

Supplementary Information for

Ceramic Membranes for Separation of Proteins and DNA by In Situ Growth of Alumina Nanofibres inside Porous Substrates

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Experimental Details

Preparation of boemite nanofibres membrane. Alumina substrates were dispersed into 50 mL of HNO₃ solution (1 wt%). They were sonicated for about 10 min, and then washed with deionized water. The substrates were dried in air at 393 K for 12 h. The boemite nanofibres membranes were prepared by a steam-assisted, solid-phase, wet-gel conversion process. Typically, 30 g of Al(NO₃)₃•9H₂O were dissolved in 100 mL of de-ionized water. Then 20 mL of an aqueous solution of tetraethylammonium hydroxide (TEAOH, 20 wt%) was added dropwise to the Al(NO₃)₃ solution at room temperature with stirring to control the desired pH value at 5.0. The resulting solid precipitates were recovered by filtration.

The alumina substrates were coated with the as-synthesized, solid, cake-like, wet gel, and then transferred into a Teflon vessel (125 mL), where 2 mL of water were poured into bottom of vessel and physically separated from the substrates. After reaction at 170°C for 72 h in the Teflon vessel, the resulting membranes were washed with de-ionized water and air dried.

Separation of Proteins and DNA. For the tangential-flow filtration, a 0.1% of protein solution ($C_{BSA}/C_{BHB}=1$) was used to test the membrane performance. The buffer solutions were prepared by mixing 0.01 M potassium dihydrogen phosphate solution and 0.01 M disodium hydrogen phosphate solution and these were then adjusted to desired pH values by adding small amounts of HCl and NaOH. In tangential-flow filtration, the fluid is pumped tangentially along the surface of the membrane (at $3 \text{ mL}\cdot\text{min}^{-1}$ in the present study), and an applied pressure (50 kPa) forces a portion of the fluid through the membrane to the filtrate side. As with conventional filtration, particulates and macromolecules that are too large to pass through the membrane pores are retained on the upstream side. However, the use of tangential flow means that the retained components do not build up at the surface of the membrane. Instead, they are swept along by the tangential flow, which makes it an ideal process for finer sized-based separations. The unique membrane produced in this work, which has its separation layer protected by the support, should be resistant to wear caused by tangential flow.

Moreover, the membrane performance was tested by depth filtration with a 0.1% ferritin solution and a 10 ng/ μL of DNA solution. The membrane was mounted in a membrane module in a depth filtration system. In each case, 10 mL of solution were suction-filtered through the fibre membrane and corresponding filtration time was recorded. A trans-membrane pressure of 50 kPa was created by a vacuum pump to drive the permeation.

Characterisation. The size and concentration of the DNA was examined by agarose-gel electrophoresis. Gels were made with 1.0 % agarose in Tris-borate-EDTA buffer containing ethidium bromide. Samples were prepared by mixing 20 μL of the DNA

solution with 4 μ L of 6 $\times$ Gel loading buffer. DNA Molecular Weight Marker (Roche) was loaded into the well of the gel as a reference. Gels were run at 60 V for 40-60 min in a Mini-Sub Cell GT (BioRad, Hercules, CA) with a PowerPac Basic power supply (BioRad). Gel images were captured by using a UV transilluminator.

The protein concentrations before and after filtration were determined by using UV-visible spectroscopy (Cary100, Varian Inc.). The protein concentration was measured, by comparison with standards, at wavelengths of 300 nm for ferritin, at 278 nm for BSA and 408 nm for BHb.

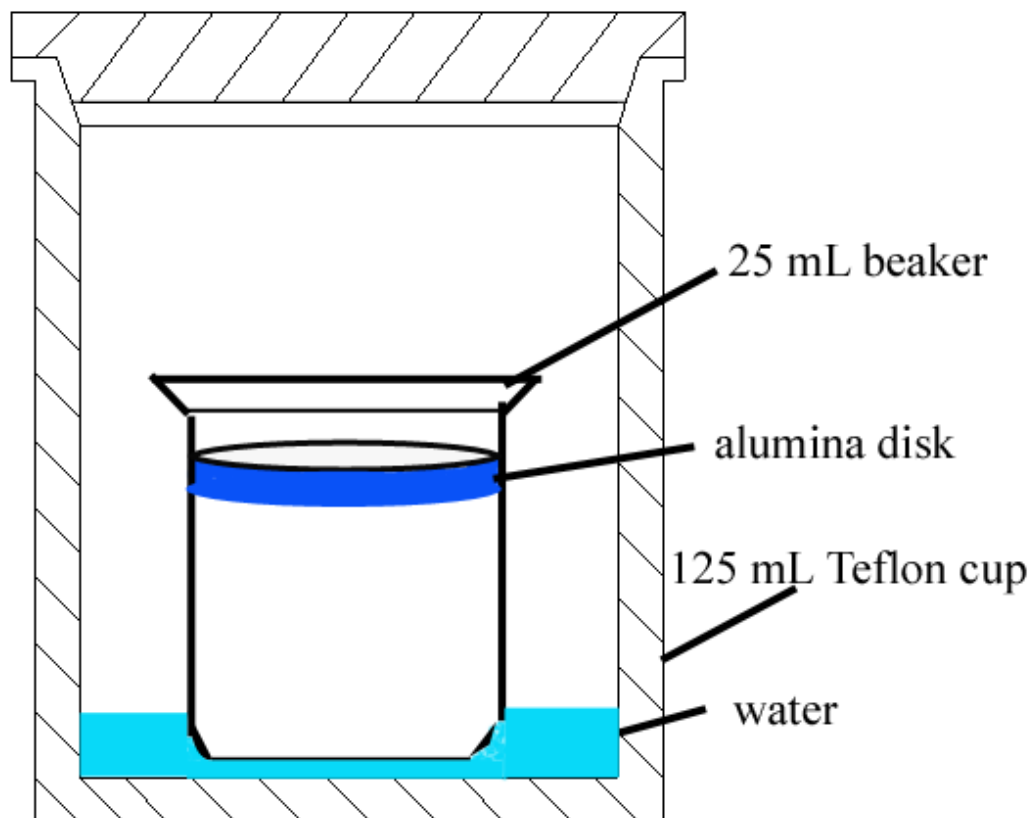


Figure S1 Schematic of the reaction set-up for the steam-assisted conversion process, during which the amorphous precipitate transformed into boehmite (AlOOH) fibres in the pores of the support. An alumina disk (the porous alumina support) loaded with amorphous precipitate was put into an autoclave with a Teflon liner (Parr, USA), and was physically separated from 2 g of water in bottom of vessel by means of a glass beaker. The steam-assisted conversion process was conducted at 170 °C for 72 h.

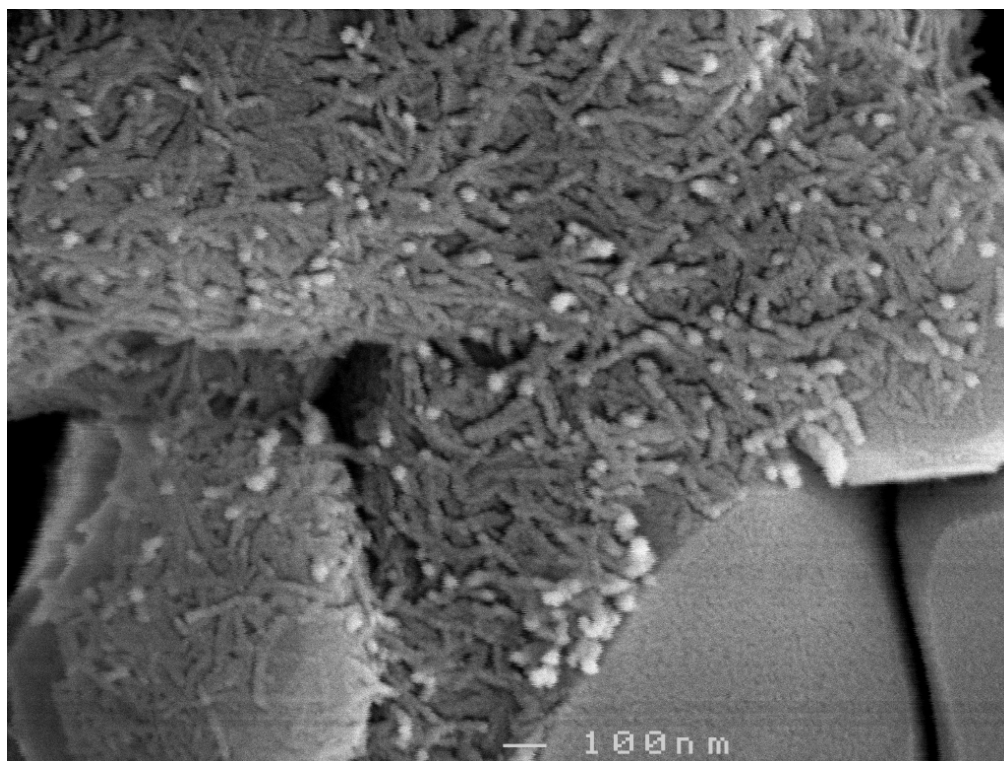


Figure S2 FESEM image of the membrane from the top. The boehmite (AlOOH) fibres, which are about 100-300 nm long and about 10 nm thick, are anchored in the pores of the support. Because of the conductive gold coating required for the SEM imaging, the fibres appear larger than their true size (as is evident from the TEM image of Figure S3).

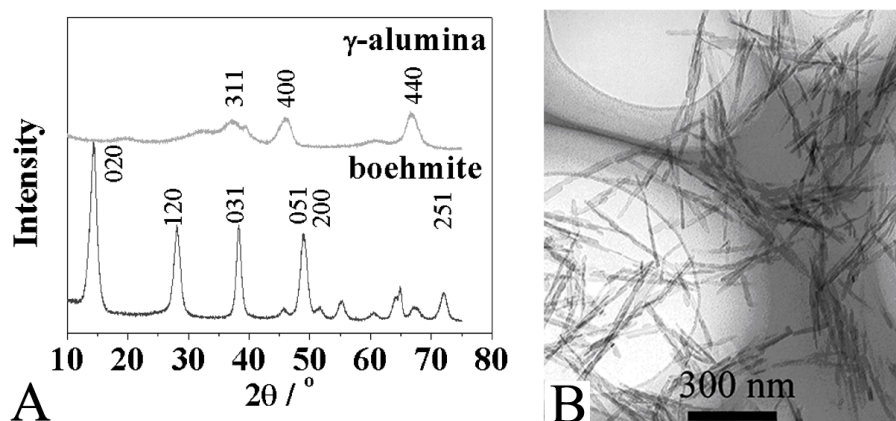


Figure S3 Crystal phase changes and morphology of nanofibres. (A) XRD patterns of the pure boehmite nanofibres (bottom) and the sample obtained by calcining the boehmite nanofibres at 500 °C (top). (B) TEM image of the nanofibres. XRD patterns of the pure fibres: the boehmite nanofibres obtained from amorphous aluminium-hydroxide precipitated under the same conditions as the *in situ* formation (bottom), and the sample obtained by calcining the boehmite nanofibres at 500 °C (top). It is evident that the boehmite phase was converted to the γ -alumina phase by the calcination process. These pure fibres were characterized because it was very difficult to recognize the peaks of boehmite or γ -alumina phases in the XRD patterns of complete membranes because of the small content of the fibres in the membranes and the strong diffraction peaks of the α -alumina support.