## Supplementary Materials: A Reliable Method for the Preparation of Multiporous Alumina Monoliths by Ice-Templating

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## Acid Source and Acid/Al Ratio



Figure S1. Small angle XRD for samples synthesized using different peptizing acids.



**Figure S2.** (**a**) Nitrogen adsorption – desorption isotherms and (**b**) pore size distributions (desorption branch) for samples synthesized using different peptizing acids.



Figure S3. Small angle XRD for samples synthesized using different concentrations of nitric acid.



**Figure S4.** (**a**) Nitrogen adsorption – desorption isotherms and (**b**) pore size distributions for samples synthesized using different HNO<sub>3</sub>/Al ratios.



**Impact of Peptizing Duration and Temperature** 

**Figure S5.** (**a**,**b**) Nitrogen adsorption—desorption isotherms and (**c**,**d**) pore size distributions for samples synthesized at (**a**,**c**) 85 °C and (**b**,**d**) 100 °C.



Figure S6. TEM images of the pseudoboehmite respectively after (a) 20 h at 85 °C and (b) 6 h at 100 °C.

Powder/Monolith Comparison



Figure S7. Comparative small angle XRD of powder and monolith synthesized under optimized conditions.



**Figure S8.** Comparative (**a**) Nitrogen adsorption—desorption isotherms and (**b**) corresponding pore size distributions of powder and monolith synthesized under optimized conditions.

## Water Content



**Figure S9.** Impact of water content on (**a**) Nitrogen adsorption—desorption isotherms and (**b**) corresponding pore size distributions.

## **Thermal Treatment**



**Figure S10.** TGA-DSC analysis of ice-templated sample after sublimation of the ice crystals. An intense exothermic peak is typical of organics combustion.

![](_page_3_Figure_8.jpeg)

**Figure S11.** Impact of the thermal treatment (**a**) Nitrogen adsorption—desorption isotherms and (**b**) pore size distribution.

![](_page_4_Figure_1.jpeg)

Figure S12. Mercury porosimetry of alumina monolith.