Electronic Supporting Information

Mechanochemically assisted hydrolysis in the ADOR process

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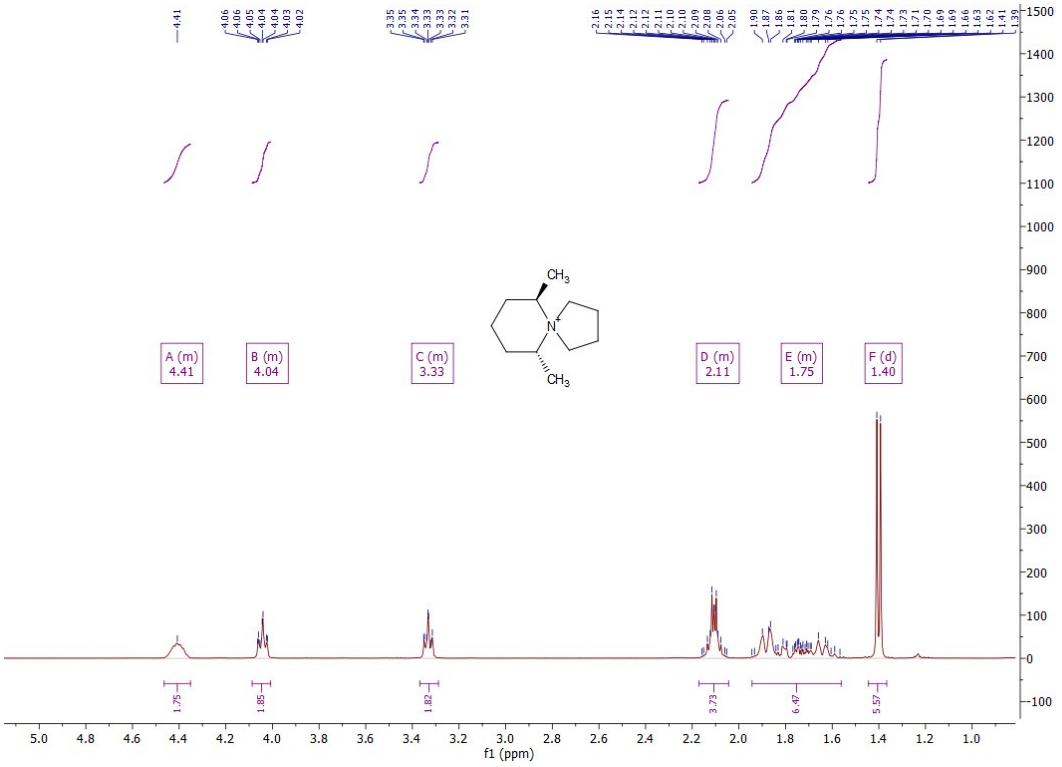

Figure S1: [1]H NMR of the OSDA (6R,10S)-6,10-dimethyl-5-azoniaspiro[4.5]decane bromide.

[1]H NMR (400 MHz, Chloroform-d) δ 4.46–4.35 (m, 2H, NC<u>H</u>), 4.09–4.01 (m, 2H, NC<u>H$_2$</u>), 3.37–3.29 (m, 2H, NC<u>H$_2$</u>), 2.17–2.04 (m, 4H, NCH$_2$C<u>H$_2$</u>), 1.94–1.56 (m, 6H, C<u>H$_2$</u>C<u>H$_2$</u>C<u>H$_2$</u>CHN), 1.40 (d, J = 6.5 Hz, 6H, C<u>H$_3$</u>).

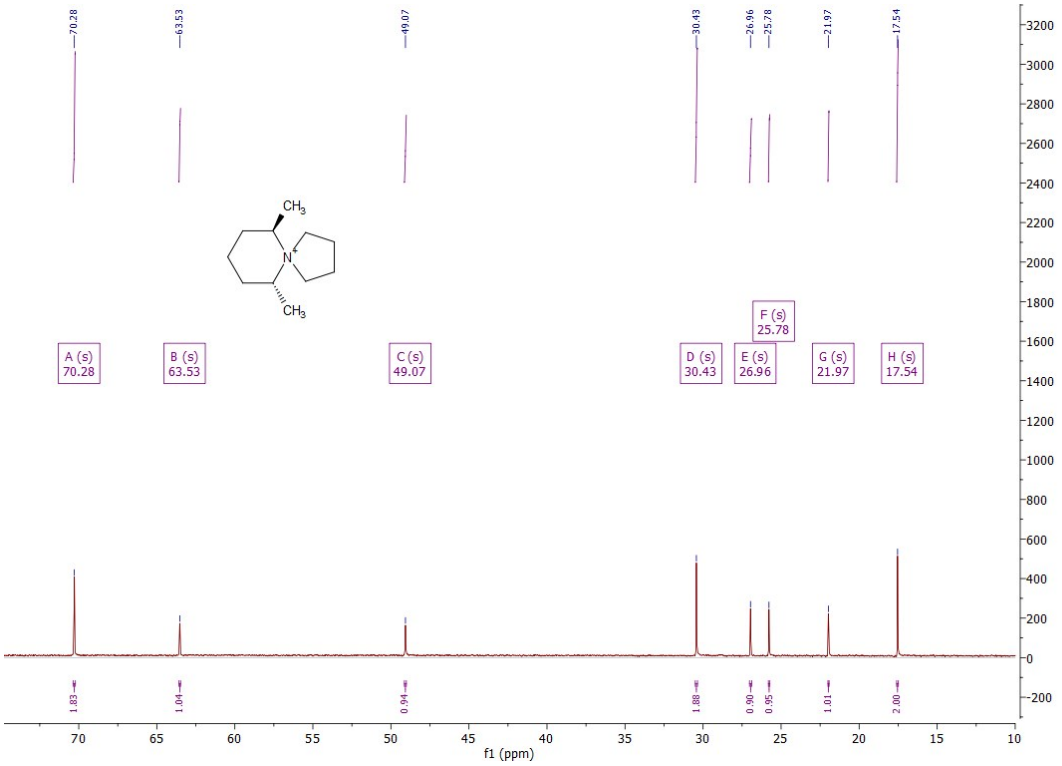

Figure S2: [13]C NMR of the OSDA (6R,10S)-6,10-dimethyl-5-azoniaspiro[4.5]decane bromide.

[13]C NMR (126 MHz, Chloroform-*d*) δ 70.28, 63.53, 49.07, 30.43, 26.96, 25.78, 21.97, 17.54.

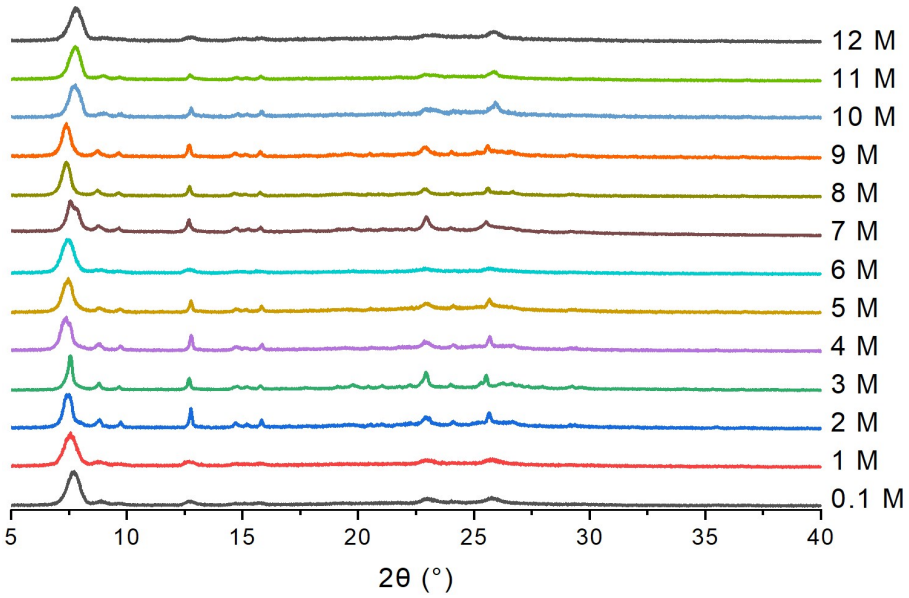

Figure S3: PXRD patterns of as made materials produced from ball milling UTL in varying concentrations of hydrochloric acid.

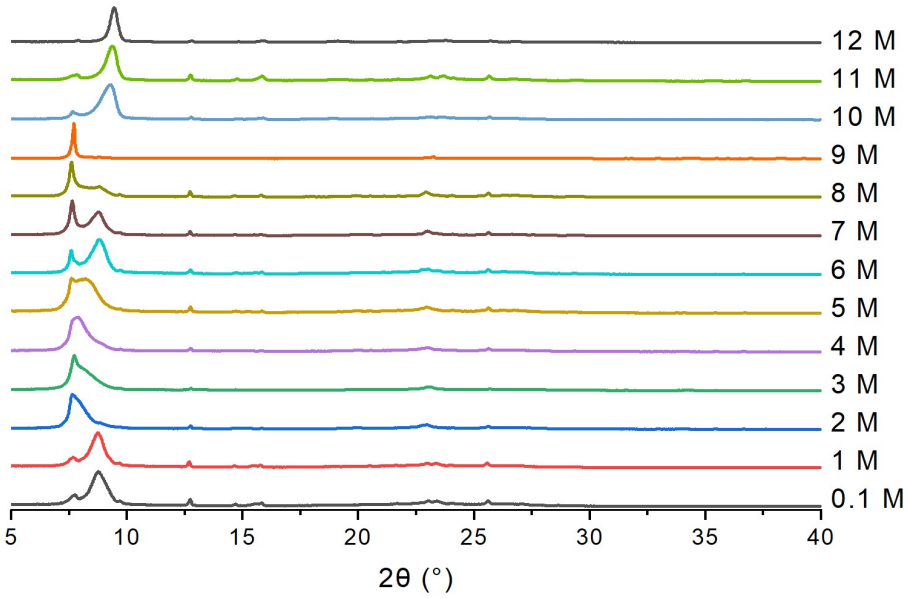

Figure S4: PXRD patterns of reassembled materials produced from ball milling UTL in varying concentrations of hydrochloric acid.

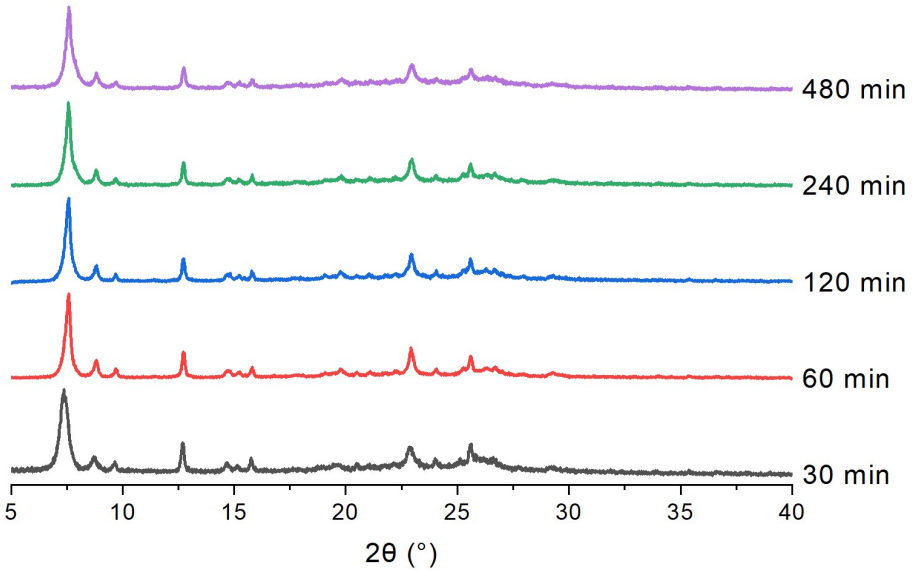

Figure 5: PXRD patterns of as made materials produced from ball milling UTL in 9 M hydrochloric acid for different times.

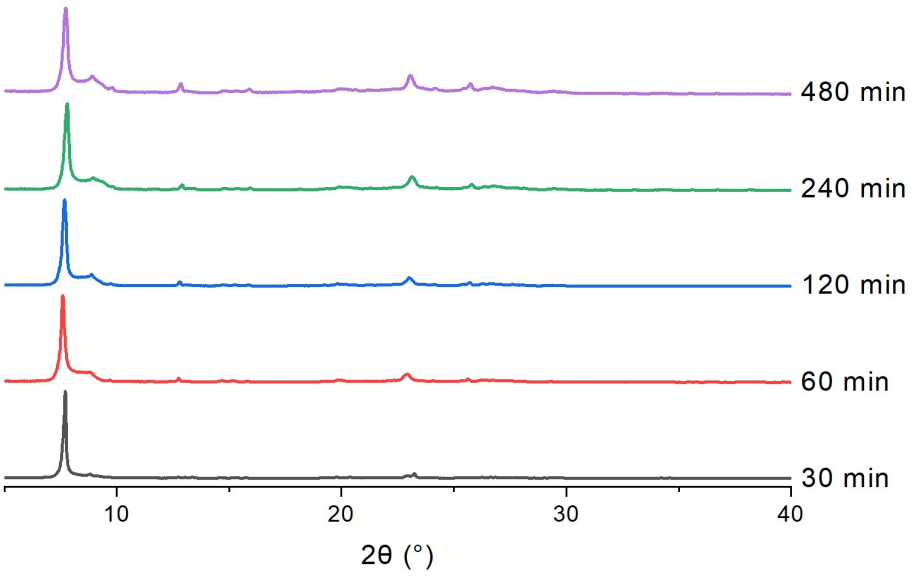

Figure 6: PXRD patterns of reassembled materials produced from ball milling UTL in 9 M hydrochloric acid for different times.

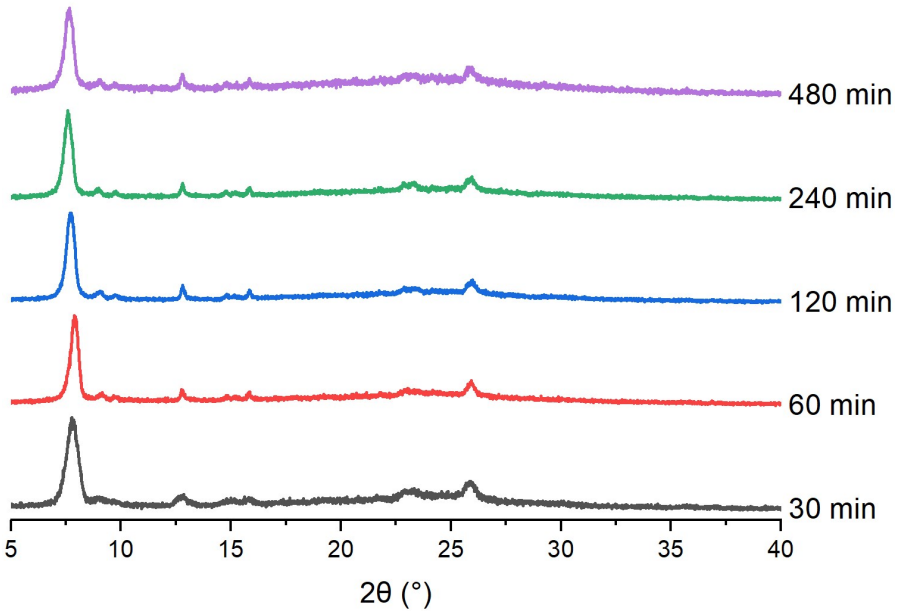

Figure S7: PXRD patterns of as made materials produced from ball milling UTL in 12 M hydrochloric acid for different times.

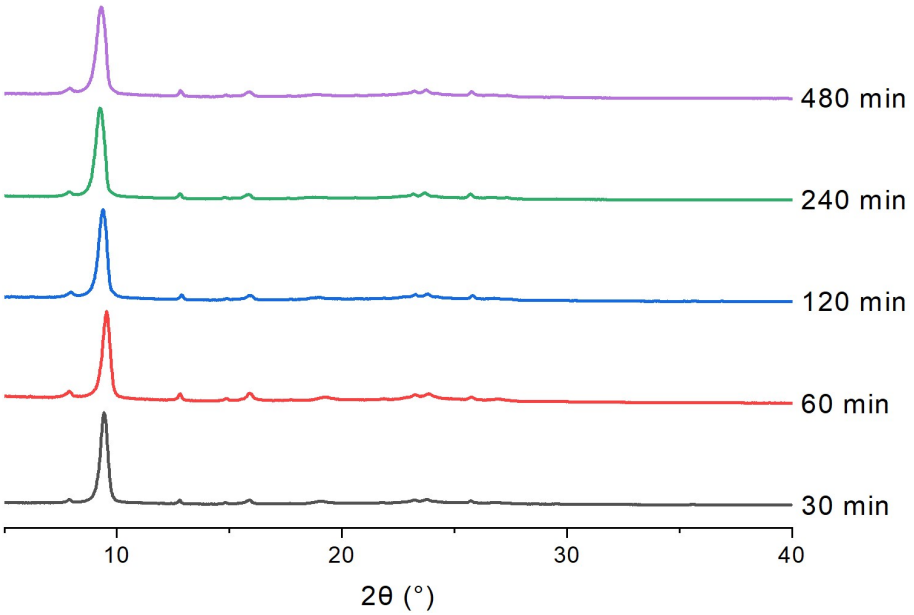

Figure S8: PXRD patterns of reassembled materials produced from ball milling UTL in 12 M hydrochloric acid for different times.

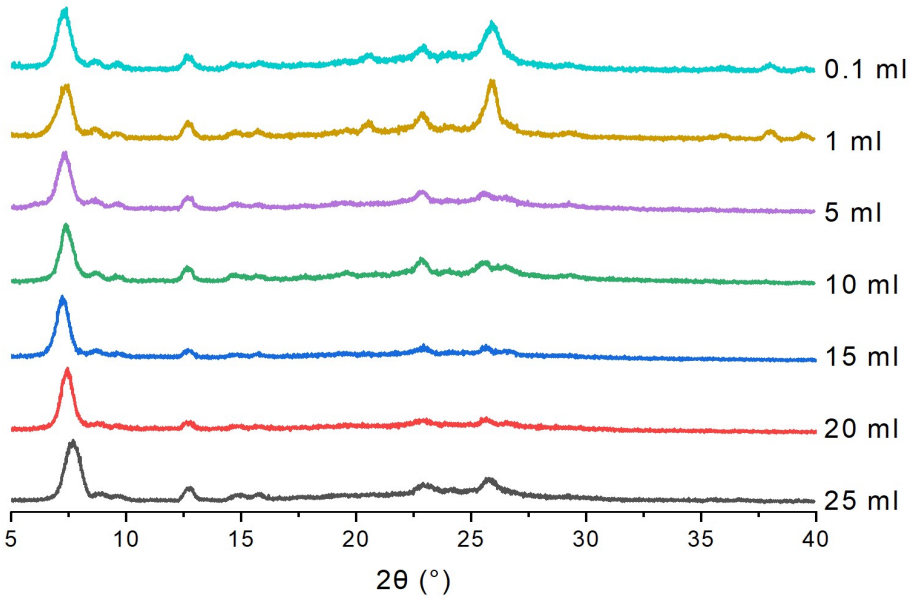

Figure S9: PXRD patterns of as made materials produced from ball milling UTL with different volumes of water.

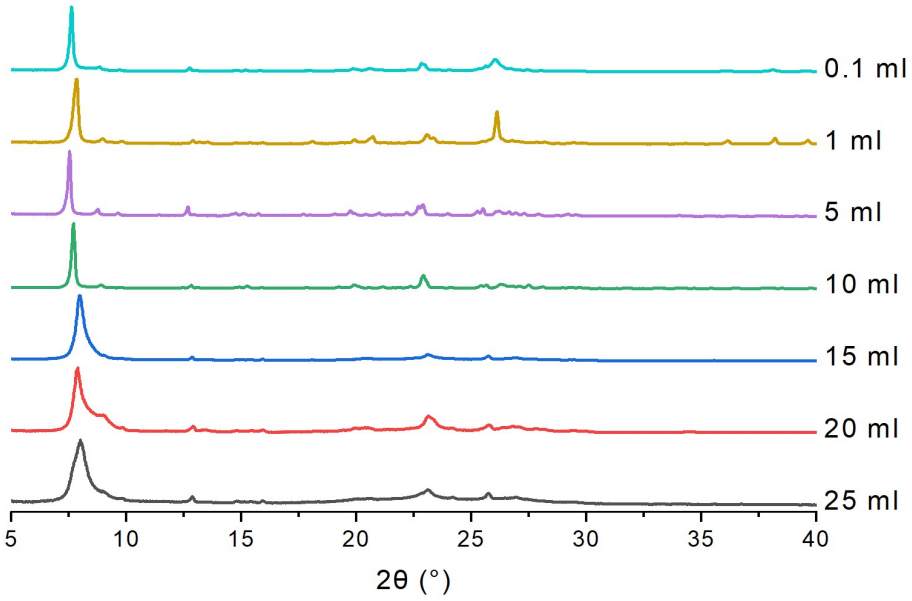

Figure S10: PXRD patterns of reassembled materials produced from ball milling UTL with different volumes of water.

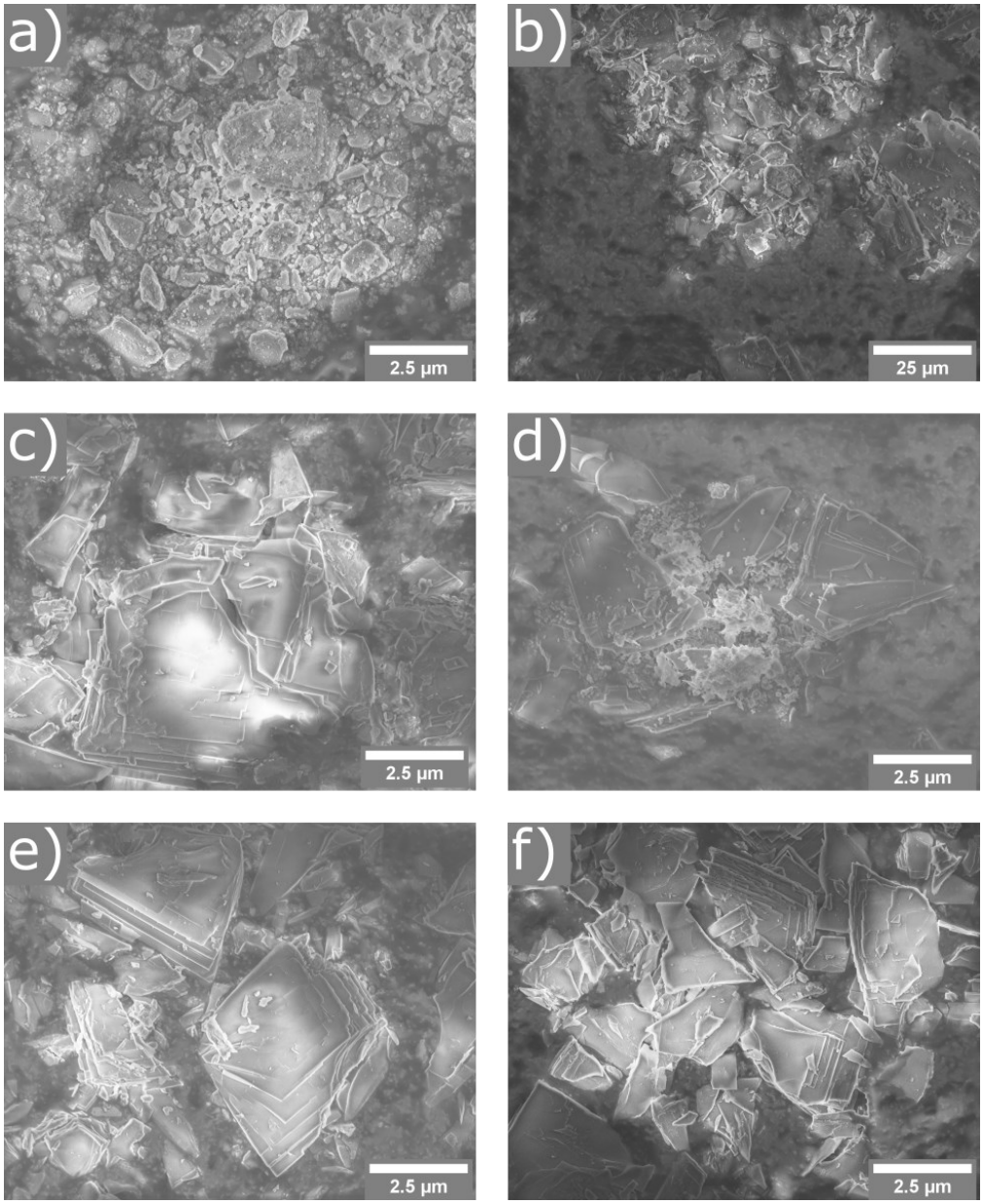

Figure S11: SEM images of various ball milled and reassembled samples.

a) 25 ml water; b) 5 ml water; c) 0.1 ml water; d) 6 M HCl; e) 9 M HCl; f) 12 M HCl.

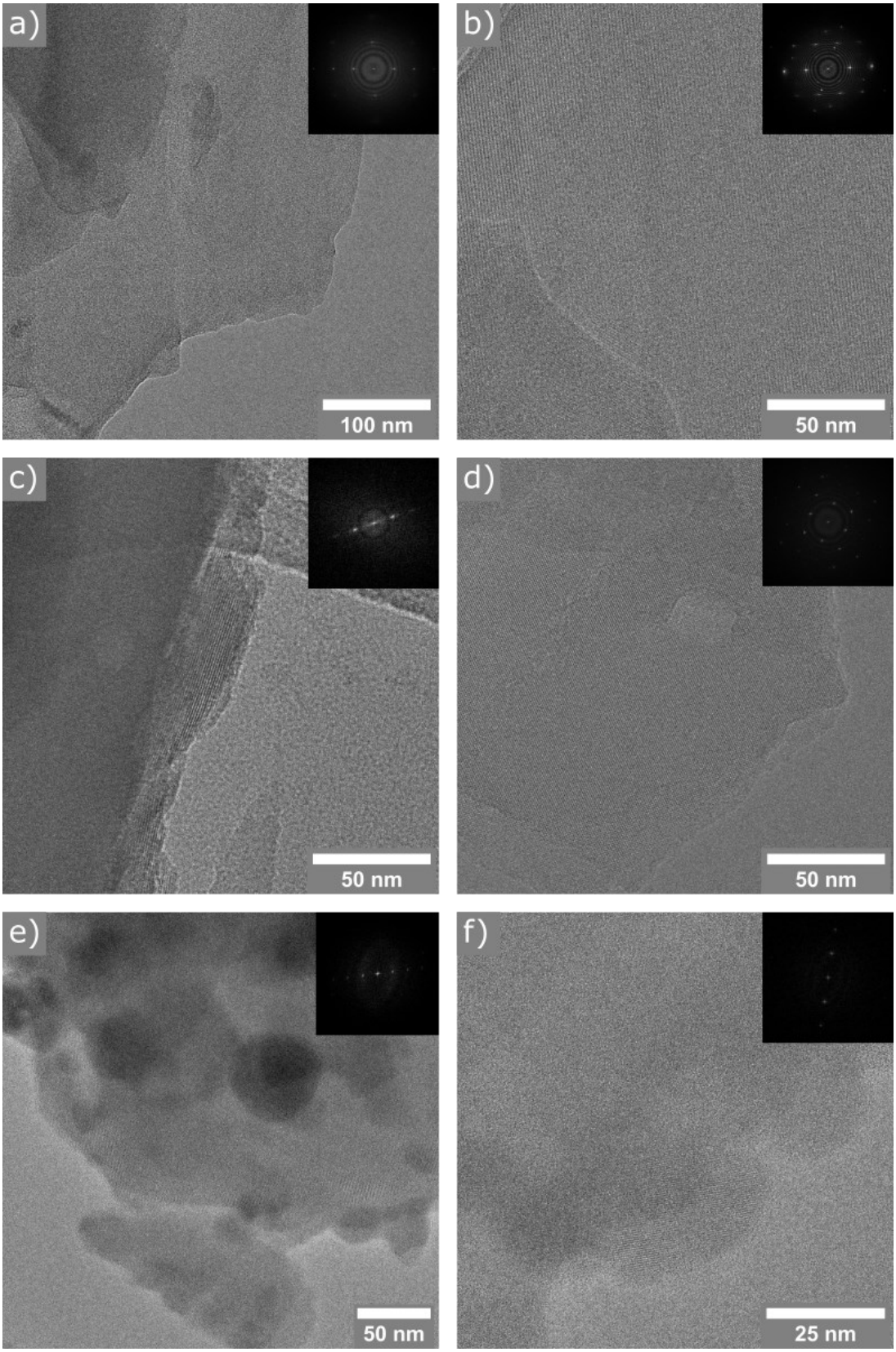

Figure S12: TEM images with FFT inserts.

a) 12 M, crystal of a minor IPC-6 phase (d-spacing 10.1 Å); b) 12 M, two overlapping crystals; c) 9 M, lattice fringes corresponding to IPC-2 (d-spacing 11.3 Å); d) 9 M, view along the [101] direction; e) 0.1 ml water, lattice fringes corresponding to IPC-2 (d-spacing 11.2 Å); f) 0.1 ml water, showing a GeO2 nanoparticle impurity.

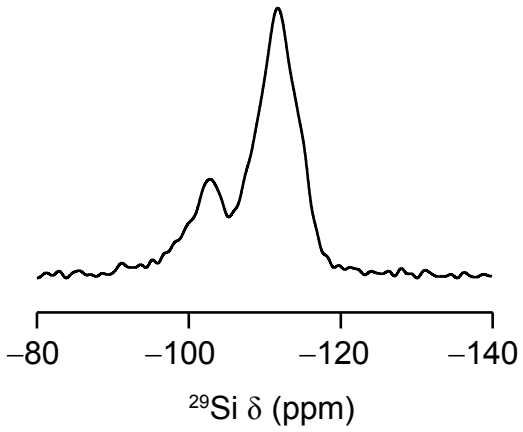

Figure 13: ^{29}Si (9.4 T, 14 kHz) MAS NMR spectrum of calcined UTL, ball milled in 40% $H_2{}^{17}O$ for 30 minutes and dehydrated under vacuum at 120 °C overnight.