

# Improved mechanochemical synthesis of MIL-100(Fe) and MIL-100(Fe)@Carbon

## Metal-Organic Framework composite

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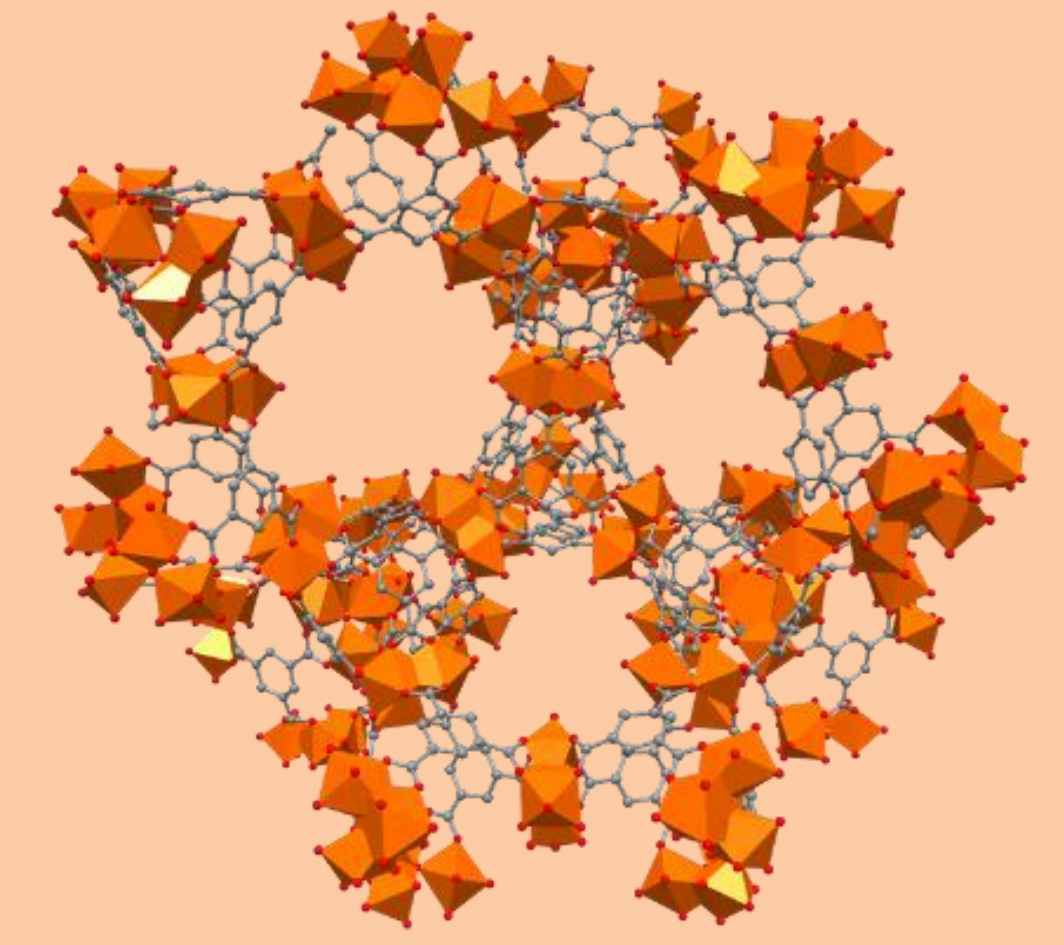
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### Introduction

MOFs are a class of crystalline solids with permanent porosity. They are coordination polymers made up of metal ions or metal clusters linked by organic ligands. MOFs are promising materials for application in the chemical world due to their high porosity and chemical tunability. However, their application is often hindered by the cost of the precursor and by the demanding synthetic conditions. One of the most promising MOFs is MIL-100 (Fe) because it is built by inexpensive and non-toxic constituent. However, its synthesis often requires toxic organic solvents and hazardous synthetic additives, high temperature and pressure.<sup>1</sup> Here we propose a new synthetic method based on grinding with the use of a ball miller for production of MIL-100 (Fe)@carbon composite (*mechanochemical composite*). This process offers advantages over the previous methods<sup>2,3,4</sup> since it is more rapid, preventing waste of time, and it uses lower temperature and less water, thus improving energy efficiency and reducing waste and resource consumption. We compared our material with two traditionally made MIL-100 composites. One made using precipitation of MOF on carbon (*in situ composite*) and other made mixing premade MIL-100 with carbon powder (*ex situ composite*).



MIL-100 (Fe) structure

### Mechanochemical synthesis of MIL-100 (Fe) composite

#### Mechanochemical mixing

- Iron salt precursor
- Trimesic acid
- Carbon

#### Water fining

- Homogeneous carbon mixture
- Distilled water

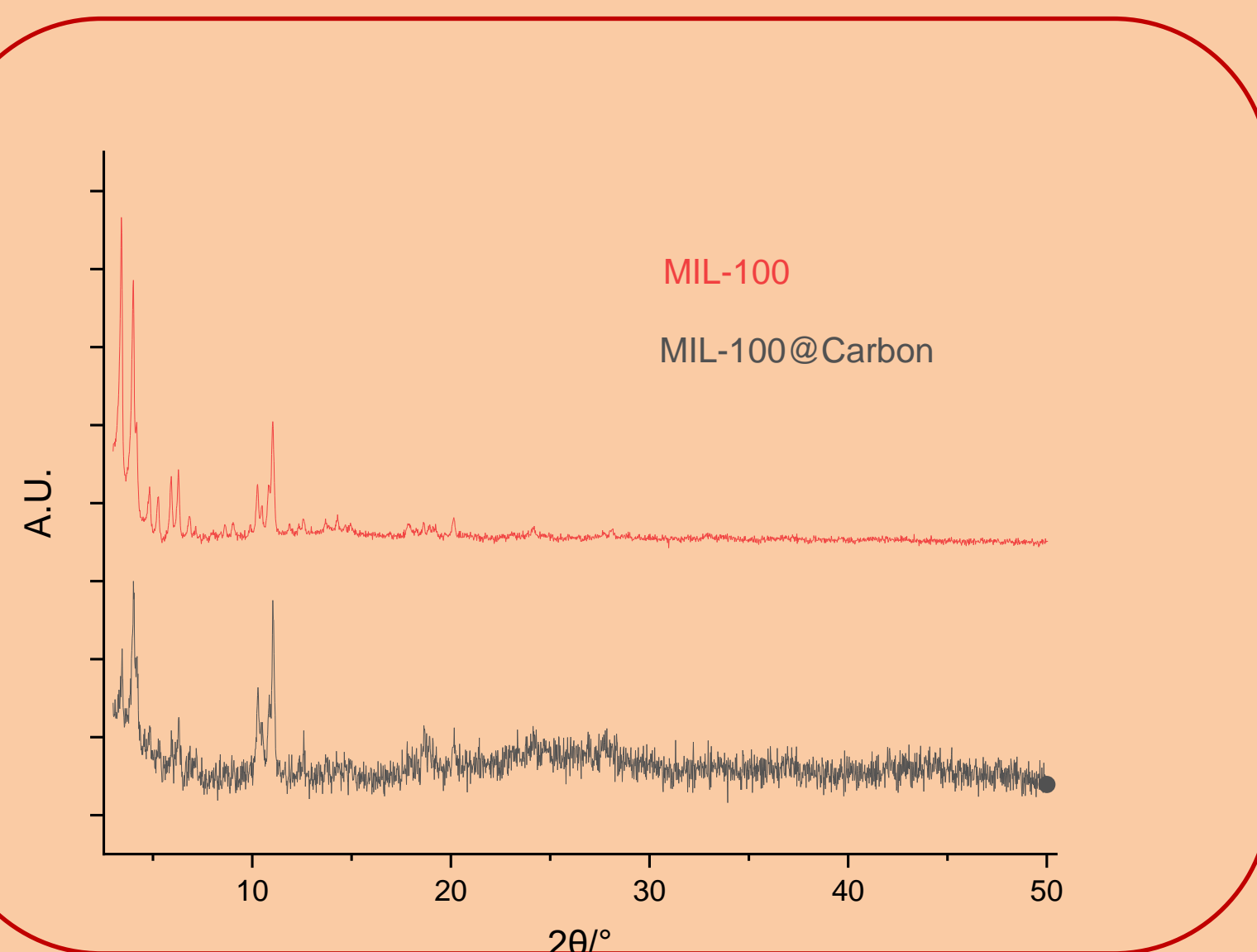
#### Drying

- Organic solvent
- Heating



Ball Miller

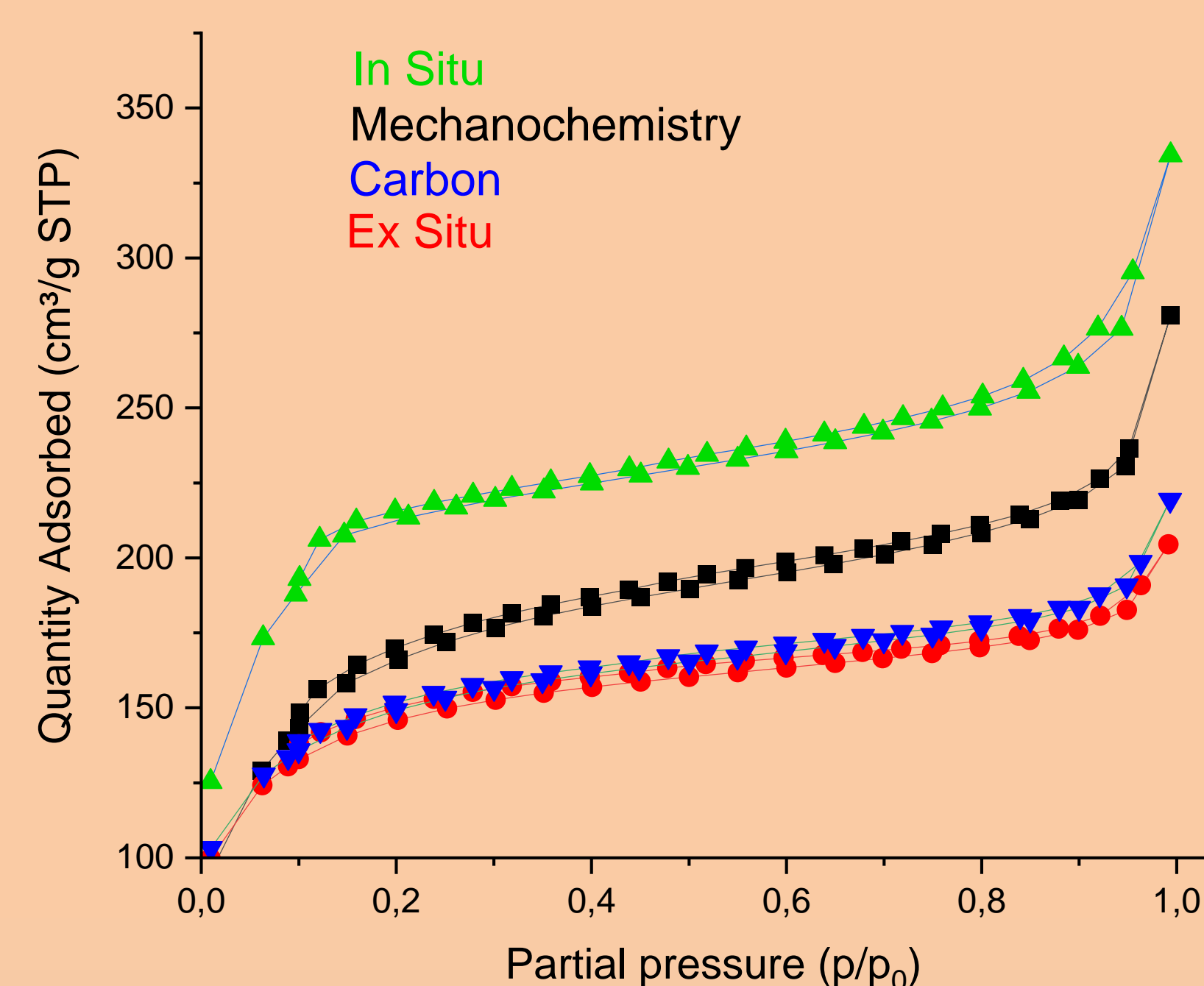
### PXRD analysis



- MIL-100 (Fe) structure can be seen using PXRD in the mechanochemical composite
- It is of lower crystallinity respect to pure MIL-100(Fe)

	MIL-100 (Fe)	MIL-100@Carbon Mechanochemistry
Space Group	Fd-3m	Fd-3m
a/Å	73.34	73.045

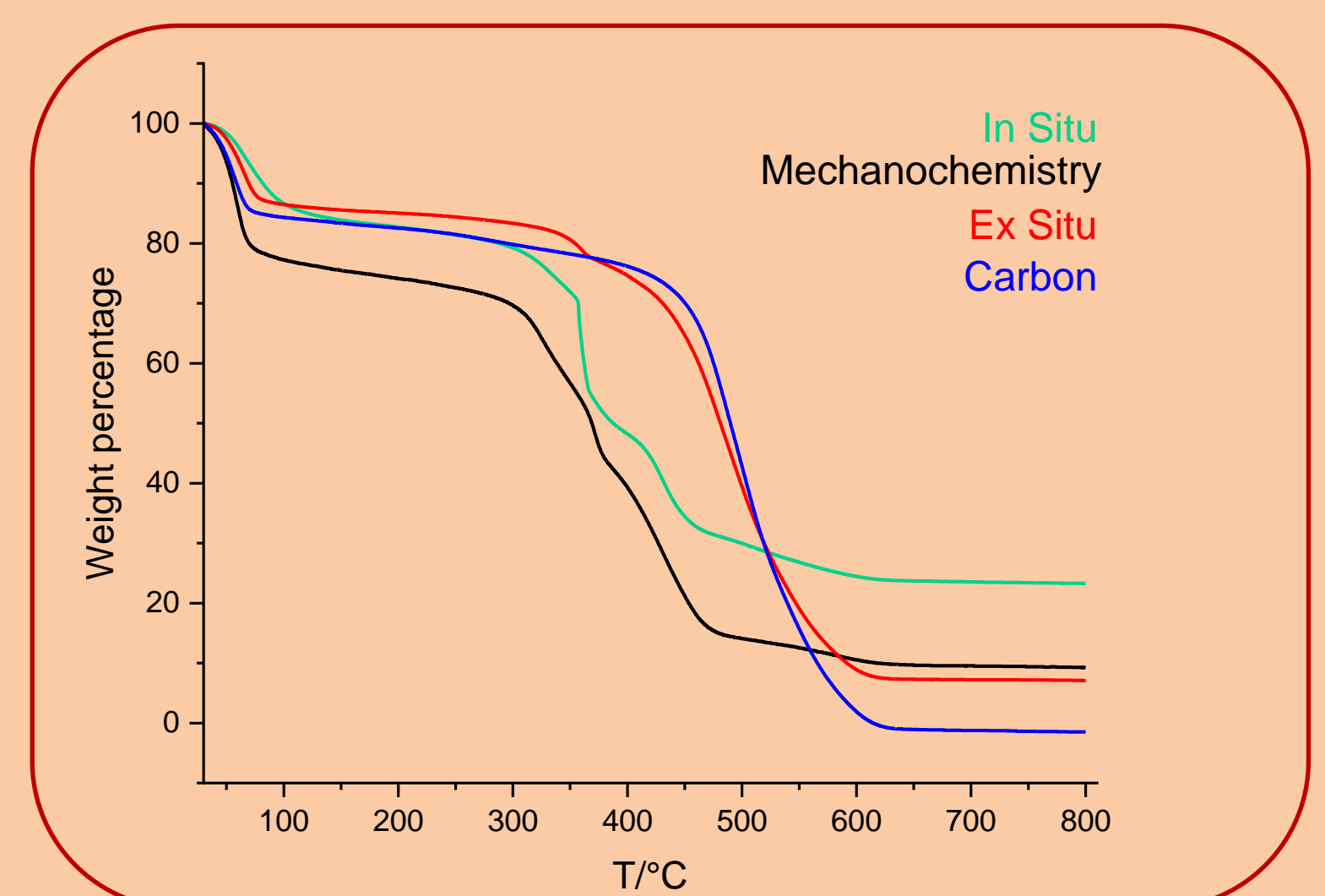
### Surface Area Characterization



- In Situ composite has the highest adsorption capacity
- The second best performing material is the mechanochemical composite
- The Ex Situ composite and Carbon have almost the same surface area, this is probably due to a unfavorable interaction between the powders during mixing

	BET (m <sup>2</sup> /g)	% micropores
In Situ	790	87
Mechanochemical	600	43
Carbon	538	38
Ex Situ	530	36

### TGA analysis



- Carbon has a smooth TGA curve with a large drop at 550 °C
- All MIL-100 (Fe)@carbon have additional drops at 300-400 °C due to MIL-100 decomposition
- The final weight of the composites is higher than carbon due to iron oxide residue
- In situ composite has higher final mass a sign of higher Iron and thus MIL-100 content
- Mechanochemical composite adsorbs more water as the first step is deeper

### Future Perspective

- Find the exact mechanism of formation of MIL-100 (Fe) under mechanochemical conditions using advanced synchrotron techniques
- Optimize the synthetic conditions of MIL-100 (Fe)@Carbon
- Study the remediation of pollutants from water using this composite

### Bibliography

#### References:

- [1] P. Horcajada et al., 2007, 27, 2820-2822
- [2] T. Steenhaut et al., 2020, 44 (10), 3847-3855.
- [3] B.E. Souza et al., 2020, 8 (22), 247-8255
- [4] K. Xiang-Jing et al., 2021, 7 (8), 1115-1139