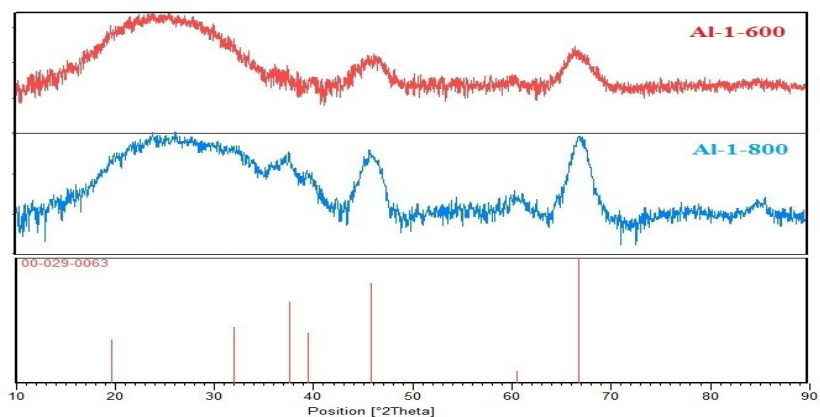


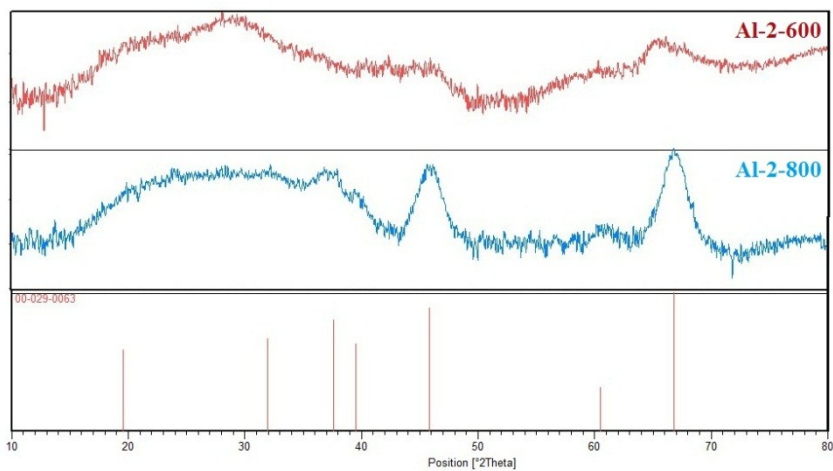
## Supplementary

### Experimental Section

Aluminum oxides were synthesized as follows: (a)  $\text{Al}(\text{O}^i\text{Pr})_3$  precursor: Freshly prepared  $\text{Al}(\text{O}^i\text{Pr})_3$  (0.05 mol) was dissolved in 2-propanol (58 mL) and heated under stirring to 83 °C. Subsequently, an aqueous solution of acetic acid (1.7 mmol in 2.7 mL  $\text{H}_2\text{O}$ ) was added to the resulting clear pale yellow solution. The temperature was then increased and the slurry was aged statically at reflux temperature for 24 h. The solvent was recovered by distillation with a rotary evaporator and the obtained sol was dried at 120 mbar and 70 °C for 3 h. (b)  $[\text{Al}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_3]$  precursor:  $[\text{Al}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_3]$  (0.06 mol) was dissolved in 2-methoxyethanol (117 mL) and heated under stirring to 125 °C. Subsequently, an aqueous solution of acetic acid (2.1 mmol in 3.2 mL  $\text{H}_2\text{O}$ ) was added to the resulting clear solution. The temperature of the mixture was then increased and the slurry was aged under static conditions at reflux temperature for 24 h. The solvent was recovered by distillation with a rotary evaporator and the obtained sol was dried at 2 mbar and 70 °C for 3 h. Calcination of the samples were carried out in static air by heating the samples to 600 and 800 °C at a rate of 1 °C/min and maintaining the final temperature for 4 h. The structures of the samples were characterized by  $\text{N}_2$  adsorption-desorption, XRD, and TEM. The  $\text{N}_2$  adsorption and desorption isotherms were measured using a Quantachrome Autosorb-iQ-MP gas sorption analyzer; the samples were previously outgassed at 250 °C for 6 h. The Brunauer–Emmett–Teller (BET) surface areas were determined from the adsorption data over different relative pressure ranges all between 0.03–0.28 while ensuring compliance with the consistency criteria.<sup>[41]</sup> The total pore volume was calculated from the amount adsorbed at a relative pressure of about 0.99. The pore diameter was obtained by NLDFT method.<sup>[42]</sup> X-ray powder diffraction patterns were recorded at room temperature with a Siemens D5000 (Cu- $K\alpha$ , germanium monochromator). For transmission electron microscopy (TEM), for each sample, aluminum oxide (20 mg) was added to absolute ethanol (100  $\mu\text{L}$ ) and sonicated at 37 kHz and 240 W for 20 min. After 10 min of sedimentation a drop of 6  $\mu\text{L}$  supernatant was placed on top of a carbon-coated 400 mesh copper grid (Quantifoil Micro Tools, Jena, Germany). Excess liquid was removed by placing a sheet of filter paper below the grid. The samples were then air dried and analyzed using a CM120 electron microscope (Philips, Eindhoven, Netherlands) operated at 120 kV. Images were acquired using a 2k TemCam F216 CMOS camera (camera and software, TVIPS, Munich, Germany).



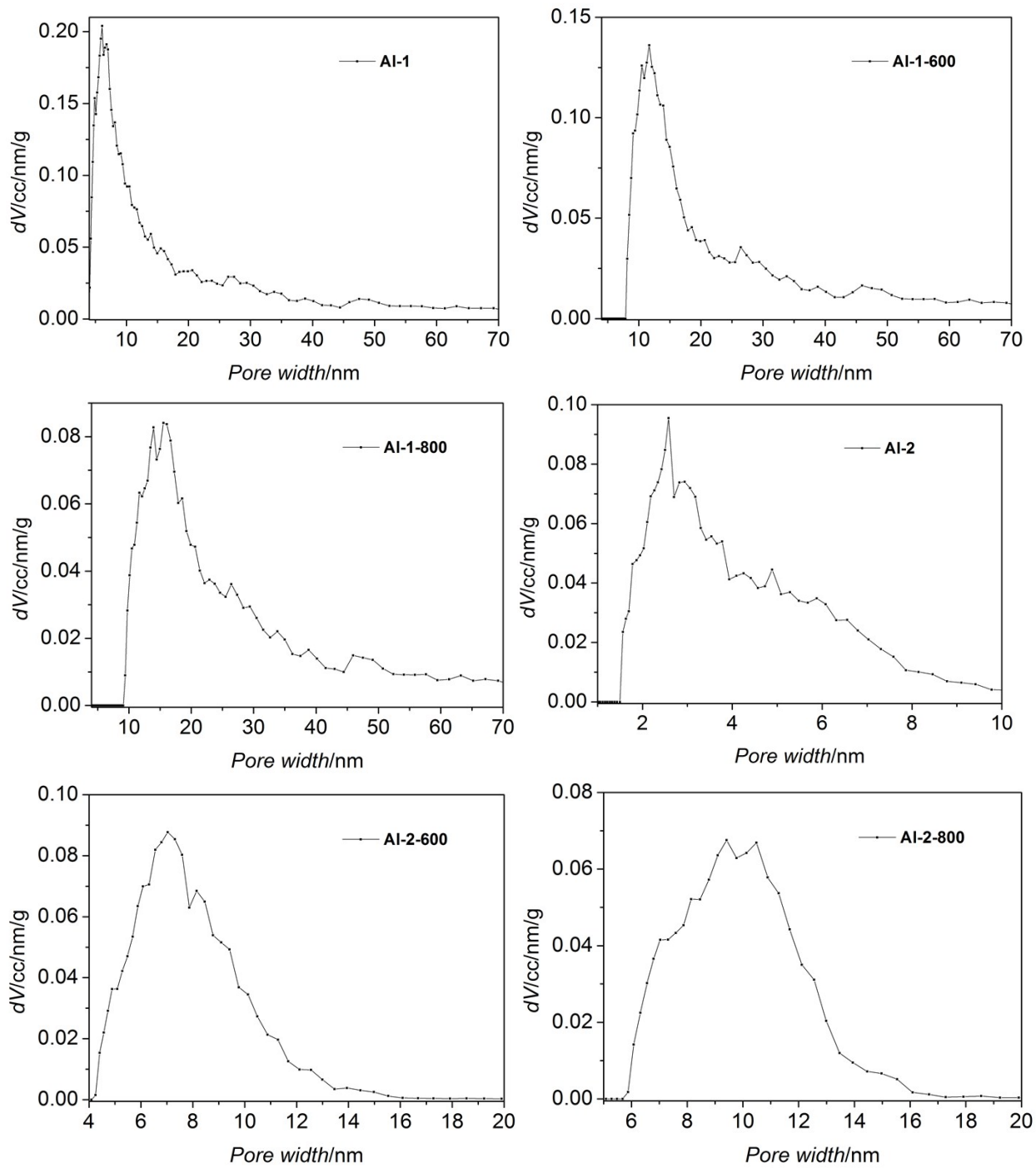
**Figure S-1** Wide-angle XRD patterns for **Al-1** calcined at 600 and 800 °C.



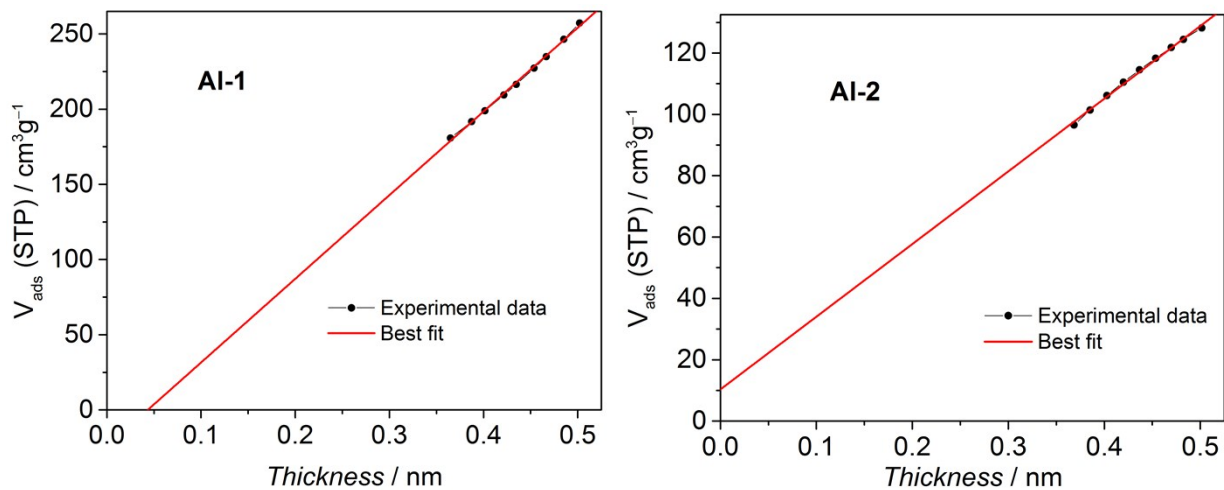
**Figure S-2** Wide-angle XRD patterns for **Al-2** calcined at 600 and 800 °C.

**Table. S-1**

	<i>P/Po range</i>	<i>C</i>	<i>Vm</i>	<i>P/Po(Vm)</i>	<i>θ(P/Po)</i>
<b>Al-1</b>	0.08-0.28	71.33	184.3	0.10	0.10
<b>Al-1-600</b>	0.03-0.12	147.57	78.8	0.08	0.08
<b>Al-1-800</b>	0.07-0.15	99.6	55.5	0.09	0.09
<b>Al-2</b>	0.05-0.2	79.96	96.6	0.10	0.10
<b>Al-2-600</b>	0.05-0.15	129.42	55.0	0.08	0.08
<b>Al-2-800</b>	0.07-0.18	105.75	34.2	0.10	0.10



**Figure S-3**



**Figure S-4. T-plots for Al-1 and Al-2 calculated by De Boer method.**

**Table. S-2: Thickness Method Summary**

Sample	Intercept	BET Area ( $\text{m}^2/\text{g}$ )	External Area ( $\text{m}^2/\text{g}$ )	Micropore Area ( $\text{m}^2/\text{g}$ )
Al-1	-24.2	802	802	-
Al-2	10.3	420	366	54