에스텔화 막반응기 공정을 위한 PVA 복합막을 통한 TFEA,MA,TFEMA/물 혼합물의 투과증발특성

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Pervaporation of TFEA, MA, TFEMA/water Mixtures through PVA Composite Membranes for Esterfication Membrane Reactor Process

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## 1. INTRODUCTION

Membrane reactor is a potential process that allows both reaction and separation in a single process using selective membranes and thus, can reduce both energy consumption and environmental pollution. Pervaporation—aided esterification, one of membrane reactors, is industrially important because of huge amount of many esters including acrylate monomers, ester—type solvents is produced by esterification. In this process, hydrophilic membrane is generally adopted to allow selective permeation of water, by—product, from reaction mixtures and thus, can increase highly the conversion of thermodynamically limited esterification.

In this study, we chose the esterification of trifluoroethanol (TFEA) with methacrylic acid(MA) as a model reaction because it produces trifluoroethyl methacrylate (TFEMA) which is industrially important monomer used in the special coating materials with waterand oil- repellency. For this process, we prepared PVA composite membranes via cross-linking reaction with glutaradehyde onto porous polyetherimde membranes. As a preliminary study to estimate the applicability of pervaporation to the esterifiction, we studied the properties for trifluoroethanol (TFEA)/methacrylic pervaporation acid(MA)/water system as a function of feed composition and operating temperature.

### 2. EXPERIMENTAL

## 2.1 Preparation of PVA composite membrane

10 wt% PVA casting solutions were prepared by dissolving PVA in water(90°C) and added to aq. glutaraldehyde solutions. The solutions were cast onto polyethersulfone membrane supporters, cured in a heating oven at 120°C for 30minutes. After drying at 120°C, The membrane was post-cross-linked in a vacuum oven under vapor phase of HCl for 4hr at 140°C.

#### 2.2 Pervaporation experiments

Pervaporation was carried out in the feed mixture of 90/10, 95/5

wt% TFEA/water, MA/water, respectively. Total permeation flux, J is calculated as follow:

$$J(kg/m^2hr) = Q/(AT)$$

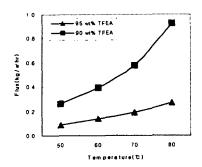
where Q, A, and Trepresent the weight of permeate(kg), effective membrane area(m2), and operating time(t), respectively. Separation factor, is calculated as follow:

$$\alpha = \frac{Y_w / Y_s}{X_w / Y_s}$$

where is the weight fraction of component i in permeate, and is that in feed. w and s denote water and TFEA, MA in the water-TFEA, water-MA mixtures.

## 3. Results and Discussion

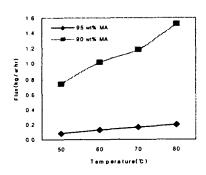
The pervaporation separation of water-TFEA, water-MA mixtures was performed by using PVA composite membranes crosslinked with glutaraldehyde. The pervaporation membrane was prepared by reacting PVA with various concentrations of glutaraldehyde. The crosslinking degree of prepared membrane was conformed by FT-IR and swelling ratio. The cross-sectional structure of the composite membrane was conformed by scanning electron microscope (SEM) showing a  $5\mu$ m active skin layer. Pervaporation separation factor and permeation flux are shown in figure  $1\sim4$ , respectively.



300 95 w% TFEA 90 w% TFEA 100 40 50 60 70 80 90 Temperature(1)

Fig 1. Permeation flux of TFEA

Fig 2. Separation factor of TFEA



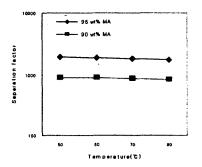


Fig 3. Permeation flux of MA

Fig 4. Separation factor of MA

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