Synthesis of switchable spin crossover compound [Fe(1-BPP)$_2$][BF$_4$] with flow progressive mixer reactor

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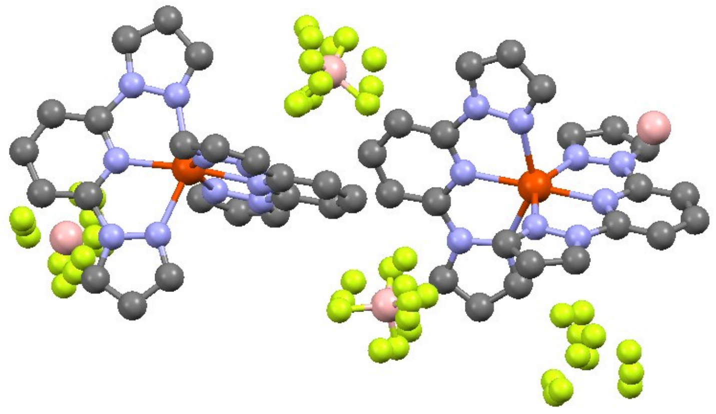
Spin crossover complexes (SCO) are characterised by a change in electronic configuration of a transition-metal centre as a response to external stimuli such as heat, light, pressure, or changes in magnetic field.

Such properties are of great interest for data storage materials due to their highly reversible switching properties. The molecular scale of those compounds are increasingly being considered as the answer to the miniaturization of the components used in the construction of working devices, but particle size control of these compounds can be difficult to exact in batch production.

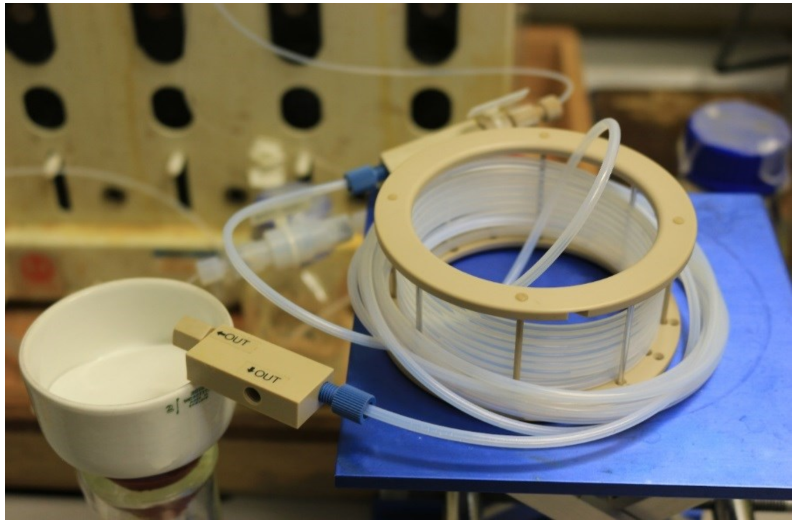
In this study is proposed a way of controlling particle size for [Fe(1-BPP)$_2$][BF$_4$] SCO compound with a progressive mixer reactor design by Vapourtec.



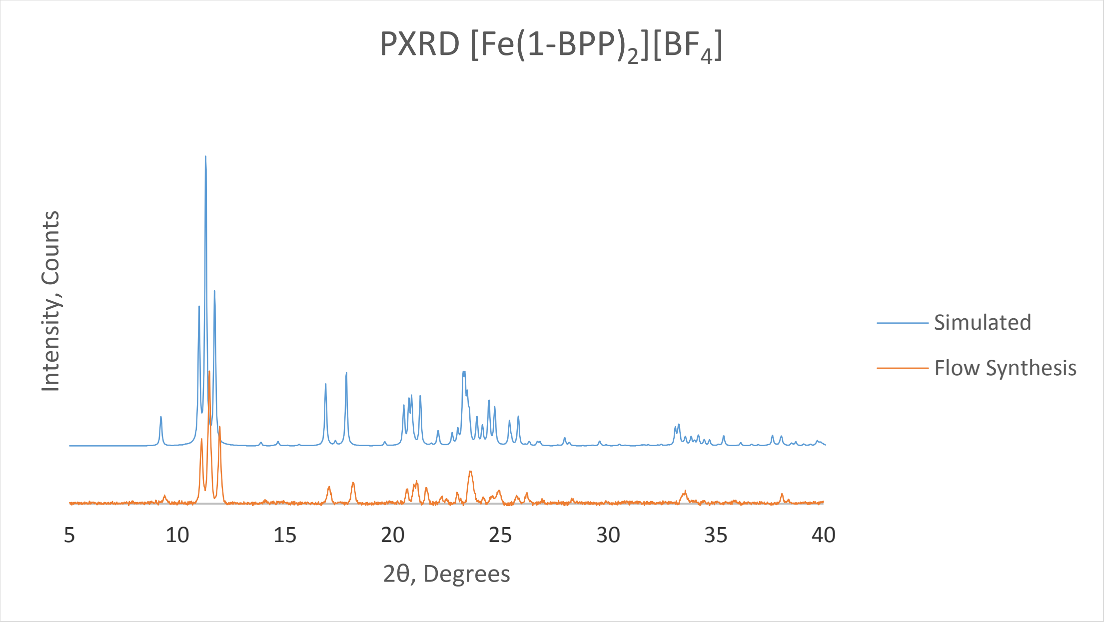
Crystal structure of SCO [Fe(1-BPP)$_2$][BF$_4$] [1]

[1] *Halcrow et al. ,Chem. Commun., 2001, 577–578*

- The anti-solvent precipitation reaction via diethyl ether of $Fe(BF_4)_2 \cdot 6H_2O$ with 2,6-Bi(pyrazol-1yl)-pyridine(1-BPP) gives $[Fe(1\text{-}BPP)_2][BF_4]$ was achieved with a tube-in-tube reactor consisting of fluoropolymer inner and outer tubes connected along their length by a series of mixing locations.
- The anti-solvent flows through the outer tubing and force precipitation of the solution (in the inner tubing) upon contact *via* the mixing locations.
- The product obtain is a fine yellow powder which switch from yellow to brown at 250K



Progressive Mixer Flow reactor at Bath University


PXRD $[Fe(1\text{-}BPP)_2][BF_4]$

Comparison between simulated and flow obtain PXRD pattern

vapourtec

METASTABLE MATERIALS
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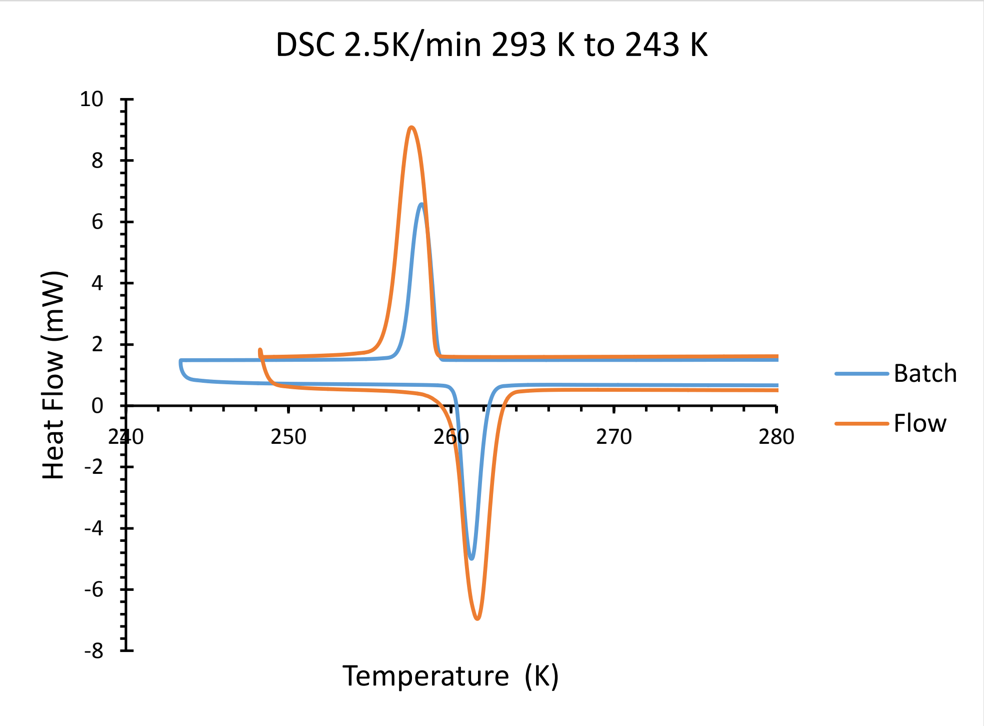
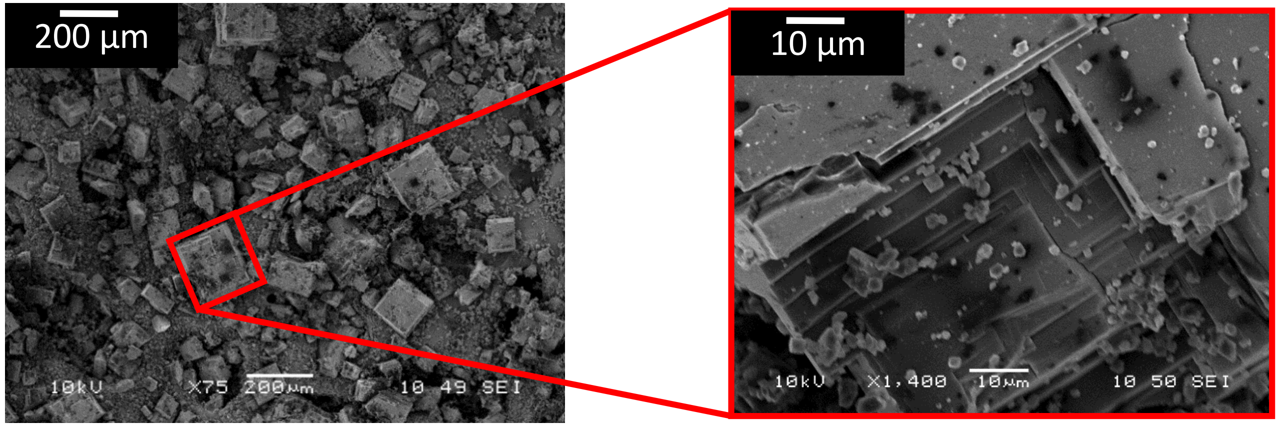
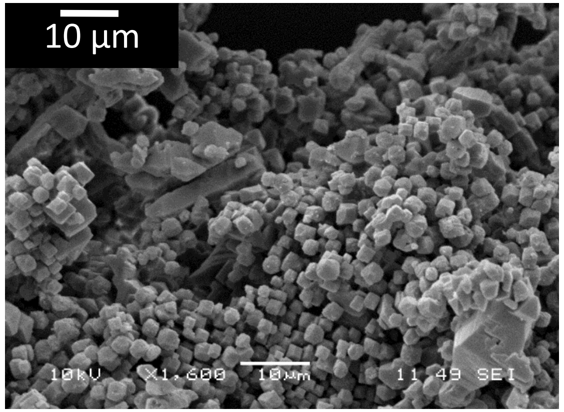
Initial results show a large improvement in particle size distribution compare to batch as shown in the pictures below



Differential scanning calorimetry (DSC) of $[Fe(1\text{-}BPP)_2][BF_4]$ comparing switching between Batch and Progressive Mixer reactor production. This graph show a switching occurring earlier with the compound produced in Flow than in batch.



Scanning electron microscope (SEM) Pictures of $[Fe(1\text{-}BPP)_2][BF_4]$ produced in batch



Scanning electron microscope (SEM) Pictures of $[Fe(1\text{-}BPP)_2][BF_4]$ produced in progressive mixer reactor