CHAPTER VII
CONCLUSIONS AND RECOMMENDATIONS

7.1 Conclusions

0.1-0.2 μm NaA zeolite was successfully synthesized using silatrane and alumatrane precursor via sol-gel process, seeding and microwave techniques. Effect of hydroxide ion concentration, seed amount as well as heating temperature on the size and morphology of the zeolite product were studied using the composition of SiO_2:Al_2O_3:xNa_2O:410H_2O; $3 \leq x \leq 6$, comparing with the direct method in which silica and alumina are used as precursors. It was found that hydroxide ion concentration affects the crystal size and heating time whereas higher amounts of seeds provide smaller sizes of NaA zeolite. The uniform of zeolite product can be synthesized using either higher temperature for short time or lower temperature for long time. The best condition for synthesizing the smallest size and the most homogeneous of NaA zeolite from the indirect method is to use the composition of SiO_2:Al_2O_3:3Na_2O:410H_2O with 3 wt% crystal seed at 80°C microwave heating for 6 h whereas the best condition for the direct method is to use the composition of SiO_2:Al_2O_3:3Na_2O:410H_2O with 3 wt% crystal seed at 60°C microwave heating for 10 h. The synthesized NaA zeolite was characterized using XRD and SEM.

The synthesized zeolite membranes were synthesized on an α-Al_2O_3 support via hydrothermal treatment (microwave or electrophoretic technique) and on secondary growth method. NaA zeolites from direct method were used as seeds for the secondary growth using vacuum seeding technique to coat on the porous tubular α-Al_2O_3 support before NaA zeolite membrane synthesis. The membrane synthesis was studied via multistage synthesis to improve quality of the membrane. The higher film thickness, the lower flux obtained. The higher temperature also caused impurity phase on membrane during synthesizing, resulting in low separation performance.

The optimum conditions were found to be 3 g/l of seed solution containing 0.5 μm zeolite crystal seed at 0.0325 MPa seeding pressure for 2 min seeding time and 363 K microwave temperature, for 15 min on the supports without an

intermediate layer and 20 min on the one with an intermediate layer. The flux and the separation factor obtained were 1.6 kg/m^2.h and 1760.5, respectively, for the substrate without an intermediate layer. The substrate with an intermediate layer shows a better flux and separation factor, 1.7 kg/m^2.h and 6532.7, respectively.

Oriented, uniform, and dense NaA zeolite membranes were also studied on an alumina substrate by seeding and electrophoretic techniques. Both the morphology and the performance of the membranes were improved. More homogeneous and more uniform crystals were formed on the alumina support surface, with a size of 1-3 μm. With increasing applied electrical potential, smaller crystal sizes and denser NaA zeolite membranes were obtained with better performance. A longer seeding time generated a too thick NaA zeolite membrane, resulting in cracks, causing poorer performance. All of the synthesized membranes were oriented-like membranes, confirmed by the strong {222} peak in the XRD pattern. The best NaA zeolite membrane was prepared using 1.5 min of seeding time on the alumina support before crystallizing at 333 K under an electrical field of 2 V, giving a separation factor > 10,000 and 0.61 kg/m^2.h in flux.

7.2 Suggestion for Further Work

The suggestions for further work are;

1. Development of a fabrication method for a thinner zeolite layer to obtain higher flux but maintain high separation factor.

2. Further developments of NaA zeolite membranes with more economically feasible fluxes are required to extend implementation of NaA zeolite membrane in the ethanol separation industry.