Supporting Information

Effective One Step Synthesis of Silica Supported 1,3-Dibutylimidazolium Acetate for Carbon Dioxide Capture

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Experimental Procedures.

Procedure for the synthesis 1, 3-dibutylimidazolium acetate

0.153 g (5.07 mmol) of paraformaldehyde was stirred in 5 mL of toluene, and cooled to 0 ^{o}C. To this 0.371 g (5.07 mmol) of n-butylamine was added dropwise, ensuring the temperature did not rise above 4 ^{o}C. After 10 min a second equivalent of n-butylamine was added to the reaction in a steady stream. 1 mL (5.07 mmol) of glacial acetic acid was added dropwise to the reaction. After this addition the solution was allowed to reach room temperature. 0.573 mL (5.07 mmol) of glyoxal and an additional 5 mL of toluene were then added and the reaction was allowed to stir at room temperature for 1 h. The solution was then heated to reflux using a Dean Stark apparatus for 3 h. Less than 1 mL of water was liberated from the reaction during this period. The black liquid was left under vacuum overnight resulting in a viscous black oil. 1H (400 MHz, $CDCl_3$): δ 0.924 (t, 6H), 1.343 (m, 4H), 1.807 (m, 4H), 1.960 (s, 3H), 4.268 (t, 4H), 7.271 (s, 2H), 10.497 (s, 1H). 13C (100 MHz, $CDCl_3$) δ 13.38 (CH_3), 19.42 (CH_2), 23.37 (CH_3), 32.09 (CH_2), 49.62 (CH_2), 121.29 (CH), 139.04 (CH), 176.61 (C).

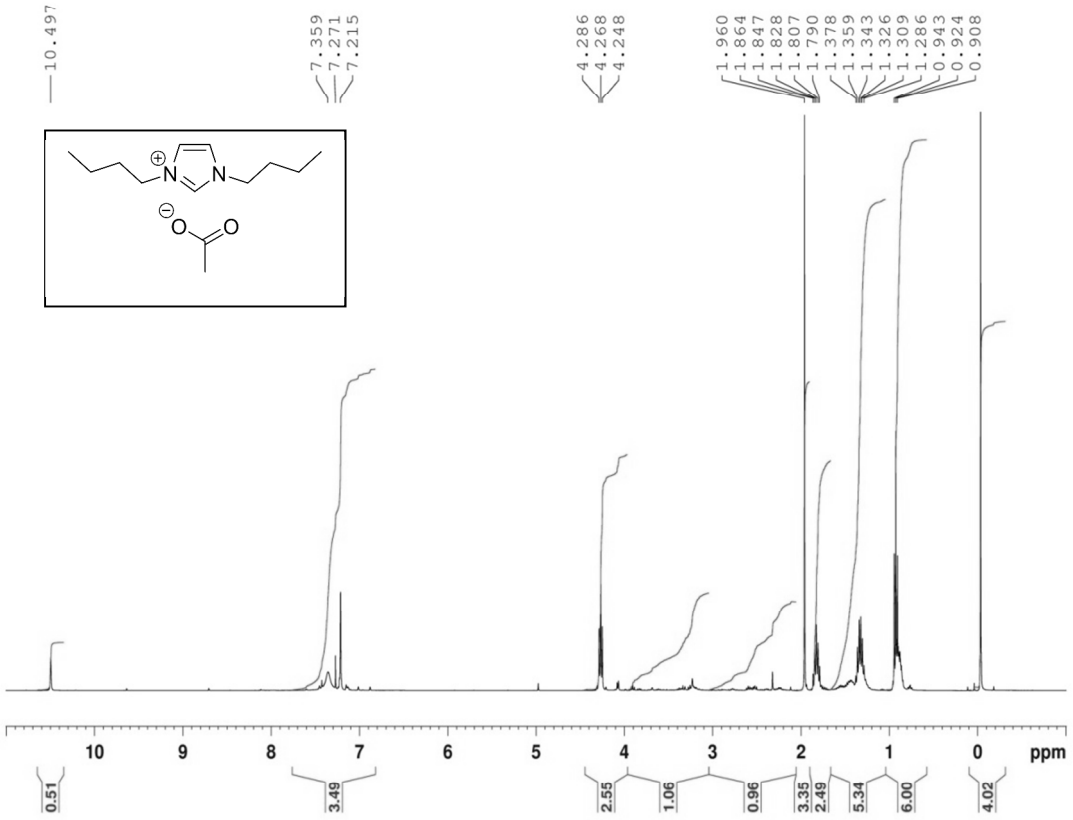

Figure S-1. ^{1}H NMR spectrum of 1, 3-dibutylimidazolium acetate in $CDCl_3$

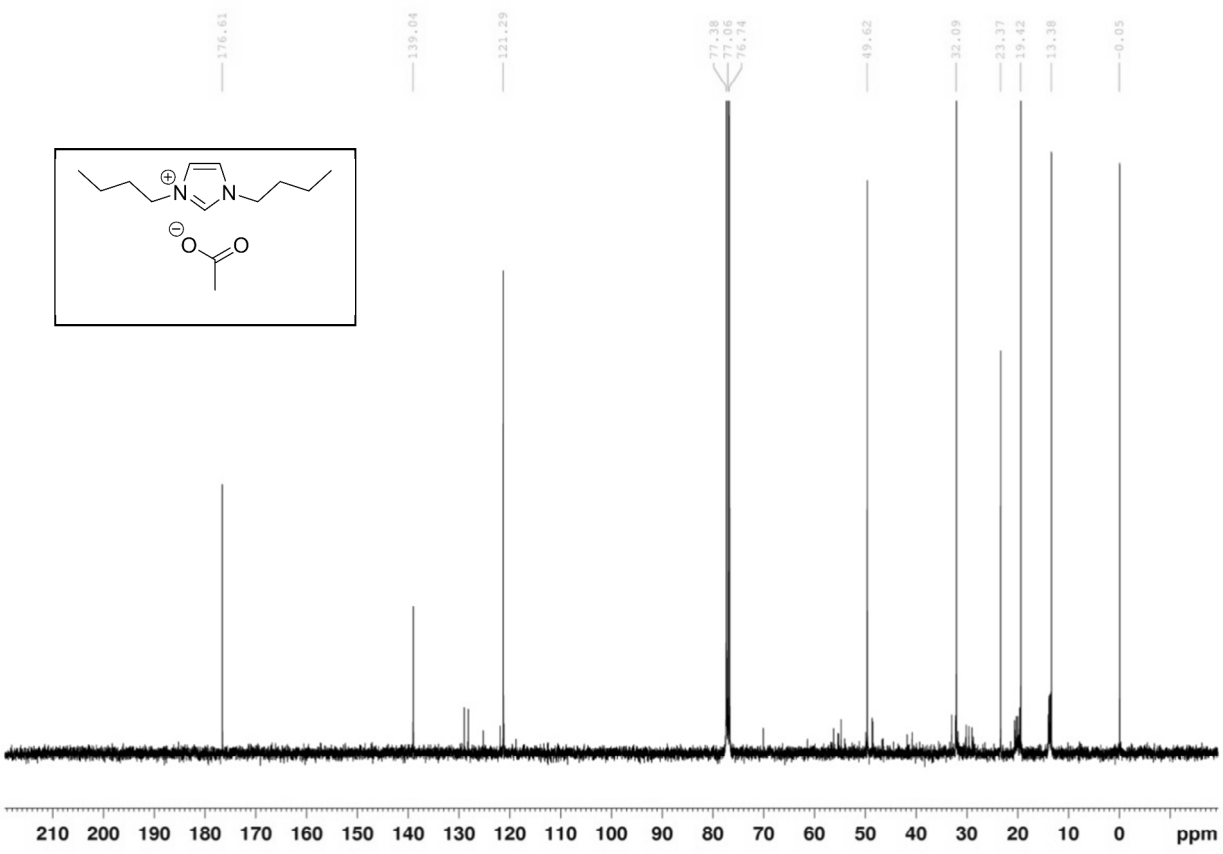

Figure S-2. ^{13}C NMR spectrum of 1, 3-dibutylimidazolium acetate in $CDCl_3$

Procedure for the synthesis of SBA-15

In a Pyrex jar, 85 mL of deionized water was added to 2.1 g of P123 and stirred at 40 ^{o}C until the P123 was completely dissolved. In a separate flask, 11.1 mL (49.8 mmol) of TEOS was added to 5.0 mL of 2.8 M hydrochloric acid and stirred vigorously for 3 h until the solution was transparent. This solution was then added directly to the previously dissolved P123 solution and stirred for 24 h. The solution was then aged at 80 ^{o}C for 24 h. The resulting slurry was vacuum filtered and washed with water and ethanol. The material was washed with ethanol using a soxhlet extractor for 3 days to remove the surfactant from the pores. The final product was air dried for a week to result in a final weight of 7.134 g. ^{29}SI MAS (79.51 MHz), δ -91.858 (Q2), -101.496 (Q3), -110.233 (Q4). IR (2 mg/ 200 mg KBr, 128 scans, 4 cm^{-1} resolution): cm^{-1} 3442, 1634, 1089, 957, 80, 468.

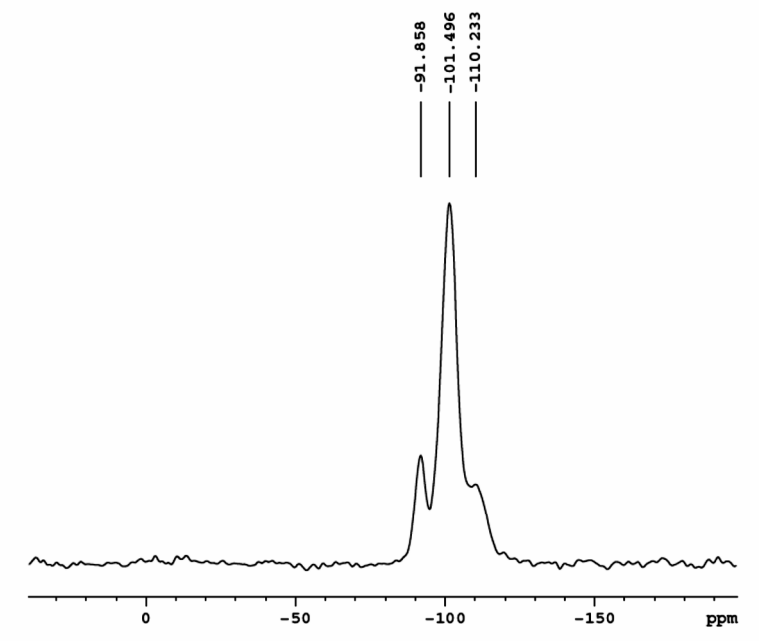

Figure S-3. ^{29}Si Solid State CP/MAS NMR Spectrum of SBA-15

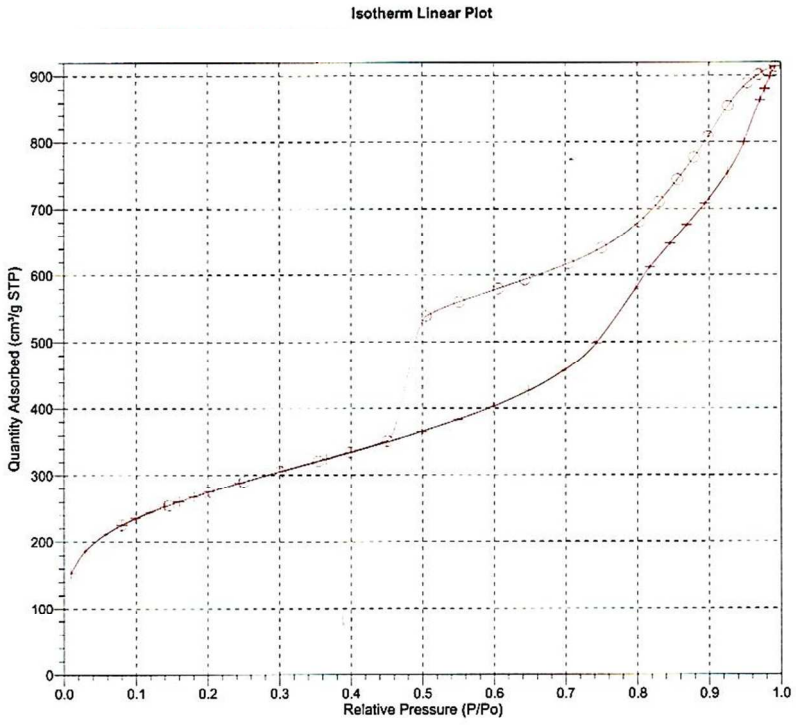

Figure S-4. Nitrogen physisorption isotherm of SBA-15

Procedure for the synthesis SBA-NH_2

0.3620 g of SBA-15 was placed in an oven at 100 ^{o}C for 3 days to drive off the water. The activated SBA-15, along with 3.82 g (17.3 mmol) of 3-aminopropyltriethoxysilane, was added to a flask containing 25 mL of toluene. The mixture was stirred and heated to reflux for 24 h. The resulting 3-aminopropyl-grafted-SBA-15 was washed with toluene and air dried for several days. After drying, the final weight was 0.3680 g. Elemental analysis showed 3.19% nitrogen grafted on the material(2.28 mmol per gram of material). ^{29}SI (79.51MHz, MAS 6.5, 11.5kHz), δ -67.713 (T3), -101.482 (Q3), -109.228 (Q4). 13C (100.65MHz, MAS 7.00, 11.5kHz), δ 10.782 (CH_2), 22.040 (CH_2), 42.824 (CH_2). IR (2 mg/ 200 mg KBr, 128 scans, 4 cm^{-1} resolution) cm^{-1} 3446, 2936, 1635, 1559, 1496, 1084, 960, 796, 465.

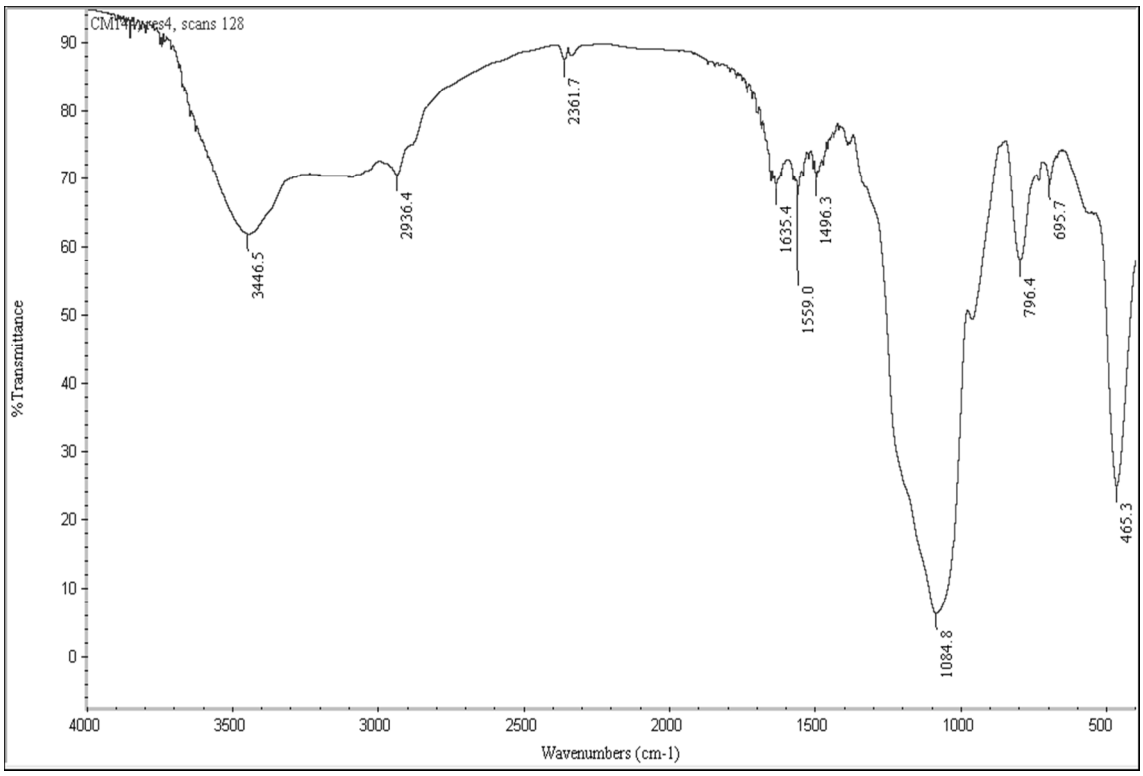

Figure S-5. IR spectra of SBA-NH_2

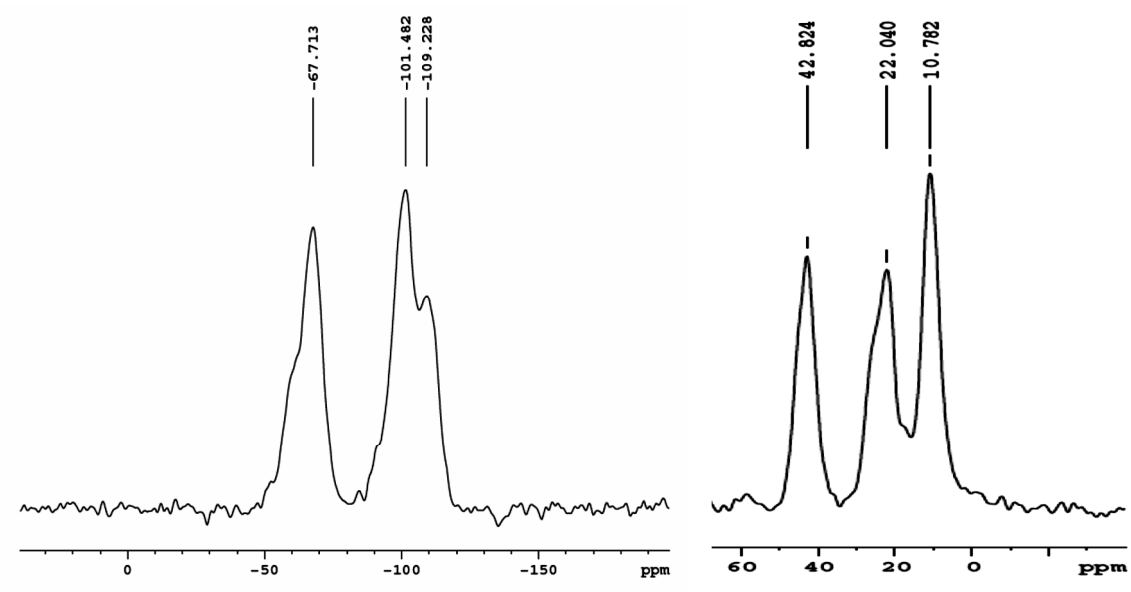

Figure S-6. ^{29}Si and ^{13}C solid state CP/MAS NMR spectrum of SBA-NH_2

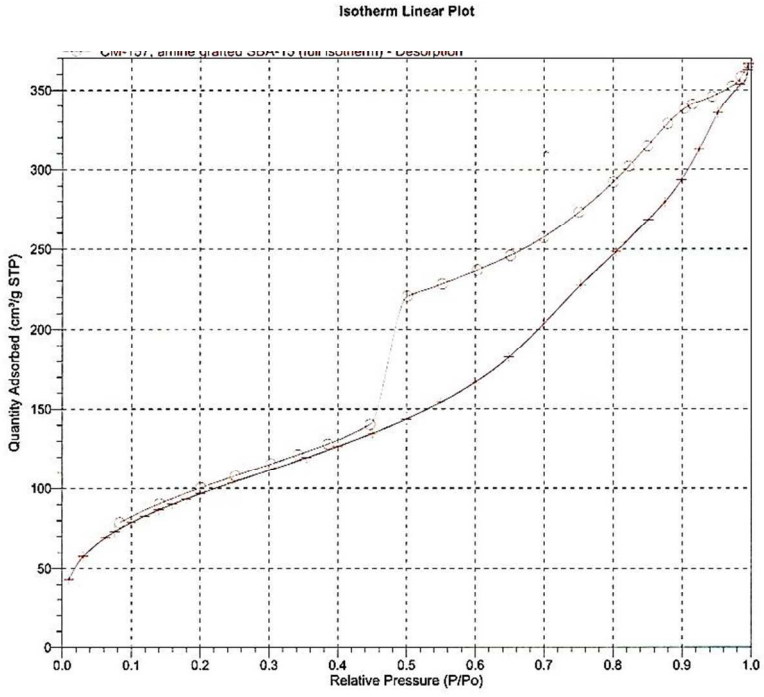

Figure S-7. Nitrogen physisorption isotherm of SBA-NH_2

Procedure for the Synthesis of 1, 3-dipropylimidazolium acetate-grafted- SBA-μ-Im

0.04 g (1.2 mmol) of paraformaldehyde was stirred in 7 mL of toluene and cooled to 0 ^{o}C. To this 0.100 g of 3-aminopropyl-grafted-SBA-15 was added. 0.25 mL (1.2 mmol) of glacial acetic acid was added dropwise to the reaction. After this the mixture was allowed to reach room temperature. 0.15 mL (1.2 mmol) of glyoxal and an additional 7 mL of toluene were then added and the reaction allowed to stir at room temperature for 1 hour. The solution was then heated to reflux using a Dean Stark apparatus for 3 h. Using rotary evaporation the solvent was removed resulting in an orange powder weighing 0.0630 g. ^{29}SI MAS (79.51MHz), δ -59.182 (T2), -66.895 (T3), -101.055 (Q3), -109.885 (Q4). 13C (100.65MHz, MAS 7.00, 11.5kHz), δ 9.5 (CH_2), 20.9 (CH_2), 44.4 (CH_2), 51.9 (CH_2), 61.3 (CH_3), 99.5, 104.5, 123.4 (CH), 136.6 (CH), 173.3 (Cq). IR (2 mg/ 200 mg KBr, 64 scans, 4 cm^{-1} resolution) cm^{-1} 3433, 2957, 1734, 1652, 1374, 1089, 801, 463.

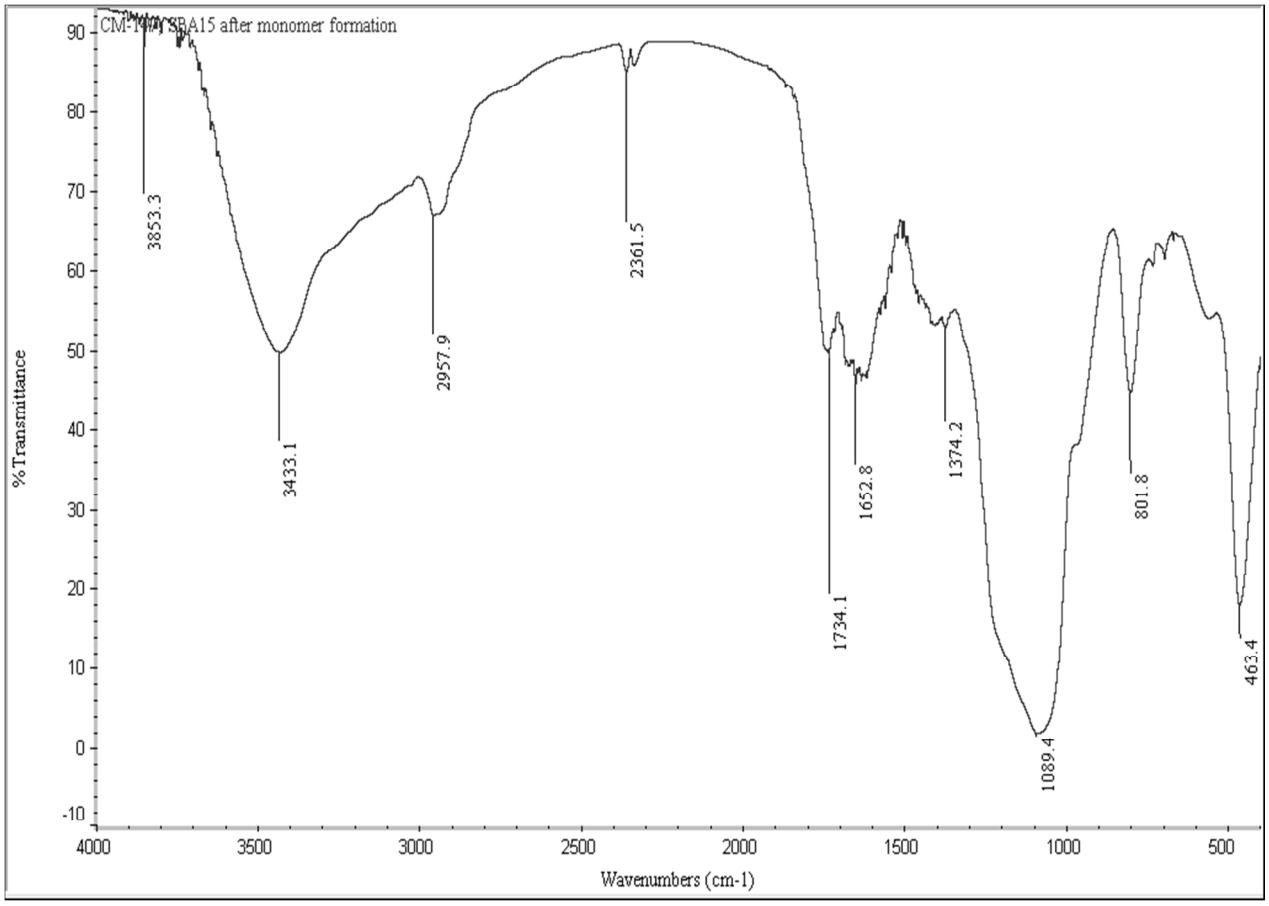

Figure S-8. IR spectra of SBA-μ-Im

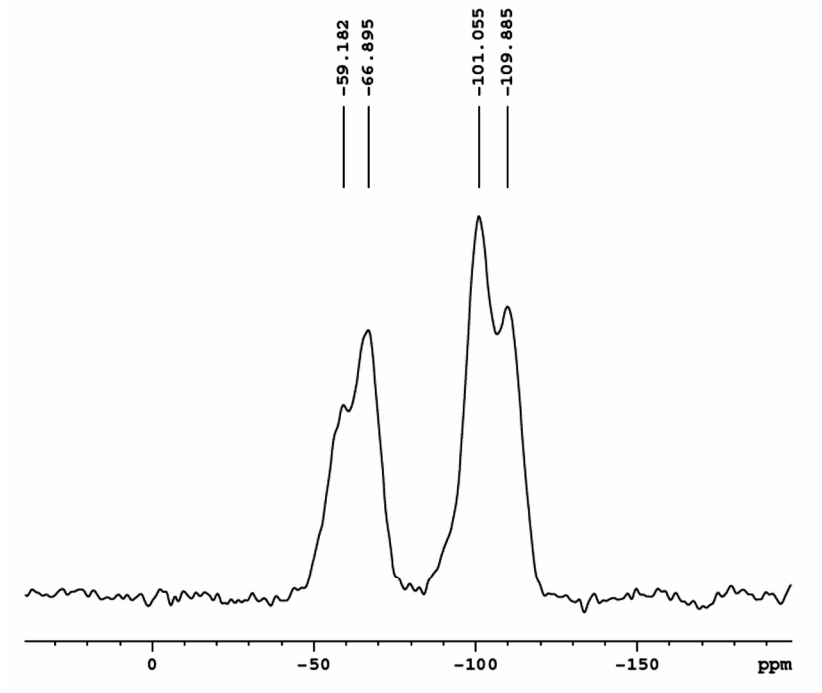

Figure S-9. ^{29}Si solid state CP/MAS NMR of SBA-μ-Im

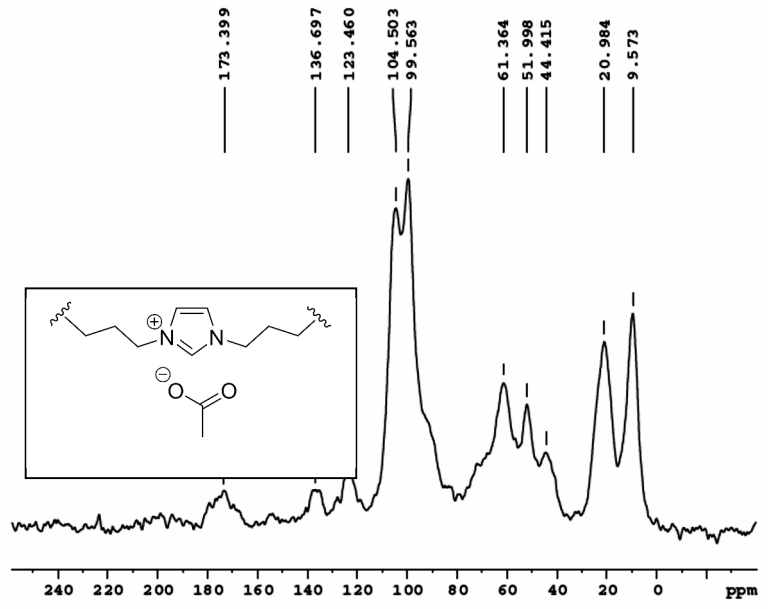

Figure S-10. ^{13}C solid state CP/MAS NRM of SBA-μ-Im

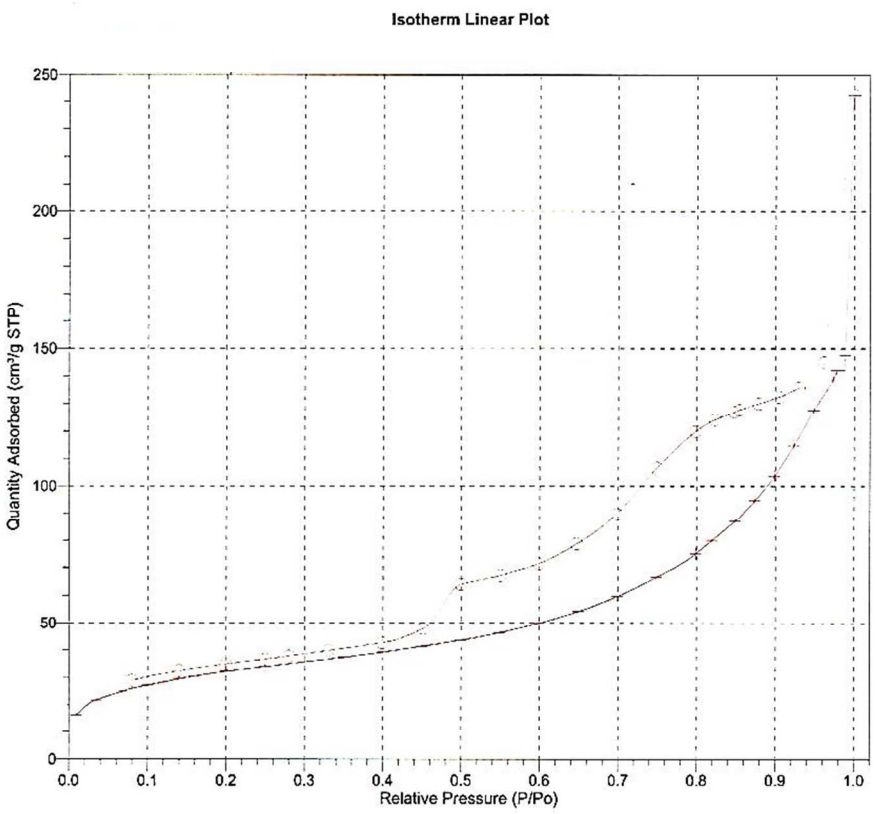

Figure S-11. Nitrogen physisorption isotherm of SBA-μ-Im

Compound	BET Surface Area (m^2/g)	BJH Pore Diameter (Å)	BJH Pore Volume (cm^3/g)
SBA-15	948	59	1.44
SBA-NH_2	356	60	0.54
SBA-μ-Im	111	61	0.22

Table S-1. Summary of nitrogen physisorption isotherms

Procedure for the synthesis of thioketone

0.0630 g of 1, 3-dipropylimidazolium acetate-grafted-SBA-15 was added to a flask containing 10 mL of methanol and stirred. 0.03 g of sodium carbonate and 0.01 g of sulfur were added and the reaction was heated to reflux for 24 h. The solution was then hot filtered to remove excess sulfur. The result was a black solid weighing 0.0643 g. Elemental analysis gave 1.17% sulfur.

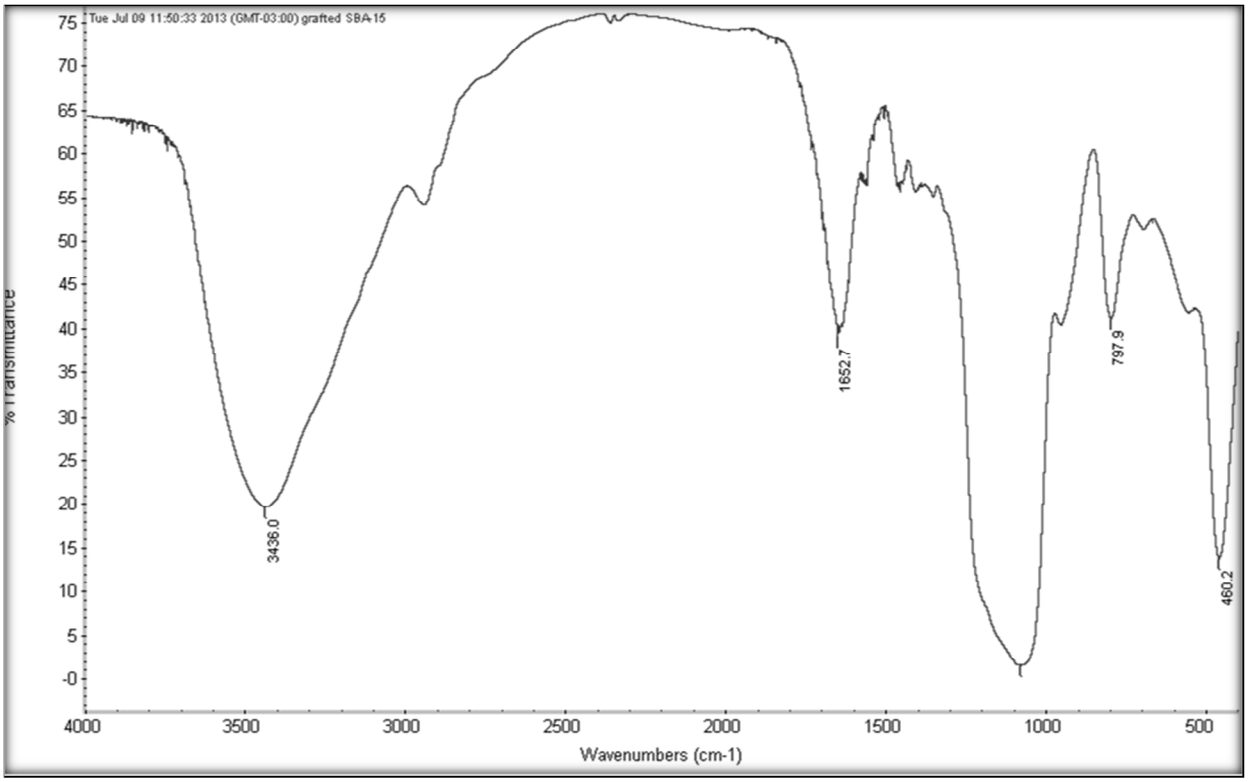

Figure S-12. IR spectra of SBA-μ-Im-CO_2