**Development of organic solvent nanofiltration membrane with high durability**

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**Highlights**

* We succeeded in the preparation of silica membranes with hydrophobic functional groups.
* The prepared membrane showed higher durability than the previously reported polymeric membrane.

**1. Introduction**

Many nanofiltration membranes, which show the molecular weight cut-off (MWCO) of 200-2000, are used for the separation of water form aqueous solutions. Development of organic solvent nanofiltration (OSN) membranes, which can permeate organic solvents, is desired for recovery and recycling solvents in pharmaceuticals and petrochemical industries. This membrane technology can greatly reduce the waste solvents which need multiple separation steps and associated costs. However, polymeric membranes generally have a low durability for organic solvents. In this research, silica was used as a membrane material in order to improve the durability for organic solvents. We investigated the effect of the type of solute on the OSN performance of the silica membranes. In addition, short-time durability of the silica membrane was confirmed.

**2. Methods**

Membrane preparation

Propyl-functionaized silica membranes (PrFS membranes) were prepared on porous supports consisted of zirconia substrates (outer diameter: **2 mm, inner diameter: **1 mm) with γ-alumina interlayers[1]. A propyltrimethoxysilane (PrTMS) silica sol was prepared by hydrolysis and copolymerization of the PrTMS. The PrTMS (0.1 mol) was added to the solution consisted of cetyltrimethyl-ammoniumbromide (CTAB) (0.008 mol) and 25 mL of EtOH with gentle stirring. 7.5 mL of 1 mol L-1HNO3 was delivered by drops into the solution to promote the hydrolysis and condensation reactions of the PrTMS. Then, the solution was stirred continuously at room temperature for 6 h. The porous support was dipped into the PrTMS solution for 60 s and then pulled out at a speed of 1 cm min–1. The support was dried at room temperature for 3 h and calcined at 453 K for 3 h. The membrane was then washed with ethanol to remove CTAB.

Nanofiltration test

Rose bengal(RB)/ethanol (EtOH), brilliant blue/EtOH, tartrazine(TZ)/EtOH and methyl orange/EtOH solution were used as the feed solution. Concentration of solutes in the feed solutions was 35 µmol l-1. The concentrations of the feed and permeate solutions were measured using an absorbance meter (ASUV 6300PC, ASONE corporation). Feed pressure was 5 bar.

**3. Results and discussion**

****The MWCO of the PrFS membrane and average permeance in the solute are shown in Fig. 1. The MWCO of the PrFS membrane was 492. The permeance was highest in the case that TZ was used as the solute. It is considered that the affinity between the solute and the membrane surface may be related to this phenomenon. Fig. 2 shows the relationships between time and permeance in RB/EtOH system. The permeance reached 200 g m-2 h-1 bar-1 in about 60 min, and was nearly constant until end of the 420 min durability test. Since the PrFS membrane is based on the siloxane network, it is considered the durability against organic solvent is relatively high.

**Fig. 1** The MWCO of the PrFS membrane and the relationships between molecular weight and permeance about PrFS membrane

**Fig. 2** The relationships between time and permeance in RB/EtOH system

**4. Conclusions**

The MWCO of the PrFS membrane was 492. The permeance was highest in the case that TZ was used as the solute. It is considered that the affinity between the solute and the membrane surface may be related to this phenomenon. The permeance was nearly constant form 60 min until end of the test in the RB/EtOH system.

**References**

1. S. Araki, et al., J. Memb. Sci. 380 (2011) 41-47