Structural and elemental influence from various MOFs on the performance of Fe@C catalysts for Fischer-Tropsch synthesis

Wezendonk, Tim A.; Warringa, Quirinus S E; Santos, Vera P.; Chojecki, Adam; Ruitenbeek, Matthijs; Meima, Garry; Makkee, Michiel; Kapteijn, Freek; Gascon, Jorge

DOI
10.1039/c6fd00198j

Publication date
2017

Document Version
Accepted author manuscript

Published in
Faraday Discussions

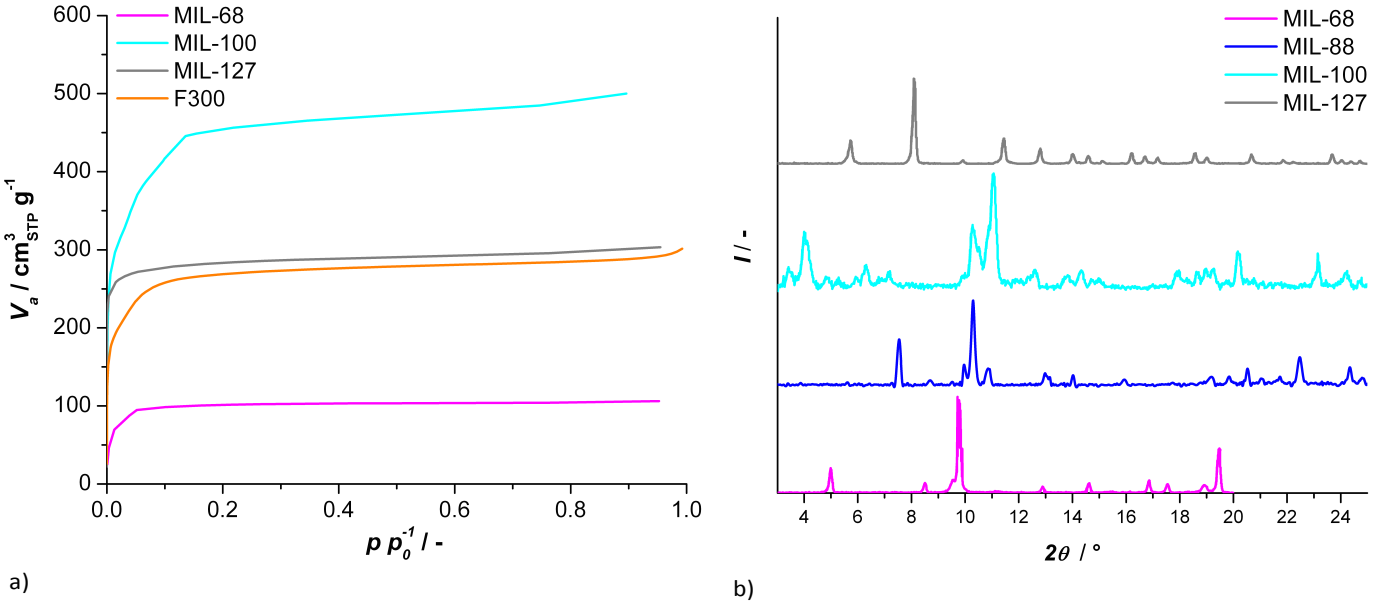
Faraday Discussions

SUPPORTING INFORMATION

Structural and elemental influence of various MOFs on the performance of Fe@C catalysts for Fischer-Tropsch synthesis

Tim A. Wezendonk[a], Quirinus S.E. Warringa[a], Vera P. Santos[b], Adam Chojecki[b], Matthijs Ruitenbeek[c], Garry Meima[c], Michiel Makkee[a], Freek Kapteijn[a] and Jorge Gascon[a]

MOF Identification



a) b)

Figure S1. a) N_2 physisorption at 77 K and b) PXRD patterns for the various MOFs. Isotherm shapes and diffraction patterns match with literature[1-12].

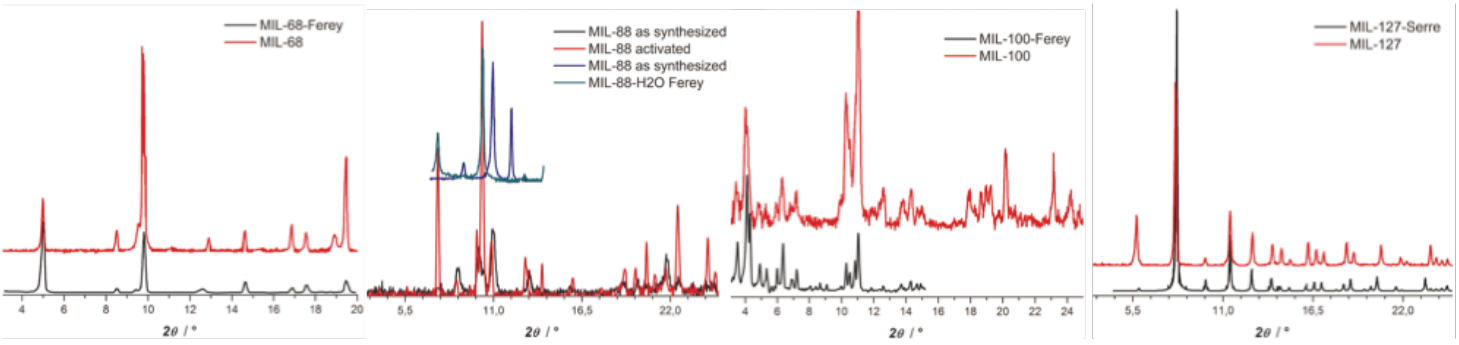


Figure S2. Comparison of the XRD angle and relative intensity for the various MOFs and reported XRD patterns from literature.

Table S1. N_2 physisorption calculations for the various MOFs, comprising BET area (S_{BET}) and total pore volume (V_p). Values agree with reported BET area and pore volume in literature.

MOF	S_{BET} $m^2\ g^{-1}$	V_p $cm^3\ g^{-1}$
MIL-68	400	0.16
MIL-100	1777	0.99
MIL-127	1131	0.47
F300	1021	0.45

Fe@C Characterization

Table S2. Data of N_2 physisorption per gram of catalyst, not taken into account the high Fe_2O_3 loading of the Fe@C materials.

Catalyst	S_{BET} $m^2\ g^{-1}$	S_{Ext} $m^2\ g^{-1}$	V_P $cm^3\ g^{-1}$	V_μ $cm^3\ g^{-1}$
Fe@C-MIL68	314	161	0.28	0.06
Fe@C-MIL88A	224	163	0.16	0.03
Fe@C-MIL100	260	136	0.18	0.06
Fe@C-MIL127	323	172	0.30	0.06
Fe@C-MIL101NH_2	283	120	0.25	0.07
Fe@C-F300	280	140	0.29	0.06

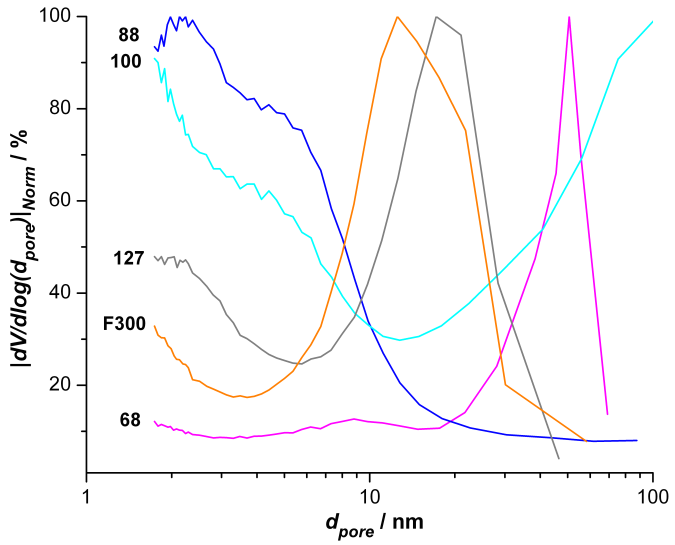


Figure S3. BJH transformation of isotherms displaying the normalized pore size distribution of Fe@C catalysts.

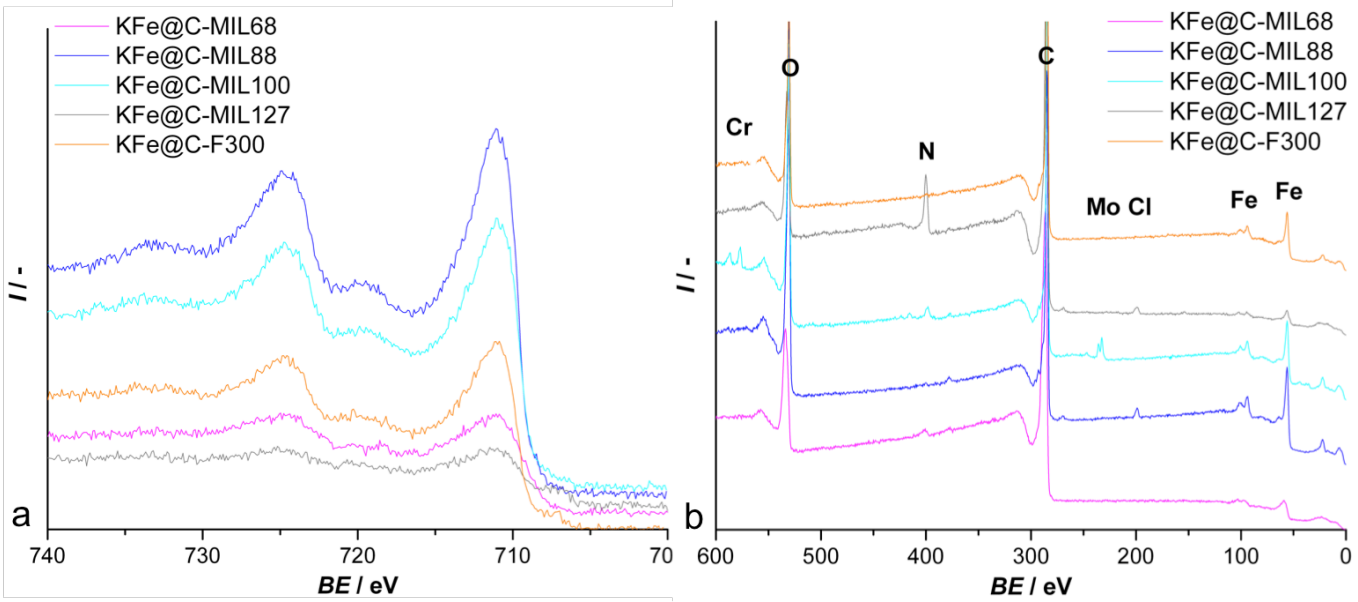


Figure S4. a) Core-level Fe*2p* spectra for passivated catalysts and b) their associating survey spectra displaying surface impurities.

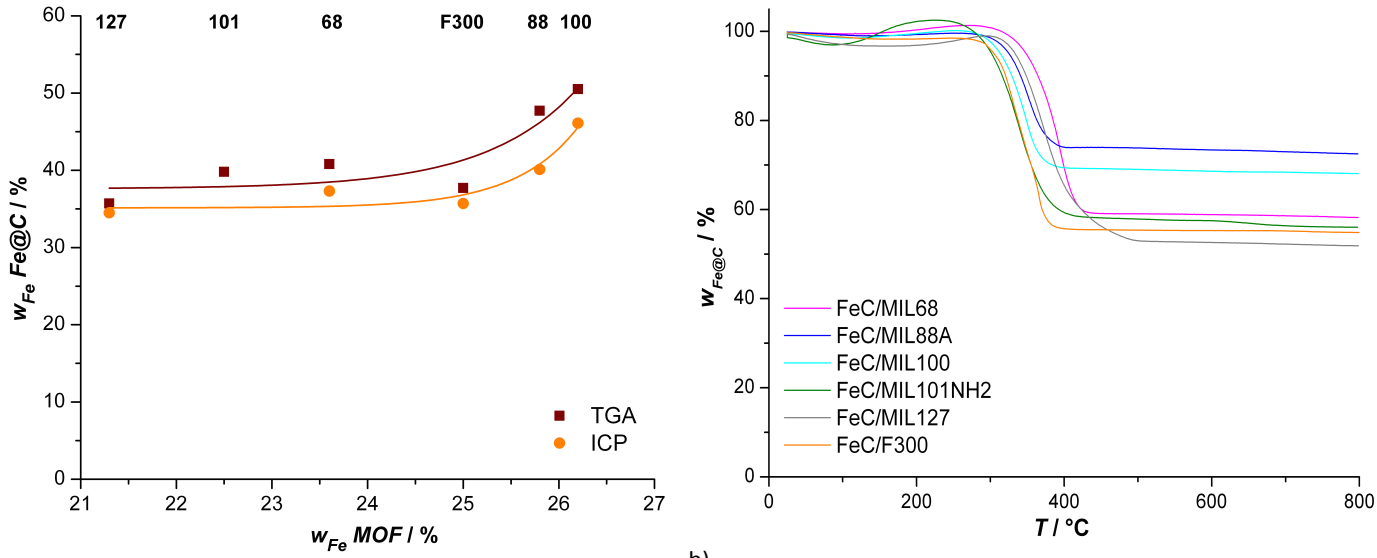


Figure S5. a) TGA and ICP analysis showing the non-linear relation between Fe loading in the MOF and in the resulting Fe@C catalyst and b) TGA profiles of Fe@C catalysts in air.

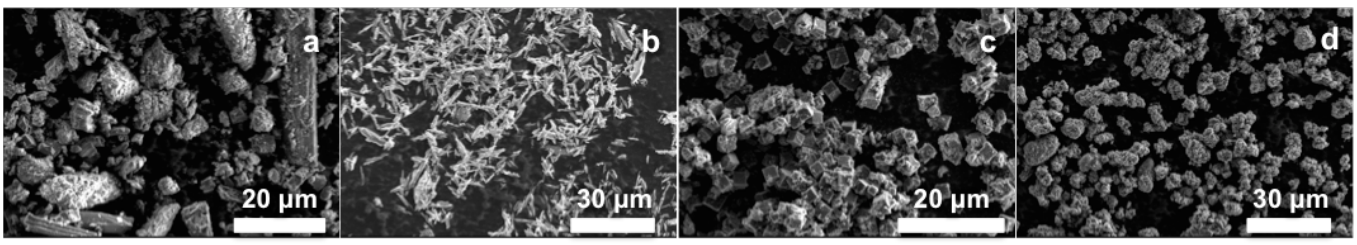


Figure S6. SEM images of pyrolyzed MOFs a) Fe@C-MIL68, b) Fe@C-MIL88, c) Fe@C-MIL127 and d) Fe@C-F300.

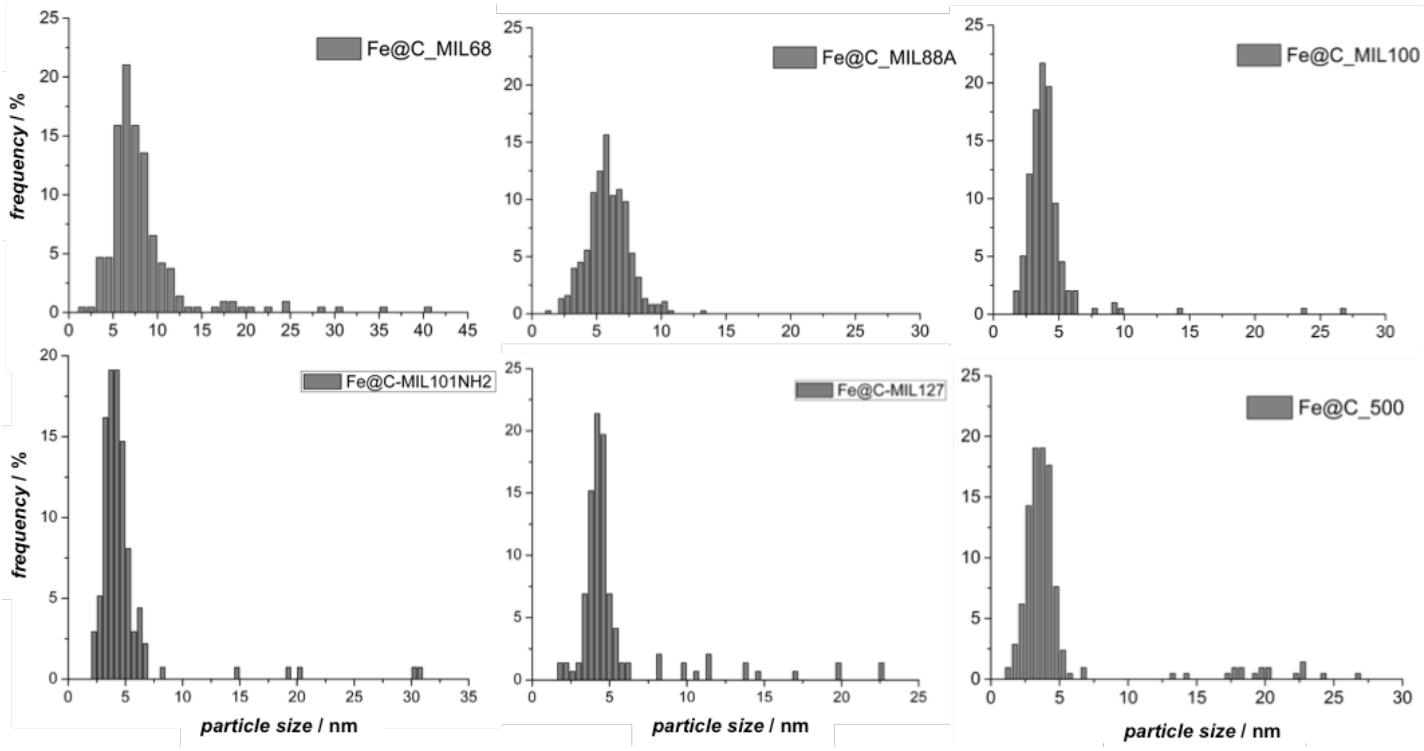


Figure S7. Particle size distribution determined by TEM in the Fe@C catalysts derived from different MOFs by pyrolysis at 500 °C.

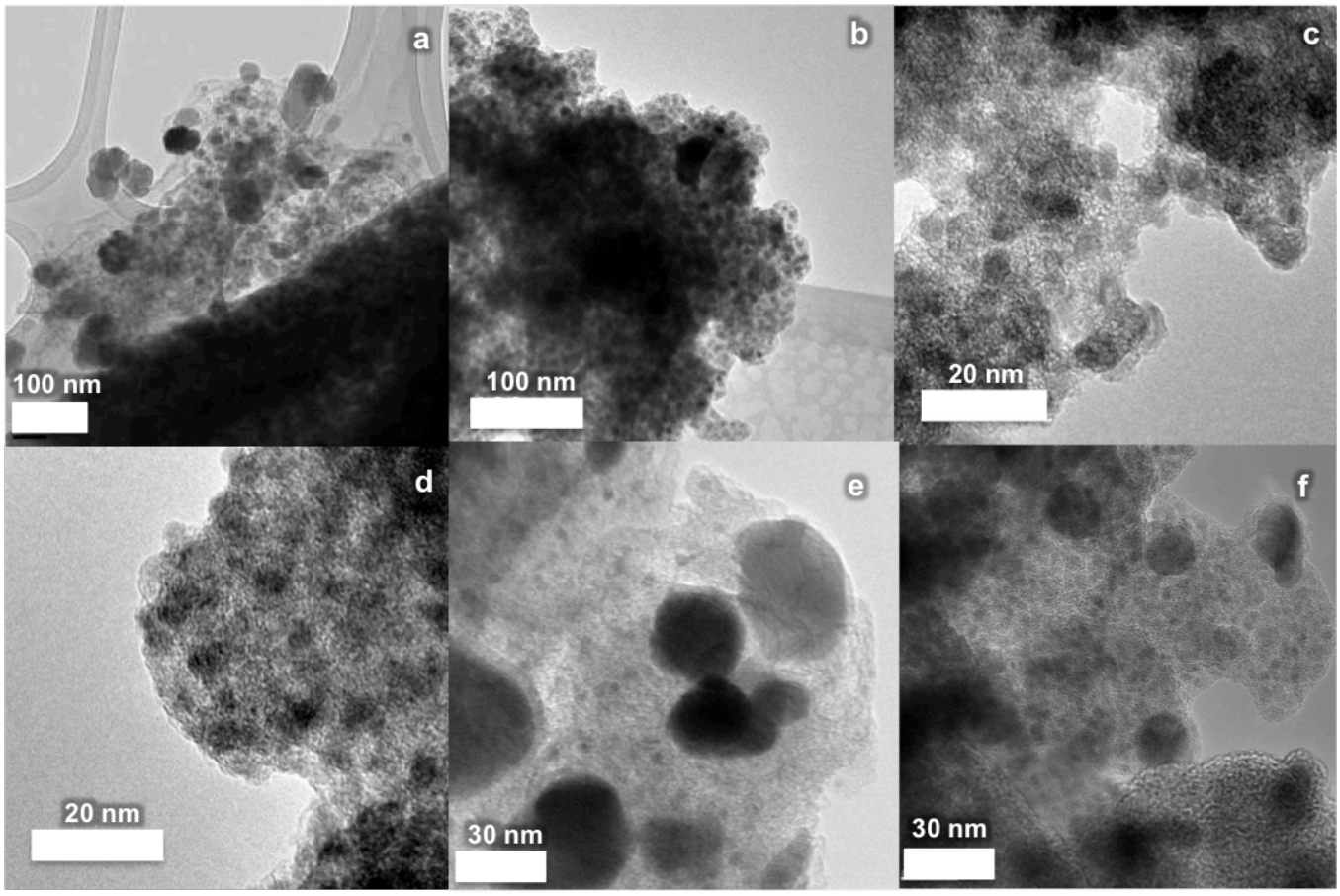
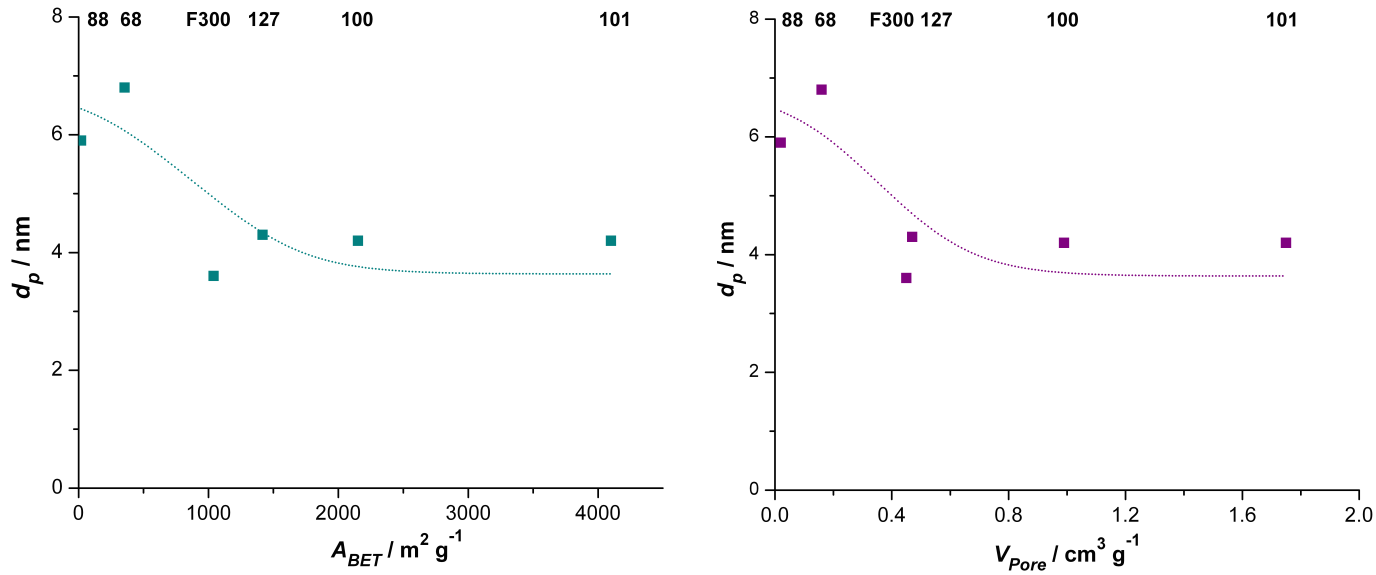


Figure S8. TEM of a) Fe@C-MIL68, b) Fe@C-MIL88, c) Fe@C-MIL100, d) Fe@C-MIL101NH2, e) Fe@C-MIL127 and f) Fe@C-F300



a) b)

Figure S9. Relation between the average Fe particle size and the BET area (a) and pore volume (b) for the Fe@C catalysts derived from the various Fe-MIL-X typologies (X= 88, 68, 127, 100 and 101) and Fe-BTC F300.

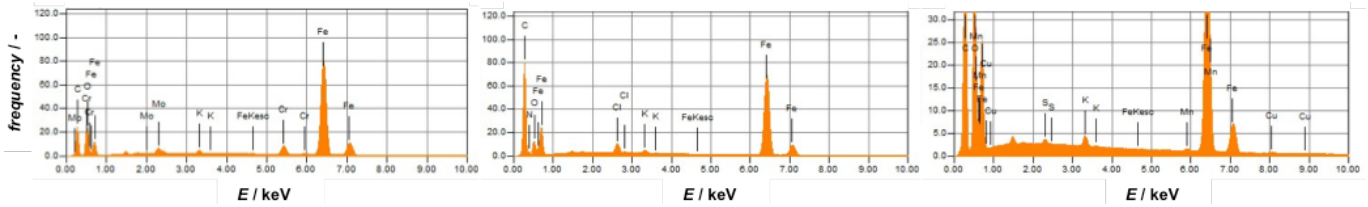


Figure S10. Elemental analysis from EDX spectra for *left*) KFe@C-MIL100, *middle*) KFe@C-MIL127 and *right*) KFe@C-F300 catalysts showing impurities in the bulk phase.

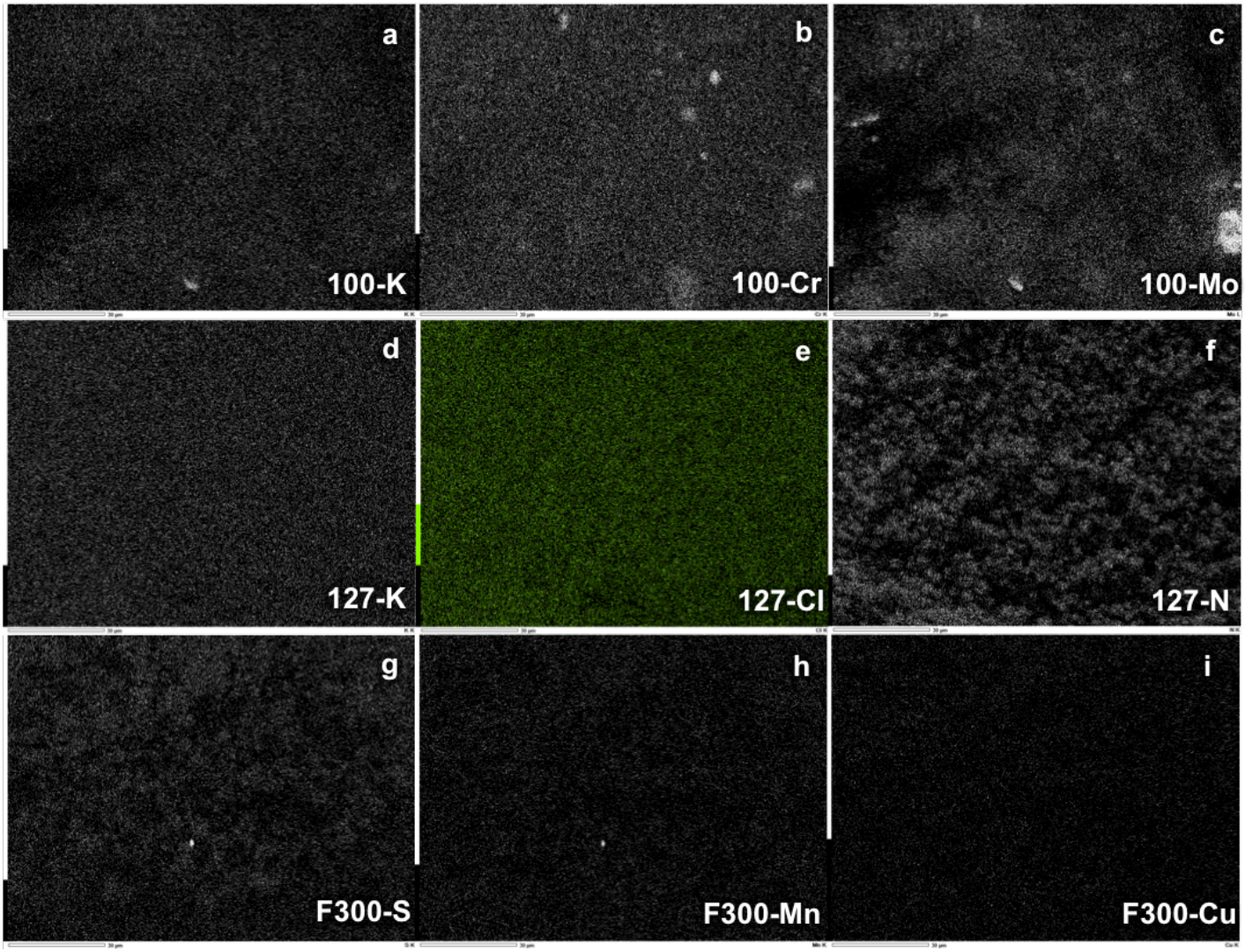


Figure S11. EDX mapping images of selected Fe@C catalysts, clearly showing agglomeration of metal impurities in the Fe@C-MIL100 sample and dispersed elements in the others.

Notes and references

1 Bauer, S.; Serre, C.; Devic, T.; Horcajada, P.; Marrot, J.; Férey, G.; Stock, N., *Inorg. Chem.* **2008,** *47* (17), 7568-7576.
2 Chevreau, H.; Permyakova, A.; Nouar, F.; Fabry, P.; Livage, C.; Ragon, F.; Garcia-Marquez, A.; Devic, T.; Steunou, N.; Serre, C.; Horcajada, P., *CrystEngComm* **2016,** *18* (22), 4094-4101.
3 Fateeva, A.; Horcajada, P.; Devic, T.; Serre, C.; Marrot, J.; Grenèche, J.-M.; Morcrette, M.; Tarascon, J.-M.; Maurin, G.; Férey, G., *Eur. J. Inorg. Chem.* **2010,** *2010* (24), 3789-3794.
4 Férey, G.; Mellot-Draznieks, C.; Serre, C.; Millange, F.; Dutour, J.; Surblé, S.; Margiolaki, I., *Science* **2005,** *309* (5743), 2040.
5 Férey, G.; Serre, C.; Mellot-Draznieks, C.; Millange, F.; Surblé, S.; Dutour, J.; Margiolaki, I., *Angew. Chem. Int. Ed.* **2004,** *43* (46), 6296-6301.
6 Horcajada, P.; Surble, S.; Serre, C.; Hong, D.-Y.; Seo, Y.-K.; Chang, J.-S.; Greneche, J.-M.; Margiolaki, I.; Ferey, G., *Chem. Commun.* **2007,** (27), 2820-2822.
7 Liu, Y.; Eubank, J. F.; Cairns, A. J.; Eckert, J.; Kravtsov, V. C.; Luebke, R.; Eddaoudi, M., *Angew. Chem. Int. Ed.* **2007,** *46* (18), 3278-3283.
8 Mellot-Draznieks, C.; Serre, C.; Surblé, S.; Audebrand, N.; Férey, G., *J. Am. Chem. Soc.* **2005,** *127* (46), 16273-16278.
9 Serre, C.; Millange, F.; Surblé, S.; Férey, G., *Angew. Chem. Int. Ed.* **2004,** *43* (46), 6285-6289.
10 Volkringer, C.; Meddouri, M.; Loiseau, T.; Guillou, N.; Marrot, J.; Férey, G.; Haouas, M.; Taulelle, F.; Audebrand, N.; Latroche, M., *Inorg. Chem.* **2008,** *47* (24), 11892-11901.
11 Volkringer, C.; Popov, D.; Loiseau, T.; Férey, G.; Burghammer, M.; Riekel, C.; Haouas, M.; Taulelle, F., *Chem. Mater.* **2009,** *21* (24), 5695-5697.
12 Cunha, D.; Ben Yahia, M.; Hall, S.; Miller, S. R.; Chevreau, H.; Elkaïm, E.; Maurin, G.; Horcajada, P.; Serre, C., *Chem. Mater.* **2013,** *25* (14), 2767-2776.

