Self Assembly of Zeolite Nanoplatelets by Dip Coating

Kumar Varoon, Michael Tsapatsis and Lorraine F. Francis

Dept. of Chemical Engineering & Materials Science
University of Minnesota, Minneapolis, MN 55455

Presented at the 15th International Coating Science and Technology Symposium,
September 13-15, 2010, St. Paul, MN[1]

Zeolites are crystalline microporous aluminosilicates with pore size in the range of 3-20Å. Due to their porous structure, they can be used as molecular sieves. Also, negatively charged alumina sites make them a robust catalyst. For both of these applications, thin zeolite films are desired on top of a suitable support. Traditionally, thin zeolite films are fabricated by hydrothermal growth of the zeolite crystals on a support. However, this technique is not suitable for large scale production of zeolite film. Also, hydrothermal growth of zeolite on a substrate is sensitive to the nucleation and growth steps; therefore, synthesis of a thin defect-free zeolite film by this technique is often challenging. On the other hand, deposition of zeolite precursor particles by coating processes allows for fabrication of extremely thin film; thereby, reducing the transport resistance of molecules through the film and saving considerable cost.

Here, we report the fabrication of a thin zeolite film by dip coating nanoplatelets of a zeolite ITQ-1 on a porous stainless steel tube. These nanoplatelets were synthesized by hydrolysis and condensation of fumed silica in the presence of structure directing agents followed by hydrothermal treatment in an autoclave at 150°C. Figure 1a shows the scanning electron microscope (SEM) image of as synthesized ITQ-1 platelets. Figure 1b shows the wide angle X-ray diffraction (WAXS) pattern of the as synthesized and calcined ITQ-1 powder. The sharp peaks in the WAXS suggest that these platelets are highly crystalline.

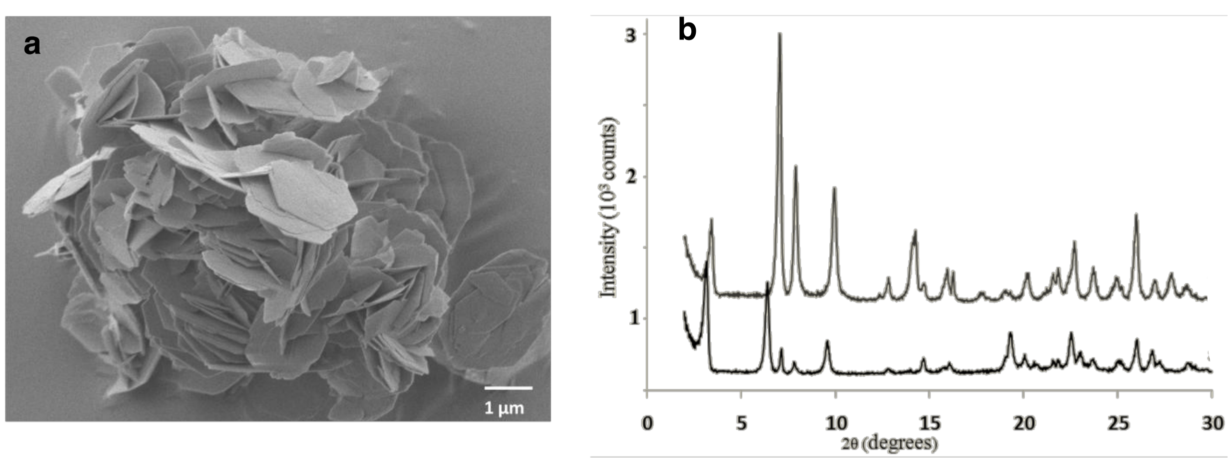

Figure 1: (a) SEM image of as made ITQ-1 zeolite platelets. (b) WAXS data on powder ITQ-1; bottom: as synthesized ITQ-1; top: calcined ITQ-1.

---

[1] Unpublished. ISCST shall not be responsible for statements or opinions contained in papers or printed in its publications.

The ITQ-1 platelets were dispersed in water and sonicated to prepare segregated colloidal suspension to facilitate their oriented deposition on a support. Porous stainless steel (SS) tube was selected as the support to allow the use of the zeolite coating for high temperature catalysis and molecular sieving (up to 600°C). The assembly of the zeolite platelets on the SS support was carried out by the dip coating process. Figure 2a shows the image of the porous SS support and Figure 2b shows the surface morphology of the support. Figure 2c shows the schematic of the linear translator used for the dip coating.

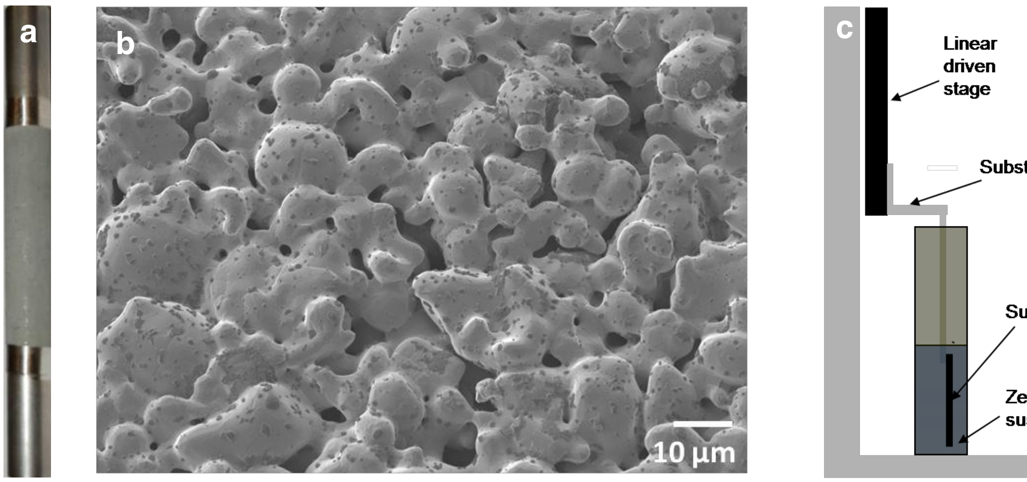

Figure 2: (a) Porous SS tubular support for the coating. (b) SEM image of the surface of the SS support. (c) Schematic of the dip coating setup.

Attempts to create a micron thick ITQ-1 film on top of porous SS support led to pinhole defects due to high surface roughness of the support (Figure 3a). To avoid pinhole defect, binder Poly(vinyl chloride) (PVA) was added to the coating. This resulted in a uniform coating of ITQ-1 without any pinhole defect (Figure 3b). However, the orientation of the platelets in the film was random. Random orientation of the zeolite crystals is detrimental to molecular sieving application; hence, an additional coating of aqueous suspension of ITQ-1 was applied on top of the first coating to obtain an oriented film. Figure 3c shows the SEM image of the oriented ITQ-1 layer. Cross-section of the film investigated under SEM revealed that the top layer of the film was oriented (Figure 3d).

To conclude, we report an oriented film of zeolite nanoplatelets on a porous SS support. Attempts to make a 1 µm thick zeolite film by dip coating aqueous suspension of ITQ-1 on the tubular SS support led to pinhole defects. Addition of a PVA in ITQ-1 solution solved the pinhole issue; however, orientation of ITQ-1 was random. Oriented deposition of ITQ-1 was obtained from aqueous suspension of ITQ-1 on top of the 'ITQ-1+PVA' layer.

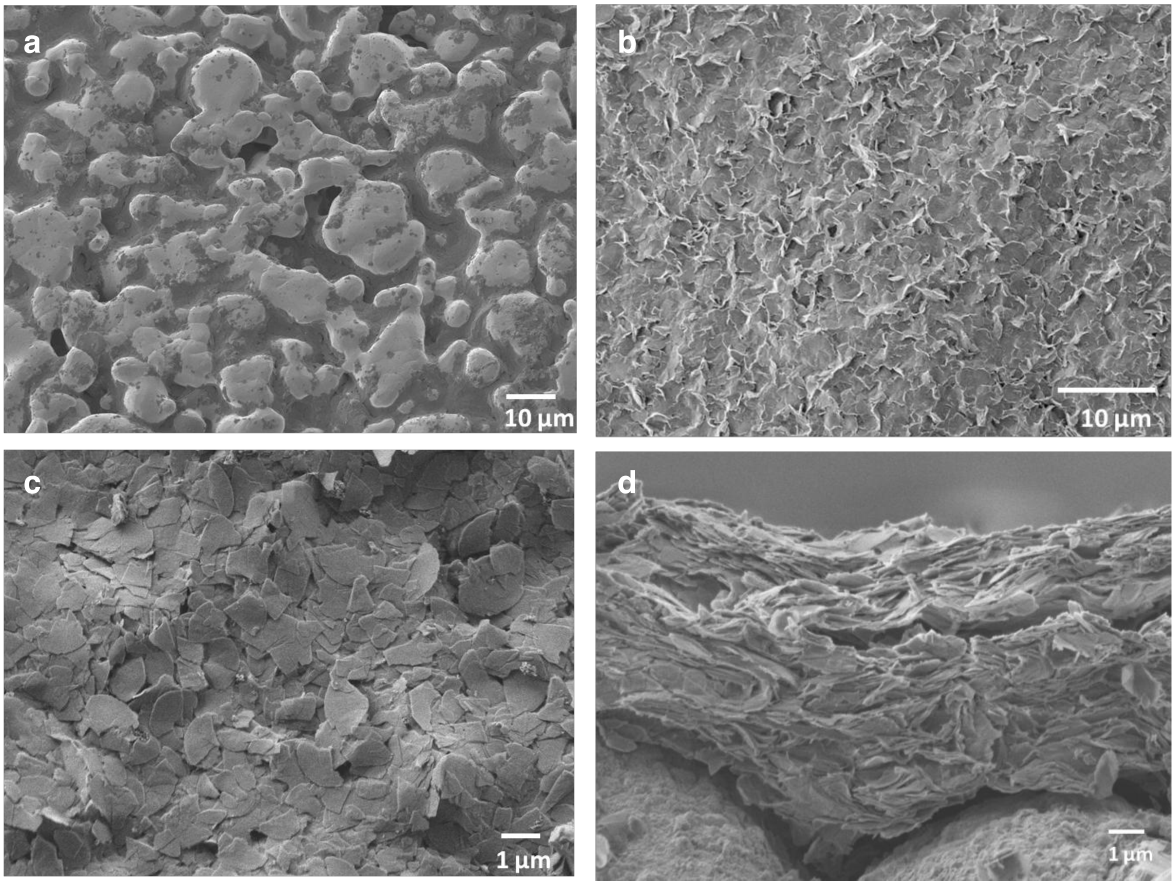

Figure 3: SEM images of (a) pinhole defects on the bare SS tube, (b) uniform coverage of ITQ-1 on the support but with random orientation, (c) oriented deposition of ITQ-1 on top of the 'PVA+ITQ-1' layer, (d) cross section of the film.