

# SYNTHESIS OF ZEOLITE A MEMBRANES: A NEW APPROACH

S.P.J. Smith\*, R.D. Sanderson\* and J.C. Jansen \*

\* Institute of Polymer Science, University of Stellenbosch, South Africa

\* Laboratory of Organic Chemistry and Catalysis, Delft University of Technology, The Netherlands

## ABSTRACT

A new approach was used to synthesize a zeolite A (NaA) membrane. The main consideration here is to achieve a continuous crystallization front of the zeolite at the interface of the individual nutrient pools. These pools are separated by the porous wall of an alumina tube performing as the interface site and as a support. The following parameters were studied, concentration, time and reversal of the source solutions. Mixtures of the nutrients were also used and the results compared.

## INTRODUCTION

In recent years numerous strategies have been adopted to produce a zeolite membrane, for example precipitation crystallization [1] and impregnation crystallization [2] of synthesis mixtures *on* and *in* a porous support, respectively, as well as sol/gel methods [3]. In most such cases the continuity and a constant thickness of the zeolite phase was difficult to achieve. Closure of pinholes requires additional nucleation and crystallization between existing crystals, which actually consume the nutrient phases rather than reacting inertly with respect to the local crystallization in the pinholes. It also resulted in the undesired thickening of the zeolite layer.

A classic CVD technique is used in this study. Individual nutrient pools are placed at both sides of the porous wall of the tube. When the nutrient pools become separated by the first crystal formation, crystallization stops in the crystallization front in the pores, resulting in a thin crystal layer. The driving force required to close pinholes is the very high concentration available in the separated nutrient pools from both sides of the pinhole. This technique, to our best knowledge, has not been previously used in membrane synthesis.

## EXPERIMENTAL

The NaA syntheses were conducted at 80°C on  $\alpha$ -alumina tubes (Atech Innovations GmbH), which had a wall thickness of 2 mm and an outer diameter of 10 mm. The inner and outer compartments, with the individual nutrient pools, were separated by the porous wall of the tube, and both had the same volume.

## RESULTS AND DISCUSSION

The results were based on the amount of crystals grown in the wall, on the outer surface and on the inner surface of the tube, as observed by SEM. A crystal concentration gradient, probably diffusion controlled, was frequently observed across the wall, with the highest concentration of zeolite A crystals on the side of the Si source. The higher the individual nutrient concentration and/or the synthesis time, the higher was the crystal concentration at the crystallization front. The mixtures yielded almost no crystal growth on either of the surfaces of the alumina tube. Hydrocarbon-separation results will be presented.

## CONCLUSIONS

Changing the concentrations of the two nutrient pools could shift the crystallization front. With this technique very high nutrient concentrations can be used. It is noted that, in contrast to the mixtures, individual nutrients wet or activate the surface of the tube, thereby mediating crystal growth on the substrate particles. This is further indicated by the fact that the nucleation point of the crystals is located on the surface of the alumina particles. The absence of crystals on the support in the case of the mixtures, indicates that the nucleation in the gel phase is so much preferred that no growth could be achieved from the alumina surface itself.

### References:

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