

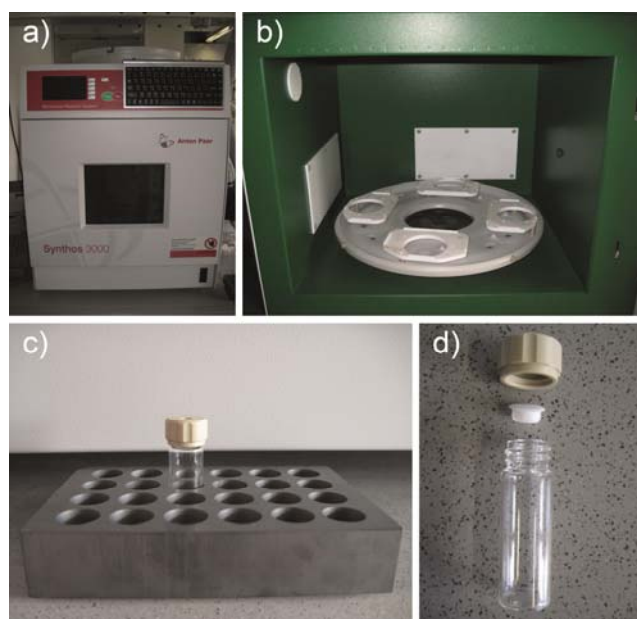
# Supporting Information

## Thermal post-synthetic modification of Al-MIL-53-COOH: systematic investigation of the decarboxylation and condensation reaction

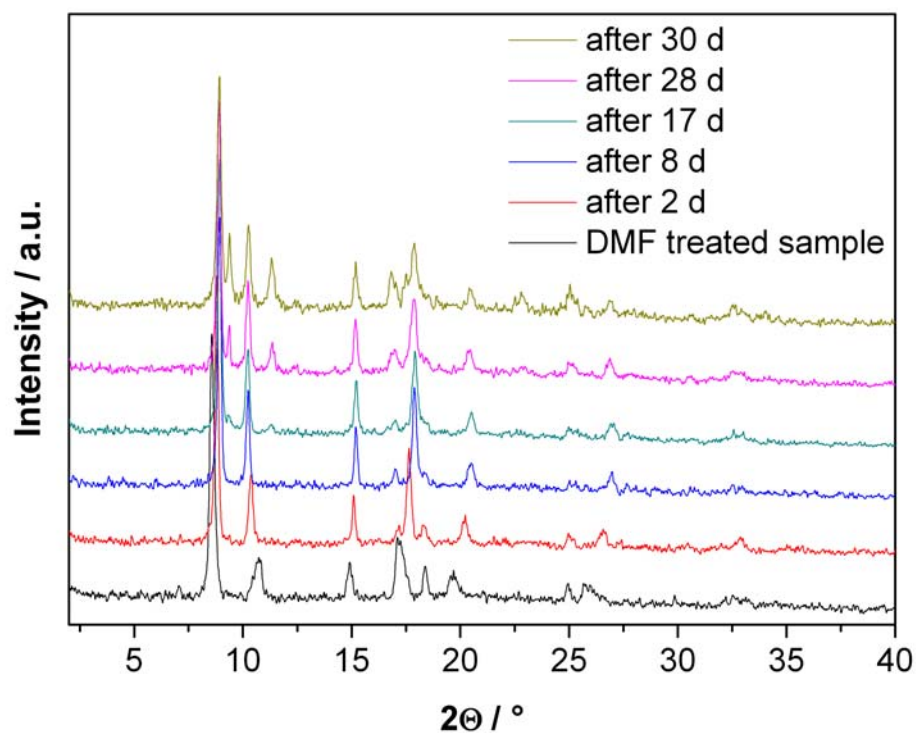
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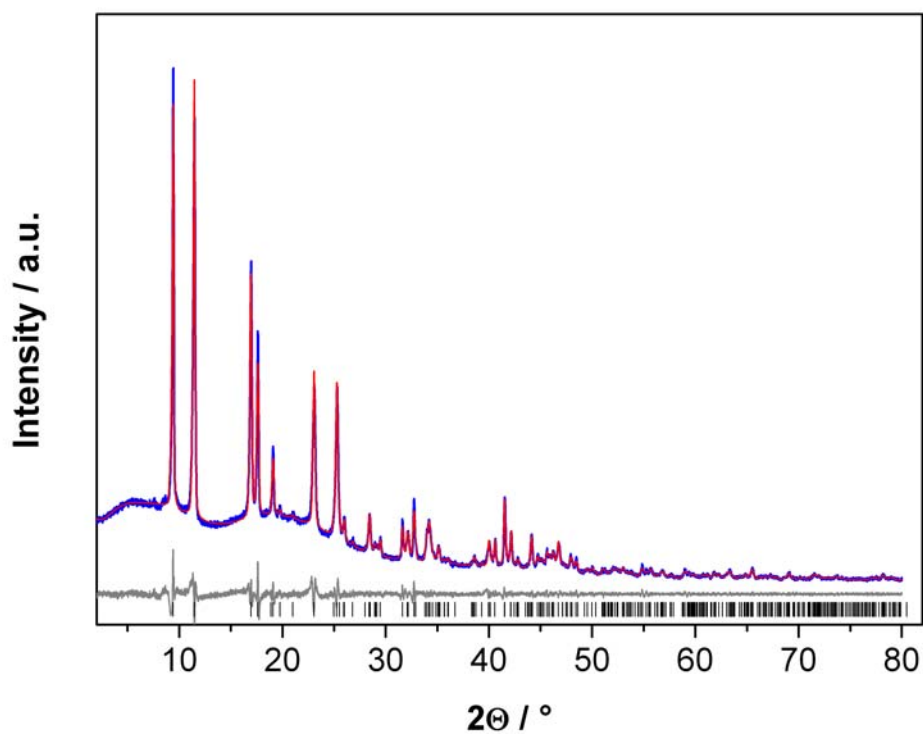
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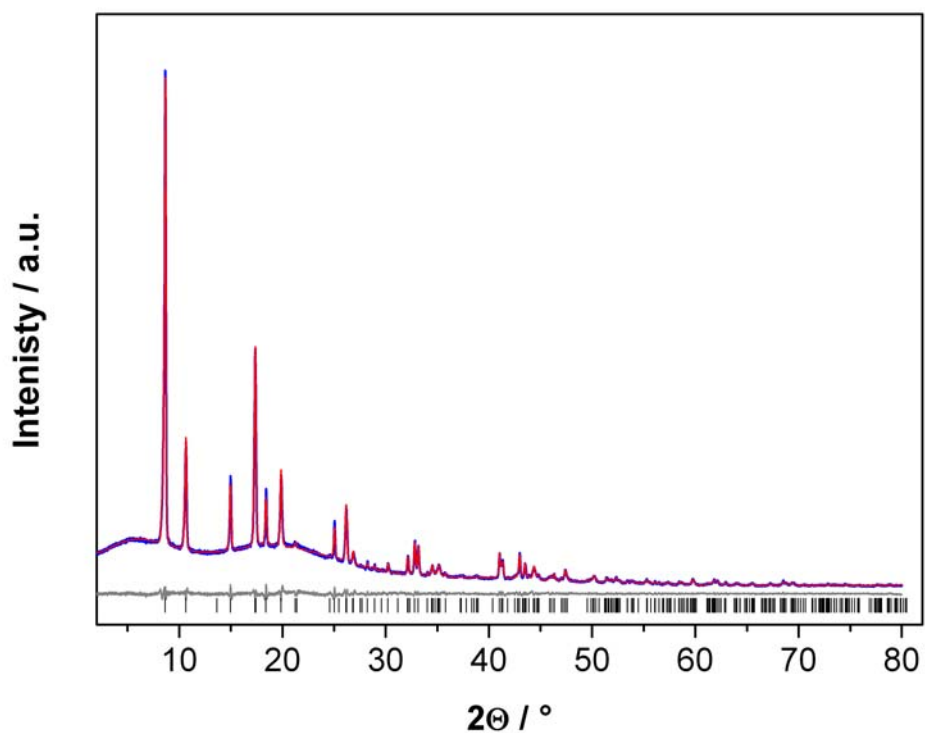
**Fig. S1** a) Anton Paar Synthos 3000 microwave oven, b) 96-position rotor, c) silicon carbide reaction block with 4 x 6 array, d) glass vial with Teflon seal and screw cap.



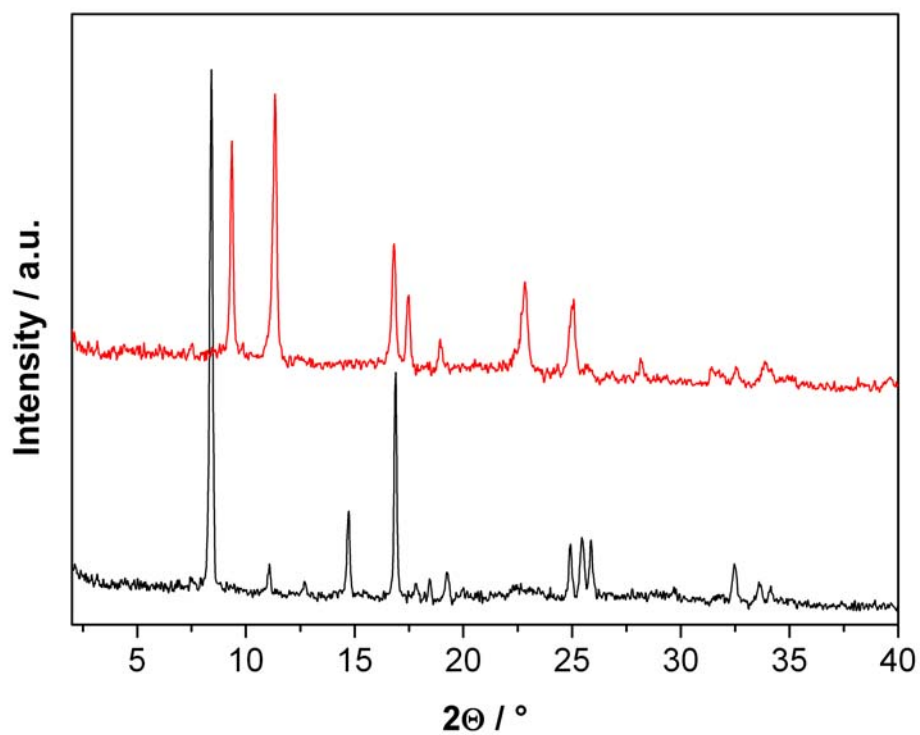
**Fig. S2** Powder patterns of Al-MIL-53-COOH(DMF) showing the structural change towards Al-MIL-53-COOH after at least 8 days under exposure to the atmosphere.



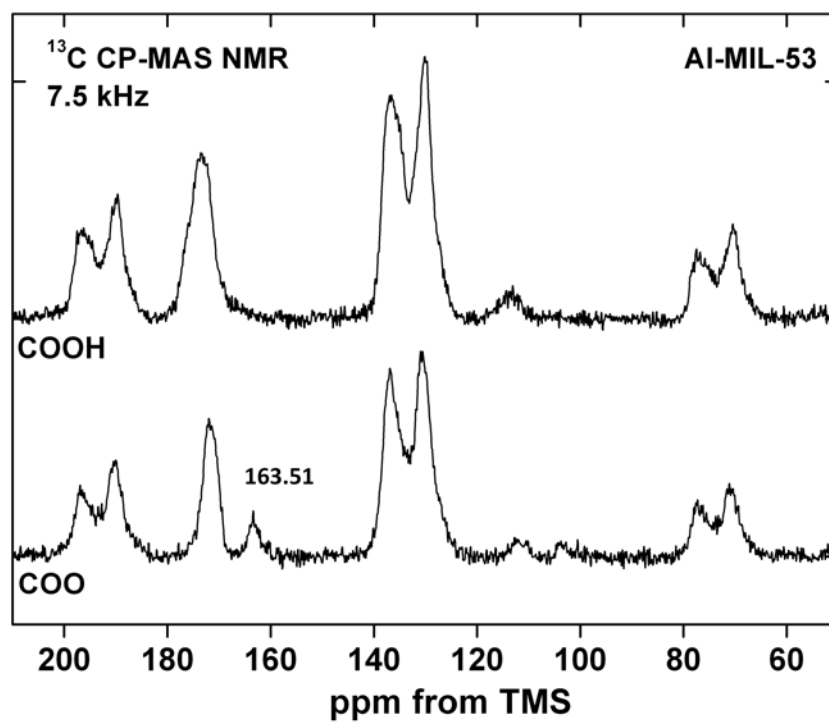
**Fig. S3** Pawley-Fit of Al-MIL-53-COOH.



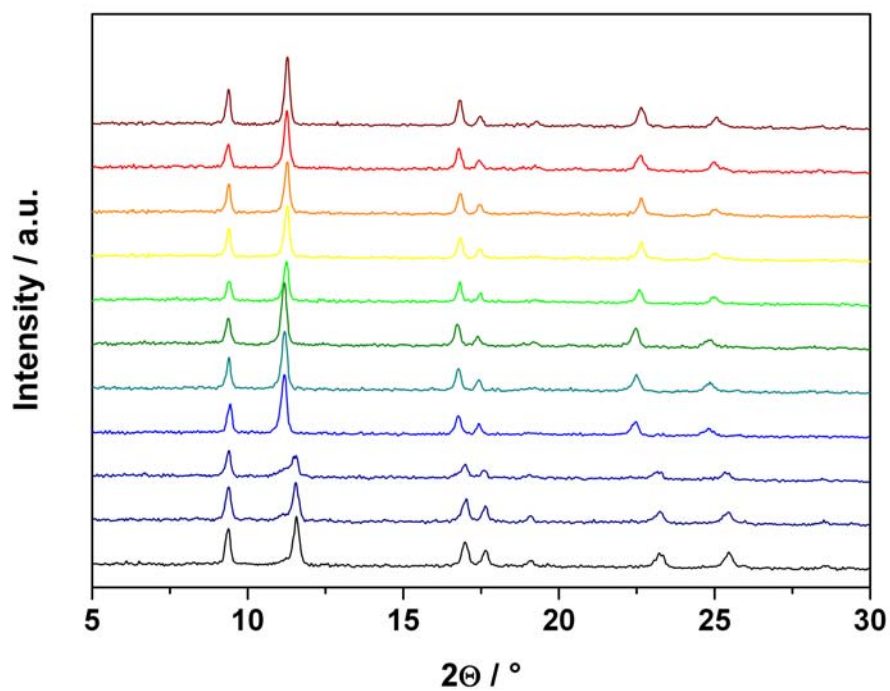
**Fig. S4** Pawley-Fit of Al-MIL-53-COOH(DMF).



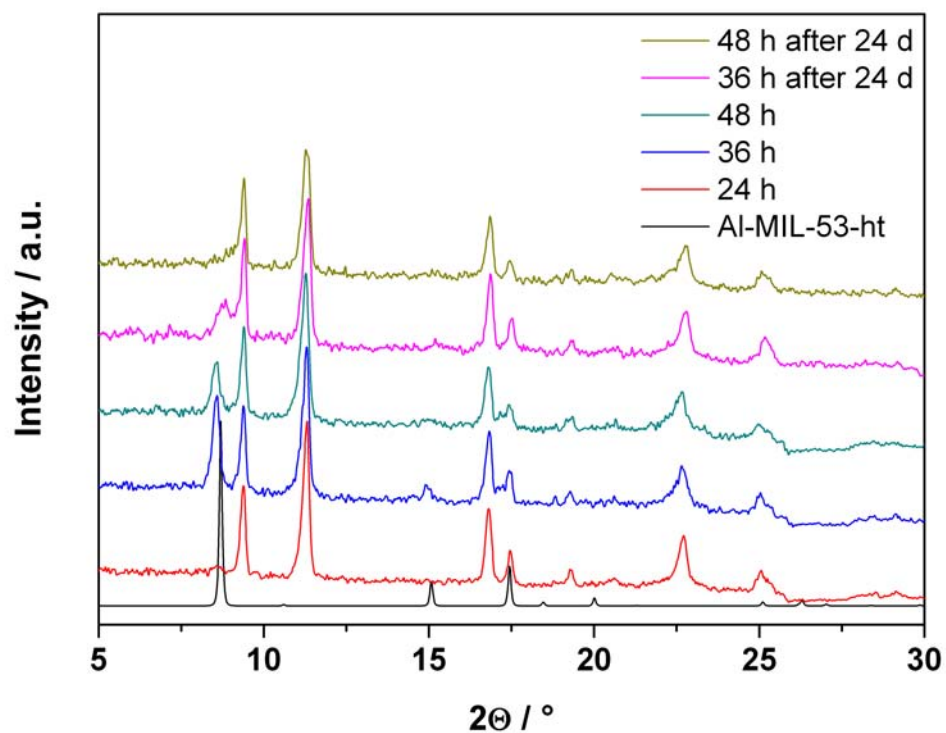
**Fig. S5** Powder patterns of the water treated sample of Al-MIL-53-COOH (black) and the in air dried product (red).



**Fig. S6** <sup>13</sup>C CP-MAS NMR spectra of the activated sample of Al-MIL-53-COOH (410 °C) (bottom) and the non-activated one (top).



**Fig. S7** Powder patterns of the thermal activated samples of Al-MIL-53-COOH for 12 h in the range of 210 °C-410 °C in 20 °C steps



**Fig. S8** Powder patterns of the thermal activated samples of Al-MIL-53-COOH at 410 °C for 24, 36 and 48 h compared with the theoretical powder pattern of Al-MIL-53.