



# The structure and pervaporation properties for acetic acid/water of polydimethylsiloxane composite membranes

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## ABSTRACT

Polydimethylsiloxane (PDMS)/organic montmorillonite (OMMT)/polyether polyethersulfone (PES) composite membranes were prepared by in situ anionic polymerization using 3-aminopropyltrimethoxy (AMEO) as a crosslinker. The morphology, thermal properties and interaction of PDMS/AMEO/OMMT membranes were characterized by using a scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FT-IR) and a thermal gravimetric analysis (TGA). The swelling behavior of membranes without PES support was investigated. The effects of AMEO content and OMMT content on separation properties were also studied. The results show that the addition of appropriate OMMT could improve the hydrophobic and pro-acetic acid properties of a membrane. The acetic acid selectivity of membranes was best when AMEO content was 0.1. The membrane, loading 2 wt.% OMMT, exhibited the highest separation factor for a feed concentration of 10 wt.% at 313 K. An increase in feed concentration resulted in the enhancement of flux and selectivity. When the feed concentration was above 20 wt.%, the separation factor of a filled membrane was larger than for an unfilled membrane. With increases in the feed temperature, the permeation flux of membranes increased. However, the acetic acid selectivity of an unfilled membrane decreased but for filled membranes initially increased before decreasing.

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## 1. Introduction

Silicone rubber is a compound that has alternate silicon and oxygen atoms, with the particular properties of being both semi-organic and semi-inorganic. The silicone rubber membranes not only have hydrophobic ability, but also fine heat and cold resistance and chemical durability which have been widely applied in pervaporation separation of organic–water mixtures. Pervaporation is a good alternative to conventional processes due to the simplicity of operation and the low working cost and reduced amounts of chemicals required [1–3]. Additionally it offers an opportunity for the separation of organic liquid mixtures, especially in azeotropic mixtures or organic contaminated industrial wastewater [4]. However, the homogeneous membrane is not meeting the practical application requirements of separation factor and permeation flux.

Polymers filled with organic particles or inorganic particles gained considerable interest owing to their enhanced mechanical, rheological properties, and carrier properties [5,6]. The mechanical properties are affected by filler type, filler concentration, and the interaction between filler and matrix. Fang et al. [7] prepared super-hydrophobic nanosilica which exhibited uniform dispersion in the PDMS matrix, and their composites also showed good

mechanical properties and distinct advantage with thermal stability compared with those of the pure silica-filled PDMS composites. The influence of the surface nature (hydrophobic and hydrophilic) and concentration of silica nanoparticles on the coalescence behavior of immiscible polydimethylsiloxane (PDMS)/polyisobutylene (PIB) which was blended under simple low-rate shear flow were investigated via optical shear techniques [8]. Yu et al. [9] prepared the hexadecyl trimethyl ammonium bromide (CTAB) filled membrane used for the separation of dilute acetic acid (ethanol) solution. The results indicated that hybrid membrane had better separation performance. Tang et al. [10] prepared fumed-silica-filled polydimethylsiloxane–polyamide composite membranes by the introduction of hydrophobic fumed silica into a PDMS skin layer. Their pervaporation performance was tested with aqueous ethanol solutions. Increasing the amount of the fumed silica resulted in significantly enhanced ethanol permeability of the membranes.

On the other hand, composite membranes are of practical importance in order to meet a sufficiently high flux requirement by reducing the thickness of the active layer. Integral composite membranes are made of one polymer forming a thin active layer upon a porous support layer. The active layer generally determines the separation performance of the membrane including the separation factor and flux, while the support layer endows the composite membrane with necessary mechanical properties [11,12]. Direct

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coating of a polymer solution onto a microporous support is the current widely used method to prepare composite membranes [13].

Acetic acid, one of the top 50 chemicals, is important in the chemical industry. It is widely used in industry of medical, synthetic fiber, coating, pesticide, food additives, dyeing weaving and so on. It is an important component of China's national economy. With the consumption of petroleum resources and the prices of petrochemical materials rising, renewable bio-resources are a worldwide concern. Using pervaporation coupled with fermentation for continuous fermentation of acetic acid, the acid can be separated from fermentation liquid. The inhibitory effect of the product would be significantly decreased to improve conversion rate and acid production rate. It can simultaneously increase the concentration of acetic acid to reduce the cost of stockpiles, transit and separation.

In this study, using 3-aminopropyltrimethoxy (AMEO) as a cross-linker, the PDMS–AMEO composite membranes were prepared using polyethersulfone (PES) as a support layer. In addition, the organic montmorillonite (OMMT) was used as filler to improve the performance of the PDMS–AMEO composite membrane by in situ anionic polymerization.

## 2. Experimental

### 2.1. Materials

Specification and suppliers of the material and reagent, used for the preparation of membranes, are shown in Table 1.

### 2.2. Membrane preparation

PDMS homogenous solution was cast on the PES support to form the active layer of composite membrane. Firstly, PDMS and AMEO were dissolved in *n*-heptane under stirring to form a homogenous solution at room temperature. The catalyst, dibutyltin dilaurate, was then added into the above solution, with a weight ratio of 0.01 to PDMS. After degassing, the solution was cast onto the PES support (the holes of which were blocked with polyethylene glycol) using a casting knife, then dried in open air and then placed in a vacuum drying oven at the desired temperature to complete crosslinking and to evaporate the residual solvent. Finally, the membranes was soaked in deionizer water making the polyethylene glycol to dissolve from the holes, dried in air for 24 h and then placed in a vacuum drying oven for 24 h at 313 K. All membrane samples were stored in a dust free and dry environment before being used in the pervaporation experiments.

For simplicity, the AMEO contents were indicated the weight ratio of AMEO to PDMS. The OMMT filled PDMS–AMEO/PES composite membranes were also prepared following an identical procedure. By following the similar procedure, PDMS–AMEO homogeneous membranes and OMMT filled PDMS–AMEO membranes (only the active layer without PES support) were fabricated for characterization purposes, except that the solutions were cast on the glass plate instead of the PES support membrane.

The preparation process of membranes is shown in Fig. 1. It shows that the AMEO not only could participate in the cross-linking reactions, but its amino-group could produce the PDMS chain onto the surface of the support layer with Van Der Waals Force. The superfluous amino-group would affect the mass transfer separation process as well.

### 2.3. Membrane characterization

#### 2.3.1. The top and cross-sectional views

The morphology and OMMT distribution of the composite membranes were observed and recorded by a QUANTA200 scanning electron microscope (SEM).

#### 2.3.2. FT-IR

Fourier transform infrared (FT-IR) spectra were obtained by a Nexus 670 (Nicolet Instrument Corporation, USA) instrument equipped with a scanning range of 400–4000  $\text{cm}^{-1}$ . The experiments were run in open air.

#### 2.3.3. TGA

Thermal degradation measurements of the homogeneous membranes and OMMT filled PDMS–AMEO membranes samples were performed using a thermogravimetric analyzer (TGA) (STA 449C, Netzsch, Germany) at a heating rate of 10  $^{\circ}\text{C}/\text{min}$  and temperature programs were run from 40  $^{\circ}\text{C}$  to 800  $^{\circ}\text{C}$  in a nitrogen environment. A nitrogen flow of 20 ml/min was utilized in order to remove all corrosive gas involved in the degradation.

#### 2.3.4. Swelling behaviour

The PDMS–AMEO membranes and OMMT filled 4 wt.% PDMS–AMEO without PES support were weighed carefully before being immersed in the feed mixture at 303 K. The swollen membrane samples were taken out from the feed mixture after a certain period of time and wiped to remove the surface liquid before being weighed. The degree of swelling ( $D$ ) was calculated by:

$$D = (m_s - m_d)/m_d \quad (1)$$

where  $m_d$  and  $m_s$  were the weights of the dried and swollen membranes, respectively.

### 2.4. Pervaporation

Pervaporation experiments were carried out on a self-regulating pervaporation membrane module. The feed solution was pumped into the membrane cell. The permeate vapour was collected in liquid nitrogen traps. The weight of permeate collected in the cold trap was measured to give the permeation flux,  $J$ :

$$J = Q/(A \cdot t) \quad (2)$$

where  $Q$  is the total amount permeated during the experimental time slot,  $t$ , and  $A$  is the effective area of the membrane. The feed and permeate compositions were analyzed by gas chromatography (6890 N).

**Table 1**  
Specification and suppliers of the materials used in the research.

Material	Specification	Supplier
PDMS	4000 mPa s	Jinan Zhonghao Chemical Co., LTD
AMEO	Analytical grade	Nanjing Xinghui Industry and Trade Co., LTD
<i>n</i> -Hexane	Analytical grade	Shanghai Chemical Co., LTD
Dibutyltin dilaurate	Analytical grade	Beijing Chemical Company
OMMT	Trademark DK1	Zhejiang Fenghong New Materials Co., LTD
PES membrane	Pore diameter 0.45 $\mu\text{m}$	Beijing Haicheng Shijie Clean Filtering Equipment Co., LTD

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