Electrospray Mass Spectrometry Investigation into the Formation of CPO-27

Electrospray ionisation mass spectrometry (ESI-MS) has been utilised to investigate the self-assembly processes occurring during the formation of the microporous metal-organic framework CPO-27-M (M = Co, Ni). The mono- and dinuclear building units \( \{M(Hxdhtp)\} \) and \( \{M_2(Hxdhtp)\} \), where Hxdhtp is the organic linker HxC8O6 and fragments thereof, were identified as key species present in the reaction mixture during the product formation. Time resolved powder X-ray diffraction analysis were used to follow the synthesis, and confirm that no other crystalline products occur in the reaction mixture prior to the crystallisation of CPO-27-Ni. When the reaction was performed at room temperature instead of the higher temperature of the solvothermal procedure, the compounds \( \{(M(H2dhtp)(H2O)4·2H2O)\} \) (M = Co, Ni) crystallized instead of CPO-27. It was confirmed that mono- and dinuclear species are key building blocks not only in the formation of CPO-27-M, but also in the formation of the 1D chain structure \( \{(M(H2dhtp)(H2O)4\} \) obtained from these room temperature reactions.

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