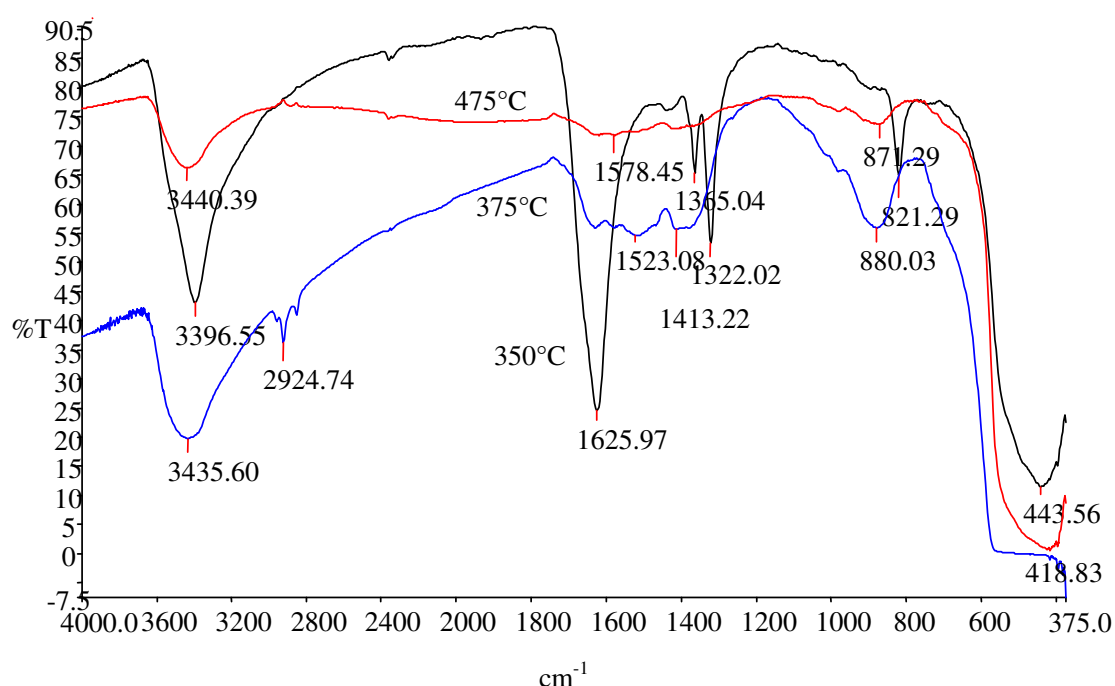


## Electronic Supplementary Information

### *FTIR data for powders prepared through rapid and slow combination, calcined at 350°C, 375°C and 475°C*

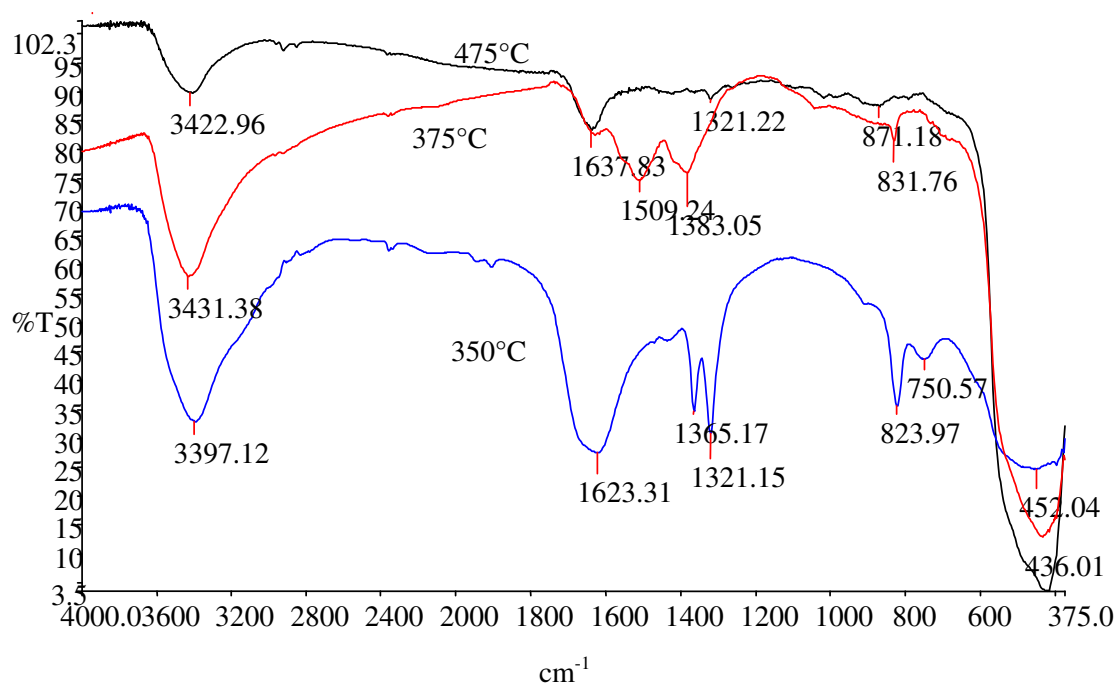
FTIR spectroscopy was performed on powders calcined at 350°C, 375°C and 475°C and the spectra are presented in ESI Fig. 1 and ESI Fig 2. Both spectra show that the symmetric carboxylic peaks at  $1365\text{cm}^{-1}$  and  $1320\text{cm}^{-1}$ , present in precursor powders when calcined to 350°C, were absent in those calcined at higher temperatures.



**ESI Fig. 1 FTIR spectra of powders prepared through rapid combination of reagents: 350°C, 375°C and 475°C**

A prominent peak related to the asymmetric  $\text{COO}^-$  bond at  $1620\text{cm}^{-1}$  was present in all powders synthesized through slow addition of reagents, this may have been due to stabilisation of the surfaces of the product particles by the counterion, or may have been indicative of unreacted reagent. Since a peak, thought to be the absorbance of atmospheric water, was present at approximately  $3400\text{cm}^{-1}$  in all powders, it is

unlikely that the changing intensity of the peak at  $1625\text{cm}^{-1}$  could have been related to water. The shape of this peak for the powders calcined to  $350^\circ\text{C}$  is also of interest.



**ESI Fig. 2 FTIR spectra of powders formed through slow combination of reagents:  $350^\circ\text{C}$ ,  $375^\circ\text{C}$  and  $475^\circ\text{C}$**

It is believed that rapid combination of reagents leads to a more homogeneous precursor than their combination over an extended time period, which it is proposed, contained carboxyl groups coordinatively bound to either hydrogen or zinc ions. The absorbance band at  $1622\text{cm}^{-1}$  was asymmetrically broadened, but exhibited a sharp apex, indicating that one resonance dominated here. For the precursor powders synthesized through slow addition of reagents, it was found that the equivalent peak was much flatter, in agreement with the hypothesis that a transient state had been preserved and oxalate molecules bound to two zinc ions *and* oxalic acid molecules were present, as well as oxalate molecules chelating zinc ions in the stoichiometric ratio of the experiment. This spectrum also contained an absorbance at  $750\text{cm}^{-1}$ , not present in the equivalent spectrum shown in ESI Fig. 1