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Seeding of porous alumina substrate with MFI zeolite nanocrystals using spin-coating technique

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Introduction:

Zeolite membranes offer a great potential for applications in many challenging separation processes. Controlled thickness of zeolite membrane allows high flux and excellent selectivity due to shape-selective properties of zeolites. Although some zeolite membranes are commercially available; producing cheap, controlled and high-throughput production of zeolite membranes still remains a challenge [1]. The controlled fabrication of zeolite membranes including synthesis time, crystal orientation and thickness under microwave heating have been reported elsewhere [2-4]. One of the approaches to control the nucleation and crystallization of MFI zeolite membrane in the secondary growth microwave-assisted hydrothermal technique is to control the seeds distribution on the surface of the support/substrate. Many methods have been explored to coat the seed crystals onto the substrate surface, including dip-coating [5], rubbing [7] and vacuum seeding [6]. Although these seeding methods improve membrane synthesis modestly, they had their own limitations. To our knowledge, no work has been reported on seeding alumina substrate with MFI zeolite seeds using spin-coating technique. In this research work, MFI zeolite seeds were synthesised by microwave-assisted hydrothermal technique and utilized for seeding the surface of a flat disk shaped alumina substrate using spin-coating seeding technique. The effect of seeding solution concentration and number of repeated spin-coating runs on the coverage of the alumina substrate surface were investigated.

Experimental:

The seed precursor sol was prepared from TEOS (Tetraethyl orthosilicate) and TPAOH (Tetrapropylammonium hydroxide solution) with the following molar ratio 1 TEOS: 0.25 TPAOH: 18 H_2O. Rotary evaporation was used for the prepared sol at 80°C in a 250 mL PFA round-bottom flask to yield a concentrated precursor sol which was then aged at 80^oC for 24hr in a sealed beaker. After that, the concentrated precursor sol was poured, after dilution 10 times in water, on the vessel reactor and placed on the microwave (synthos 3000) for 120 min at 160^oC. Then the synthesized solution was centrifuged at 14000 rpm 5 times, after each time the solution were washed with DI water to bring the pH number to 7. The MFI zeolite seeds were diluted in water and then deposited on the surface of 1-inch diameter alpha-alumina substrate using spin-coating technique with 3000 RPM for 60 seconds followed by drying at 80^oC for 3hours. The concentration of the seed solution and the number of repeated spin-coating can play a vital role on the uniformity and coverage of the seeds on the surface of alumina substrates which in turn will affect the growth and morphology of MFI zeolite membrane.

Results and Discussion:

Figure 1(g) shows the particle size distribution of the synthesised MFI zeolite seeds. The average particle size is around 89 nm. Figure 1(a) displays the surface of a porous alpha-alumina substrate. SEM images of the seeded substrate with 0.1wt% concentration of MFI zeolite seeds solution obtained after one run and 10 runs of spin-coating are shown in figure 1 (b) and (c) respectively. It is clear from Figure 1 (b) that there is a poor coverage of the seeds on the alumina surface. Furthermore, most of these seeds try to fill the pores between the alumina grains with little contribution on covering the grains of the substrate. On the other hand, when the number of repeated spin-coating runs increased to ten times, beside of filling the pores the seeds seems to be able to cover some of the outer big alumina grains as shown in

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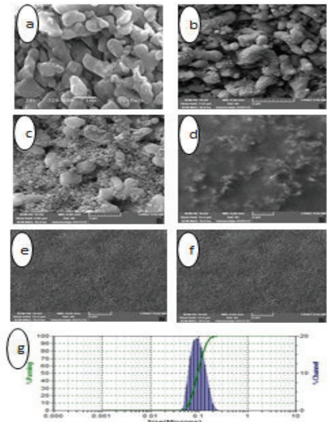

Figure 1 SEM images of (a) surface of alumina substrate, (b) Spin-coating with solution of 0.1wt% seeds concentration for 1 run , (c) Spin-coating with solution 0.1wt% seeds concentration for 10 runs, (d) Spin-coating with solution 1wt% seeds concentration for 1 run. (e) Spin-coating with solution 1wt% seeds concentration for 2 runs, (f) Spin-coating with solution 1wt% seeds concentration for 3 runs, (g) Particle size distribution of MFI zeolite seeds.

figure 1(c). Although, the number of repeated spin-coating increased from one to ten times the MFI zeolite seeds did not fully cover the alumina substrate surface. As a result, the concentration of 0.1%wt MFI zeolite seeds solution is not practical because it requires more than 10 runs of repeated spin-coating in order to reach full coverage for this type of substrate. However, as the concentration was increased from 0.1 wt % to 1 wt %, there was a clear enhancement in the coverage of MFI zeolite seeds as shown in figure 1(d,e&f). In figure 1(d), alumina surface can barely be seen after spin-coating of 1wt% concentration of MFI seeds solution after only one run. Therefore, increasing the number of spin-coating runs completely covered the surface of the substrate with a continuous layer of seeds thus enabling to eliminate the difference between different roughness and morphology of the substrates as shown in figure 1 (e&f). Consequently, this type of surface modification will have an important role on the formation of a continuous and defect free MFI zeolite active layer/membrane.

Conclusion:

The results of this study indicate that spin-coating is a viable technique to deposit a continuous layer of MFI zeolite nanocrystal seeds on the surface of porous alumina substrate. The influences of MFI zeolite seeds concentration as well as number of spin-coating runs on the coverage of the 1-inch diameter alumina substrate have been investigated. It is shown that 1wt% seed solution concentration with one run or more gave a full coverage.

The next stage of this research work is to grow and evaluate MFI zeolite layers using the different seeded substrates prepared with different seed concentrations and spin-coating runs. This study will allow developing a good understanding on the impact of seeding on fabrication of MFI zeolite layer. Further work is underway to achieve good coverage by selecting an optimum value of dielectric constant, different sizes of MFI seeds and substrate pre-treatment.

Keywords: MFI Zeolite Seeding, Spin-Coating, Secondary Growth, Zeolite membrane