

Supplementary information

Towards efficient polyoxometalate encapsulation in MIL-100(Cr): influence of synthesis conditions

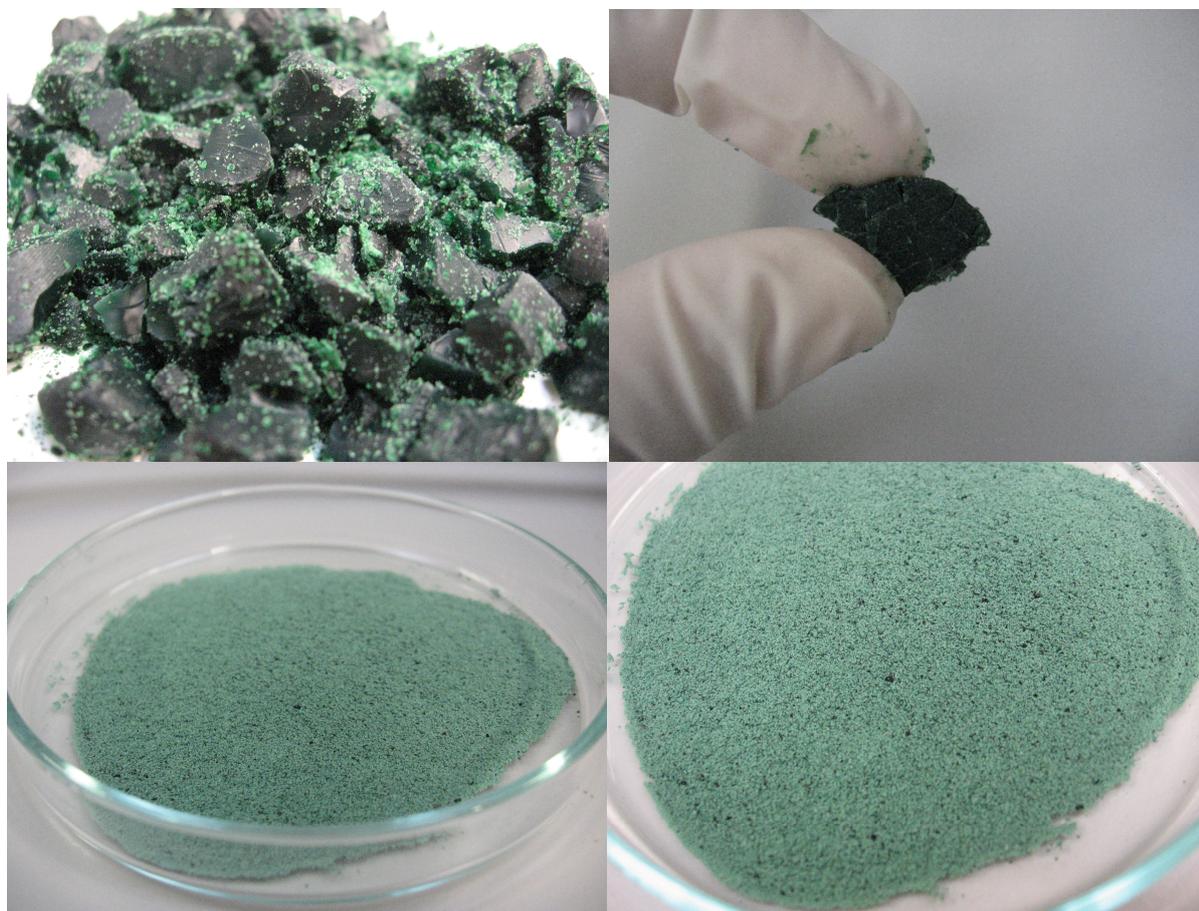
Jana Juan-Alcañiz*, Maarten G. Goesten, Enrique V. Ramos-Fernandez,
Jorge Gascon* and Freek Kapteijn

Catalysis Engineering - ChemE, Delft University of Technology

Julianalaan 136, 2628 BL Delft, The Netherlands.

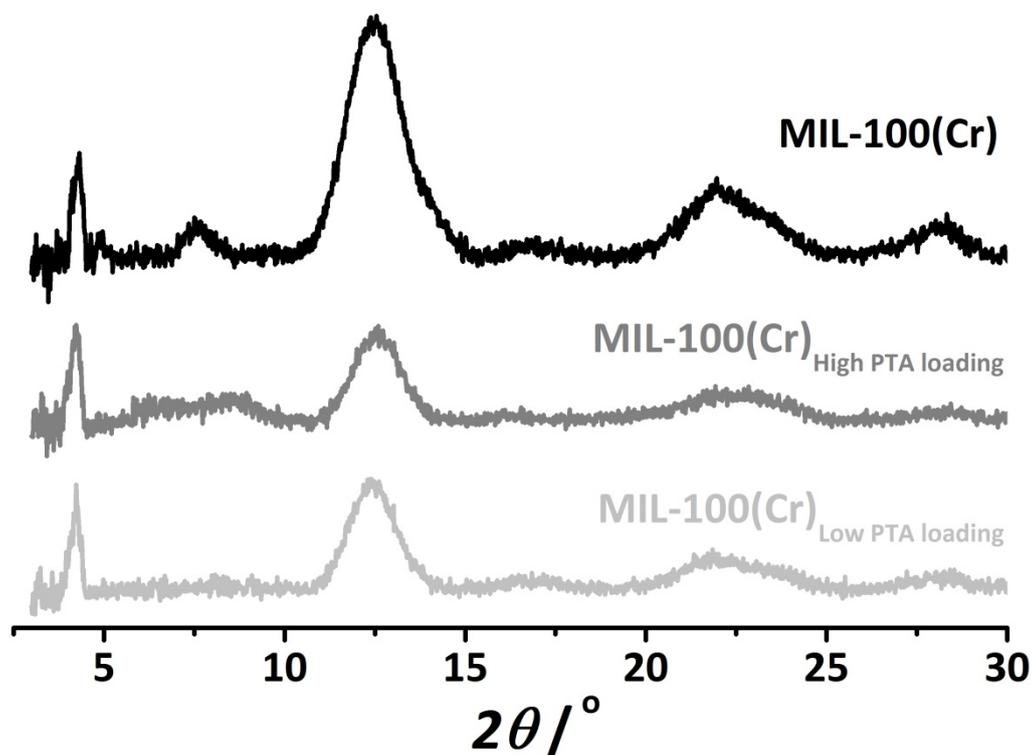
*j.juanalcaniz@tudelft.nl, *j.gascon@tudelft.nl

Figure 1.- Images of MIL-100(Cr)-DMF synthesized under conventional heating. (*Top*) Large aggregates and gel-like structure. (*Bottom*) Dried material at 433K overnight



The top pictures are taken from the sample, MIL-100(Cr)-DMF, as-synthesized. It can be observed large agglomerates of particules in a gel-like shape. In addition, smaller and green lighter particles are observed. However, these systems are, as well, aggregates of smaller particles with a gel-like structure. Lighter colour is obtain due to the size of the aggregate and probably the decreased in DMF within the pores. This phenomenon can be better observed in the right-top picture, where a gel aggregate is holded. After drying the sample in the oven overnight at 433K, the sample is transformed into a powder like can be observed in the bottom pictures. After crashing the dried material, all big and small agglomerates are transformed into fine powder, which is the one used to characterize this system.

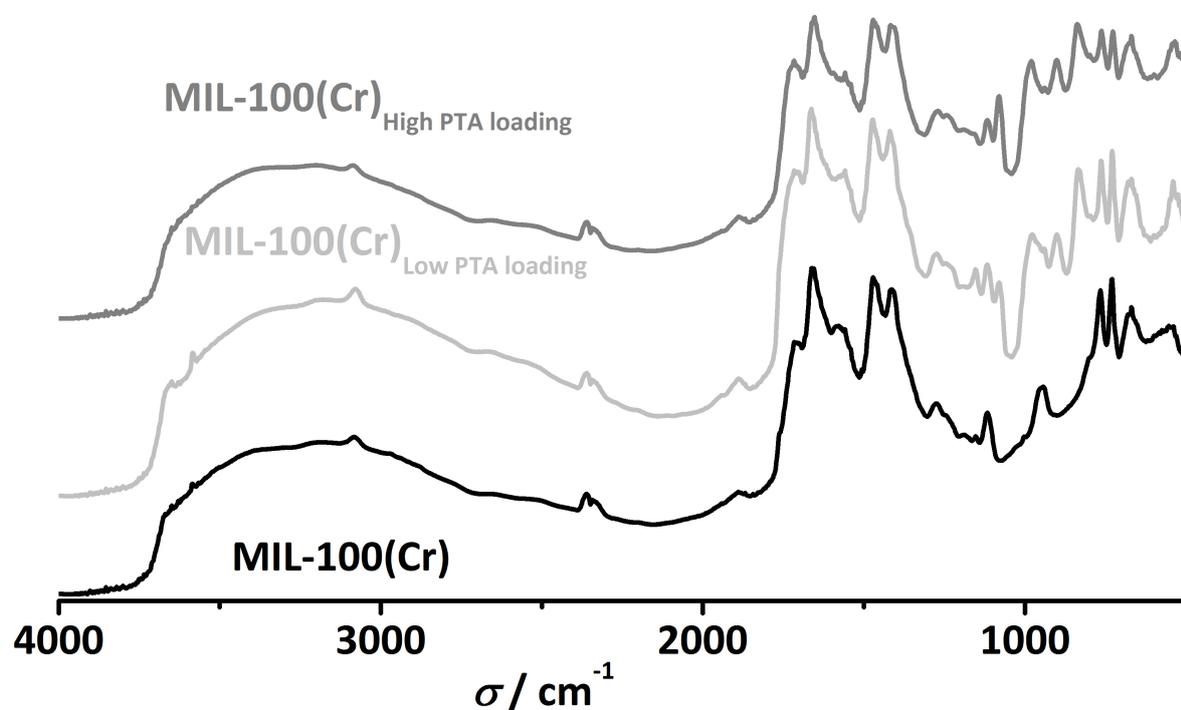
Figure 2.- XRD patterns of MIL-100(Cr) biphasic/microwave systems (CoK α radiation). (Black) Bare sample. (Dark grey) High PTA loading MIL-100(Cr)-biphasic. (Light grey) Low PTA loading MIL-100(Cr)-biphasic



CoK α radiation has been used to measure all three patterns. No significant differences are observed in the case of PTA encapsulated in MIL-100(Cr) under biphasic/microwave synthesis conditions. Nevertheless, lower diffraction intensities are observed due to attenuation of the signal by PTA within the cavities.¹ Broad reflections are due to small particle size of the samples produced under microwave irradiations, and the short time of the synthesis leading to an early growing stage.

Figure 3.- DRIFT spectra of MIL-100(Cr)-biphasic/microwave. (Black) Bare sample; (Dark grey) High PTA loading MIL-100(Cr)-biphasic; (Light grey) Low PTA loading MIL-100(Cr)-

biphasic



No significant differences are observed, apart from the ones mentioned in the zoomed in region of 1200-500 cm^{-1} described in the main text. Possible differences can be seen in the OH region, due to recording of the spectrum without pretreatment of the samples. However, differences in solvent occlusion or other interactions can be neglected.

References

- (1) Juan-Alcañiz, J.; Goesten, M.; Martinez-Joaristi, A.; Stavitski, E.; Petukhov, A. V.; Gascon, J.; Kapteijn, F. *Chemical Communications* **2011**, *47*, 8578.