



Hyperbranched polymer composite membrane using water as solvent for separating aromatic/aliphatic hydrocarbon mixtures



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ARTICLE INFO

Article history:

Received 10 January 2017

Received in revised form 6 February 2017

Accepted 6 February 2017

Available online 8 February 2017

Keywords:

Hyperbranched polymer

Water as solvent

Pervaporation membrane

Aromatic/aliphatic separation

ABSTRACT

The separation of aromatic/aliphatic mixtures is significant in chemical industry. Pervaporation has attracted increasing attention due to its low energy consumption and environmentally friendly process. However, the formation of membranes mostly use toxic organic solvents, whereas in this study water was used instead to prepare membrane in separating aromatic/aliphatic mixtures. W3000 were dispersed in water to form micelles, which were then deposited onto the surface of the tubular porous ceramic substrate through the negative pressure-driven assembly method. The emulsion and composite membranes were characterized by TEM, SEM and FTIR. The crosslinking density of W3000 was also tested using NMR XLD analyzing system. The membranes were used for separating aromatic/aliphatic mixtures through pervaporation. The results showed that compared with the membrane prepared by DMF, the pervaporation separation index (PSI) of the composite membrane formed using water as solvent was 3.3-fold increased for separating toluene/*n*-heptane mixtures. The as-prepared composite membranes showed a better comprehensive pervaporation performance. This facile strategy may have a potential in the application of pervaporation for separating aromatic/aliphatic mixtures in industry.

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1. Introduction

The separation of aromatic/aliphatic mixtures is of great significance in the chemical and petrochemical industries, such as naphtha reforming, gasoline purification, and cyclohexane recovery from catalytic hydrogenation of benzene [1,2]. However, effective separation of aromatic/aliphatic mixtures is difficult because of their close physical and chemical properties [3]. Compared with traditional separation techniques such as extractive distillation and azeotropic distillation, membrane techniques are preferred alternatives because of its efficiency, energy conservation, eco-friendliness, and facile scale-up [4]. Pervaporation, as a membrane process for liquid-liquid separation, has been researched for decades in separating aromatic/aliphatic mixtures, yet there has been no reported use in industrialized application. A reason is that the separation performance of the membrane is still ineffective [5]. Moreover, the synthesis process of the membrane materials is complex [6,7], and harms the environment and human health. Both these two problems limit the application of the pervaporation in

industry. In order to promote the development of pervaporation in separating aromatic/aliphatic mixtures, a facile membrane preparation method is necessary.

The membrane materials commonly used in the separation of aromatic/aliphatic mixtures are organic polymers, including polyimides, polyurethanes, poly(ether amide)s, polyacrylates, poly(methyl methacrylate)s, and poly(ether-block-amide) [8–16]. All of these polymers are dissolved in organic solvent to form homogeneous solution during the membrane formation process. However, most of the organic solvents are toxic, such as *N,N*-dimethylformamide (DMF), *N,N*-dimethylacetamide (DMA), *N*-methyl-2-pyrrolidone (NMP), and tetrahydrofuran (THF) [17]. The use of these toxic solvents will lead to serious environmental and economic impacts, and thus reduce the benefit of membrane technology. Many membrane scientists have been aware of this problem. In thus, some non-toxic organic solvents, such as polyethylene glycol (PEG) [18], dimethyl sulfoxide (DMSO) [19–21], dioctyl sebacate (DOS) [22], triethylene glycol (TEG) [23,24], triethylphosphate(TEP) [25–27], ionic liquids (ILs) [28,29], and supercritical CO₂ (ScCO₂) [30,31], are developed to be feasible substitutes in some certain membrane preparation process.

However, some of the non-toxic solvents still have a few drawbacks, because they could be a toxic solvent under certain

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conditions. Therefore, water is considered alternatively as an ideal dispersion media during the membrane preparation process. Moreover, using water as a solvent brings easy recovery and quite low investment. There have been some reports of using water as a solvent for the preparation of membranes in other separation fields. For example, Lively et al. used water as solvent to prepare membrane for CO₂ capture [32]. They prepared a defect-free lumen-side barrier layer on hollow fiber substrate by polyvinylidene chloride (PVDC) latex. The latex film formation mechanism was also schematically illustrated. In pervaporation membrane field, water was used as solvent to prepare membrane in the separation of furfural and butanol/water mixtures [33,34]. Polydimethylsiloxane (PDMS) were emulsified in water with the addition of surfactant to prepare membranes, which exhibited good performance especially with enhanced permeate flux. Therefore, it is quite promising that emulsion of polymer in water can be used to prepare membrane for different separation fields.

In this study, water was used as the solvent without any surfactant to prepare membranes for separating aromatic/aliphatic mixtures. Hyperbranched polymer (Boltorn W3000) was chosen as the membrane material. W3000 is a macromolecule containing hyperbranched polyester as the core, which grafted by hydrophobic fatty acid chains and hydrophilic polyethylene glycol chains. The enrichment of hydroxyl and carboxyl groups in W3000 lead to affinity with the benzene ring, which is helpful for the preferential permeate of aromatic compounds [35]. The W3000 micelle was deposited onto the tubular ceramic porous substrate through the negative pressure-driven assembly method. The morphology and structure of the composite membranes were characterized by scanning electron microscope (SEM), transmission electron microscope (TEM), Fourier transform infrared spectroscopy (FTIR) and crosslinking density measuring apparatus (XLD). The effect of the preparation conditions (concentration of W3000, crosslinking temperature and crosslinking time) on the separation performance was also investigated and compared with the membrane prepared by organic solvent DMF. This facile method could provide the opportunity for application of pervaporation in separating aromatic/aliphatic mixtures.

2. Experimental

2.1. Materials

Hyperbranched polymer (Boltorn W3000, Mw 9000) was provided by the Perstorp Specialty Chemicals AB (Sweden) [36]. Alumina (Al₂O₃) substrates were obtained from JieXi LiShun Technology Co., Ltd, China. The inner and outer diameters of the tubular substrates are 8.5 and 13.5 mm respectively. N,N-dimethylformamide (DMF), toluene, *n*-heptane, benzene, cyclohexane and iso-octane were purchased from Beijing Chemical Factory. 3-aminopropyl-trimethoxysilane (Mw 179.29) was purchased from Aldrich Co., Ltd, America. Deionized water was home-made by a water purification system. All chemicals were used as received without further purification.

2.2. Preparation of W3000 membrane

The alumina tubular substrates were pre-treated by a silane coupling agent (3-aminopropyl-trimethoxysilane). Pretreatment of the ceramic tube was performed using immersion of a mixture of ethanol (EtOH) solution (EtOH:H₂O = 95:5) and 2 g/L silane coupling agent at room temperature for 2 h, followed by rinsing with deionized water and drying in air at 110 °C for 2 h in a vacuum oven. W3000 was added into the deionized water and stirred at 40 °C for 1 h to form the emulsion. Different concentrations of W3000 emulsion were prepared and their influence on the separation performance was studied. The W3000 micelles were assembled onto the surface of the pre-treated alumina tubular substrates through the negative pressure-driven assembly method. As shown in Fig. 1, one end of the pre-treated tubular substrate was sealed by a rubber plug, and the other end was connected to vacuum pump. The substrate was then immersed into the W3000 emulsion and the vacuum was formed in the lumen side. The W3000 micelles were thus deposited onto the outer surface of the ceramic substrate due to the transmembrane pressure. The as-formed composite membrane was then thermal crosslinked in an oven. The membrane prepared by immersion method using water as the solvent was prepared for comparison. The preparation process was as follows: the pre-treated tubular substrate was

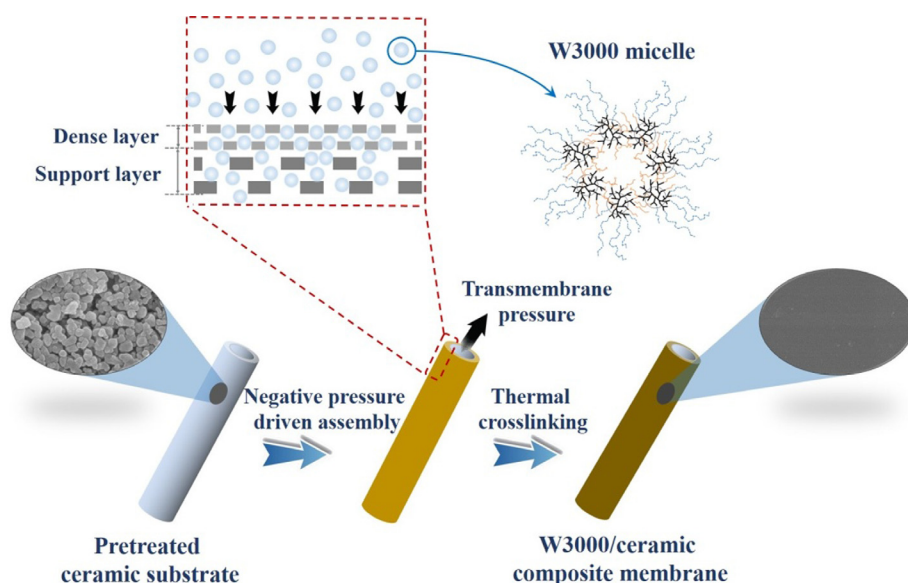


Fig. 1. Illustration of membrane forming of W3000 micelles in the porous ceramic substrate.

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