

A high-pressure synthesis of hydrothermally stable periodic mesoporous crystalline aluminosilica materials

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Supporting information:

High-pressure experiment using multi-anvil apparatus. The experiments at a pressure of 4 GPa were carried out using a multi-anvil assembly and a 1500 ton hydraulic press. The sample was encapsulated in a platinum capsule (2.5 x 3 mm). The capsule was placed in an alumina sleeve surrounded by a cylindrical Re heater, and a zirconia sleeve for thermal insulation. This assembly was placed inside a Cr₂O₃ doped MgO octahedron with an edge length of 18 mm. The temperature was measured using a W/Re thermocouple (type C) which was implemented inside the assembly. The octahedron was placed inside eight corner truncated tungsten carbide cubes with an edge length of 11 mm. The resulting cubic assembly was placed into the press. The sample was compressed to the desired pressure at a rate of 2 GPa/h at room temperature and then heated to the final temperature at a rate of 100 °C/min and kept at the target pressure-temperature for 3 h. After that, the pressure was released at a rate of 2 GPa/h. After normal pressure was reached, the sample was extracted from the capsule for characterization.

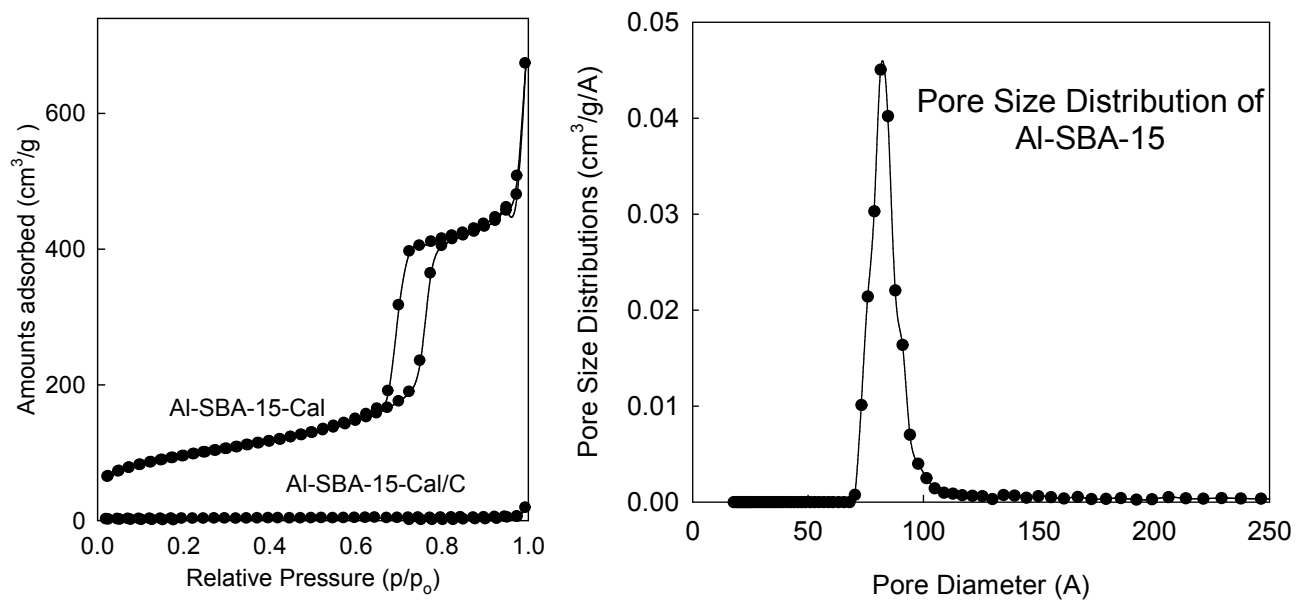


Fig. S1 Nitrogen adsorption isotherm and pore size distribution of mesoporous aluminosilica and mesoporous aluminosilica filled with carbon.

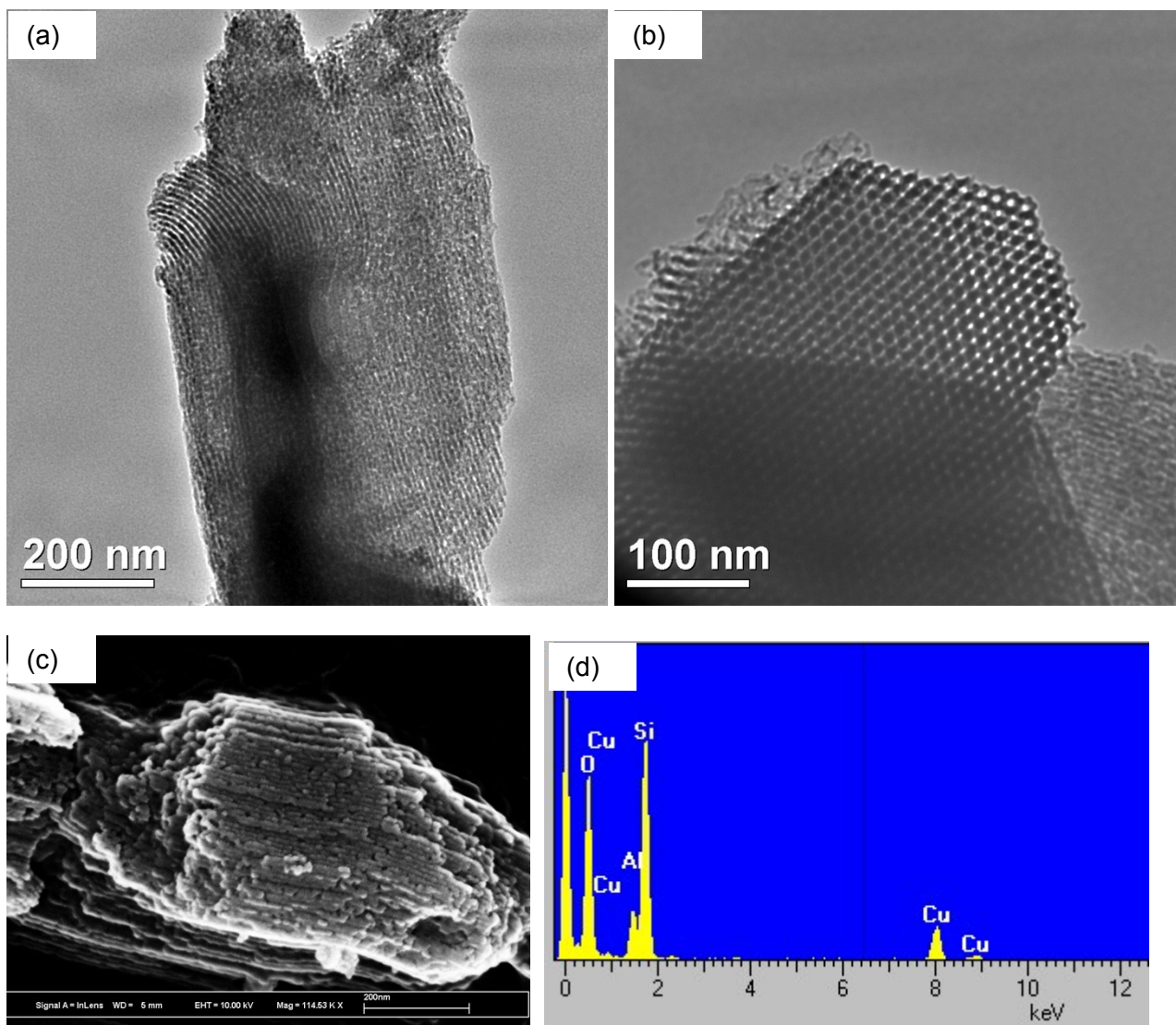


Fig. S2 TEM (a, b), SEM (c) image and EDX (d) pattern of mesoporous aluminosilica. The presence of Cu in EDX is due to use of TEM grid which consists of copper.

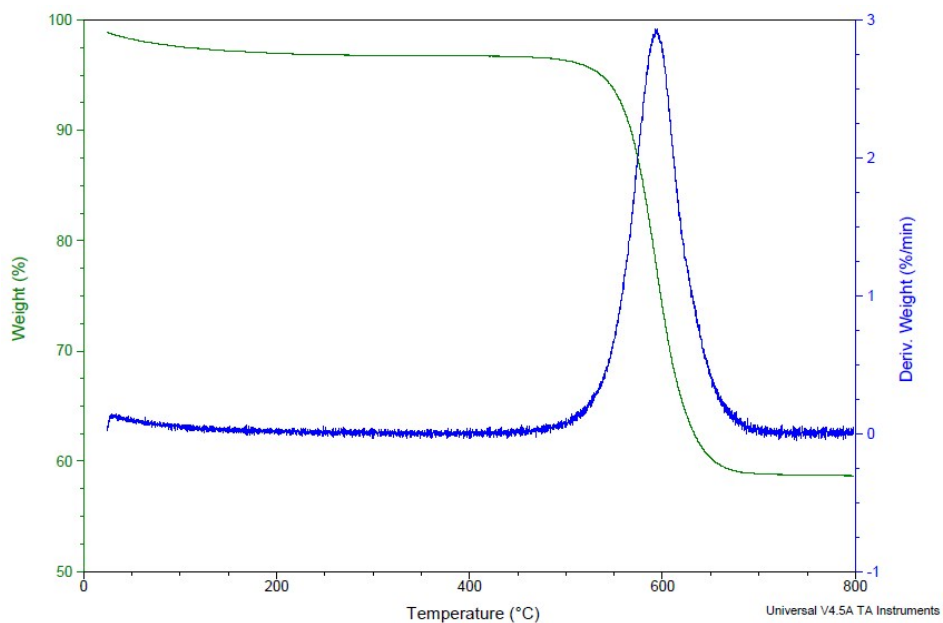


Fig. S3 Thermogravimetric (TGA) analysis of aluminosilica/carbon composite under air atmosphere. The weight loss at ~ 550-600 °C is due to elimination of carbon.

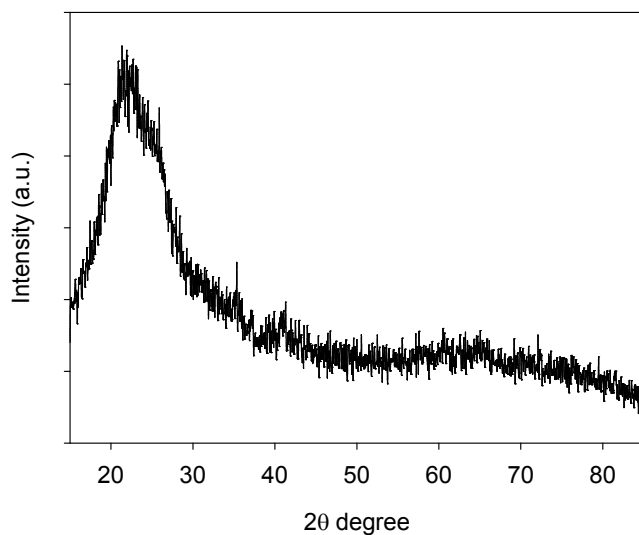


Fig. S4 XRD pattern for the calcined aluminosilica/carbon composite treated at 1 GPa and 750 °C for 6h. The broad peak is due to amorphous aluminosilica.

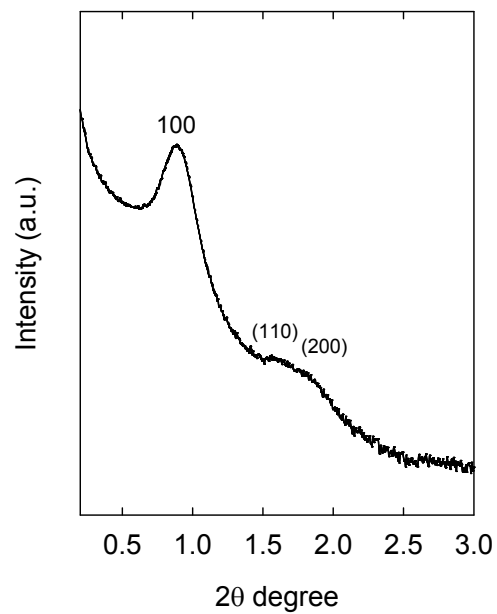


Fig. S5 SAXS pattern for the calcined aluminosilica/carbon composite treated at 1 GPa and 750 °C for 6h.

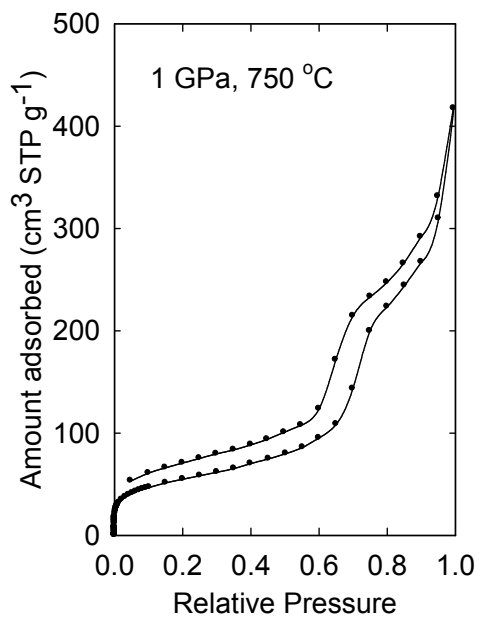


Fig. S6 Nitrogen adsorption isotherm for material obtained at a pressure of 1 GPa and temperature of 750 °C for 6 h. The materials was calcined under air before analysis.

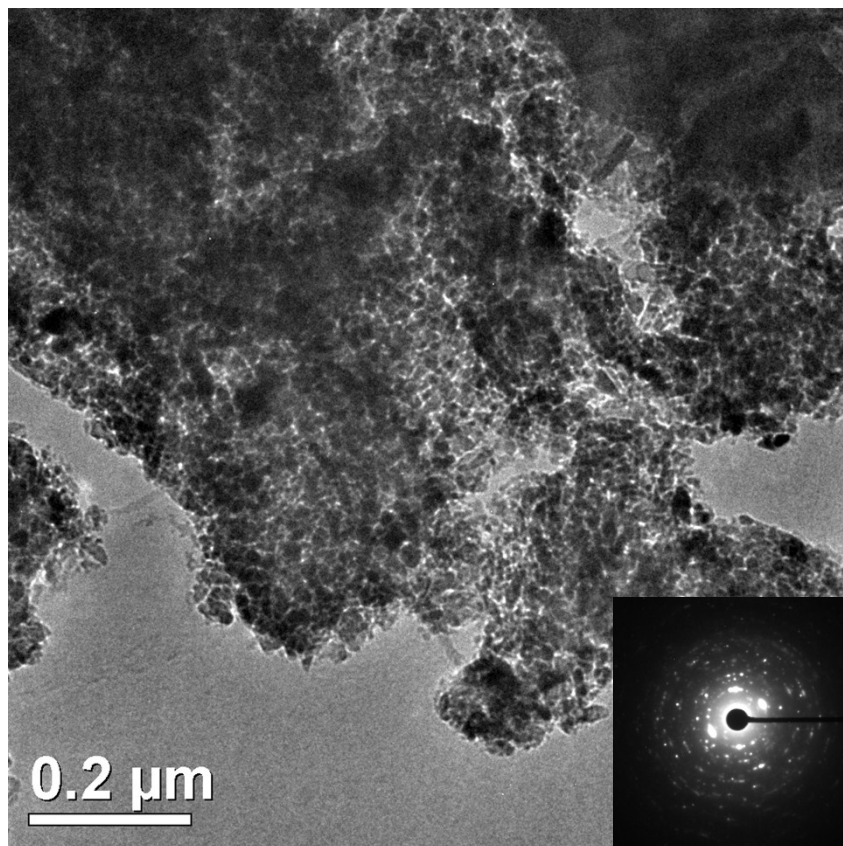


Fig. S7 TEM images for calcined aluminosilica treated at 2 GPa and 750 °C for 6 h. Inset shows corresponding SAED.

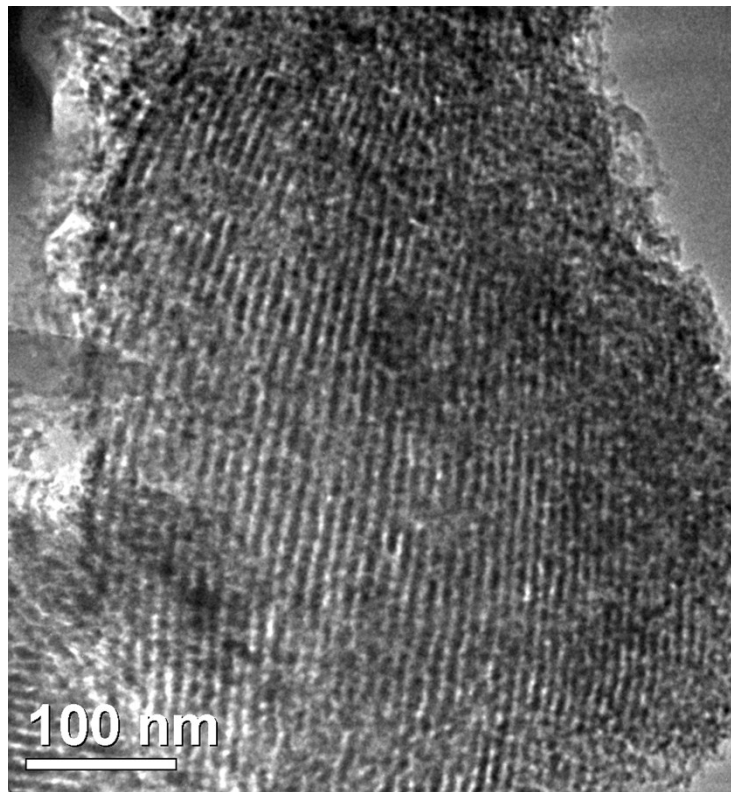


Fig. S8 TEM image for calcined aluminosilica material obtained at 1 GPa and 750 °C for 6h.

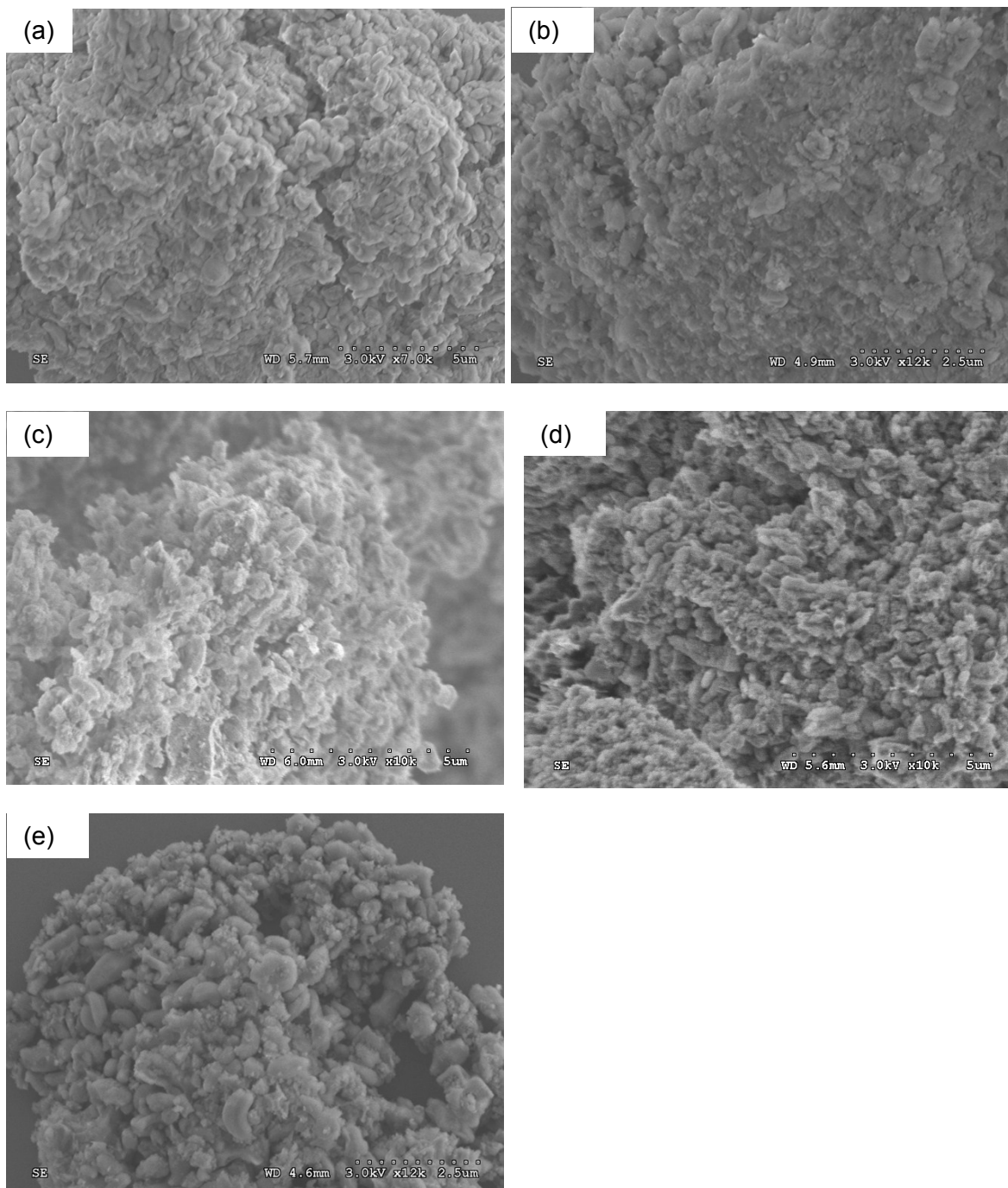


Fig. S9 SEM images for calcined aluminosilica treated at 2 GPa and (a) 350, (b) 550, (c) 650 and (d) 750 °C for 6 h and for calcined material obtained at 1 GPa and 750 °C (e). Scale bar are (a) 5 μm, (b) 2.5 μm, (c) 5 μm, (d) 5 μm, and (e) 2.5 μm respectively.

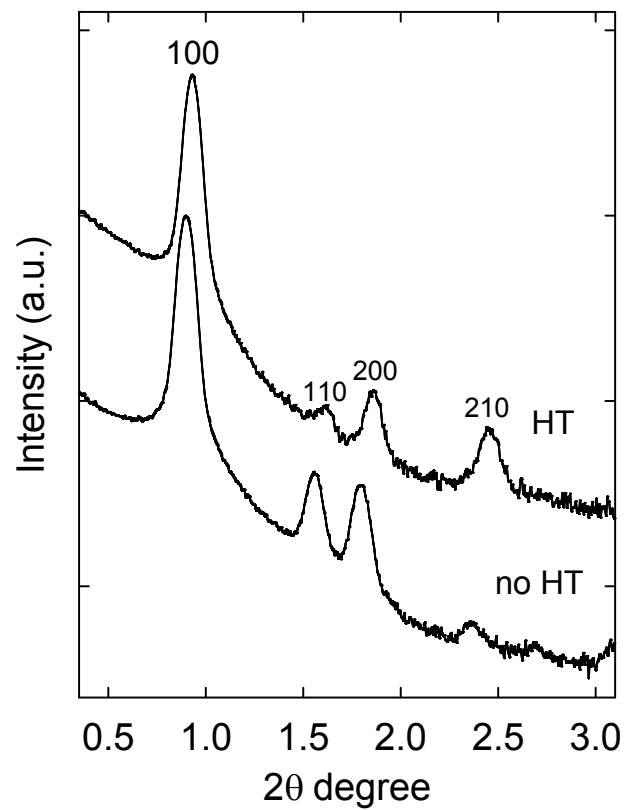


Fig. S10 SAXS patterns for the amorphous mesoporous aluminosilica before and after hydrothermal treatment at 800 °C under pure steam for 2 h.