

## Electronic supplementary information

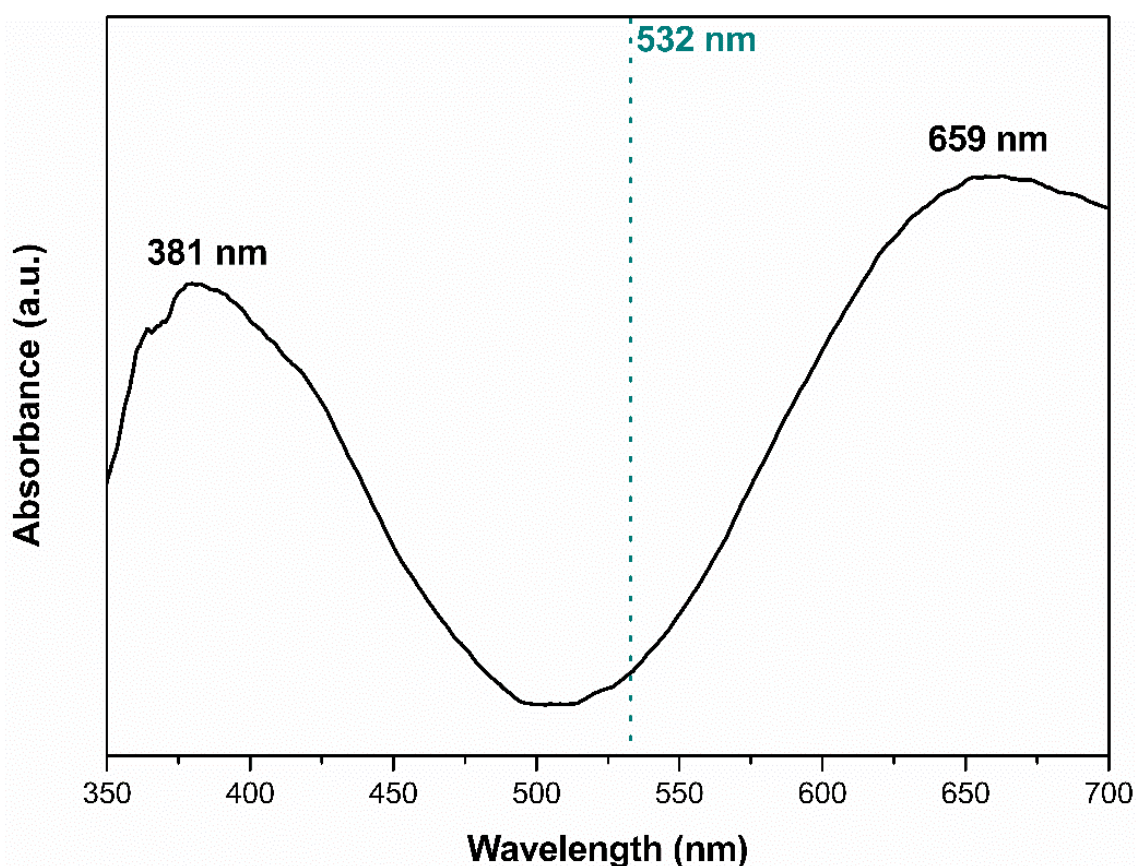
### Thermal decomposition of layered double hydroxide as a bottom up approach for the synthesis of metallic nanoparticles embedded in carbon structures

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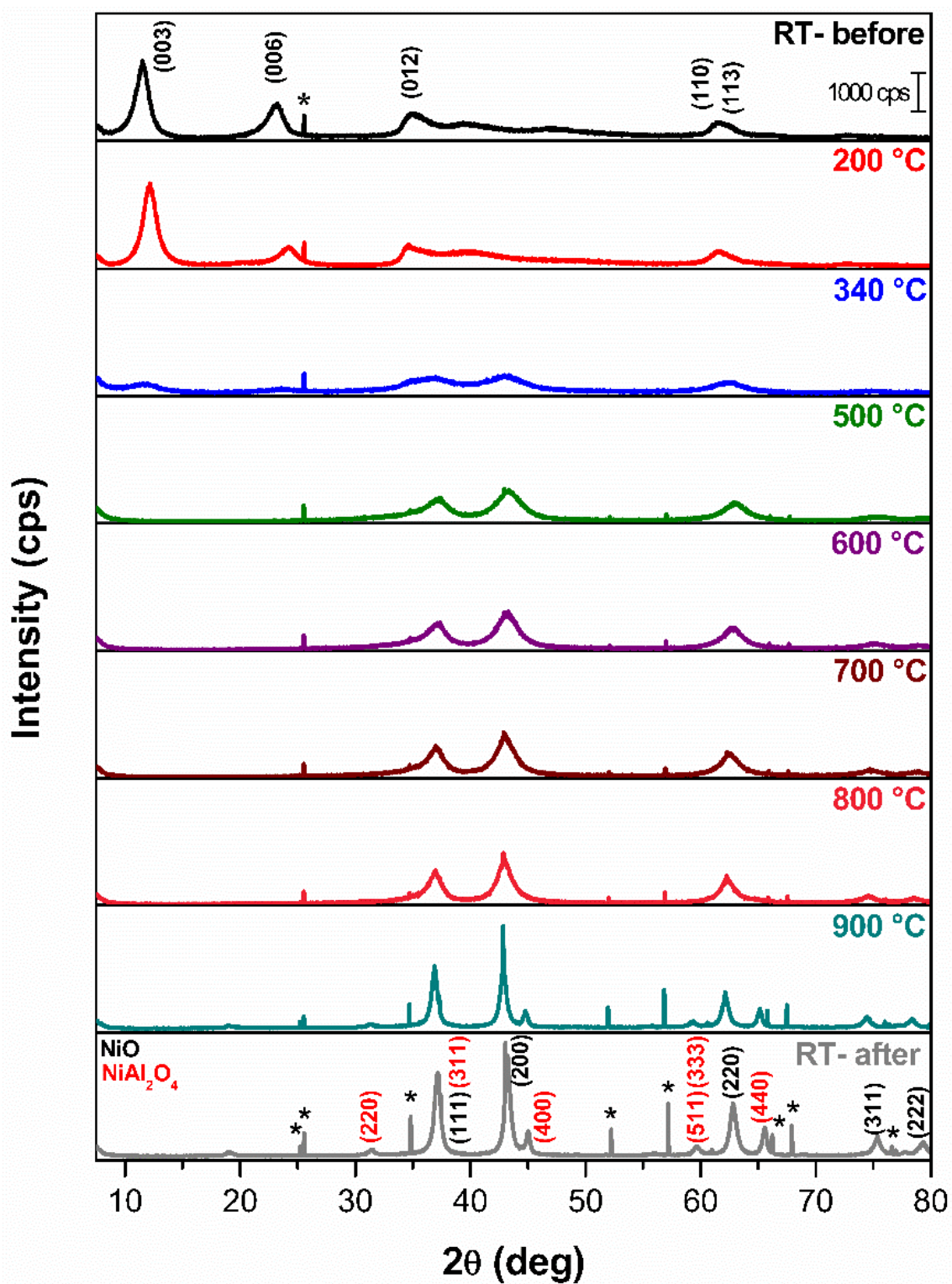
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**Fig. S1** Ultraviolet-visible electronic absorption spectrum of LDH-Cl (curve smoothed by Adjacent-Averaging method). The material was mixed with barium sulfate ( $\text{BaSO}_4$ ) and the solid state spectrum recorded in spectrophotometer Shimadzu UV-2401PC equipped with a integrating sphere.



**Fig. S2** HT-XRD patterns of LDH-Cl recorded under He atmosphere, under same conditions used for LDH-CMC sample. \* sample holder.

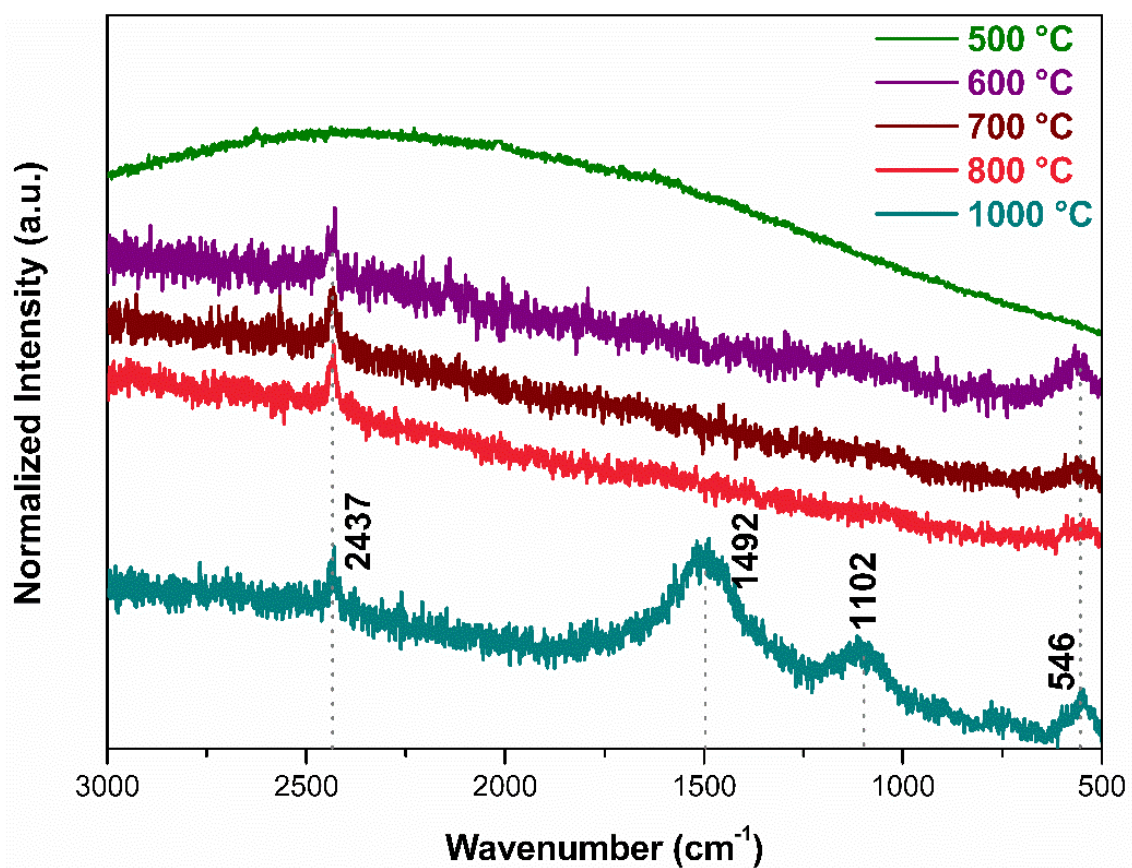
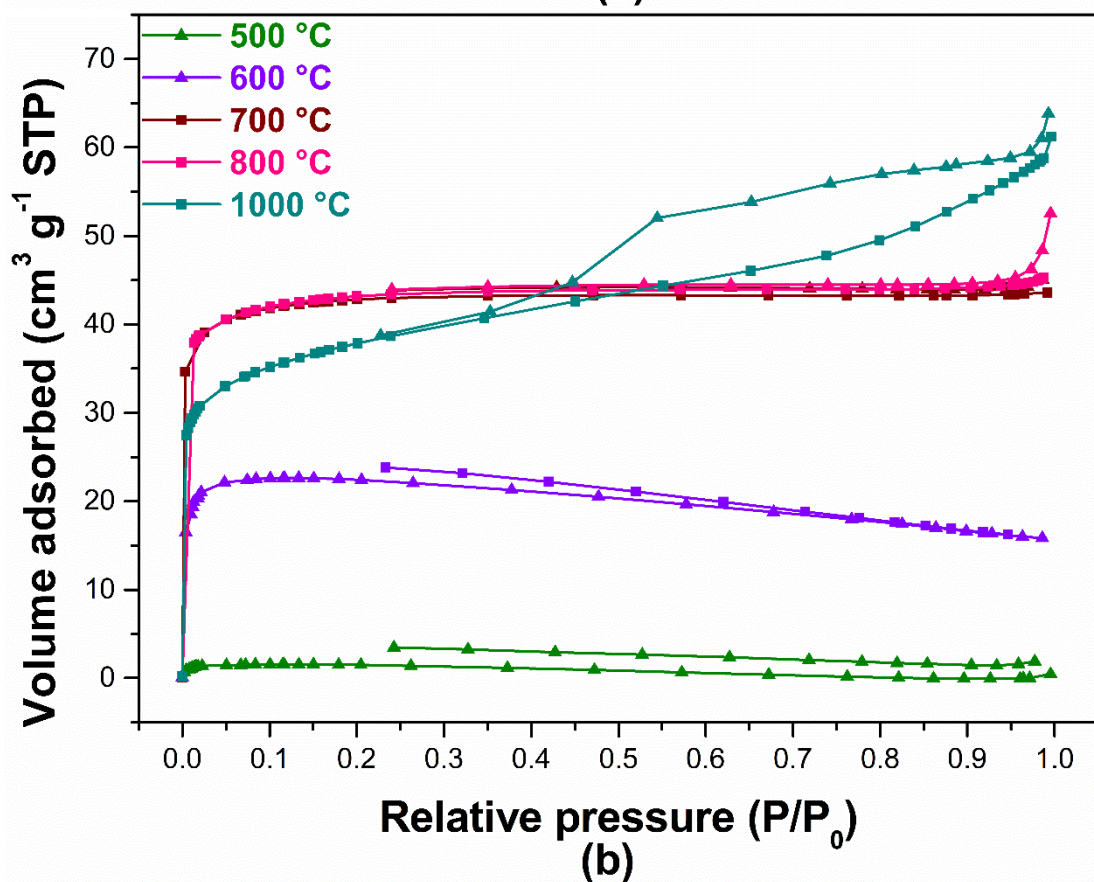
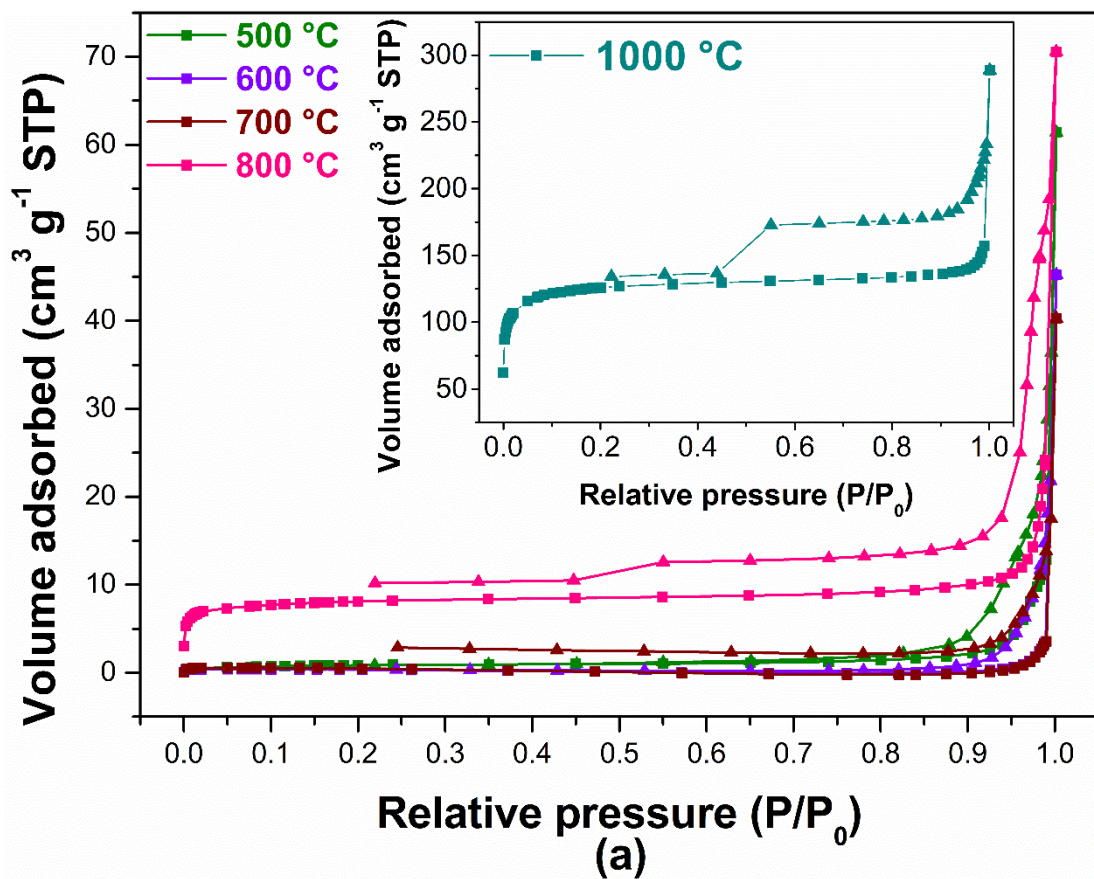


Fig. S3 Raman spectra of LDH-Cl-X series recorded using  $\lambda_{\text{ex}}$  of 532 nm.



**Fig. S4**  $N_2$ -adsorption/desorption isotherms of CMC-X (a) and LDH-CMC-X (b) samples series. *Inset* shows the curve of CMC-1000.

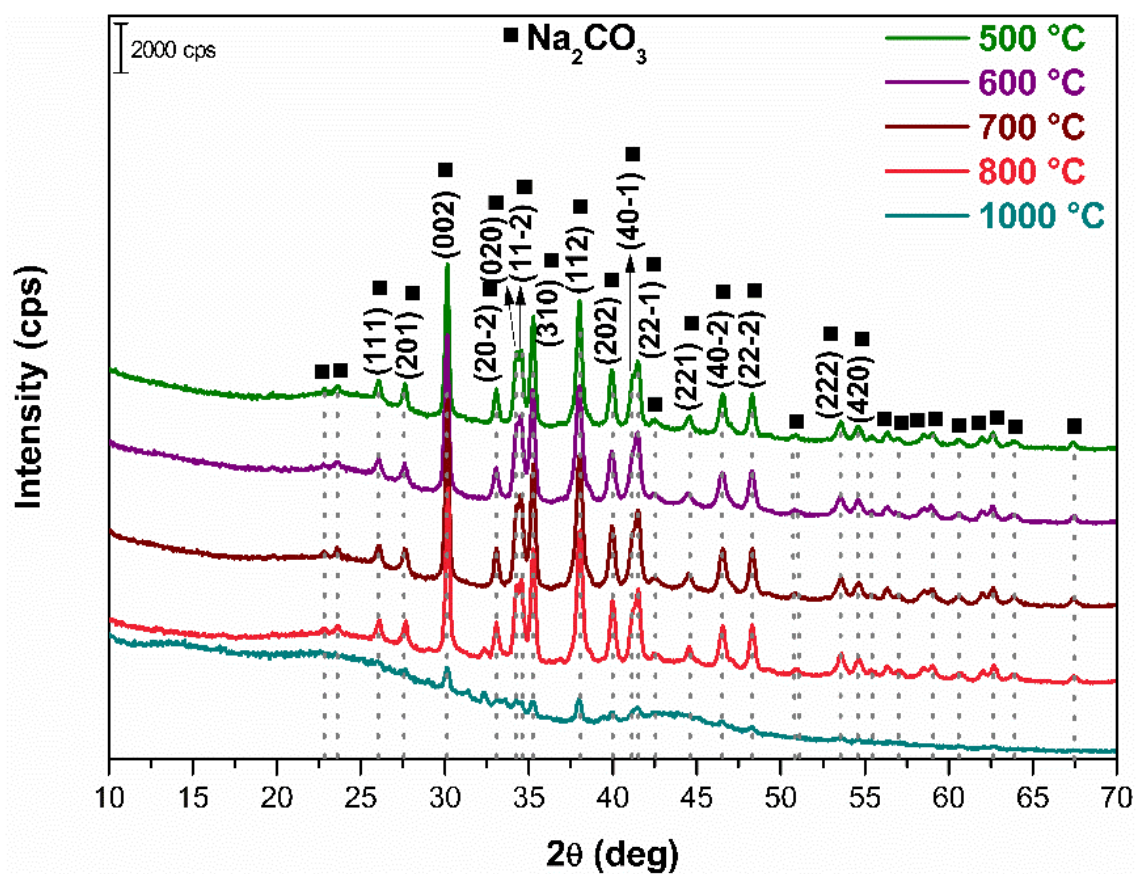
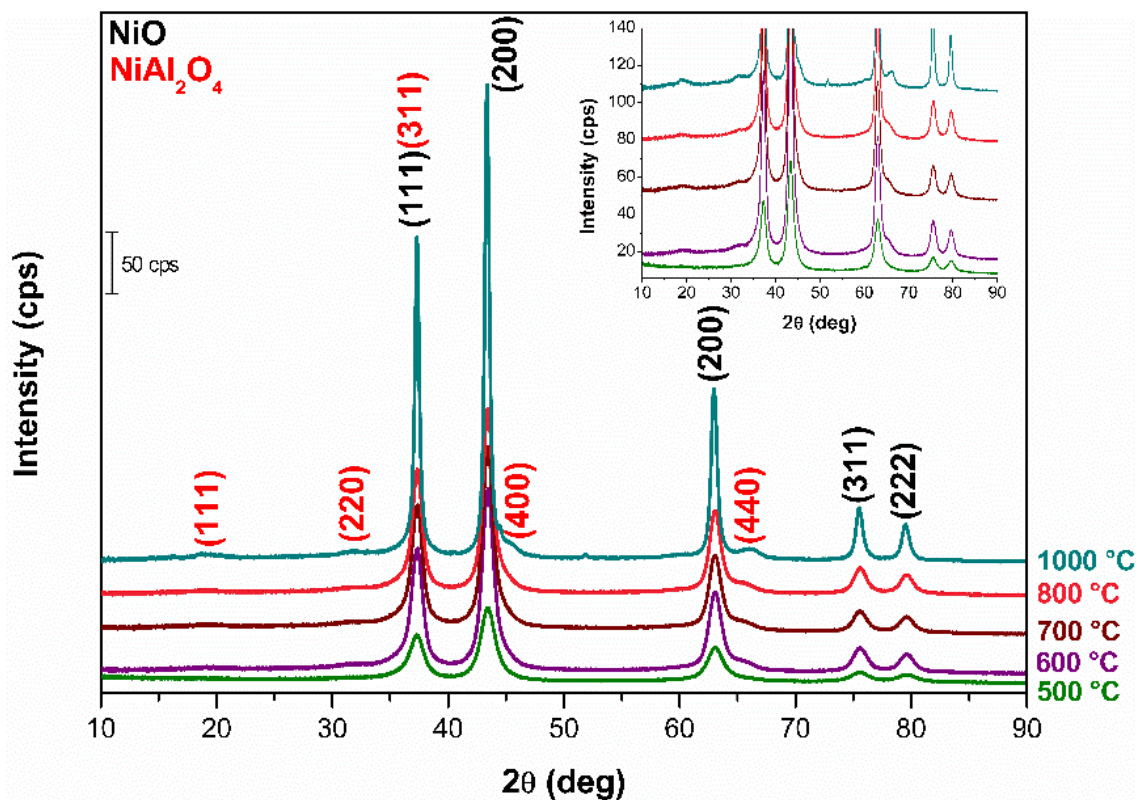


Fig. S5 XRD patterns of CMC-X materials pyrolyzed at the indicated temperature value.



**Fig. S6** XRD patterns of residues from LDH-CMC-X thermal analysis under air atmosphere. *Inset*: detailed diffractograms.

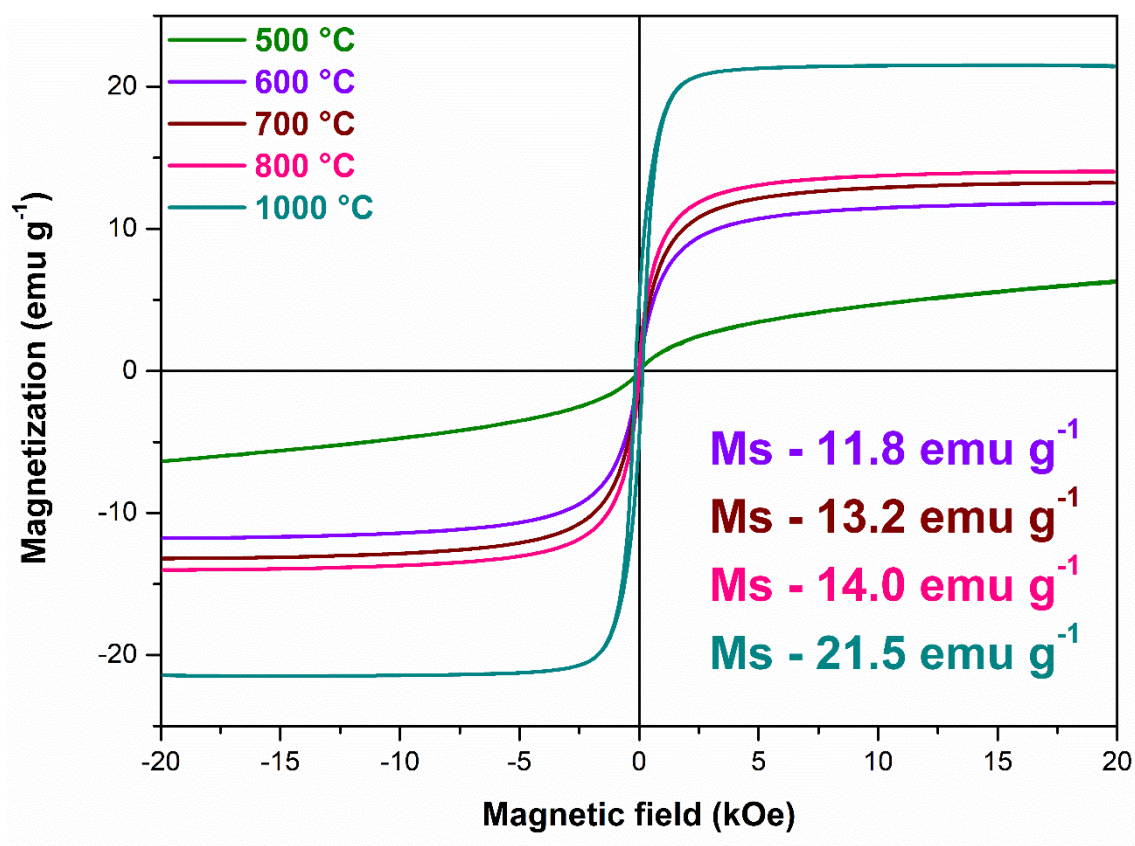
**Table S1** LDH-CMC-X residual mass from TG curves recorded under air atmosphere and experimental mass percentages of nickel and carbon.

Pyrolysis temperature (°C)	Residual mass <sup>a</sup> (%)	%Ni <sup>b</sup>	%C <sup>c</sup>
600	81.4	21.5	13.7
700	83.9	24.1	14.3
800	85.8	25.5	13.6
1000	92.1	39.0	13.1

<sup>a</sup> Values obtained in the 200 - 900 °C range, in order to discount mass loss from dehydration process.

<sup>b</sup> Values estimated from magnetization saturation ( $M_S$ ) of bulk nickel ( $55.1 \text{ emu g}^{-1}$ ),<sup>1</sup> and the corresponding values of each sample (see Fig. S6), calculated by:  $\%Ni = \frac{M_S}{M_S(\text{bulk})} \cdot 100\%$ .

<sup>c</sup> Data from elemental chemical analysis.



**Fig. S7** LDH-CMC-X magnetization curves recorded at room temperature, containing the values of saturation magnetization ( $M_s$ ) for the materials pyrolyzed from 600 to 1000 °C. The magnetization values were normalized by total mass of each material.

#### Reference

- 1 J. Crangle and G. M. Goodman, *Proc. R. Soc. A Math. Phys. Eng. Sci.*, 1971, **321**, 477–491.