Resin-assisted Solvothermal Synthesis of Metal-Organic Frameworks

Yi Du*, Amber L. Thompson* and Dermot O’Hare*

The powder XRD patterns of a bulk sample of 1, 2 and 3 were identical to the powder patterns generated by the solution to the single crystal structure solutions. Comparison reactions were carried out by replacing the Fe$^{2+}$/Co$^{2+}$/Ni$^{2+}$-resin precursors by same molar amount of the appropriate metal nitrate salts. None of XRDs matched with the single crystal data for compound 1, 2 and 3.

Figure S1. (a) Powder XRD for single crystal compound 1; (b) simulated powder pattern from single crystal analysis.
Figure S2. Powder XRD for (a) single crystal compound 1 using Fe$^{2+}$-resin as metal precursors; (b) powder product using Fe(NO$_3$)$_2$ as metal precursors.

Figure S3. (a) Powder XRD for single crystal compound 2; (b) simulated powder pattern from single crystal analysis.
Figure S4. Powder XRD for (a) single crystal compound 2 using Co\textsuperscript{2+}-resin as metal precursors; (b) powder product using Co(NO\textsubscript{3})\textsubscript{2} as metal precursors.

Figure S5. (a) Powder XRD for single crystal compound 3; (b) simulated powder pattern from single crystal analysis.
Compound 1 is thermally stable until *ca.* 270 °C following two major weight losses corresponding to the structural coordinated 2,6-NDC ligands decomposition. Compound 2 is thermally stable until *ca.* 200 °C, then followed by two steady weight losses. The first weight loss corresponds to the two DMF molecules and the two NHMe₂ molecules. The second weight loss, between 440 °C and 590 °C, corresponds to the decomposition of the 2,6-NDC ligands. The TGA of 3 shows three weight loss events; the first weight loss (*ca.* 4%) corresponds to half of the unbound dmf molecules. The second and the third weight losses correspond to the decomposition of structural dmf, NMe₄⁺ molecules and 1,3,5-BTC ligands.