Supporting Information

Achieving Extreme Thermal Stability of High-Silica FAU through Trace Water-Assisted Steam Treatment

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EXPERIMENTAL PROCEDURES

Steam/Heat Treatments

The sample was subjected to steam/heat treatments at high temperatures using a custom-built apparatus (Figure S1), with further details of the apparatus provided elsewhere.[5] The desired temperature (*T*) was reached after a ramping period of 75 min and maintained for 24 h or 48 h before naturally cooling to room temperature. A specific volume of water was introduced via a syringe pump and mixed with high purity dry air to achieve *x* vol% steam at atmospheric pressure. The high purity gas cylinder (dry air) was purchased from Tokyo High Pressure Yamazaki Co., Ltd.

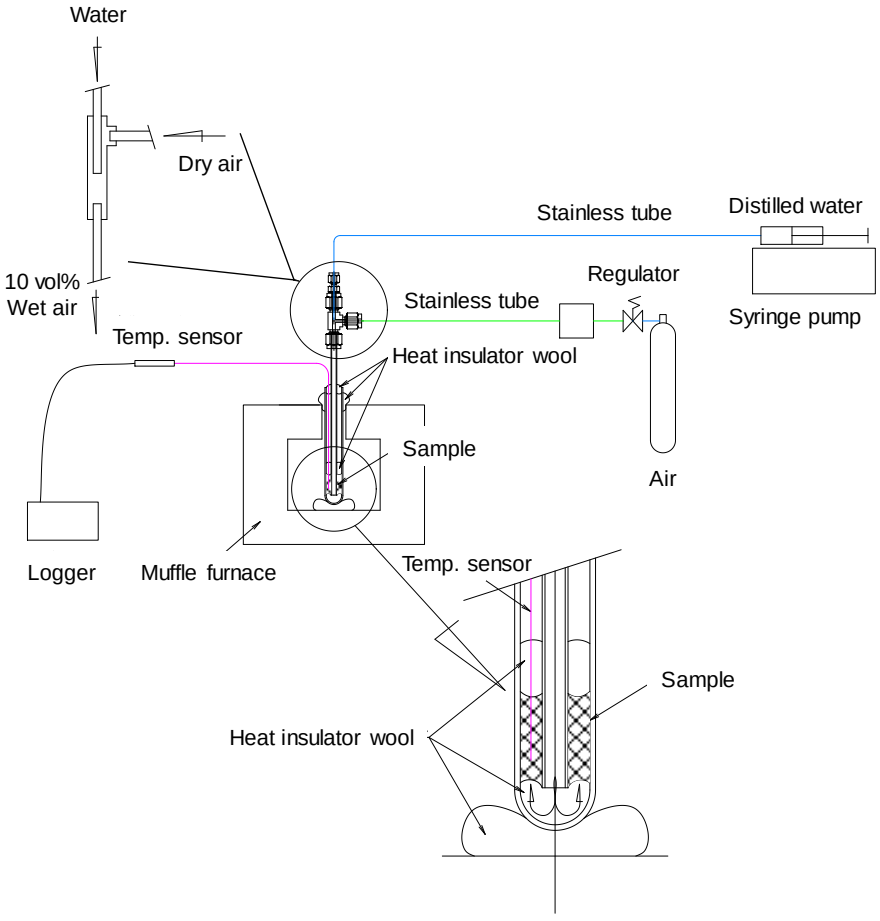

Figure S1 The custom-built apparatus for the steam treatment.[5]

Heat Resistance Tests

Heat resistance tests were conducted using a muffle furnace, set at 1150°C. The temperature ramped up over 4 h and was maintained for an additional 20 h. Each test batch consisted of 2 to 6 samples, each weighing 0.25 g, with FAU-as-purchased samples included in every test run as a reference standard to ensure consistency and minimize potential errors during testing and analysis. All treated samples underwent testing at least twice to ensure the reliability and consistency of the results obtained. The relative crystallinity value of each sample reported in this study represents the average value derived from its repeated tests.

Characterizations

Powder XRD patterns were acquired utilizing an X-ray diffractometer (Ultima IV, Rigaku Corp.), employing Cu Kα radiation with specific parameters set at 40 kV and 40 mA, resulting in a wavelength (λ) of 0.15406 nm. The diffraction patterns were collected across an angular range spanning $2\theta = 3 \sim 50°$, with a scanning rate set at 10° per minute and angular intervals of 0.02° maintained. FT-IR spectra were obtained utilizing a JASCO FT/IR-6600 spectrometer, outfitted with a mercury cadmium telluride detector. Prior to spectral acquisition, activation of samples was conducted at 500°C for a duration of 1 h under a controlled N_2 flow environment. Subsequently, the spectra were recorded at a temperature of 25°C, employing the diffuse reflectance mode. Morphological observations were performed using a JEOL JSM-IT800 SEM at 5 kV acceleration voltage, with osmium coating. Nitrogen adsorption-desorption measurements were conducted using an Anton Paar Autosorb-iQ2-MP instrument at -196 °C. ^{29}Si MAS NMR spectra were acquired on a JEOL NMR spectrometer (Delta) at 14.10 T using a 3.2mm ZrO_2 rotor

with spinning at 15 kHz. ^{1}H dipole decoupling ^{29}Si MAS NMR spectra were obtained with ^{1}H irradiation. For each sample, 1024 acquisitions were obtained with a pulse delay of 30 s and a p/2pulse of 2.63 ms duration. ^{29}Si cross polarization (CP) MAS spectra were collected with a contact time of 2 ms for 1024 scans and 4 s relaxation delay. Chemical shifts were referenced to tetramethylsilane (TMS) at 0 ppm.

RESULTS AND DISCUSSION

Table S1 The relative crystallinity[a] of treated samples compared to FAU-as-purchased.

Sample Name	Relative Crystallinity (%)
FAU-as-purchased	100
FAU-700°C-5%-48h	94
FAU-700°C-3%-48h	95
FAU-700°C-1%-24h	98
FAU-650°C-2%-24h	98
FAU-650°C-1%-48h	98
FAU-650°C-1%-24h	98
FAU-650°C-1%-12h	99
FAU-650°C-0.3%-24h	98
FAU-650°C-0%-24h	99
FAU-600°C-7%-48h	95
FAU-600°C-5%-48h	97
FAU-600°C-3%-48h	97
FAU-600°C-1%-24h	98
FAU-600°C-0.3%-24h	99
FAU-600°C-0%-24h	99
FAU-550°C-1%-24h	98
FAU-550°C-0.3%-24h	98
FAU-550°C-0%-24h	100
FAU-500°C-7%-48h	97
FAU-500°C-5%-48h	97

[a] The relative crystallinity of each sample in this study was evaluated using powder XRD diffraction, focusing on peaks in the 2θ range of 20° to 30° through deconvolution and integration techniques. In addition, all the relative crystallinity values reported in this study are relative to the FAU-as-purchased.

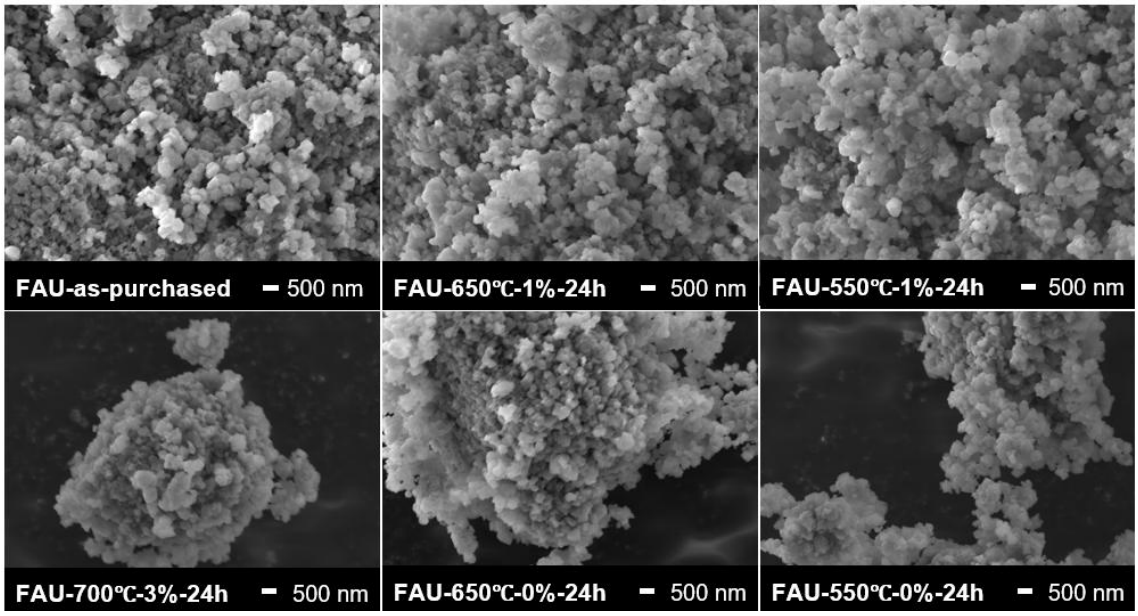

Figure S2 The SEM images of selected FAU samples before and after steam treatments.

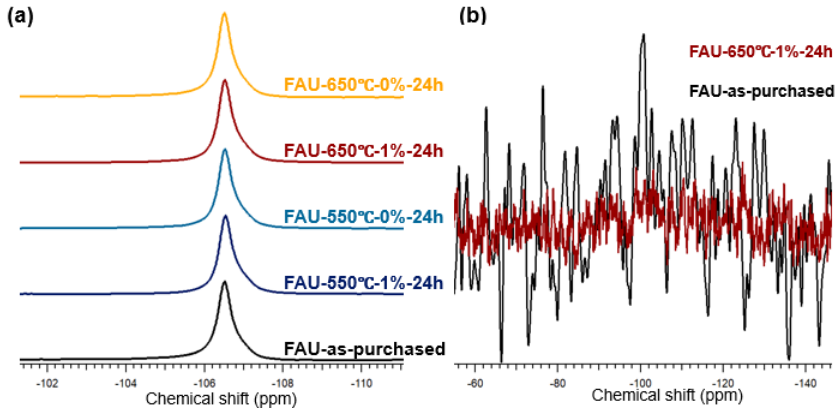

Figure S3 ^{29}Si single-pulse MAS NMR (a) and CP MAS NMR (b) spectra of FAU-as-purchased and selected treated samples.

Table S2 Surface area and *t*-plot micropore volume of FAU-as-purchased and selected treated samples.

Sample name	**Surface area (m^2/g)**	***t*-plot micropore volume (cm^3/g)**
FAU-as-purchased	822	0.28
FAU-650℃-1%-24h-tested	561	0.19
FAU-650℃-0.3%-24h-tested	502	0.17
FAU-650℃-0%-24h-tested	505	0.17
FAU-as-purchased-tested	224	0.073