Study on the effect of seed particle size toward the formation of NaX zeolite membranes via vacuum-assisted seeding technique

Liyana Salwa Mohd Nazir, Yin Fong Yeong and Thiam Leng Chew

ABSTRACT
In this work, we report the effect of seed particle size towards the formation of NaX zeolite membranes through single and multi-stage hydrothermal growth methods. A seed layer was firstly deposited onto the α-alumina support via vacuum-assisted seeding technique prior to hydrothermal growth of the membrane layer. The morphology and crystallinity of the membranes were characterized by using scanning electron microscope (SEM) and X-ray diffraction (XRD), respectively. From the results obtained, by using smaller seed particles of 750 nm, the growth of the membrane layer was enhanced due to minimum gaps found in between the seed particles. The N2 gas permeation results showed that the multistage synthesis method has successfully reduced the defects of the membranes. However, it was observed that the presence of zeolite NaP impurities in the membrane layer became more significant along with the increase of the synthesis stage.

1. Introduction

Over the past decades, zeolite membranes have shown great potential in various separation applications compared to the other types of inorganic membranes. Zeolite is a crystalline, aluminosilicate material with well-defined framework and pore structure. The characteristics of zeolites, such as molecular sieving ability, catalytic and selective sorption properties, thermal and chemical stabilities, have made them potential candidate in various applications including gas separation, pervaporation, solvent dehydration, membrane reactors, coating materials, catalytic micro reactors, gas sensors, and fuel cells [1–7]. Zeolite membrane was firstly prepared by Suzuki in 1987, where a continuous zeolite layer was synthesized on the porous support [8]. In 1998, Mitsui Engineering and Shipbuilding Co. Ltd in Japan was the first company to apply NaA zeolite membrane for pervaporation dehydration in industrial-scale separation plant [9]. Subsequently, the applications of zeolite membranes have been extensively explored and reported in the literature [4,5,10–14].

FAU-type zeolite membrane has received attention from researchers due to its unique performance in separation processes. NaY and NaX zeolite membranes which categorized under FAU type are low silica zeolites with 12 membered-ring and pore size of 7.4 Å. NaY and NaX zeolite membranes are highly CO2 selective.

CONTACT Yin Fong Yeong (yinfong.yeong@utp.edu.my) Chemical Engineering Department, Universiti Teknologi Petronas, Bandar Seri Iskandar, Perak 32610, Malaysia © 2021 The Author(s). Published by Informa UK Limited, trading as Taylor & Francis Group on behalf of The Korean Ceramic Society and The Ceramic Society of Japan. This is an Open Access article distributed under the terms of the Creative Commons Attribution-NonCommercial License (http://creativecommons.org/licenses/by-nc/4.0/), which permits unrestricted non-commercial use, distribution, and reproduction in any medium, provided the original work is properly cited.
due to their polar characteristic. Besides, these membranes showed high performance in CO₂/CH₄, CO₂/N₂, and CO₂/H₂ gas separation [15–19], and demonstrated promising performance in pervaporation process [20–24]. Nevertheless, FAU-type zeolite membrane does not exhibit molecular sieving property mainly attributed to its large pore size, as compared to zeolite membranes with smaller pore size such as CHA (~3.4 Å) and MFI-types (~5.6 Å). The separation property of FAU-type zeolite membrane is usually achieved by the difference in adsorption and diffusion mechanisms of the permeates. Even so, high permeation rate is one of the advantages demonstrated by FAU-type zeolite membrane, as compared to small pore zeolite membranes [16]. In terms of membrane formation, zeolite membranes are commonly prepared by using hydrothermal growth method. The reaction mixture is usually heated at a constant temperature under autogenous pressure in an autoclave reactor for several hours or even days, depending on the type of zeolite to be synthesized [11]. Compared to the other method such as chemical vapor deposition, hydrothermal synthesis method is preferable because this method is relatively easier and involved simpler equipment setup.

In fact, large number of variables could affect the formation of zeolite membranes such as molar composition of the synthesis gel, reactant source, Si/Al ratio, alkalinity, water content, inorganic cation, organic template, temperature, and aging duration [10]. It is important to produce zeolite membrane with continuous, defects-free, and nearly bi-dimensional layer in order to enhance the transportation of permeate through the zeolite pores [4]. The existence of defects will significantly affect the separation performance of the zeolite membranes [25]. Defects are defined as intercrystalline spaces (or non-zeolitic pores) which are larger than the zeolite pores [26]. Defects formed on the zeolite membranes should be minimized in order to achieve molecular sieving and preferential adsorption properties of the zeolite layer [26].

Throughout the years, there have been some progress in the synthesis of FAU-type zeolite membrane. However, the control of defects and zeolite phase purity remain challenging. Furthermore, the synthesis of FAU-type zeolite membrane by using vacuum coating as seeding method is hardly found in the literature. The vacuum-assisted seeding technique is favorable for the synthesis of zeolite membranes because this technique usually produces a homogeneous seed layer which can subsequently minimize the defects formed on the membrane. In this study, we report the synthesis of NaX zeolite membranes via vacuum seeded hydrothermal growth method. The effects of seed particle size and synthesis stage toward the formation of NaX zeolite membranes are investigated.

2. Methodology

2.1. Synthesis of NaX zeolite seeds

NaX zeolite seeds with different particle sizes were synthesized via in-situ crystallization method as described in the literature [16]. The starting solution was prepared by mixing sodium silicate, sodium aluminate, sodium hydroxide (NaOH) and deionized (DI) water. Seed S1 was synthesized by using a mixture solution with molar composition of Al₂O₃: 4.8SiO₂: 17Na₂O: 975H₂O and solution aging duration of 4 h. Meanwhile, seeds S2 and S3 were synthesized by using the same solution mixture with a molar composition of Al₂O₃: 4.8SiO₂: 19Na₂O: 975H₂O, but with different solution aging durations of 4 h and 48 h, respectively. All synthesis mixtures were heated in an oven for 24 h at 90°C. After that, the resultant particles were filtered and repeatedly washed with DI water until the pH value of the filtrate was less than 10. The particles were then collected and dried in an oven overnight at 90°C.

2.2. Preparation of zeolite seed layer via vacuum seeding method and synthesis of NaX zeolite membranes

For the preparation of the zeolite seed layer on α-alumina disc-type support, zeolite suspension was firstly prepared by dispersing 0.50 g of NaX zeolite seeds in 100 mL of DI water, followed by ultrasonic treatment for 60 min. The seeds were deposited onto the support via dead-end vacuum filtration method. Prior to the deposition of seed layer, the support was washed with DI water and dried in an oven.

On the other hand, the synthesis of NaX zeolite membranes was carried out via secondary hydrothermal growth method by following the procedure as described in the literature [16]. The synthesis solution was prepared by mixing sodium silicate, sodium aluminate, NaOH, and DI water with molar composition of Al₂O₃: 4.8SiO₂: 17Na₂O: 975H₂O. After aging for 4 h under stirring, the synthesis solution was poured into an autoclave reactor containing α-alumina disc-type support coated with the zeolite seed layer. Then, the autoclave reactor was heated in an oven for 24 h at 90°C. After synthesis, the membrane collected from the reactor was washed with DI water and subsequently dried at ambient temperature. For multi-stage hydrothermal synthesis, similar procedure was applied from stage one up to stage three. Table 1 shows the membranes prepared in this work.

### Table 1. Zeolite seeds and membranes prepared in this work.

| Seeds | Seed Particle sizes (nm) | Seeded supports | Membranes |
|-------|--------------------------|-----------------|-----------|
| S1    | 5500                     | NaX-1           | NaX-1-M1  |
| S2    | 2700                     | NaX-2           | NaX-2-M2  |
| S3    | 750                      | NaX-3           | NaX-3-M3  |
2.3. Characterization and $N_2$ permeation test

The morphology of the synthesized membranes was characterized by using scanning electron microscope, SEM (Hitachi TM 3000), and the crystallinity of the membranes was verified by using X-ray diffraction (XRD) (Bruker D8). The defects of the synthesized membranes were measured through $N_2$ permeation test by using the permeation test setup as illustrated in Figure 1. Firstly, pure $N_2$ gas was fed into the setup at 1 bar and room temperature. Then, the $N_2$ gas permeating through the membrane was measured by using bubble flow meter (in ml/s). The permeance, $P$ (mol m$^{-2}$ s$^{-1}$ Pa$^{-1}$) of $N_2$ gas is then calculated by using equation 1 as follows [27]:

$$P = \frac{J}{\Delta p}$$  \hspace{1cm} (1)

where $J$ is the flux of $N_2$ gas (mol m$^{-2}$ s$^{-1}$), $\Delta p$ is the partial pressure difference of gas across the membrane (Pa).
Figure 3. SEM images of NaX zeolite membranes growth on seeded supports (a) surface morphology (b) cross-section view.
3. Results and discussion

3.1. Effect of seed particle size on the growth of NaX zeolite membranes

Figure 2 shows the SEM micrographs of zeolite seed layer deposited on the disc support. Referring to Figure 2, all seeded supports of NaX-1, NaX-2, and NaX-3 exhibit homogenous seed layers. On the other hand, the SEM micrographs of NaX zeolite membranes growth on these seeded supports are shown in Figure 3(a). It can be seen from Figure 3(a) that homogenous zeolite membrane layers are obtained. From Figure 3(a), intercrystalline gaps are more visible for the membrane synthesized by using seed S1 (NaX-1-M1). Meanwhile, membranes prepared by using seed S2 (NaX-2-M2) and seed S3 (NaX-3-M3) showed better zeolite crystals intergrowth. Besides, as shown in Figure 3(a), zeolite crystals formed on the membranes are randomly oriented with well-intergrown layer. However, large octahedral-shaped crystals were observed for NaX-2-M2 and NaX-3-M3 membranes. These crystals might be generated from the synthesis solution during the membrane growth and eventually fall onto the surface of the membrane. These results are consistent with the results reported by Engstrom et al. [28], whereas, they prepared silicalite-1 membranes on gold surface by using zeolite seeds with particle sizes of 60, 165, and 320 nm, respectively. They found that the zeolite grain packing was improved, and the membrane layer became smoother when smaller seed was used during the deposition of the seed layer onto the support.

Figure 3(b) shows the cross-section SEM micrographs of the membranes obtained. Although the NaX zeolite membranes were prepared by using seeds with different sizes, referring to Figure 3(b), all three samples exhibit similar membrane thickness of 17 to 19 μm. The cross-section SEM images shown in Figure 3(b) also displayed the demarcation line between the support and membrane layers. Obviously, penetration of zeolites into the pores of the support was not found. According to Xu et al. [29], in order to inhibit the growth of zeolites in the pores of the support, the zeolite seeds from the seed layer should not penetrate into the pores of the support. Also, the size of the seeds used needs to be larger than the pore size of the support. In this work, α-alumina disc with a pore size of 100 nm was used as support and the size of the seeds used are larger than 100 nm. Therefore, penetration of zeolite seeds into the pores of the support is hardly occurred.

The NaX membranes obtained were proceeded for further analysis by using XRD and the results are shown in Figure 4. It can be seen from Figure 4 that all three membranes exhibit NaX zeolite structure with high degree of crystallinity. NaX-1-M1 membrane prepared by using seed S1 (5500 nm) demonstrated minor peaks belonging to the NaP phase. NaP is known as one of the competing phases of NaX, which may grow as spontaneous product during the synthesis of NaX zeolite [30]. On the other hand, pure NaX phase was obtained for NaX-2-M2 and NaX-3-M3 membranes. A previous study reported that the Na2O/Al2O3 ratio

| NaX membranes      | N2 permeance (x10^-6 mol m^-2 s^-1 Pa^-1) |
|--------------------|------------------------------------------|
| NaX-1-M1 (This work)| 207.52                                   |
| NaX-2-M2 (This work)| 77.38                                    |
| NaX-3-M3 (This work)| 55.93                                    |
| Hasegawa et al. (2002) [16] | 4–9                                   |
| Kusakabe et al. (1999) [17] | ~10                                   |
| Gu et al. (2003) [15]   | 0.2–5.4                                  |

*Membranes synthesized via stage one hydrothermal growth method.
of the synthesis solution used affected the purity of the zeolite formed [31]. It is noted that the Na$_2$O/Al$_2$O$_3$ ratio of the synthesis solution used in this work are varied to produce seed particles with different sizes. Seed S1 was prepared by using synthesis solution with Na$_2$O/Al$_2$O$_3$ ratio of 17, whereas seeds S2 and S3 were...
prepared by using synthesis solution with Na$_2$O/Al$_2$O$_3$ ratio of 19. Meanwhile, the aging duration for synthesizing seeds S1, S2, and S3 were 4, 4, and 48 h, respectively. This could be the main reason for the formation of impurities on NaX-1-M1 membrane. Furthermore, according to Liu et al. [32], synthesis solution with lower Na$_2$O/Al$_2$O$_3$ ratio (lower alkalinity) may inhibit the formation of zeolite crystals. By using a solution mixture with lower alkalinity, the dissolution of silicate in the solution will be reduced and subsequently lead to the poor conversion of the synthesis solution to zeolites. Therefore, the formation of NaP phase as impurities was obtained for NaX-1-M1 membrane.

The defects of NaX zeolite membranes synthesized in this work were evaluated by using N$_2$ gas permeation test. According to Hasegawa et al. [16], NaX membrane with minimum defects should attain N$_2$ gas permeance within the range from 4 to 9 × 10$^{-8}$ mol m$^{-2}$s$^{-1}$Pa$^{-1}$. Table 2 shows the N$_2$ permeation test results of the NaX membranes synthesized in this work and those values reported in the literature. Referring to Table 2, all three NaX membranes exhibit higher N$_2$ permeance values than that of the literature data. These results show that the membranes obtained in this work did not fall within the minimum defects range. It can be also observed from Table 2 that the membranes prepared by using smaller seeds demonstrated lower N$_2$ permeance values. This result exhibits that the defects formed on NaX-3-M3 membrane were less pronounced as compared to the NaX membranes synthesized by using larger seeds (NaX-1-M1 and NaX-2-M2). The formation of defects on the membranes could also be attributed to the absence of the closely packed seed layer and therefore, caused the formation of larger grains which resulted in less intergrown membrane layer [33].

Furthermore, referring to the SEM images shown in Figure 3(a), NaX-1-M1 membrane demonstrates intercrystalline gaps in between the zeolite crystals formed on the membrane layer. This could be due to the larger seeds used for the formation of seed layer, and thus created more gaps in between the particles which affected the growth of the zeolite membrane. On the other hand, zeolite membrane growth on the seed layer formed by using smaller seeds exhibited greater membrane intergrowth. This is mainly due to the presence of lesser gaps between the seeds. In addition, smaller seeds contained larger surface area and thus, can provide adequate contact area to the synthesis solution for the growth of the membrane.

### 3.2. Multi-stage growth of zeolite NaX membranes

In the previous section, although one-stage hydrothermal synthesis method was able to produce continuous NaX membranes, these membranes were not able to achieve minimum defects level based on the N$_2$
permeance results obtained (Table 2). Therefore, multi-stage hydrothermal synthesis method was applied on the formation of NaX zeolite membranes.

Figure 6. Continued.

Figure 5(a) shows the SEM micrographs of NaX-1-M1 membrane after every synthesis stage, up to three stages. It can be observed from the images that
the zeolite intergrowth was improved and the intercrystalline gaps were reduced after second and third synthesis stages. Similar results were also observed for NaX-2-M2 and NaX-3-M3 membranes as shown in Figure 5(b, c), respectively. Referring to Figure 5, the size of the crystals formed on the membrane layers increases with the number of synthesis stage. This result indicates that the growth of zeolite layer was improved after each of the synthesis stage. Nevertheless, it can be found from Figure 5 that non-uniform membrane layers were observed along with the synthesis stage. This result could be attributed to

---

**Figure 6.** SEM cross section images of NaX zeolite membranes synthesized at different stages (a) NaX-1-M1, (b) NaX-2-M2 and (c) NaX-3-M3.

**Figure 7.** XRD patterns of NaX-1-M1 membranes synthesized at different stages. Solid black circle (●) represents NaP phase.
the formation of fresh crystals from the synthesis solution, which eventually fell and incorporated onto the membrane layer.

Figure 6 shows the cross-section SEM images of NaX-1-M1, NaX-2-M2, and NaX-3-M3 membranes. From Figure 6, increment in membrane thickness of about 7, 9, and 13 µm from synthesis stage one to stage three was obtained for NaX-1-M1, NaX-2-M2, and NaX-3-M3 membranes, respectively, mainly due to the growth of the zeolite crystals. Besides, as shown in Figure 5, the size of the crystals formed on the membrane layer increases with the synthesis stage, which subsequently contributed to the increment of the membrane thickness. Furthermore, it was also found that the thickness of the membrane layer increased as the particle size of the seeds used to form the seed layer decreased. This result indicates that higher zeolite growth rate can be achieved by using smaller seeds, mainly attributed to the higher surface area of the seeds that exposed to the reactant mixture during synthesis.

In order to confirm the crystallinity and structure of the zeolite membranes produced, the membranes were further characterized by using XRD. The XRD patterns of the membranes synthesized via multi-stage hydrothermal growth are shown in Figures 7–9. Referring to Figures 7–9, NaX zeolite is the dominant

Table 3. N$_2$ permeance results of NaX membranes synthesized via stage two and stage three hydrothermal growth method.

| Membrane | Synthesis stage | N$_2$ permeance (x10$^{-8}$ mol m$^{-2}$ s$^{-1}$ Pa$^{-1}$) |
|-----------|-----------------|----------------------------------------------------------|
| NaX-1-M1  | two             | 89.67                                                    |
|           | three           | 4.31                                                     |
| NaX-2-M2  | two             | 29.84                                                    |
|           | three           | 1.74                                                     |
| NaX-3-M3  | two             | 26.46                                                    |
|           | three           | 1.19                                                     |

Figure 8. XRD patterns of NaX-2-M2 membranes synthesized at different stages. Solid black circle (●) represents NaP phase.

Figure 9. XRD patterns of NaX-3-M3 membranes synthesized at different stages. Solid black circle (●) represents NaP phase.
phase for all the membrane layers. However, it can be observed from Figures 7–9 that the NaP phase is presented in all three synthesis stages for NaX-1-M1 membrane. Meanwhile, for NaX-2-M2 and NaX-3-M3 membranes, insignificant NaP phase was found at the second and third synthesis stages. It was also observed that the peak's intensity of NaP phase increased with the synthesis stage which indicated that the NaP zeolite formed on the membrane became more noticeable as the membrane underwent more synthesis stages.

Table 3 shows the \( \text{N}_2 \) permeance results obtained for the membranes synthesized via stage two and stage three hydrothermal growth method. From Table 3, the \( \text{N}_2 \) permeance of all membranes reduced with the number of synthesis stages, which indicates that the defects were reduced after each synthesis stage. After the third synthesis stage, the \( \text{N}_2 \) permeance values obtained were \( 4.31 \times 10^{-8} \text{ mol m}^{-2}\text{s}^{-1}\text{Pa}^{-1} \), \( 1.74 \times 10^{-8} \text{ mol m}^{-2}\text{s}^{-1}\text{Pa}^{-1} \), and \( 1.19 \times 10^{-8} \text{ mol m}^{-2}\text{s}^{-1}\text{Pa}^{-1} \) for NaX-1-M1, NaX-2-M2, and NaX-3-M3 membranes, respectively. These values are within or even lower than the range of the minimum defects of NaX membrane \( (4.9 \times 10^{-8} \text{ mol m}^{-2}\text{s}^{-1}\text{Pa}^{-1}) \) reported by Hasegawa et al. [16]. The presence of small percentage of NaP phase on the membrane layer after the third synthesis stage could contribute to the reduction of the gas permeance value, mainly due to the smaller pore size of NaP zeolite (~2.9 Å) as compared to the pore size of NaX zeolite (~2.4 Å). However, since NaX zeolite was identified as the dominant phase in the membrane layer, the influence of NaP phase on the \( \text{N}_2 \) gas permeance can be considered small.

Overall, the mechanism of membrane layer growth via secondary hydrothermal synthesis method involves the growth of seed layer deposited on the support, followed by the transformation of the precursor composed of seed crystals into a dense form [34]. In this study, it can be concluded that the size of the seed affected the growth of NaX zeolite membranes and thicker membranes with lower defects level were formed by using smaller seeds. Figure 10 illustrates the effect of seed particle size on the formation of NaX zeolite membranes.

4. Conclusion

The effects of synthesis parameters including seed particle size and synthesis stage toward the formation of NaX zeolite membranes was successfully investigated in this work. The results showed that the multi-stage synthesis technique has reduced the defects level of the membrane via \( \text{N}_2 \) gas permeation test. \( \text{N}_2 \) gas permeation results indicated that, after the third stage hydrothermal synthesis, the gas permeance values achieved were comparable with the reported values of \( \text{N}_2 \) permeance for NaX membranes with minimum defects. However, increasing the hydrothermal synthesis stage has increased the impurity phase in the form of zeolite NaP in the membrane layer.

Acknowledgments

This work was financially aided and supported by the Petroleum Research Fund (PRF), Universiti Teknologi PETRONAS, and PETRONAS Research Sdn. Bhd. Besides, the technical support provided by CO2 Research Centre (CO2RES), Institute of Contaminant Management, Universiti Teknologi PETRONAS is duly acknowledged.

Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This work was supported by the Petroleum Research Fund (PRF) [Cost Center: E025 CO2502015331].
References

[1] Pina MP, Mallada R, Arruebo M, et al. Zeolite films and membranes. Emerging applications. Microporous Mesoporous Mater. 2011;144(1–3):19–27.

[2] Arruebo M, Mallada R, Pina MP. Zeolite membranes: synthesis, characterization, important applications, and recent advances. In: Pabby AK, Rizvi SSH, Sastre AM, editors. Handbook of membrane separation: chemical, pharmaceutical, food, and biotechnological applications. Boca Raton, FL: CRC Press; 2009. p. 269–323.

[3] Wang Z, Yan Y, Zeolite Thin Films and Membranes: From Fundamental to Applications. In: Xiao FS, Meng X, editors. Zeolites in Sustainable Chemistry. Green Chemistry and Sustainable Technology. Springer, Berlin, Heidelberg. 2016. p. 435–472.

[4] Kosinov N, Gascon J, Kapteijn F, et al. Recent developments in zeolite membranes for gas separation. J Membr Sci. 2016;499:65–79.

[5] Caro J, Noack M, Kolsch P, et al. Zeolite membranes – state of their development and perspective. Microporous Mesoporous Mater. 2000;38:3–24.

[6] Feng C, Khulbe KC, Matsura T, et al. Recent progress in zeolite/zeotype membranes. J Membr Sci Res. 2015;1:49–72.

[7] Collins F, Rozhkovskaya A, Outram JG, et al. A critical review of waste resources, synthesis, and applications for Zeolite LTA. Microporous Mesoporous Mater. 2020;291:109667.

[8] Suzuki H. Composite membrane having a surface layer of an ultrathin film of cage-shaped zeolite and processes for production thereof,” United States patent US 4,699,892. 1987 Oct 13.

[9] Wee SL, Tye CT, Bhatia S. Membrane separation process – pervaporation through zeolite membrane. Sep Purif Technol. 2008;63:500–516.

[10] Coronas J, Santamaria J. Separations using zeolite membranes. Sep Purif Methods. 1999;28(2):127–177.

[11] Caro J, Noack M. Zeolite membranes – recent developments and progress. Microporous Mesoporous Mater. 2008;115(3):215–233.

[12] Lin YS. Microporous and dense inorganic membranes: current status and prospective. Sep Purif Technol. 2001;25:39–55.

[13] Raso R, Tovar M, Lasobras J, et al. Zeolite membranes: comparison in the separation of H2O/H2/CO2 mixtures and test of a reactor for CO2 hydrogenation to methanol. Catal Today. 2020. DOI:10.1016/j.cattod.2020.03.014 in press.

[14] Liu B, Kita H, Yogo K. Preparation of Si-rich LTA zeolite membrane using organic template-free solution for methanol dehydration. Sep Purif Technol. 2020;239:116533.

[15] Gu X, Dong J, Nenoff TM. Synthesis of defect-free FAU-type zeolite membranes and separation for dry and moist CO2/N2 mixtures. Ind Eng Chem Res. 2005;44(4):937–944.

[16] Hasegawa Y, Tanaka T, Watanabe K, et al. Separation of CO2–CH4 and CO2–N2 systems using ion-exchanged FAU-type zeolite membranes with different Si/Al ratios. Korean J Chem Eng. 2002;19(2):309–313.

[17] Kusakabe K, Kuroda T, Uchino K, et al. Gas permeation properties of ion-exchanged faujasite-type zeolite membranes. AIChE J. 1999;45:1220–1226.

[18] Weh K, Noack M, Sieber I, et al. Permeation of single gases and gas mixtures through faujasite-type molecular sieve membranes. Microporous Mesoporous Mater. 2002;54:27–36.

[19] Fujiki J, Yogo K. Effect of counter cations on the hydrothermal conversion of FAU-type zeolites into ABW or ANA and their potential applicability for CO2/N2 separation. J Chem Eng Jpn. 2020;53(1):17–23.

[20] Zhou C, Zhou J, Huang A. Seeding-free synthesis of zeolite FAU membrane for seawater desalination by pervaporation. Microporous Mesoporous Mater. 2016;234:377–383.

[21] Li S, Tuan VA, Falconer JL, et al. X-type zeolite membranes: preparation, characterization, and pervaporation performance. Microporous Mesoporous Mater. 2002;53(1–3):59–70.

[22] Zhu G, Li Y, Zhou H, et al. Microwave synthesis of high-performance FAU-type zeolite membranes: optimization, characterization and pervaporation dehydration of alcohols. J Membr Sci. 2009;337(1-2):47–54.

[23] Sato K, Sugimoto K, Nakane T. Synthesis of industrial scale NaY zeolite membranes and ethanol permeating performance in pervaporation and vapor permeation up to 130°C and 570 kPa. J Membr Sci. 2008;310(1–2):161–173.

[24] Kita H, Inoue T, Asamura H, et al. NaY zeolite membrane for the pervaporation separation of methanol – methyl tert-butyl ether mixtures. Chem Commun. 1997;1:45–46.

[25] Maghsoudi H. Defects of zeolite membranes: characterization, modification and post-treatment techniques. Sep Purif Rev. 2016;45(3):169–192.

[26] Wu T, Diaz MC, Sheng Y, et al. Influence of propane on CO2/CH4 and N2/CH4 separations in CHA zeolite membranes. J Membr Sci. 2015;473:201–209.

[27] Chew TL, Ahmad AL, Bhatia S. Ba-APSO-34 membrane synthesized from microwave heating and its performance for CO2/CH4 gas separation. Chem Eng J. 2011;171(3):1053–1059.

[28] Engstrom V, Mihailova B, Hedlund J, et al. The effect of seed size on the growth of silicate-1 films on gold surfaces. Microporous Mesoporous Mater. 2000;38:51–60.

[29] Xu X, Yang W, Liu J, et al. Synthesis of NaA zeolite membranes from clear solution. Microporous Mesoporous Mater. 2001;43(3):299–311.

[30] Cundy CS, Cox PA. The hydrothermal synthesis of zeolites: history and development from the earliest days to the present time. Chem Rev. 2003;103:663–701.

[31] Ma J, Shao J, Wang Z, et al. Preparation of zeolite NaA membranes on macroporous alumina supports by secondary growth of gel layers. Ind Eng Chem Res. 2014;53(14):6121–6130.

[32] Liu L, Du T, Li G, et al. Using one waste to tackle another: preparation of a CO2 capture material zeolite X from laterite residue and bauxite. J Hazard Mater. 2014;278:551–558.

[33] Xomeritakis G, Gouzinis A, Nair S, et al. Growth, microstructure, and permeation properties of supported zeolite (MFI) films and membranes prepared by secondary growth. Chem Eng Sci. 1999;54:3521–3531.

[34] Matsuda M, Zeolite membrane. In: Naito M, Yokoyama T, Hosokawa K, et al., editors. Nanoparticle technology handbook. Amsterdam, Netherlands: Elsevier BV; 2018. p. 539–542.