Membrane Reactor Process of Esterification of Trifluoroethanol through PVA pervaporation membrane.

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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 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 an industrially important monomer used in the special coating materials with water- and 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 pervaporation properties for trifluoroethanol (TFEA)/methacrylic acid(MA)/water system as functions of feed composition and operating temperature.

2. EXPERIMENTAL

2.1 Membrane preparation

10 wt% PVA casting solutions were prepared by dissolving PVA in water(90℃) and crosslinked with aq. glutaraldehyde solutions The solutions were cast onto polyetherimide membrane supporter, which were cured in a heating oven at 100℃ for 20minutes to make the PVA composite membranes..

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:

$$J(kg/m^2hr) = Q/(A \times T)$$

where Q, A, and T represent the weight of permeate(kg), effective membrane area(m^2), 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 ㎛ active skin layer. Pervaporation separation factor and permeation flux are shown in figure 1~4, respectively.

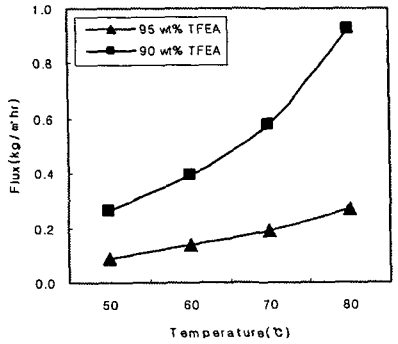

Fig 1. Permeation flux of TFEA

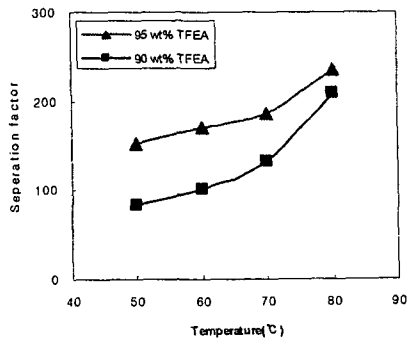

Fig 2. Separation factor of TFEA

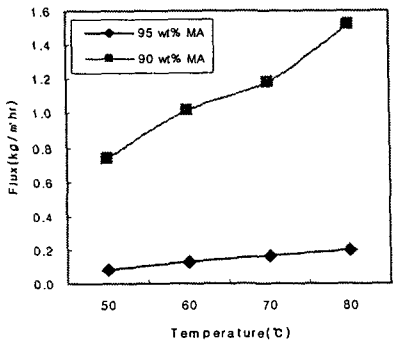

Fig 3. Permeation flux of MA

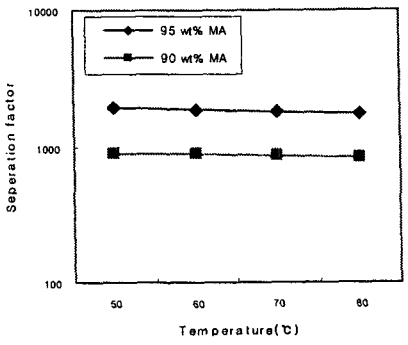

Fig 4. Separation factor of MA

Acknowledgement: This study is supported by CDRS(Carbon Dioxide Reduction & Sequestration R&D Center) under the Ministry of Science & Technology 21st Century Frontier R&D Program. www.cdrs.re.kr

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