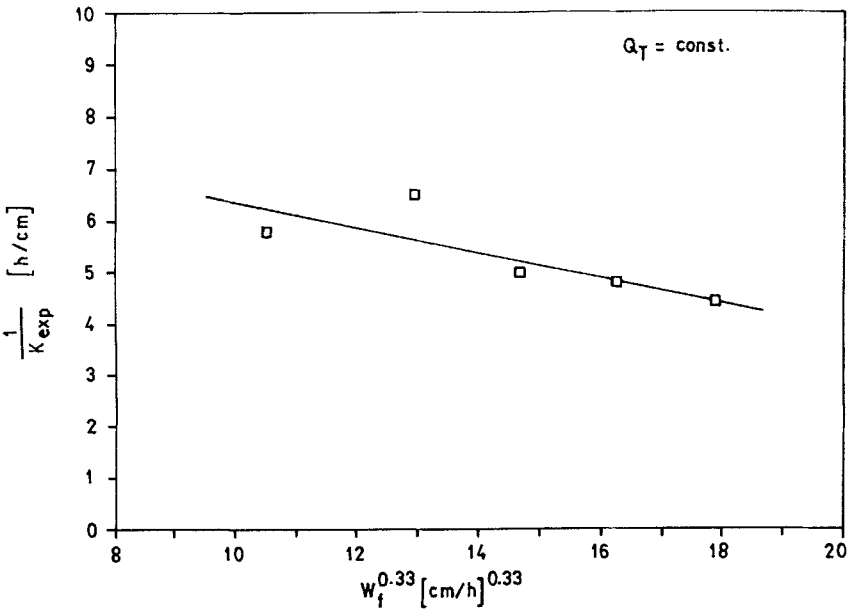


FIG. 1 Applicability of Leveque's equation to tube-side mass transfer.

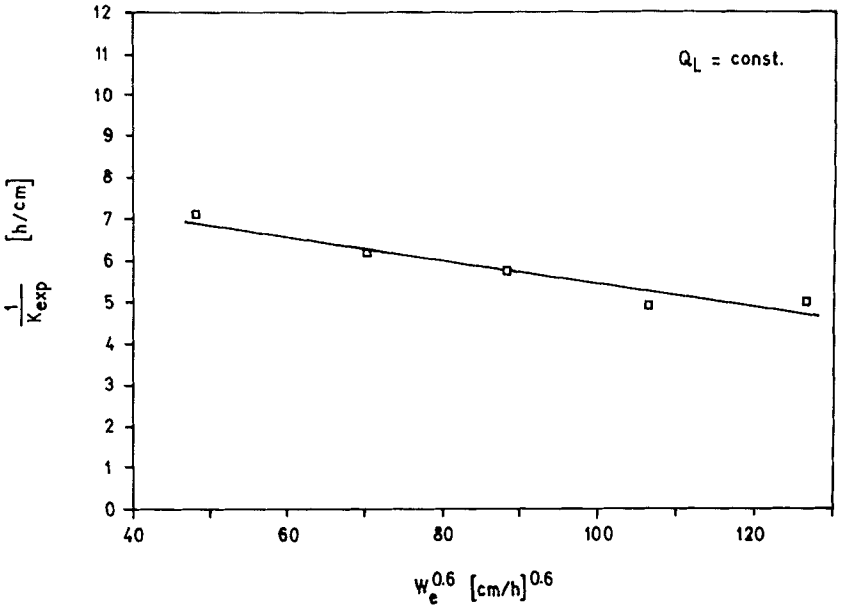


FIG. 2 Applicabily of Prasad–Sirkar equation to shell-side mass transfer.

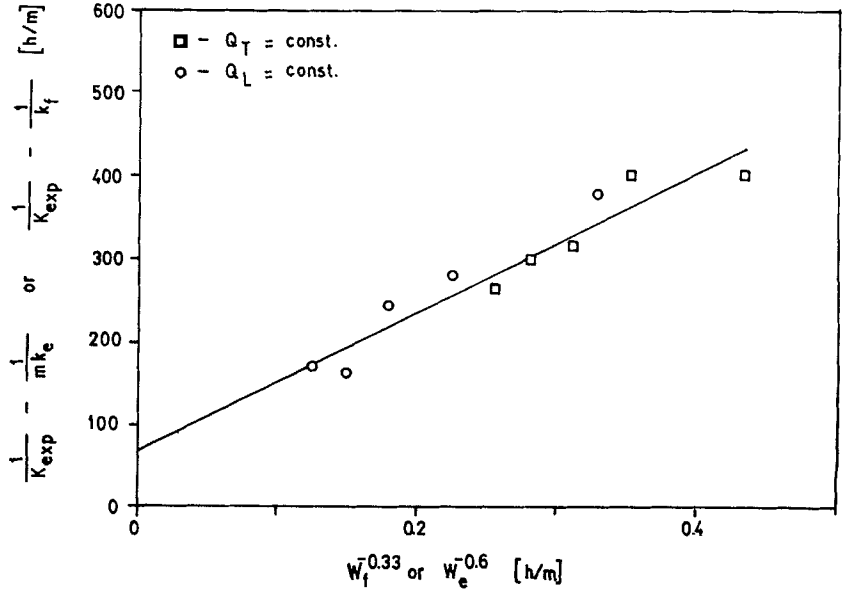


FIG. 3 Wilson's plot for determining the mass transfer coefficient of the membrane.

a high transfer area per unit volume. Furthermore, a ceramic membrane also provides high resistance to aggressive solvents like those used in this experimental work.

NOTATION

C	solute concentration
C_0	initial solute concentration
D	diffusion coefficient
d	tube diameter
d_e	equivalent diameter
K	overall mass transfer coefficient
k_e	mass transfer coefficient in extractant (heavy phase)
k_f	mass transfer coefficient in feed (light phase)
L	membrane tube length
m	distribution coefficient
ν	kinematic viscosity
Q_L	volumetric flow rate of light phase (feed)
Q_T	volumetric flow rate of heavy phase (extractant)
t	time
V_L	reservoir volume of light phase
V_T	reservoir volume of heavy phase
w_e	extractant velocity
w_f	feed velocity

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Received by editor December 1, 1992
Revised June 30, 1993