

## Electronic Supplementary Information

### Optimization of the time and temperature of the microwave-assisted amination of the phenylene-PMO

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## Table of Contents

**Figure S1.**  $^{13}\text{C}$  CP-MAS NMR spectra of PMO, NO<sub>2</sub>-PMO\_C and NO<sub>2</sub>-PMO\_x/y materials.

**Figure S2.** FTIR (ATR) spectra of PMO and NO<sub>2</sub>-PMO\_x/y materials.

**Figure S3.** FTIR (ATR) spectra of PMO, NO<sub>2</sub>-PMO\_15min/60°C and NH<sub>2</sub>-PMO\_15min/y materials.

**Table S1.** Texture parameters and physical properties of PMO and NO<sub>2</sub>-PMO\_x/y materials.

**Figure S4.** PXRD of Ph-PMO and NO<sub>2</sub>-Ph-PMO\_15min synthesized at different reaction temperatures during 15 minutes.

**Figure S5.** -196 °C N<sub>2</sub> isotherms and PSD curves of PMO and NO<sub>2</sub>-PMO\_x/y materials.

**Figure S6.** TGA of PMO and NO<sub>2</sub>-PMO\_x/y materials.

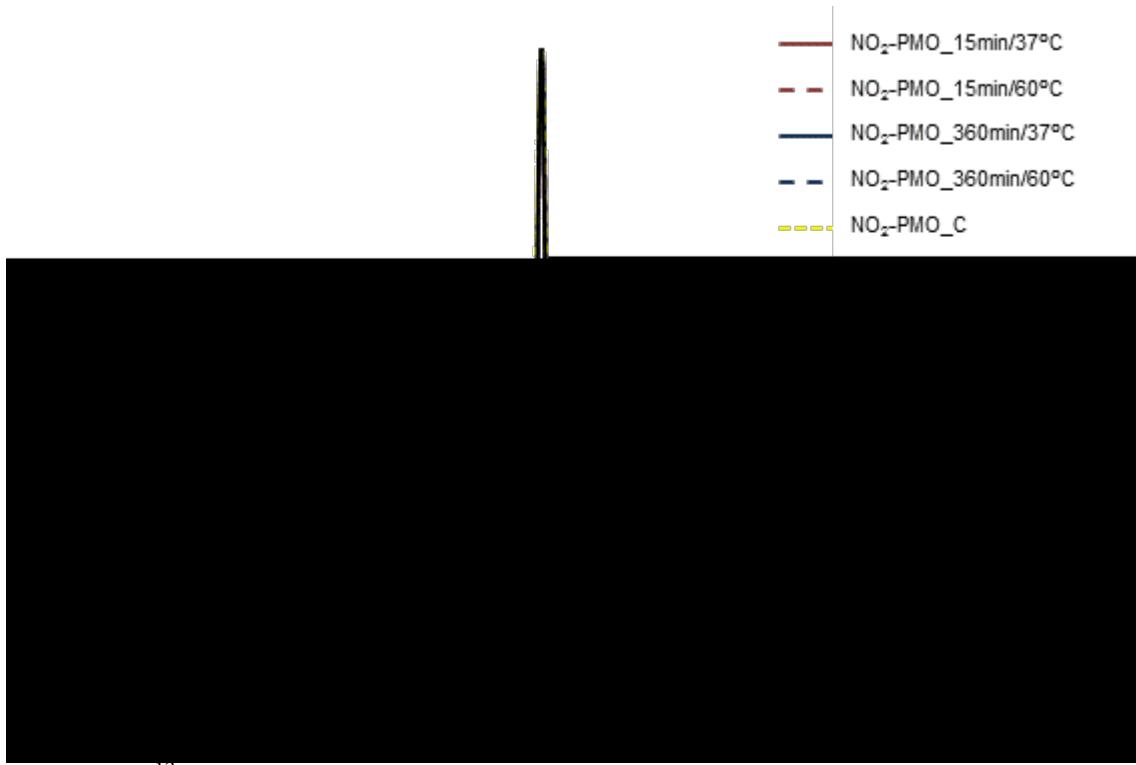
**Figure S7.** SEM images for NO<sub>2</sub>-PMO\_C, NO<sub>2</sub>-PMO\_15min/37°C and NO<sub>2</sub>-PMO\_360min/60°C.

**Figure S8.**  $^{29}\text{Si}$  MAS and CP-MAS NMR spectra of NH<sub>2</sub>-PMO\_15min/60°C.

**Table S2.** Texture parameters and physical properties of PMO, NO<sub>2</sub>-PMO\_15min/60°C and NH<sub>2</sub>-PMO\_15min/y materials.

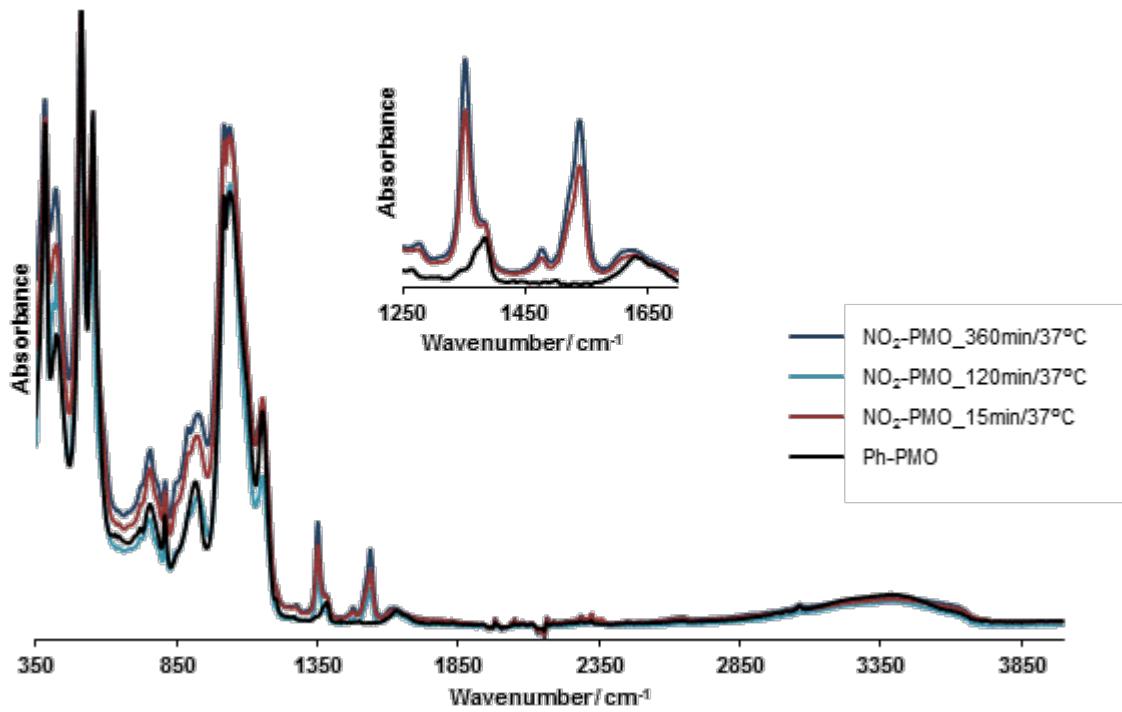
**Figure S9.** -196 °C N<sub>2</sub> isotherms and PSD curves of NO<sub>2</sub>-PMO\_15min/60°C, NH<sub>2</sub>-PMO\_15min/37°C\_C, NH<sub>2</sub>-PMO\_15min/37°C and NH<sub>2</sub>-PMO\_15min/90°C.

**Figure S10.** TGA of PMO, NO<sub>2</sub>-PMO\_15min/60°C, NH<sub>2</sub>-PMO\_15min/37°C and NH<sub>2</sub>-PMO\_15min/90°C materials.

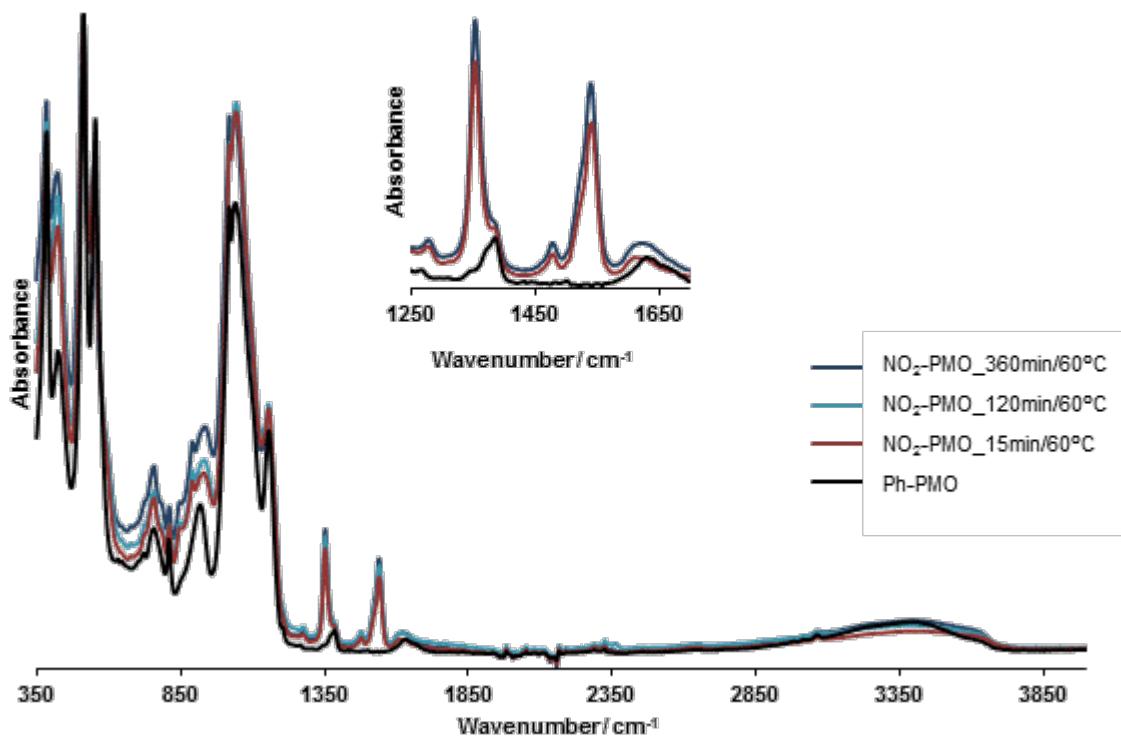


**Figure S1.**  $^{13}\text{C}$  CP MAS NMR spectra for PMO and  $\text{NO}_2\text{-PMO}_{-x/y}$  samples obtained under different synthetic conditions. \* denotes spinning sidebands.

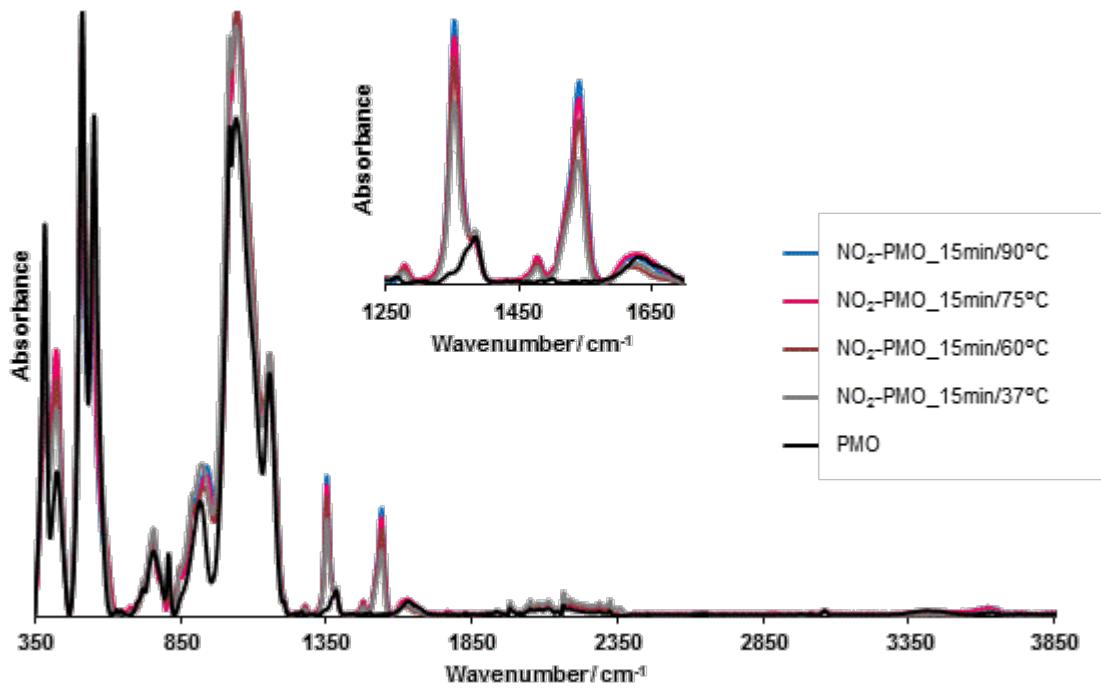
a)



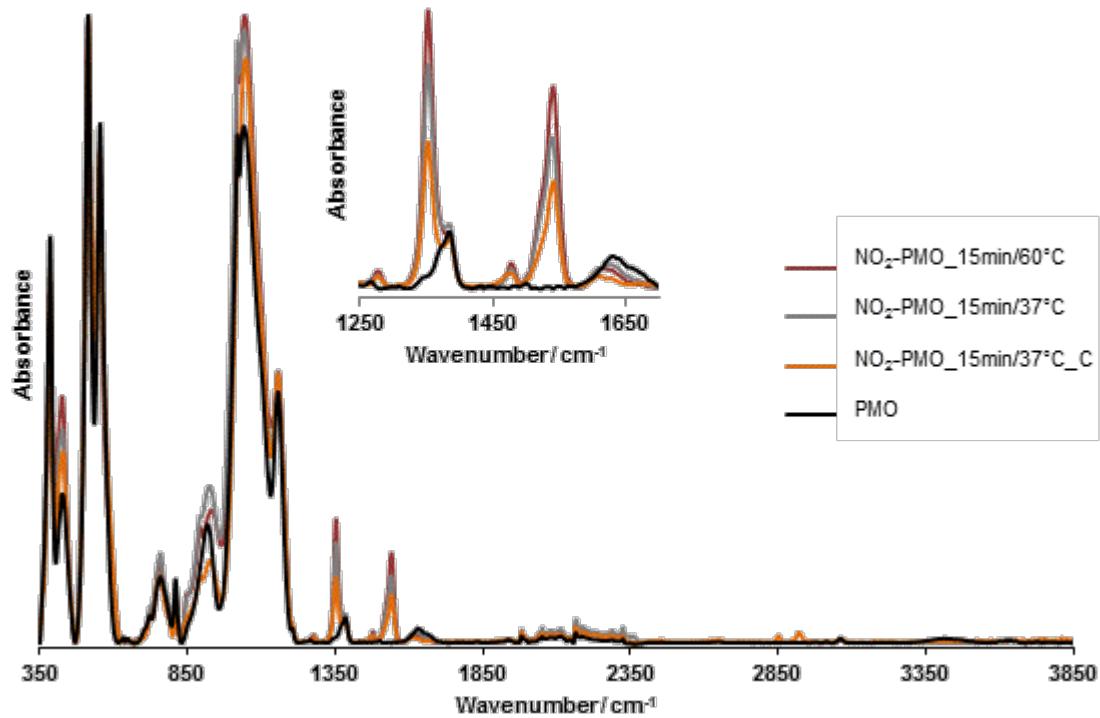
b)



c)

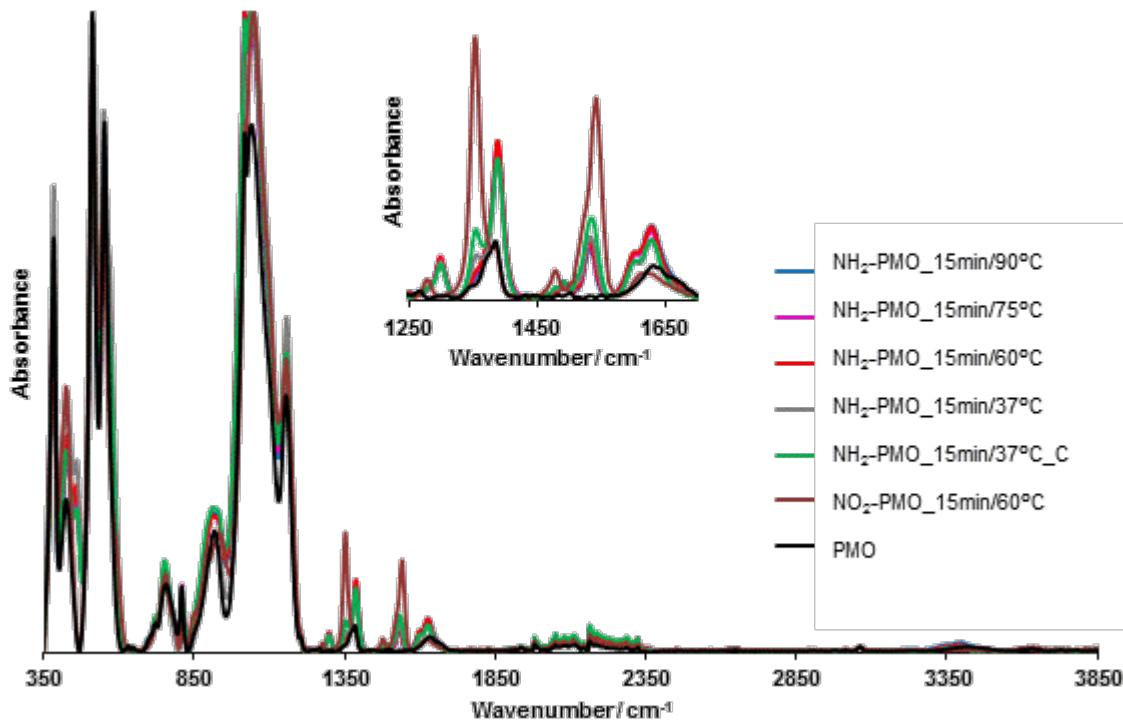


d)



**Figure S2.** FTIR (ATR) spectra of PMO, NO<sub>2</sub>-PMO<sub>x/y</sub> at a) 37 °C and b) 60 °C for different reaction times, c) NO<sub>2</sub>-PMO<sub>15min/y</sub> at different temperatures and d) NO<sub>2</sub>-PMO<sub>15min/y</sub> at different temperatures using microwave and conventional heating. The insets display the region 1250 – 1700 cm<sup>-1</sup> magn

ified.

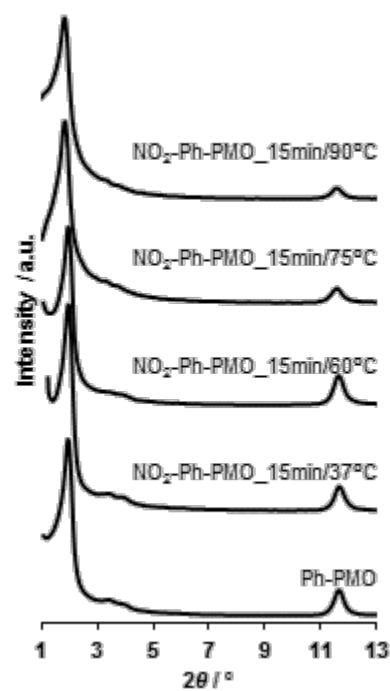


**Figure S3.** FTIR (ATR) spectra of PMO, NO<sub>2</sub>-PMO\_15min/60°C and NH<sub>2</sub>-PMO\_15min/y at different temperatures. The insets display the region 1250 – 1700  $\text{cm}^{-1}$  magnified.

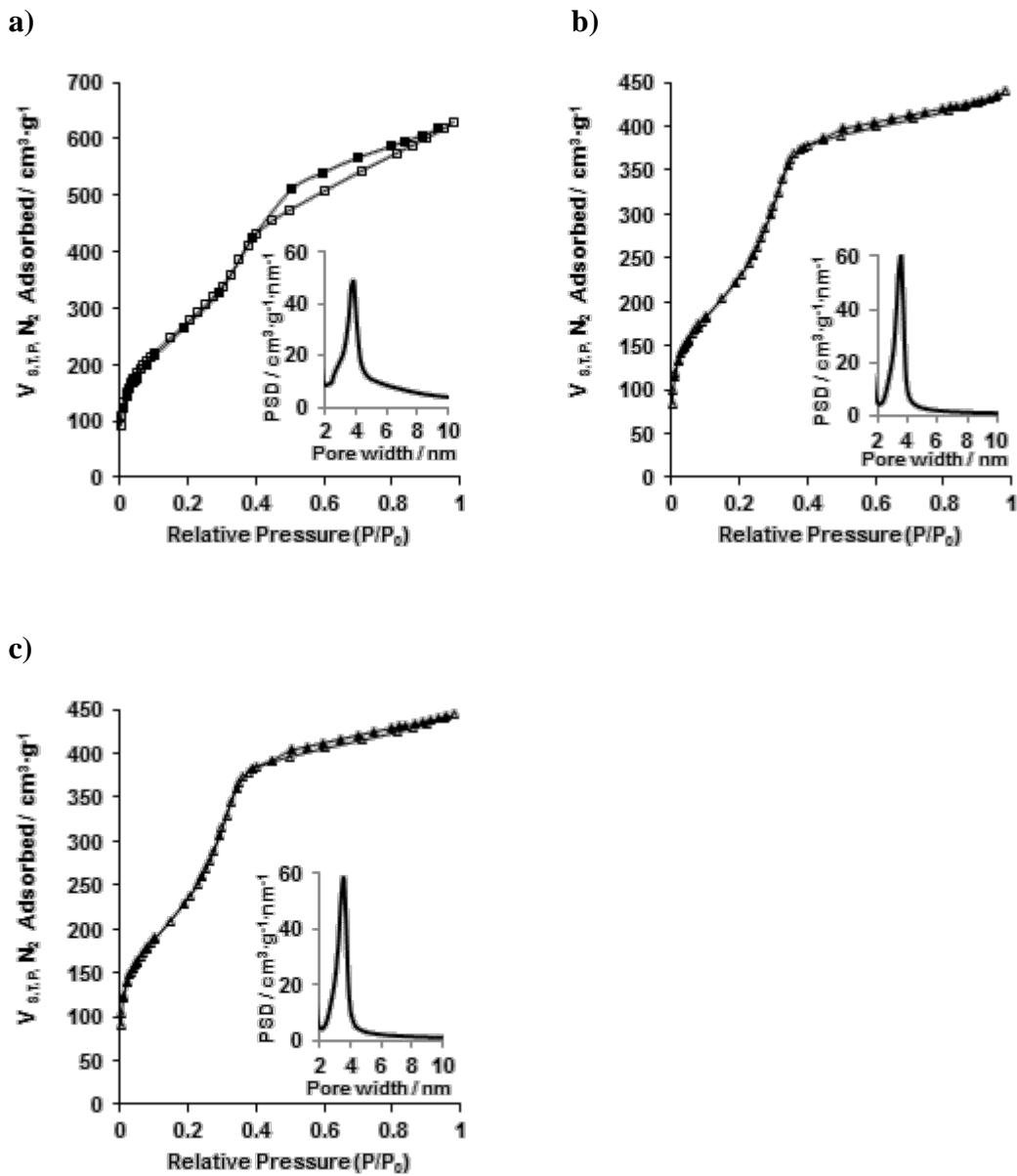
**Table S1.** Textural parameters and physical properties of PMO and NO<sub>2</sub>-PMOs\_x/y.

Sample	$d_{100}$ / nm	$a$ / nm <sup>a</sup>	$S_{\text{BET}}$ / $\text{m}^2 \text{g}^{-1}$	$V_p$ / $\text{cm}^3 \text{g}^{-1}$	$d_p$ / nm <sup>b</sup>	$b$ / nm <sup>c</sup>
PMO	4.55	5.25	920	0.66	3.58	1.67
NO <sub>2</sub> -PMO_15min/37°C	4.70	5.42	788	0.62	3.58	1.84
NO <sub>2</sub> -PMO_120min/37°C	4.50	5.20	757	0.58	3.51	1.69
NO <sub>2</sub> -PMO_360min/37°C	4.65	5.36	626	0.49	3.51	1.85
NO <sub>2</sub> -PMO_15min/60°C	4.70	5.42	698	0.57	3.54	1.88
NO <sub>2</sub> -PMO_120min/60°C	4.55	5.25	743	0.57	3.51	1.74
NO <sub>2</sub> -PMO_360min/60°C	4.55	5.25	808	0.63	3.51	1.74
NO <sub>2</sub> -PMO_15min/75°C	4.82	5.57	730	0.61	3.69	1.88
NO <sub>2</sub> -PMO_15min/90°C	4.82	5.57	776	0.65	3.55	2.02

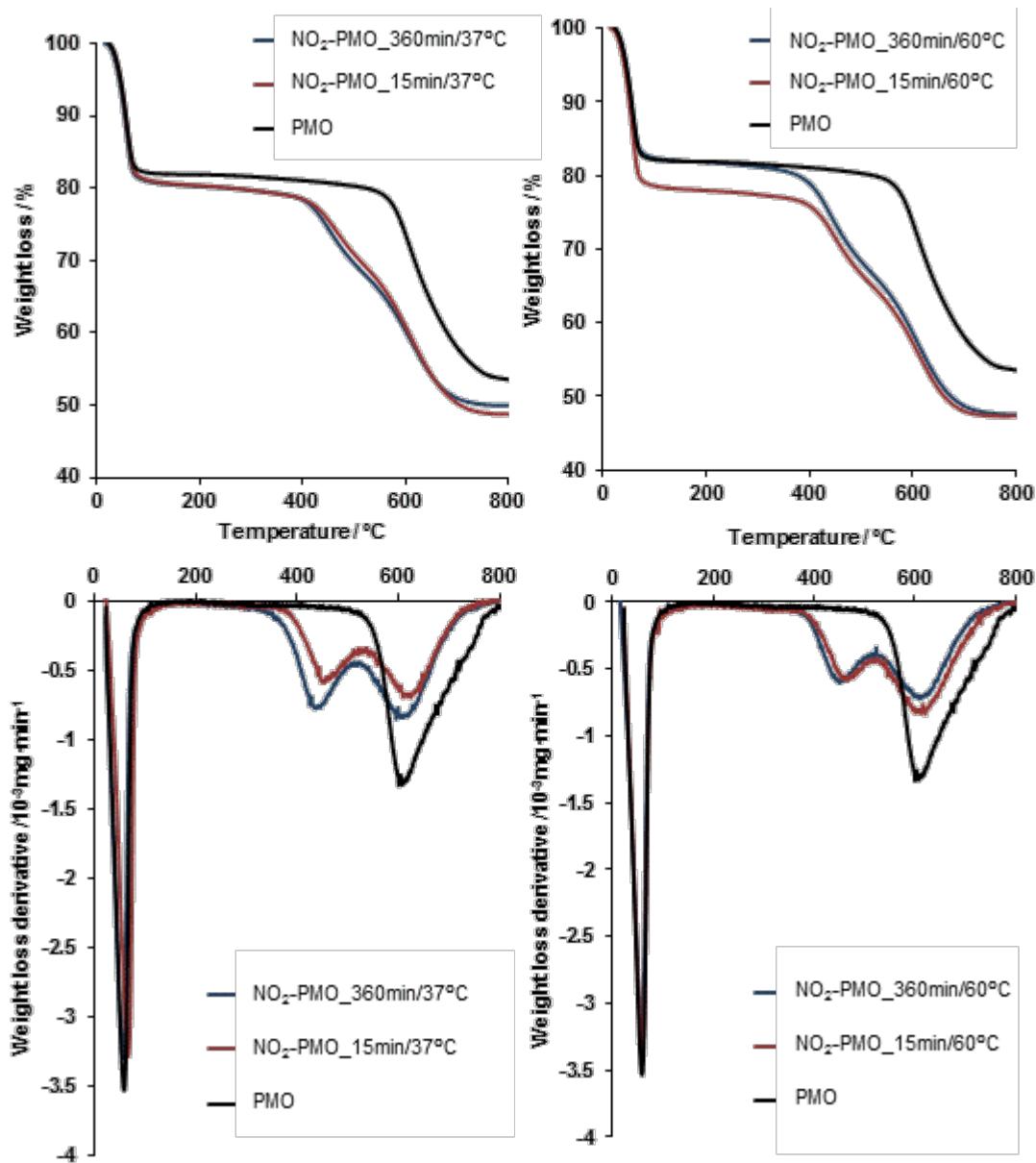
<sup>a</sup>Unit cell parameter calculated as  $(2d_{100}/\sqrt{3})$ . <sup>b</sup>Pore width obtained from the BJH method with the corrected Kelvin equation, i.e. KJS-BJH method at the maximum of pore size distribution calculated on the basis of adsorption data. <sup>c</sup>Pore wall thickness calculated as  $(2d_{100}/\sqrt{3} - d_p)$ , where the first term is the unit cell parameter.



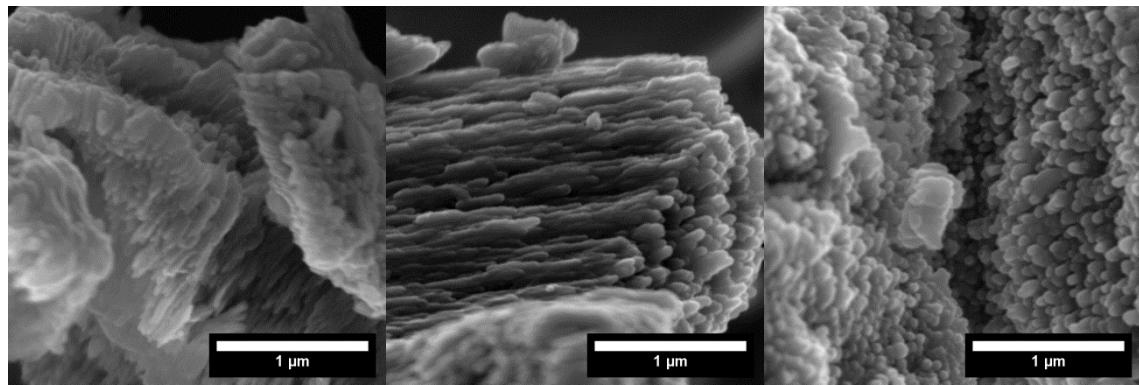
**Figure S4.** X-ray diffraction patterns of Ph-PMO and NO<sub>2</sub>-Ph-PMO\_15min synthesized at different reaction temperatures during 15 minutes.



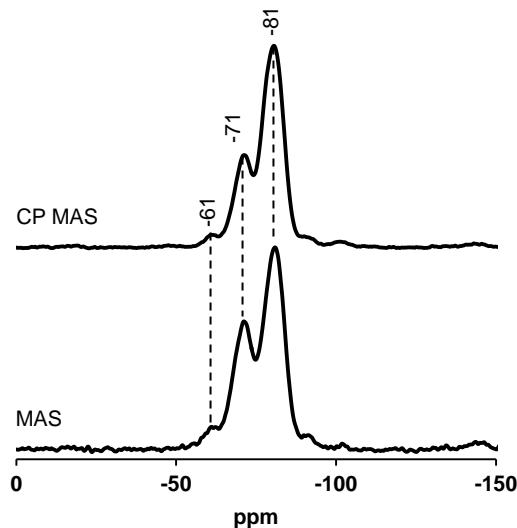
**Figure S5.** -196 °C nitrogen type IV isotherms of: a) PMO; b) NO<sub>2</sub>-PMO\_15min/37°C and c) NO<sub>2</sub>-PMO\_360min/60°C. Empty/filled symbols correspond to adsorption/desorption. The insets display the characteristic narrow PSD curves of PMO materials.



**Figure S6.** Weight loss curves (up) and weight loss derivatives (down) for PMO and  $\text{NO}_2$ -PMO.



**Figure S7.** Scanning electron microscopy images for NO<sub>2</sub>-PMO\_C (left), NO<sub>2</sub>-PMO\_15min/37°C (middle) and NO<sub>2</sub>-PMO\_360min/60°C (right).

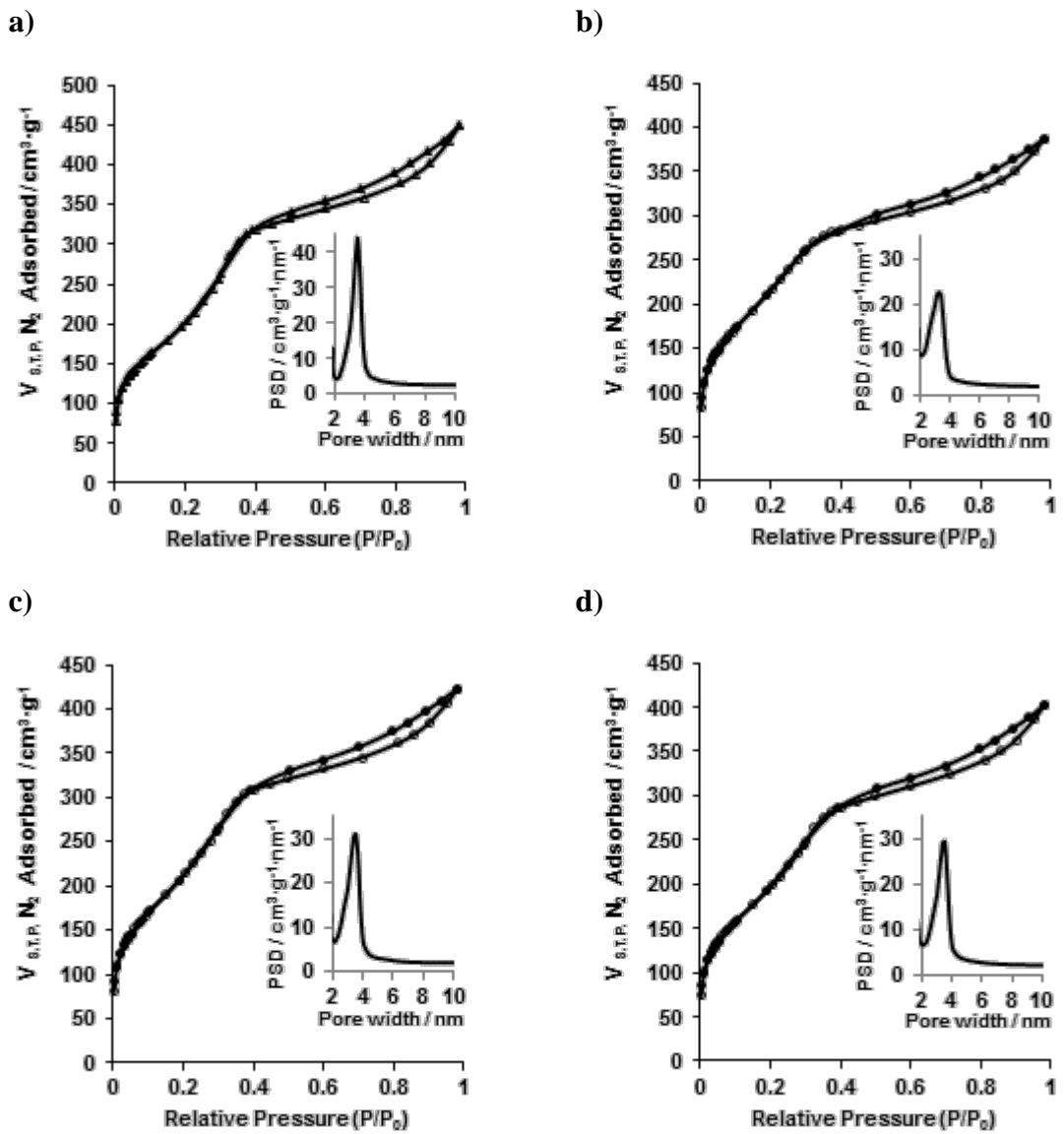


**Figure S8.** <sup>29</sup>Si MAS and CP MAS NMR spectra of the NH<sub>2</sub>-PMO\_15min/60°C.

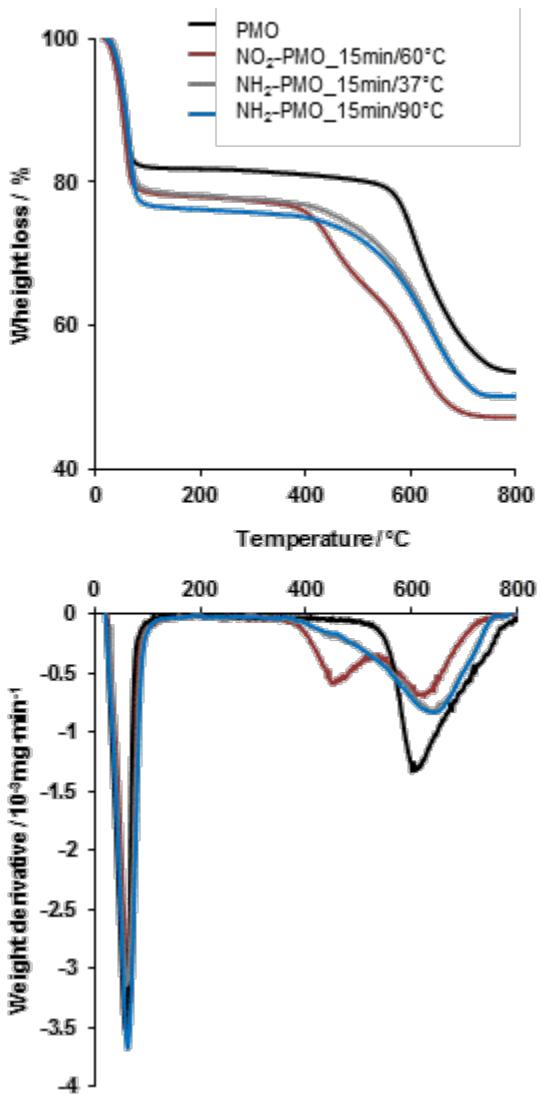
**Table S2.** Physical properties of PMO, NO<sub>2</sub>-PMO\_15min/60°C and NH<sub>2</sub>-PMO\_15min/y materials.

Sample	$d_{100}$ / nm	$a$ / nm <sup>a</sup>	$S_{\text{BET}}$ / m <sup>2</sup> g <sup>-1</sup>	$V_p$ / cm <sup>3</sup> g <sup>-1</sup>	$d_p$ / nm <sup>b</sup>	$b$ / nm <sup>c</sup>
PMO	4.55	5.25	920	0.66	3.58	1.67
NO <sub>2</sub> -PMO_15min/60°C	4.70	5.42	698	0.57	3.54	1.88
NH <sub>2</sub> -PMO_15min/37°C_C	4.46	5.15	752	0.44	3.27	1.88
NH <sub>2</sub> -PMO_15min/37°C	4.37	5.05	722	0.44	3.40	1.65
NH <sub>2</sub> -PMO_15min/60°C	4.41	5.10	699	0.44	3.40	1.70
NH <sub>2</sub> -PMO_15min/75°C	4.37	5.05	688	0.43	3.40	1.65
NH <sub>2</sub> -PMO_15min/90°C	4.46	5.15	686	0.44	3.54	1.61

<sup>a</sup>Unit cell parameter calculated as  $(2d_{100}/\sqrt{3})$ . <sup>b</sup>Pore width obtained from the BJH method with the corrected Kelvin equation, i.e. KJS-BJH method at the maximum of pore size distribution calculated on the basis of adsorption data. <sup>c</sup>Pore wall thickness calculated as  $(2d_{100}/\sqrt{3} - d_p)$ , where the first term is the unit cell parameter.



**Figure S9.**  $-196\text{ }^\circ\text{C}$   $N_2$  isotherms of a) NO<sub>2</sub>-PMO\_15min/60°C (triangles); b) NH<sub>2</sub>-PMO\_15min/37°C\_C (circles); c) NH<sub>2</sub>-PMO\_15min/37°C and d) NH<sub>2</sub>-PMO\_15min/90°C (circles). Empty/full symbols correspond to adsorption/desorption. The insets display PSD curves.



**Figure S10.** Weight loss curves (up) and weight loss derivatives (down) for PMO,  $\text{NO}_2\text{-PMO}_{15\text{min}/60^\circ\text{C}}$ ,  $\text{NH}_2\text{-PMO}_{15\text{min}/37^\circ\text{C}}$  and  $\text{NH}_2\text{-PMO}_{15\text{min}/90^\circ\text{C}}$ .