

Pervaporation through NaA Zeolite Membranes - A Review

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Abstract— The article concerns with pervaporation process through NaA zeolite membranes. This kind of membrane is known for its separation performance in removing water from organic compound mixture. Extremely good water adsorption level and the feature of molecular sieving are the main attributes that makes them very good in water removal. Recent studies show that NaA zeolite membrane has very high separation factor (more than 10000) and reasonable flux (up to 5 kg m²/h). Despite these good separation performances, NaA zeolite membranes suffer some drawbacks concerning its durability under highly acidic condition and high temperature.

Index Terms— azeotrope separation, membrane, zeolite

I. INTRODUCTION

Pervaporation is a method to separate liquid mixtures which is depend on partial vapor pressure of the compounds. To increase the driving force, the permeate side uses vacuum condition. The growing uses of pervaporation are especially in energy application, when it is used to overcome the azeotrope condition of water/ethanol mixture, separation of organic compound and water mixtures such as 2,2,2-trifluoroethyl alcohol (TFEA) [24], isopropanol [7,15,17,20,23] and acetic acid [25]. In some cases, pervaporation is also used to separate hydrocarbons [27].

The pervaporation can be explained in this way: The vapor is enriched in the preferentially permeating component and is condensed for future processing. Meanwhile, the retentate is enriched in the non-preferentially permeating component. The retentate stream can be either recycled or used for other processes [26].

When the pervaporation process was still in infancy, polymeric membranes were much more common put into use. The sole reason was its high reproducibility, cheap, and relatively easy to use. Usually, “thermally resistant” polymer such as polyimide was used. Yet this type of membranes was not ideal because it is not really resist even at slightly higher temperature (about 100°C or above), thus resulting to proneness to swell. The swelling membrane turns to decrease the performance significantly as the membrane structure will take more liquid.

Manuscript received April 4, 2013.

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Zeolite has been used in many applications, such as catalyst [1], ion exchanger, and adsorbent. Meanwhile, zeolite membranes have also been used for pervaporation both industrially and in laboratory studies. These membranes are polycrystalline zeolite layers deposited on porous inorganic supports. Zeolite membranes are significantly structurally stable both physically and chemically than polymer membranes. Most zeolite membranes are resistant to low pH (with some exceptions) and able to perform good separation even in temperatures up to 1270 K [4, 10]. Moreover, zeolite membranes do not swell and have uniform molecular size pores, allowing molecular sieving. Despite its advantages, zeolite membranes are inferior to polymer membranes in some ways: they are more expensive to produce and more brittle.

II. NAA ZEOLITE MEMBRANES

A. NaA Zeolite Membranes Characteristics

Zeolite A with sodium cations, denoted as NaA, has formula of Na₁₂[(AlO₂)₁₂(SiO₂)₁₂] · 27H₂O, and contains cages with orthogonal 3-D oriented apertures of approximately 0.4 nm. The pore dimension is changed to 0.45 or 0.30 nm, when the zeolite is ion exchanged with Ca²⁺ or K⁺, respectively [17] which is close to or smaller than the molecular kinetic diameters of short-chain alkanes. As a membrane, NaA zeolite was very promising molecular sieve membrane due to their hydrophilicity. These features make NaA zeolite very good in terms of removing water substances out of the mixtures. NaA zeolite shows excellent hydrophilic characteristics because of its low silica/alumina ratio. The aluminum content of A-type zeolites is high (Si/Al = 1), making them hydrophilic [34]. Bowen et al. mentioned NaA zeolite has a greater average affinity for water than methanol [26]. Water-NaA affinity has heat of adsorption of 100 ± 25 kJ/mol, whereas methanol-NaA has only 85 ± 20 kJ/mol. This difference is enough to explain why NaA zeolite is so hydrophilic. Hydrophilic zeolite membranes like NaA have effectively dehydrated alcohols with high separation factors (Table 1).

Like any other polycrystalline zeolite membranes, NaA zeolite membrane also contains transport pathways in intercrystalline regions, or non-zeolite pores. Kondo and Kita (2010) proposed the theoretical consideration based on their experiments (see Fig 1), in which the zeolitic pores in zeolite layer are assembling in a very fine and narrow non-zeolitic pore opened to the support tube through the zeolite layer [7]. In PV (see Figure 1), the feed solution evaporates at Boundary 1 near the membrane surface. The water molecules in the feed are selectively adsorbed in zeolitic pores in the zeolite layer on

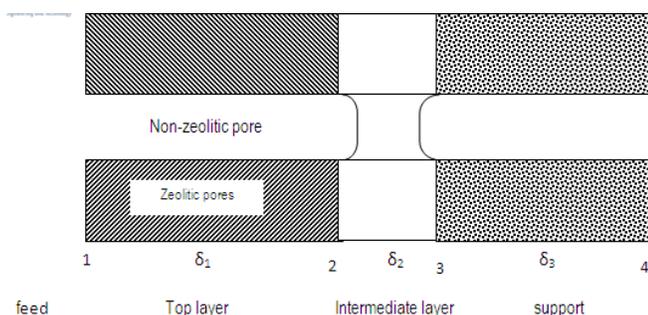


Fig. 1. Schematic diagram of flow model occurring in a zeolite membrane. The feed is at the left hand side, whereas permeate is in the opposite (beyond the support)

the membrane surface, and then transported to the non-zeolitic pore through the zeolitic pores by surface diffusion. Subsequently, at narrower space (Boundary 2) in the non-zeolitic pore, the capillary condensation occurs at lower relative pressure of vapor, and then the space (δ_2 length) filled by the condensate. Subsequently, on the condition that the permeation side was kept under vacuum, the condensate evaporates in Boundary 3 and diffuses into the permeation side. The condensate significantly inhibits permeation of other components by blocking them from entering the pore.

B. NaA Zeolite Membranes Synthesis'

NaA zeolite membranes are most often prepared by hydrothermal synthesis [2-13, 16-17, 31-33]. Hydrothermal synthesis involves crystallization of a zeolite layer onto a porous support from a gel that is usually composed of water, amorphous silica, a source of tetrahedral framework atoms other than silica, a structure directing organic template, and sodium source (usually Na_2O). This gel is placed in contact with the support in an autoclave. The operation time, temperature, and gel compositions for crystallization depend on the zeolite. Supports are generally alumina tubes or discs, although other ceramics, and other materials have been used, such as mullite [3,7], $\alpha\text{-Al}_2\text{O}_3$ -boehmite [4], UV-radiated TiO_2 wafer [8], porous Ni sheet [13]. Xu et al. even used hollow fiber supports [3] with considerable result. Alumina ($\alpha\text{-Al}_2\text{O}_3$) supports typically have pore diameters between 100-200 nm.

During in situ crystallization, zeolite crystals nucleate grow on the support surface. Crystals sometimes nucleate in the bulk solution, but this is not preferred because it will form framboid-like zeolite structure in the surface, making the surface rougher and thicker the supposedly thin NaA zeolite layer, thus lowering the flux significantly [12,15,17]. The faster seed transfer to the support surface, the better the thin layer surface will be.

Nucleation in the bulk is less likely for dilute gels [2,15]. Techniques have been developed to prepare NaA zeolite membranes with organic template molecules [9, 20], as this type of membranes are usually prepared without a template, but if a template is used, the zeolite structure forms around the organic template molecules, making the pores. Addition of template also induces formation of smaller, more interlocked crystalline.

Seed crystals are added to the support prior to the crystallization step to provide sites for zeolite growth and

improve control of crystal growth. Using seed crystals is referred to as two-step crystallization. This method is usually used to prepare high-performance, thin layer membranes. Dip coating [4], electric charge [19], vacuum seeding [23], microwave [14, 22] and the usage of larger-pore support [10] increased seed crystal adherence and improved membrane quality.

C. NaA Zeolite Membranes Separation Performances

As mentioned previously, NaA zeolite membranes are nearly ideally suited for organic dehydration because they are highly hydrophilic and their pore diameter (0.4 nm) is smaller than almost all organic molecules but larger than water. These aforementioned properties allow preferential permeation of water over organic compounds with separation factors that are often over 1000 and sometimes higher than 10,000. These high separation factors are sensitive to permeate concentration because the water concentrations are often higher than 98%.

NaA zeolite membranes are very selective to water and its fluxes are relatively high compared to other zeolite membranes. Meanwhile, mordenite (in this article used as benchmark zeolite membrane), which is known for its resistance against acidic condition, is not even the same as NaA zeolite. Whilst NaA zeolite membranes' ethanol fluxes and separation factors are about 3-4 $\text{kg m}^2/\text{h}$ and 10000 respectively, compared to mordenite membrane was 1.17 $\text{kg m}^2/\text{h}$ and 6800 [20]. This caused by pore size and how interlocked the crystallines are. NaA zeolite membranes, prepared with appropriate concentration and seeding time, are very well interlocked, leaving only small defects on its surface [18,19].

D. NaA Zeolite Membranes Durability

For application in separation processes, the membrane must be defect-free, dense and uniform. Recently it has been reported that the NaA zeolite membrane has only a low thermal stability. Caro et al. [28] suggested the mismatch of thermal expansion coefficients as one of the reasons the NaA zeolite membrane shows such a low gas-separation performance. Noack et al. [29] reported that LTA zeolite in the wet state shows a strong contraction ($-50 \times 10^{-6}/\text{K}$) between 25 and 100 $^\circ\text{C}$, a strong expansion ($+50 \times 10^{-6}/\text{K}$) between 100 and 150 $^\circ\text{C}$, and a weak contraction ($-5 \times 10^{-6}/\text{K}$) between 150 and 450 $^\circ\text{C}$. Considering the average pervaporation processes are operated in temperature range of 50-100 $^\circ\text{C}$ and vapor permeation between the strong expansion temperature, these are clearly affected the endurance of NaA membrane under such circumstances. Moreover, NaA membranes made using small-sized pore support are even weaker because they tend to build thin intermediate layer, because the size of the pores are small enough to be plugged by the seeds [10].

Some methods were proposed to lessen the effects. Cho et al. [10] proposed what is called "control of intermediate layer structure". It is basically a way to thicken the intermediate layer using large pore support, so the contraction force and shear by the contraction of NaA layer can be shared to the support (in case of pervaporation). Das et al. [4], propose another way to reduce cracks on NaA zeolite membranes by

utilizing addition of boehmite to reinforce the intermediate layer. All these methods showed improved thermal stability.

Smaller seed particles and larger support pores are proved beneficial to the formation of physically better NaA membranes, but they do have drawbacks. Yang et al. [32] found that smaller seed particle (0.3 μm) induces low-quality zeolite like hydroxysodalite (pore size of 2.8 μm) instead of NaA. It was not clear whether such low quality membranes induced by submicron-size seeds were attributed to over-crystallization, since the smaller seeds generally required short crystallization time.

Although the NaA-type zeolite membrane shows excellent performance, the acid stability of the membrane is reportedly poor. Unfortunately, only limited publications are available in the literature. Hasegawa et al. [5] studied the permeation fluxes through the NaA-type zeolite membrane was monitored using the real-time monitoring system to study the influence of acid on the permeation properties. At the end of the experiment, it could be concluded that the NaA zeolite layer is virtually destroyed, separated from its support, making the membrane practically useless for pervaporation operations. As in 2012, there has been some experiment related to endurance of zeolite membrane under acidic condition, mainly intended to replace the existing NaA zeolite membranes with more acid-resistant ones, such as mordenite [21], merlinoite and phillipsite [30].

E. NaA Zeolite Membranes Reproducibility

Although progress in improving separations suggests that zeolites may have further uses in large-scale pervaporation, the only current large-scale commercial use of NaA zeolite membranes we are aware of is in organic dehydration. Mitsui Engineering & Shipbuilding Co. in Japan has implemented A-type zeolite membranes for this application commercially in 2001. The Mitsui Engineering & Shipbuilding Co. zeolite membrane pervaporation plant uses 20–30 μm thick NaA zeolite membranes on porous, tubular ceramic supports, and processes alcohols up to 530 L/h with separation factors as high as 10,000, and increased the alcohols purity from 10 wt.% water to 0.2 wt.% water content.[31].

In 2008, Aguado et al. [17] reported a continuous NaA zeolite membrane production, by continuously flowing and practically immersing the support tube in “nutrient”, yet the result was not satisfactory. The NaA zeolite membrane was not properly interlocked and even the polycrystalline had not yet formed as it hoped to be. Meanwhile, Sato and Nakane [12] proposed a much-higher performance reproducible fabrication method for high-flux NaA zeolite membrane has been developed for industrial mass production. The experiment itself was undertaken by using dip coating, therefore in the mass-production scale it would be still had difficulty because it cannot produce membranes with very large surfacecommercially.

Another research conducted by Pina et al. [16] concluded that zeolite NaA membranes have been synthesized by secondary growth on the external surface of α -alumina tubular supports using a semicontinuous system in which fresh gel was periodically supplied to the synthesis vessel. Compared to traditional batch methods, the procedure developed in this work provides a better control of the synthesis and

crystallization conditions and is easier to implement at an industrial scale. The membranes obtained by the semi-continuous method displayed reasonable separation performance in the pervaporation of ethanol/water mixtures (e.g., a separation factor of 3600 at a water permeation flux of 3.8 kg/h.m^2).

F. Future Trends

The NaA zeolite membrane technology is still evolving. Advances in the following areas have potential to improve understanding and effectiveness of pervaporation through zeolite membranes:

- An improvement of flux with making thinner NaA zeolite upper layer
- New techniques of secondary growth
- Endurance of NaA zeolite membranes under high temperature and low pH.
- The mass-production of NaA zeolite membrane with both good separation performance and good reproducibility, including robust, cheaper (cost per product), and easiness to produce. This includes preparation of NaA zeolite layer on the inner side of the tube using rotating processes [33].
- Improvement on modeling and simulations of transport through zeolites at high coverages [26]
- A better understanding about fouling in the NaA zeolite membrane, as they are very adsorptive

III. CONCLUSION

Pervaporation through NaA zeolite membranes has advantages for separating azeotropes, close-boiling mixtures, and thermally sensitive compounds, but only for removing the species present in low concentration because heat transfer becomes important if large quantities are removed. NaA zeolite membranes have additional advantages in separating mixtures employing high hydrophilicity molecular size differences and/or adsorption differences. Despite the good separation performances, NaA zeolite membranes suffer some drawbacks concerning its durability under highly acidic condition and high temperature.

TABLE 1
Comparisons of the pervaporation properties of some as-synthesized zeolite membrane

Membranes	Supports	Membrane synthesis	Membrane type	Mixtures (A/B)	T (K)	Concentration (A % wt)	$u_{A/B}$	Flux ($\text{kg/m}^2\text{h}$)	Remarks	References
NaA	Mullite-alumina-cristoballite	Dip coating, secondary growth, hydrothermal	Tubular	H ₂ O/EtOH	368	5	>5000	2.35	-	[2]
NaA	α -alumina	Rubbing, secondary growth, hydrothermal	tubular	H ₂ O/EtOH	393	10	47000	1.59	-	[6]
T-type	Mullite	Dip coating, secondary growth, hydrothermal	-	H ₂ O/i-PrOH	348	10	10000	2.01	-	[7]
NaA	Mullite	Dip coating, secondary growth, hydrothermal	-	H ₂ O/EtOH	408	5	30000	8.34	Vapor permeation	[7]
NaA	TiO ₂ (UV-radiated)	UV irradiation	-	H ₂ O/EtOH	303	-	54000	-	-	[8]
NaA	α -alumina	Secondary growth, hydrothermal	-	H ₂ O/i-PrOH	343	5	4700	1.67	TMAOH template addition	[9]
NaA	α -alumina	Rubbing, secondary growth, hydrothermal	-	H ₂ O/TPEA	333	40	250000	1.25	-	[11]
NaA	α -alumina	Dip coating, secondary growth, hydrothermal	Tubular	H ₂ O/EtOH	348	10	10000	5.6	-	[12]
NaA	α -alumina	Dip coating, secondary growth, hydrothermal	Tubular	H ₂ O/EtOH	418	10	>10000	31	Vapor permeation	[13]
NaA	Porous Ni sheet	Slip coating, secondary growth, hydrothermal	-	H ₂ O/EtOH	348	10	>10000	4	-	[13]
NaA	α -alumina	Microwave seeding	-	H ₂ O/EtOH	363	10	10000	0.8	-	[14]
NaA	α -alumina	Hydrothermal vacuum seeding	-	H ₂ O/EtOH	333	10	10000	1	-	[14]
NaA	α -alumina	Hydrothermal seeding in electric field	-	H ₂ O/i-PrOH		5	3281	1.24	Electric charge 1 V	[15]
NaA	α -alumina	Dip coating and rubbing, secondary growth, hydrothermal	Tubular	H ₂ O/EtOH	383	10	>10000	2.6	seed size 1 μm	[32]
NaA	α -alumina	Dip coating, secondary growth, hydrothermal	Hollow fiber	H ₂ O/EtOH	348	10	>10000	6.3	Inner thin layer	[33]
Mordenite	α -alumina	Secondary growth, hydrothermal	Tubular	H ₂ O/i-PrOH	323	50	6800	1.17	Using template TEABr	[20]
NaA	α -alumina	Hydrothermal vacuum seeding	Tubular	H ₂ O/i-PrOH	343	5	>10000	1.67	-	[23]

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