Supplementary Information for:

Synthesis of Hydrophobic MIL-53(Al)-nanoparticles in Low Weight Alcohols: Systematic Investigation of Solvent Effects

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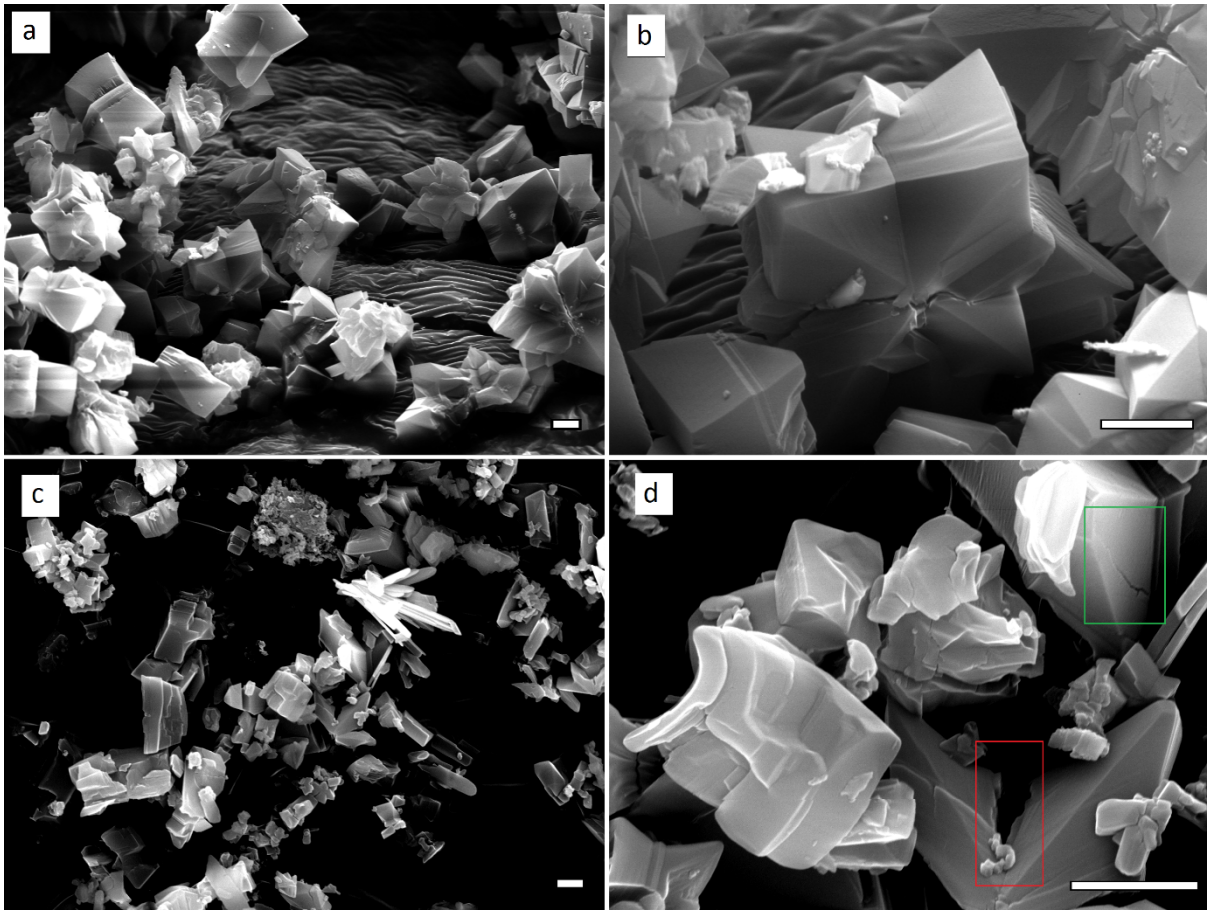

Figure S1: SEM images of MIL-53(H_2O). AsSyn sample is shown at low magnification (a) and higher magnification (b). MIL-53 grows in water in orthorhombic shape from individual seeds leading to micrometre sized particles. After activation, the overall size, morphology and crystallinity of the particles does not change, but cracks (d, green square) and breaking of intergrown crystals (d red square) was observed. Scale bar: 1 µm.

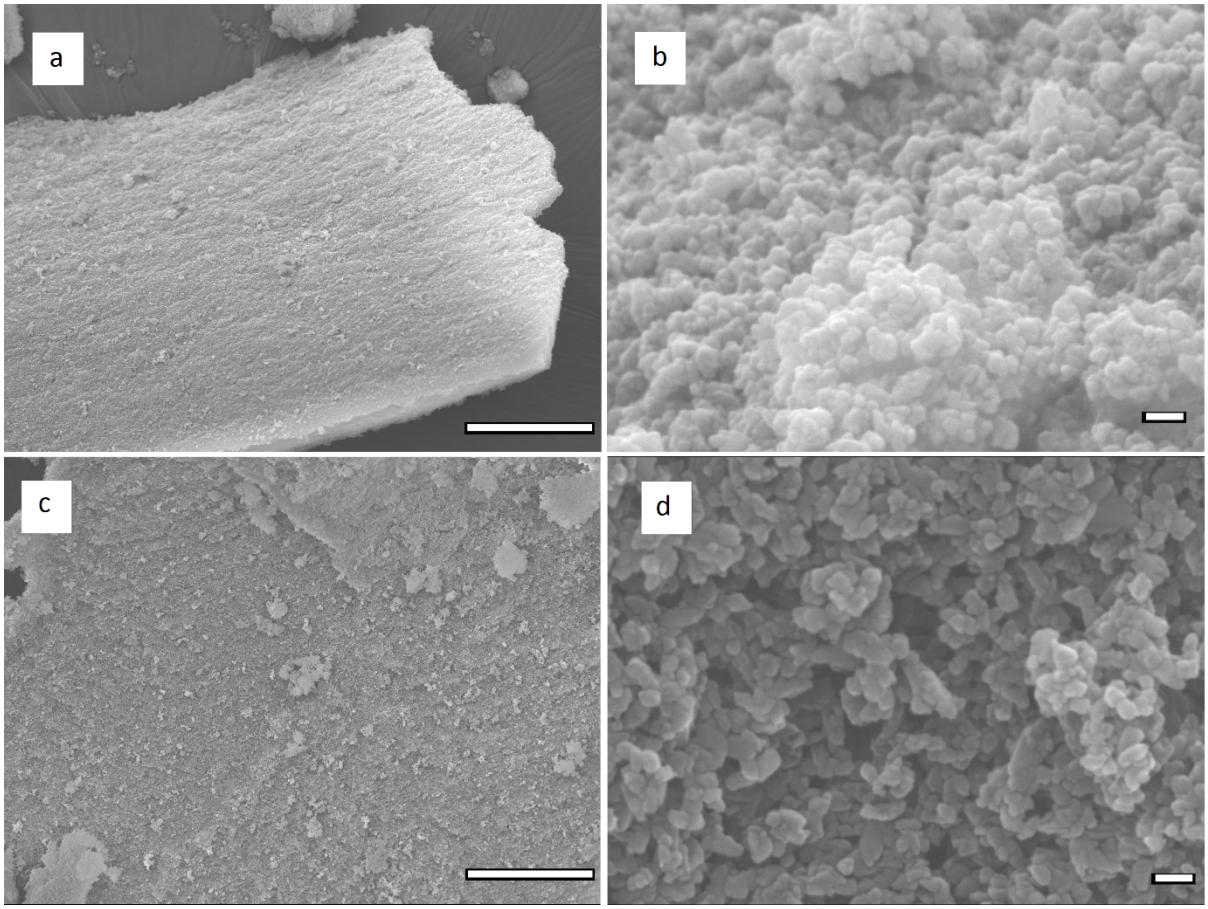

Figure S2: SEM images of MIL-53(DMF). For the AsSyn sample the agglomeration of particles to several micrometre is observed in low magnification (a). In higher magnification, the MIL-53(DMF) particles have a spherical shape and size of 41±17nm. After activation, (c for lower magnification and d for higher magnification) no difference was observed. Scale bar in a and c: 10 µm; Scale bar in b and d: 100 nm.

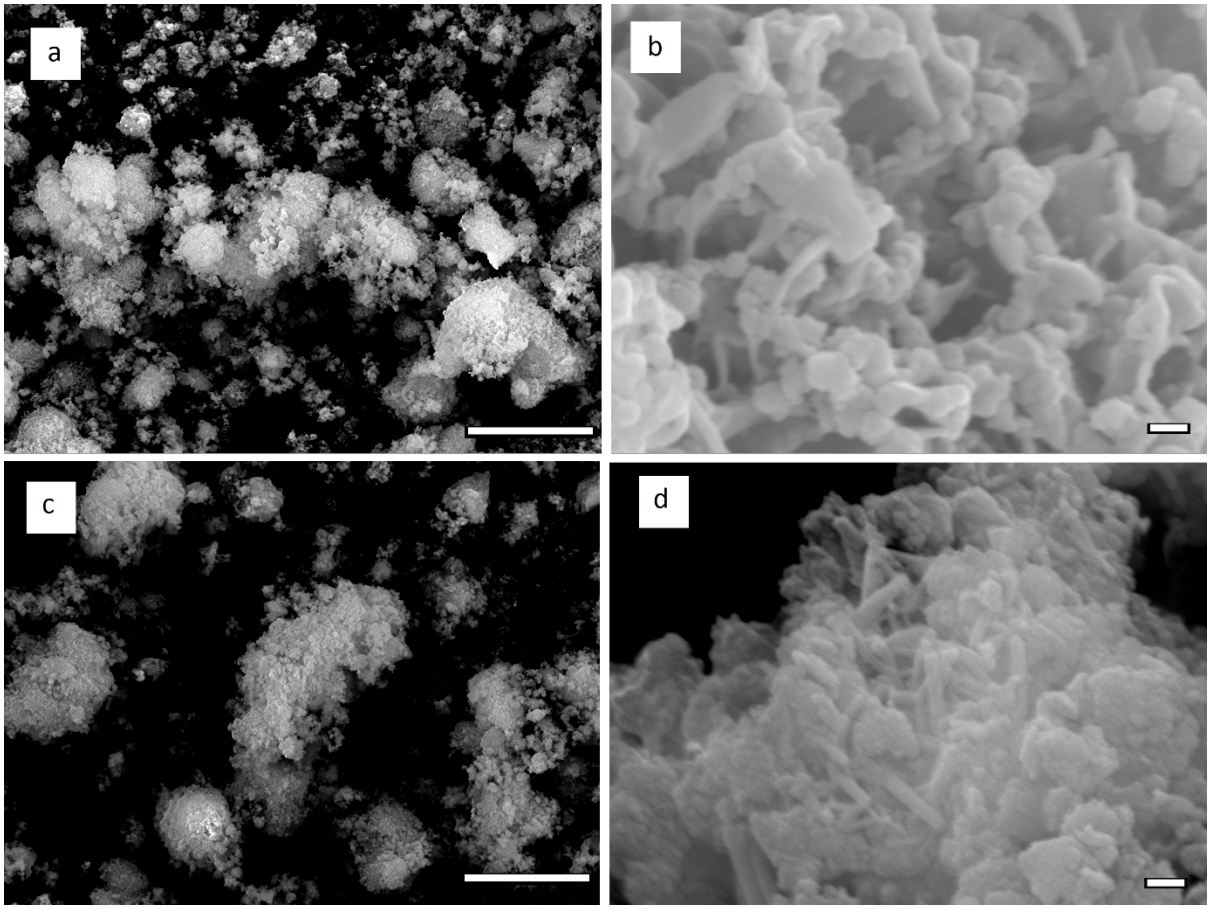

Figure S3: SEM images of MIL-53(MeOH). For the AsSyn sample the agglomeration is observed in low magnification (a). In contrast to MIL-53(DMF), the nano-sized particles are a mixture of spherical particles with size of 34±15nm and rod-shaped particles with a length of 178±42nm and a width of 33±8nm. After activation, (c for lower magnification and d for higher magnification) particles size or morphology has not changed. Scale bar in a and c: 10 µm; Scale bar in b and d: 100 nm.

Table S1: H_2BDC solubility at room temperature and at reaction temperature of 150°C (for methanol solubility at 160°C). The values are given as g per 100g solvent.

Solvent	Solubility at 25°C	Solubility at reaction temperature
Water	0.0065[S1]	0.0332[S2]
DMF	7.4[S3]	14.49[S4]
Methanol	0.11[S5]	2.9[S6]

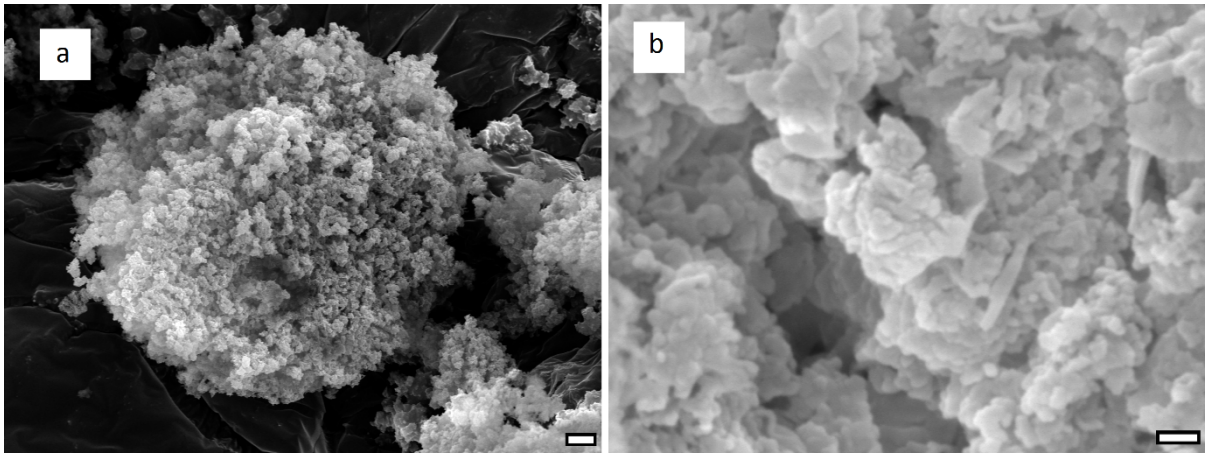

Figure S4: SEM images of MIL53-(EtOH)-AsSyn. The shown sample was activated at elevated temperature. The sample tends to agglomerate (left, a) and show similar particle morphology to MIL53-(MeOH) (right, b). Scale bar in a: 1 µm, Scale bar in b: 100 nm.

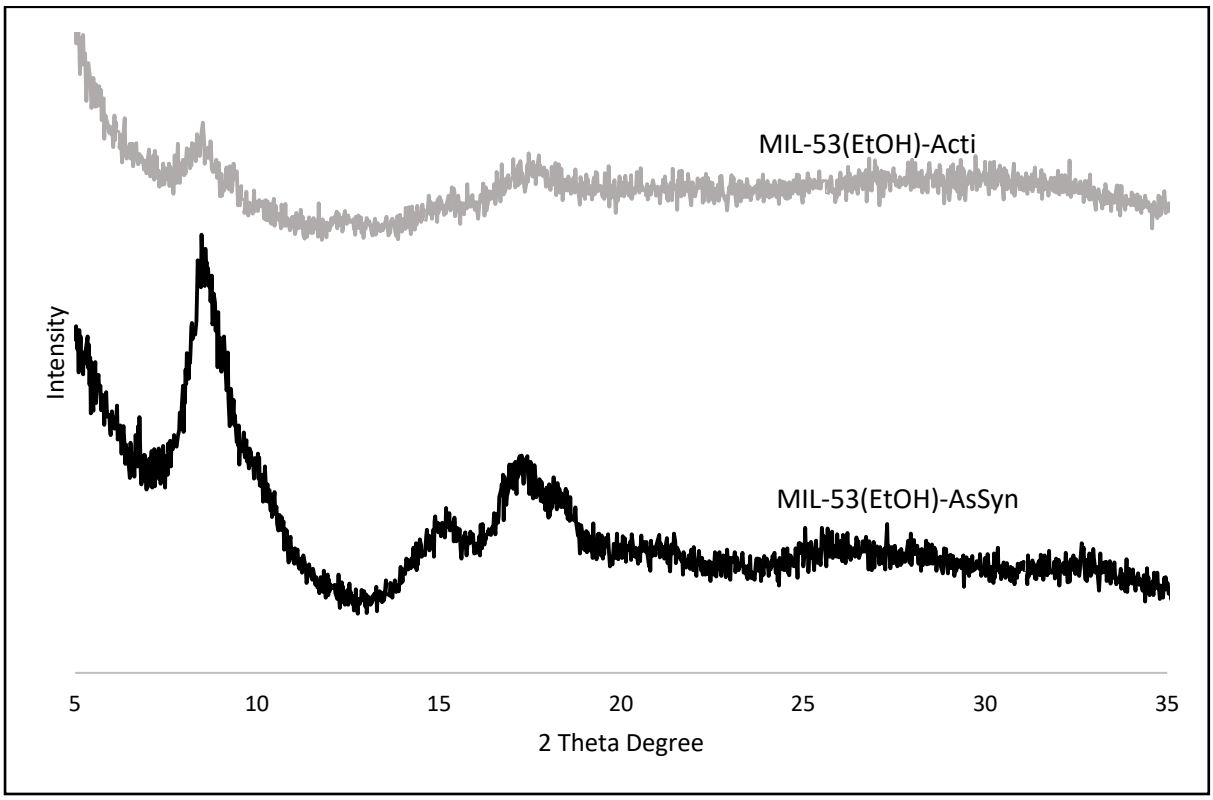

Figure S5: XRD pattern of MIL-53(EtOH). In the AsSyn sample (black) the characteristic peaks for the large pore form can be identified. After activation (grey) MIL-53 decomposes into an amorphous product.

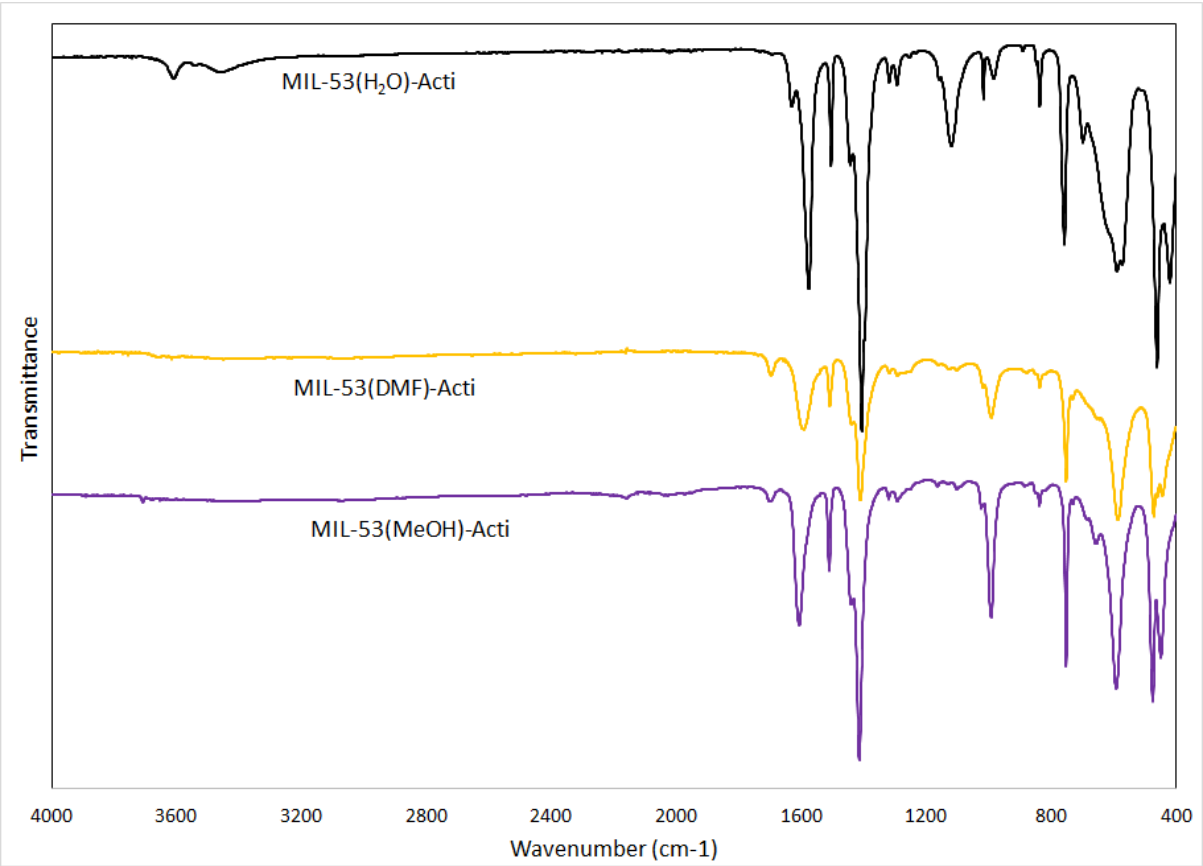

Figure S6: FTIR spectrum of MIL-53(H_2O)-Acti (black), MIL-53(DMF)-Acti (yellow) and MIL-53(MeOH)-Acti (purple).

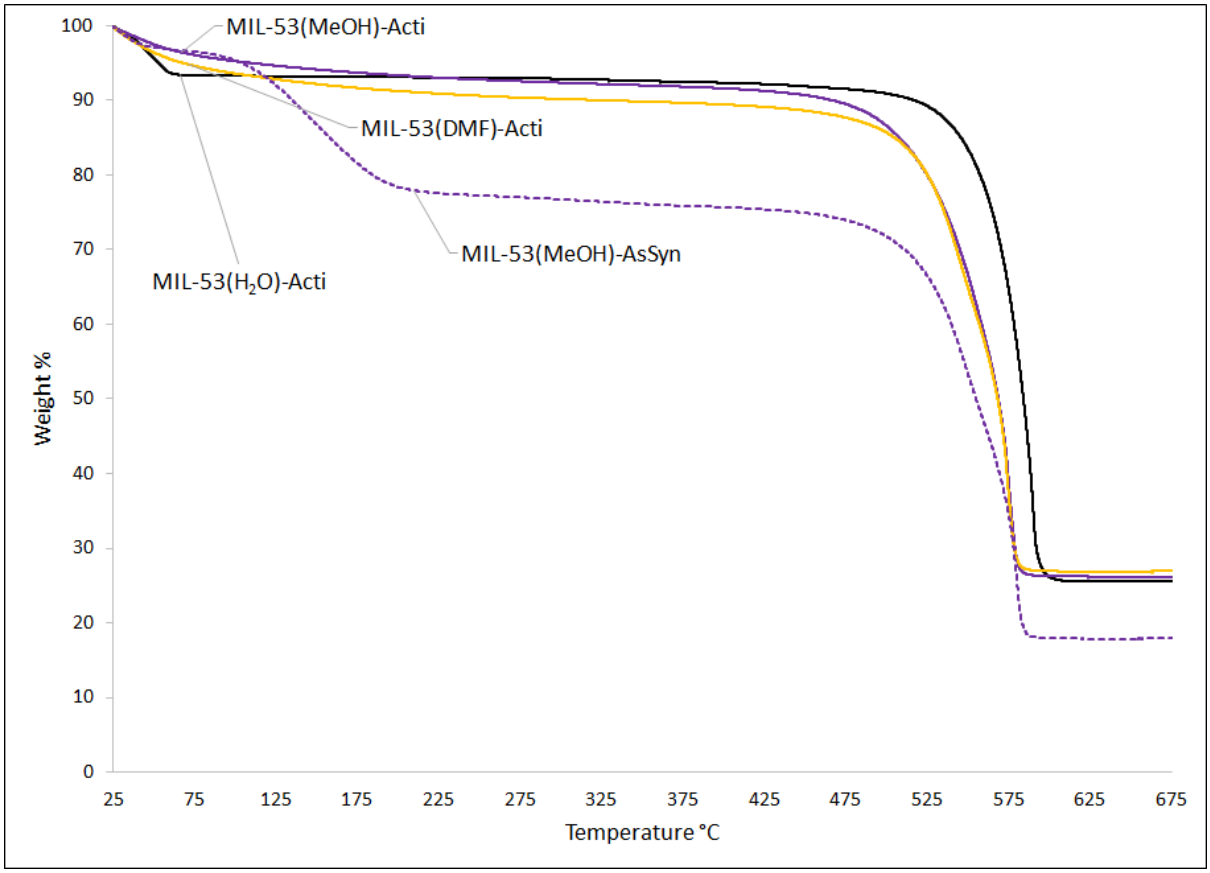

Figure S7: TGA curves of activated MIL-53 prepared in water (black), DMF (yellow) and methanol (purple).TGA curve of MIL-53(MeOH)-AsSyn is shown as purple, dotted line

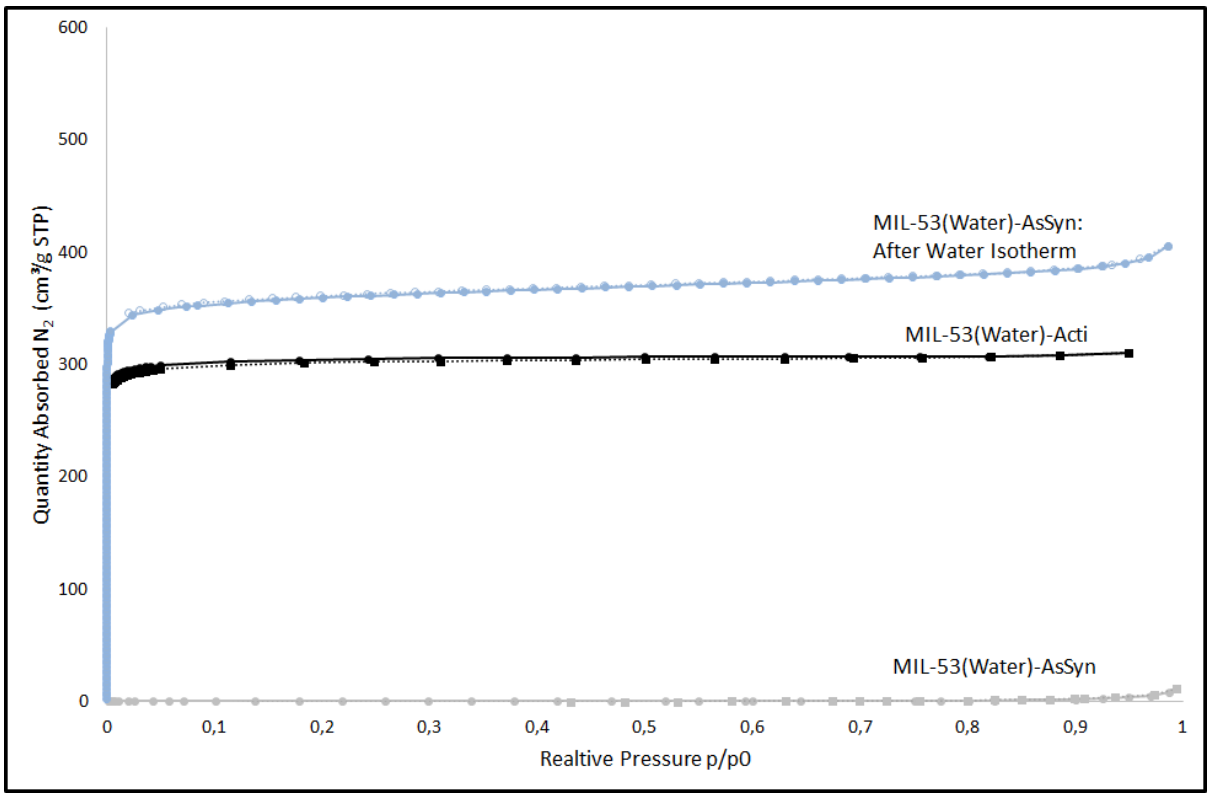

Figure S8: N_2 isotherms for MIL-53(H_2O)-AsSyn (grey), MIL-53(H_2O)-Acti (black) and MIL-53(H_2O)-Acti after measurement of water isotherms (blue). Full line: Adsorption; Dotted line: Desorption.

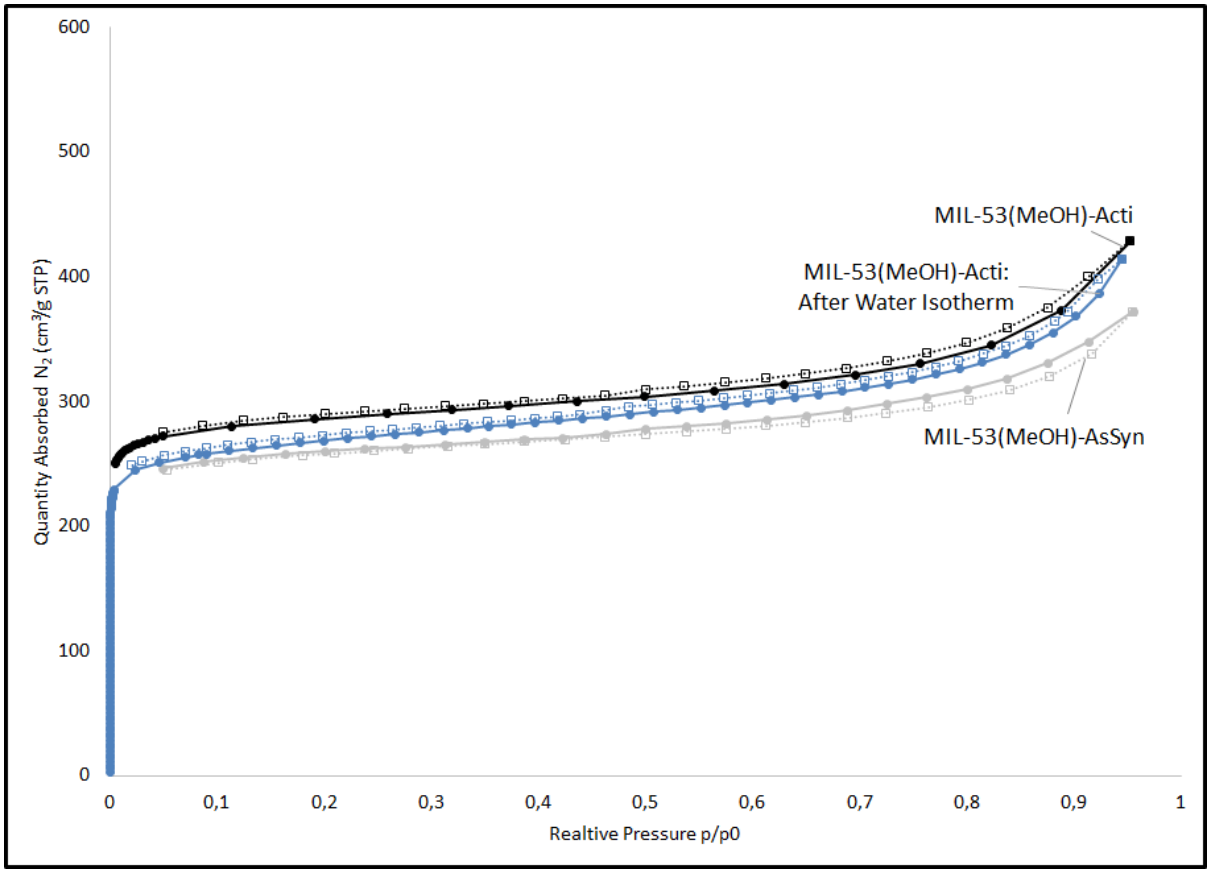

Figure S9: N_2 isotherms for MIL-53(MeOH)-AsSyn (grey), MIL-53(MeOH)-Acti (black) and MIL-53(MeOH)-Acti after measurement of water isotherms (blue). Full line: Adsorption; Dotted line: Desorption.

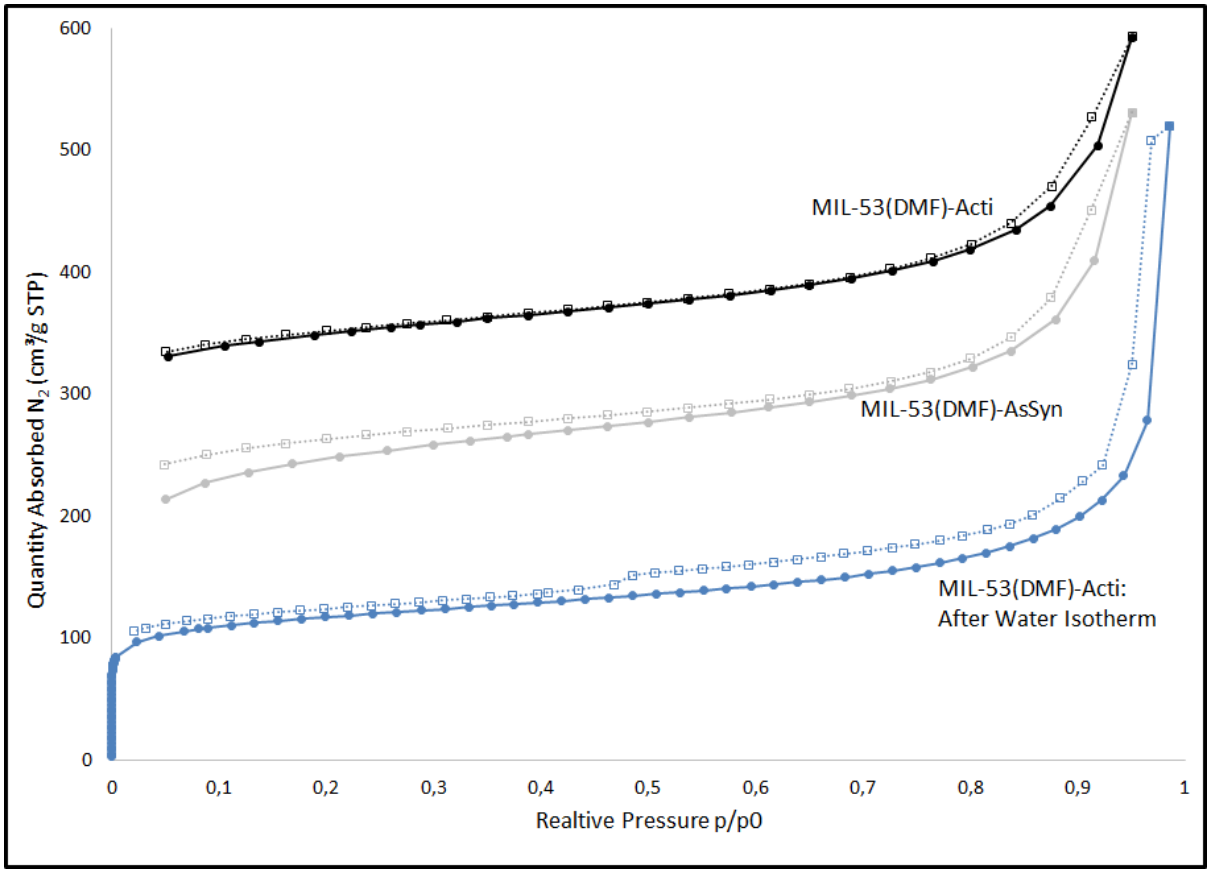

Figure S10: N_2 isotherms for MIL-53(DMF)-AsSyn (grey), MIL-53(DMF)-Acti (black) and MIL-53(DMF)-Acti after measurement of water isotherm (blue). Full line: Adsorption; Dotted line: Desorption.

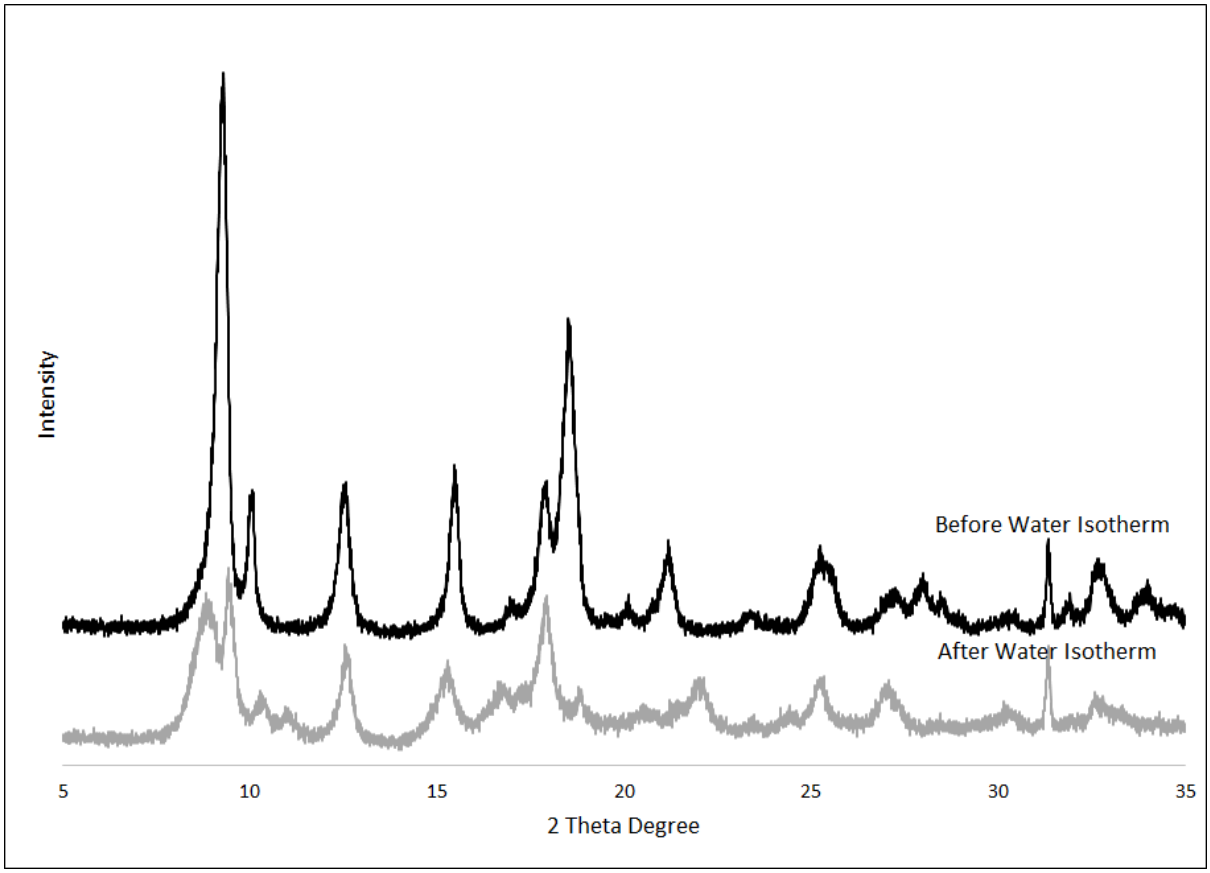

Figure S4: XRD pattern of MIL-53(DMF)-Acti before (black) and after (grey) measurement of water isotherms.

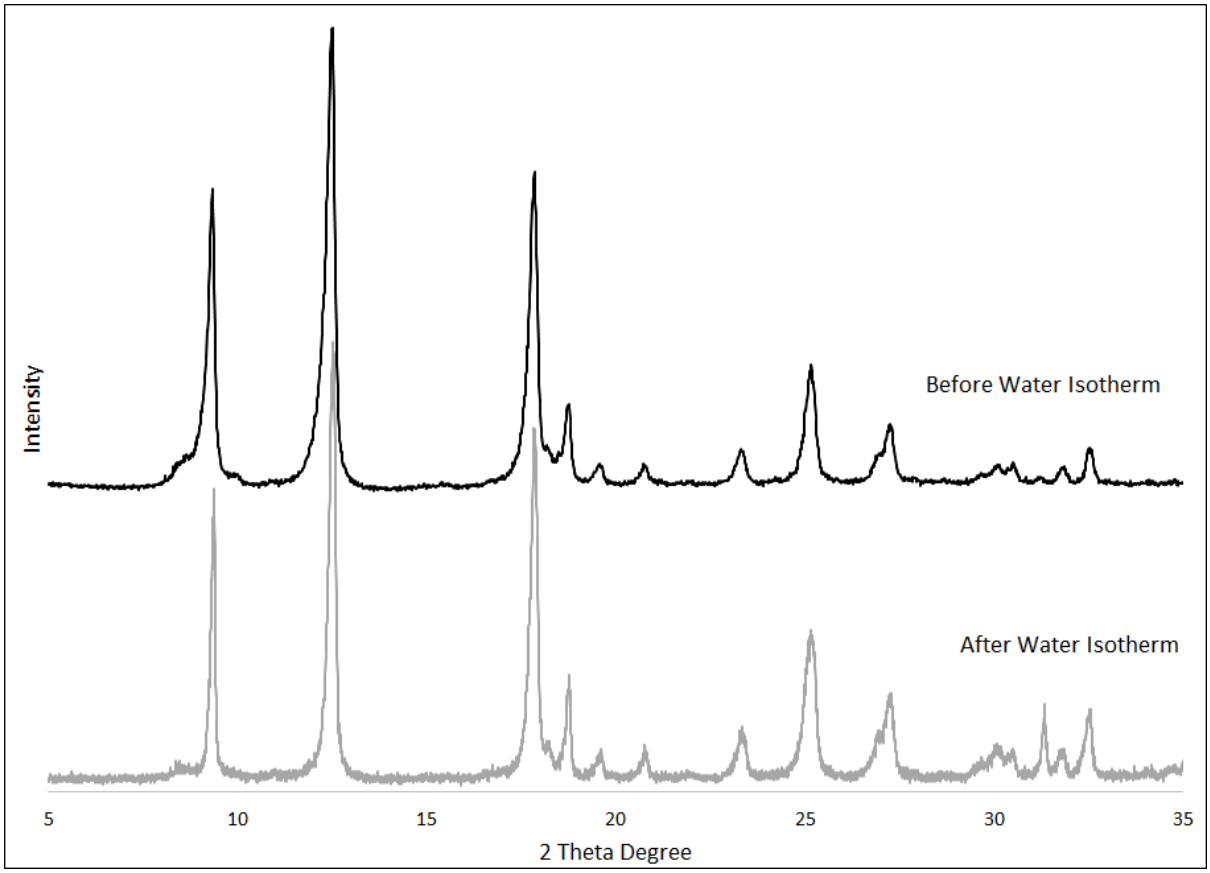

Figure S5: XRD pattern of MIL-53(H_2O)-Acti before (black) and after (grey) measurement of water isotherms.

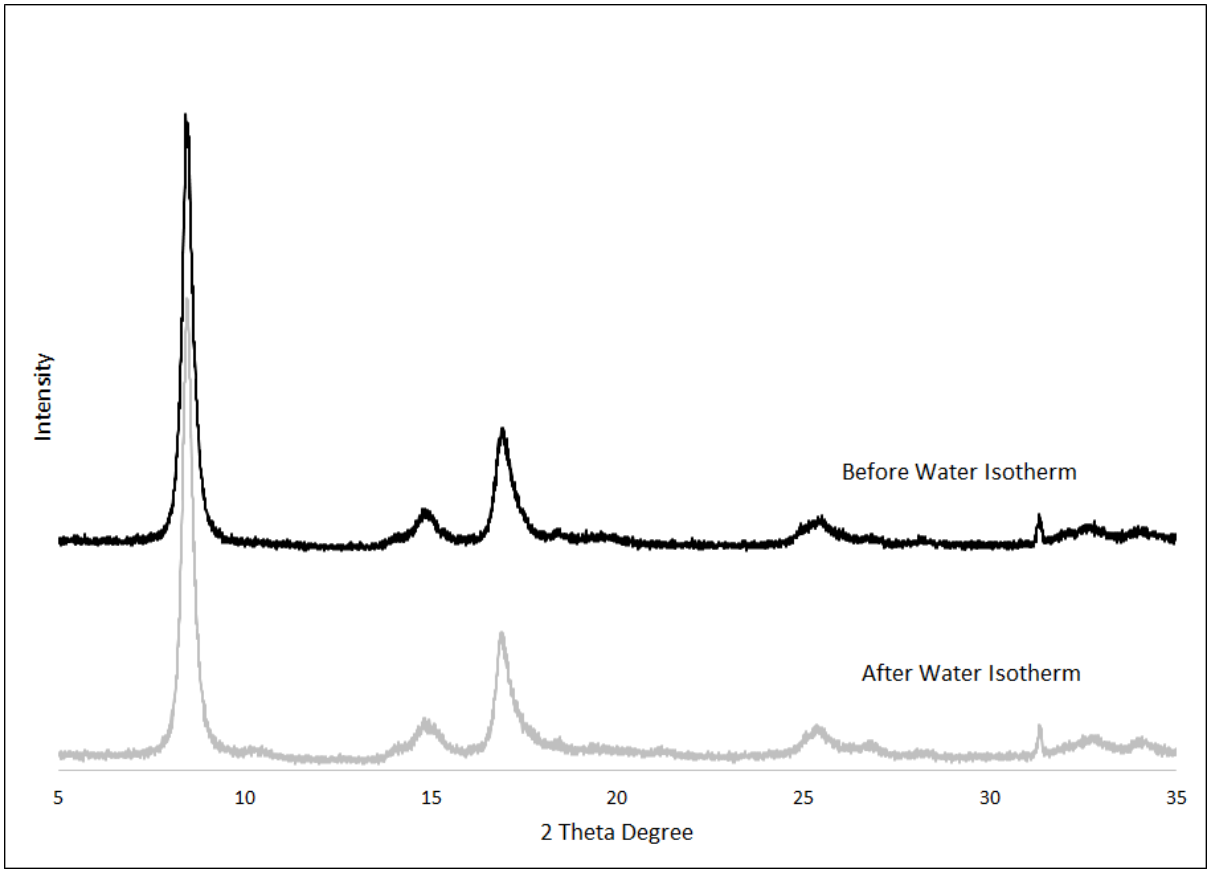

Figure S6: XRD pattern of MIL-53(MeOH)-Acti before (black) and after (grey) measurement of water isotherms.

Table S2: BET-area and micropore volume after measurement of water isotherms.

Compound	BET-area(m^2/g)	t-plot micropore Volume (cm^3/g)
MIL-53(H_2O)-Acti	1021	0.48
MIL-53(DMF)-Acti	377	0.18
MIL-53(MeOH)-Acti	862	0.34

References

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