

## Supplementary Information

### Size control of NiO nanoparticles for hydrogen storage application

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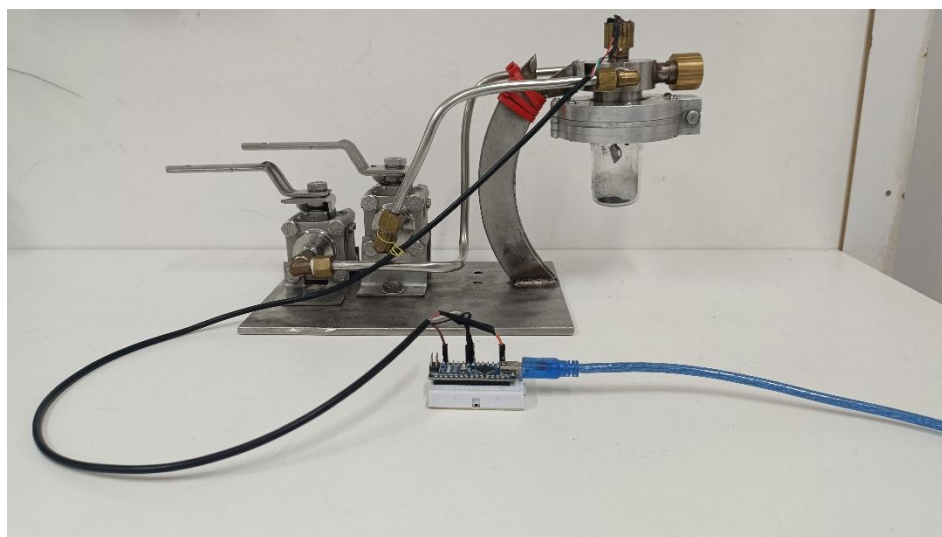


Figure S1 – Picture of the homemade reactor used in the H<sub>2</sub> adsorption reaction measurements.

Table S1 – H<sub>2</sub> amount as a function of time for the reactor without sample.

Time (min)	P (atm)	T (°C)	H <sub>2</sub> percentage (%)
5	1.0037	25.3	0.9635
30	1.0092	24.0	0.9639
60	1.0117	23.7	0.9637
999	1.0095	22.7	0.9611
1003	1.0077	22.7	0.9591
1030	1.0070	22.9	0.9565
1050	1.0054	23.0	0.9578
1060	1.0042	23.1	0.9595

Table S2 – H<sub>2</sub> adsorption measurements of activated carbon.

Time (min)	P (atm)	T (°C)	H <sub>2</sub> percentage (%)	Weight %
1	0.9951	21.1	0.9579	0.000
6	0.9941	21.0	0.9549	0.009
30	0.9920	20.5	0.9576	-0.009
60	0.9905	20.3	0.9563	-0.007
90	0.9888	20.3	0.9561	-0.006
120	0.9870	20.2	0.9556	-0.005
150	0.9858	20.3	0.9555	-0.005
170	0.9838	20.3	0.9538	0.004
180	0.9825	20.4	0.9535	0.005
210	0.9809	20.5	0.9546	0.002
220	0.9793	20.5	0.9552	-0.002
240	0.9773	20.6	0.9538	0.007
250	0.9757	20.6	0.9459	0.042
254	0.9740	20.7	0.9525	0.014

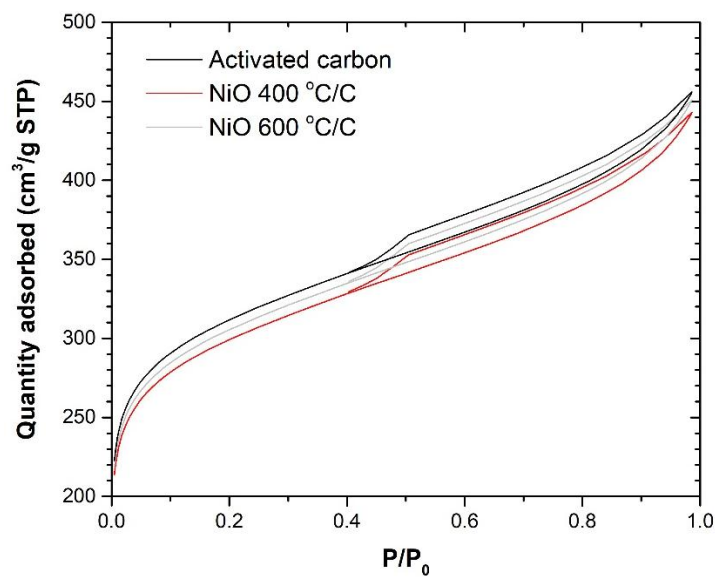


Figure S2 – N<sub>2</sub> adsorption-desorption isotherms of activated carbon and NiO 400 °C and NiO 600 °C nanoparticles supported on activated carbon.

Table S3 – Specific surface area and pore volume values derived from the N<sub>2</sub> adsorption-desorption isotherms.

Sample	BET surface area (m <sup>2</sup> /g)	BJH adsorption average pore width (Å)	BJH desorption average pore width (Å)
Activated C	1161	35	65
NiO 400 °C/C	1111	35	65
NiO 600 °C/C	1137	35	65

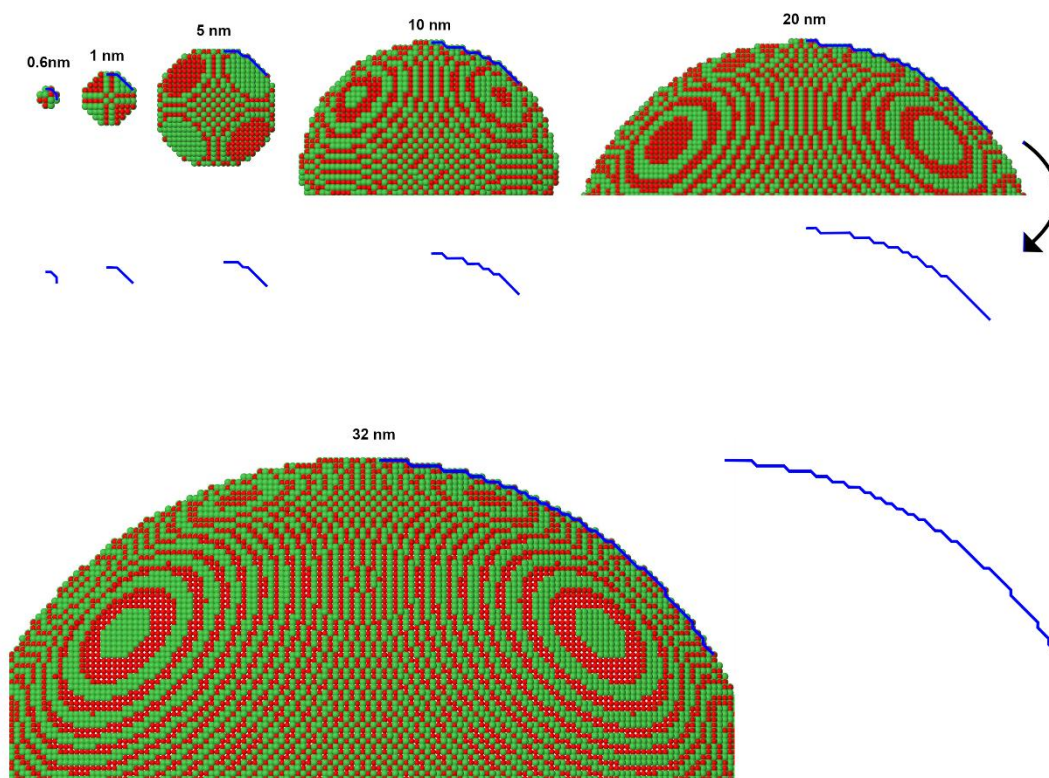


Figure S3 – Schematic representation of the NiO clusters used in the DFT calculations highlighting the changes on the surface curvature with the cluster size (blue line).

Table S4 – Lognormal distribution parameters obtained from the SAXS analysis of the NiO nanoparticles. The parameters are defined from the lognormal distribution curve where

$$y(x) = Nx^{-p} e^{-(\ln(x) - \ln(\mu))^2 / 2\sigma^2}$$

Sample	N	p	$\mu$	$\sigma$	Diameter (nm)	Standard deviation (nm)
NiO 400 °C	0.99	1	5.78	0.42	13	5.6
NiO 450 °C	1.04	1	7.50	0.44	17	7.6
NiO 500 °C	1.00	1	10.10	0.41	22	9.4
NiO 550 °C	0.99	1	12.35	0.42	27	11.9
NiO 600 °C	0.98	1	14.50	0.41	32	13.5

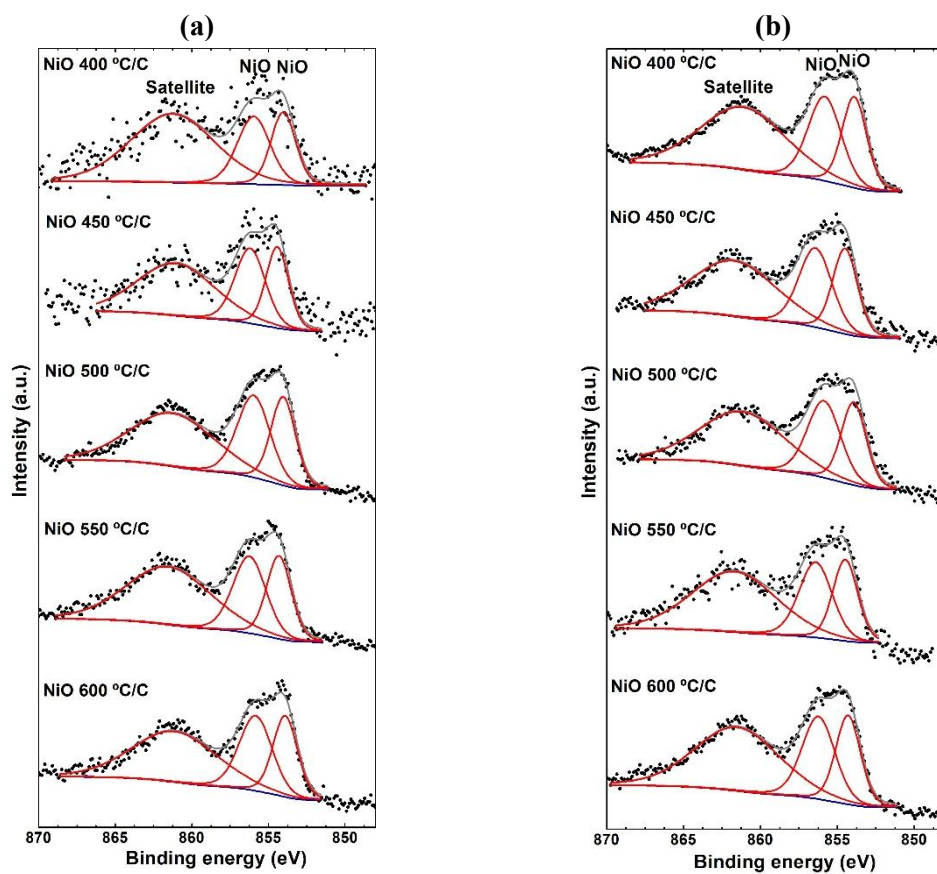


Figure S4 – XPS measurements at the Ni  $2p_{3/2}$  region of the 8 wt.% NiO/C nanoparticles (a) before and (b) after the  $H_2$  adsorption experiment.

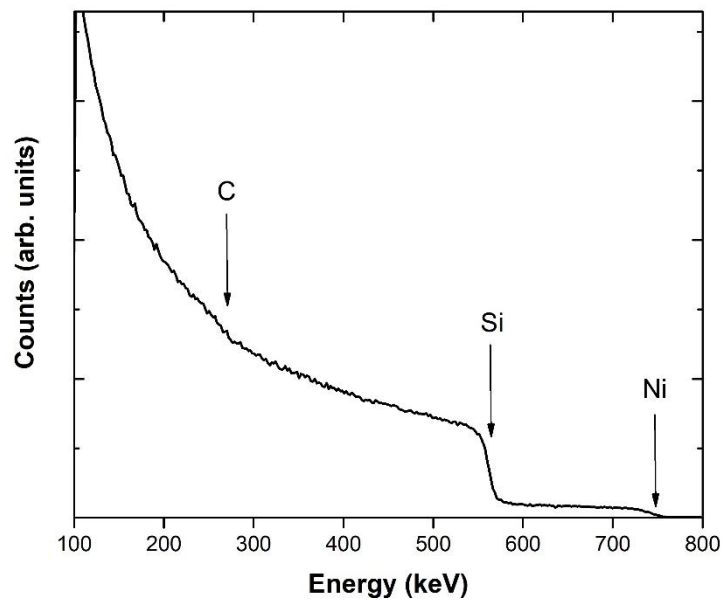


Figure S5 – Typical RBS measurement of the 1 wt.% NiO 550 °C/C nanoparticles film prepared over a Si substrate.

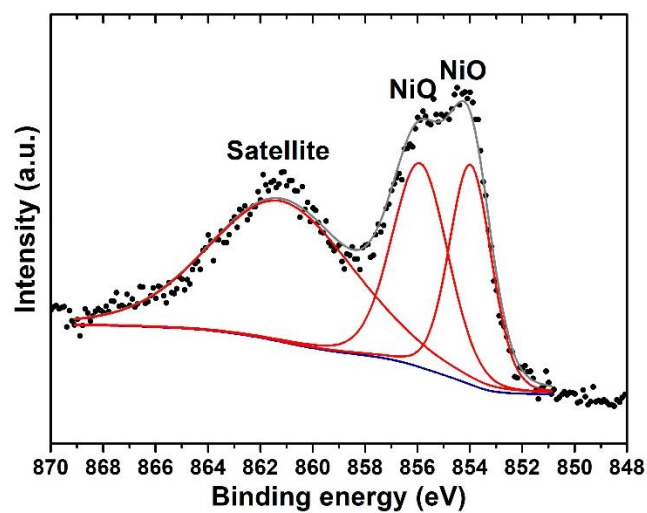


Figure S6 – XPS measurement at the Ni  $2p_{3/2}$  region of the 8 wt.% NiO 400 °C/C nanoparticles immediately after the H<sub>2</sub> adsorption experiment, with a short atmosphere exposition (around 5 min).

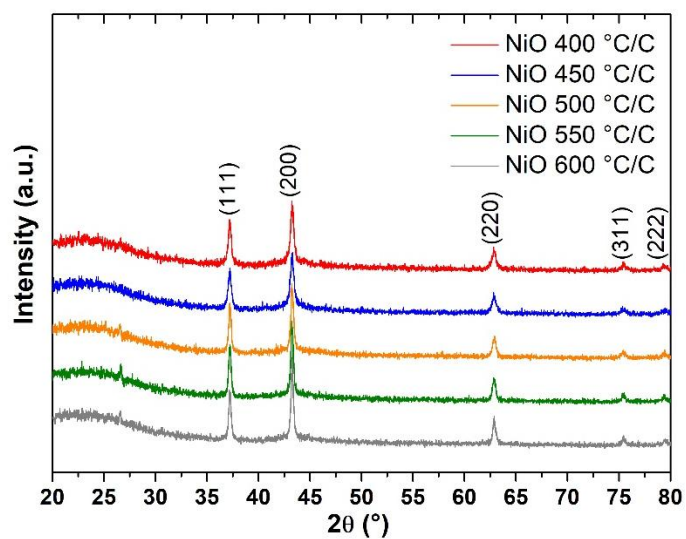


Figure S7 – XRD measurements of the 8 wt.% NiO/C nanoparticles after H<sub>2</sub> adsorption measurements.

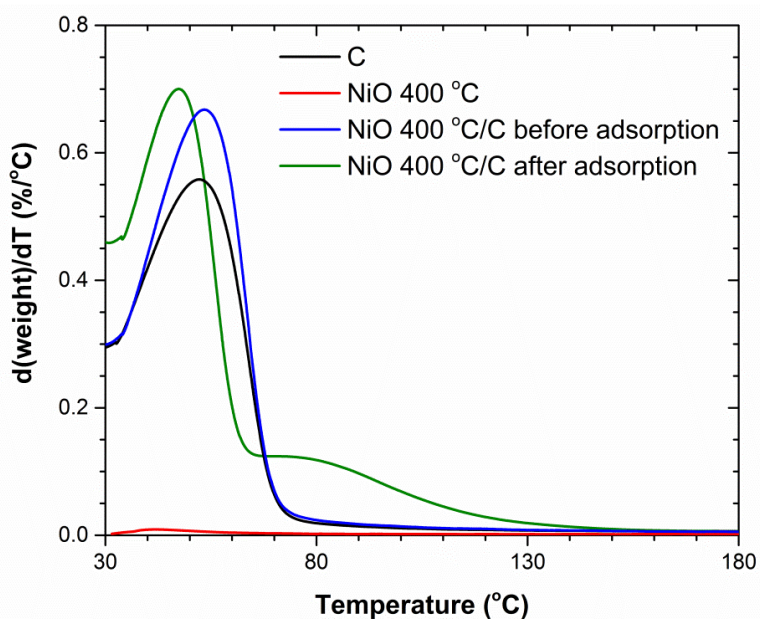


Figure S8 – TGA measurement showing the derivative of the weight with relation to the temperature of the samples before and after H<sub>2</sub> adsorption together with the activated carbon alone for comparison purposes.

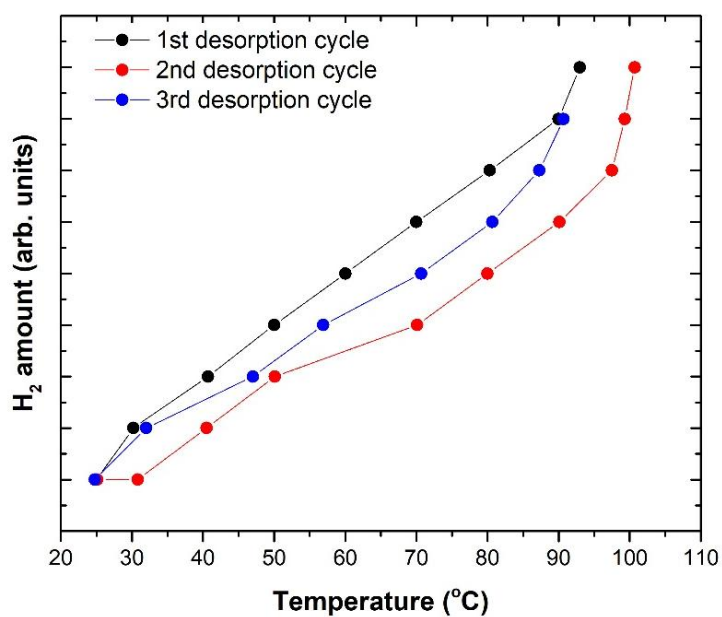


Figure S9 – H<sub>2</sub> desorption profile during heating under Ar atmosphere to around 95 °C of NiO 400 °C/C sample.

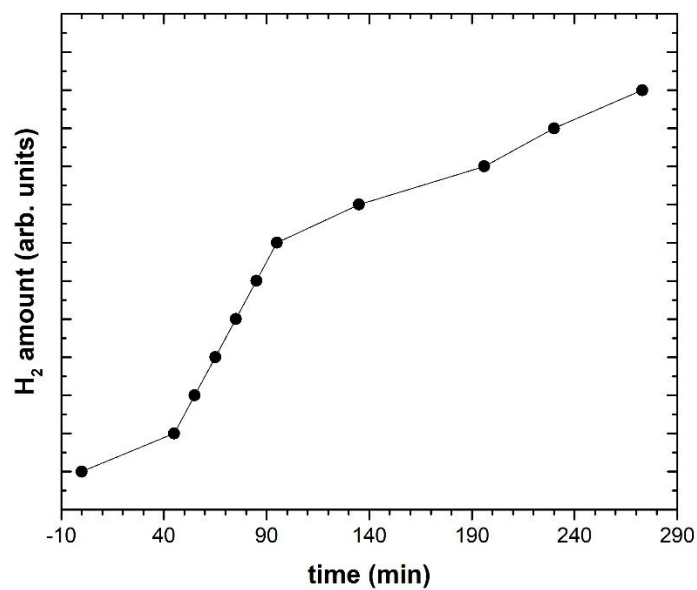


Figure S10 – H<sub>2</sub> desorption profile at a fixed temperature of 95 °C as a function of time for the NiO 400 °C/C sample.

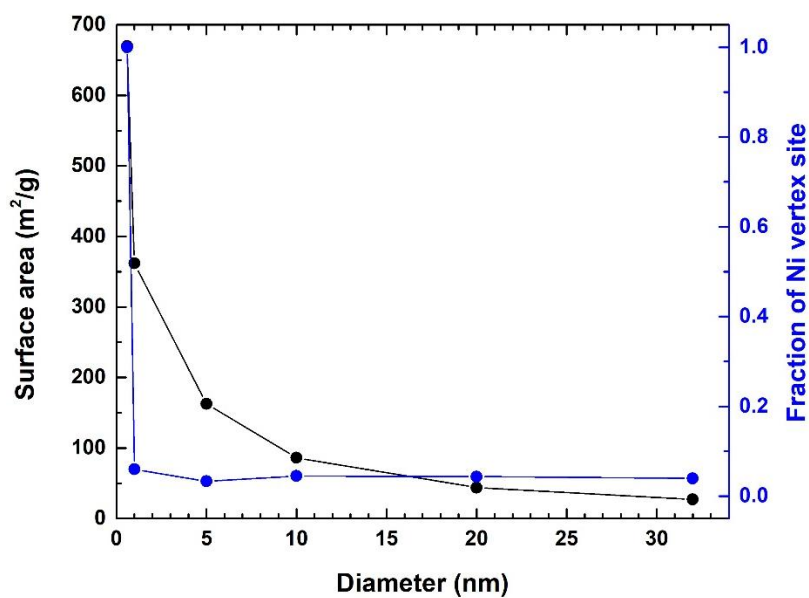


Figure S11 – Calculus of the specific surface area and fraction of Ni vertex site at the surface as a function of the NiO nanoparticle size.

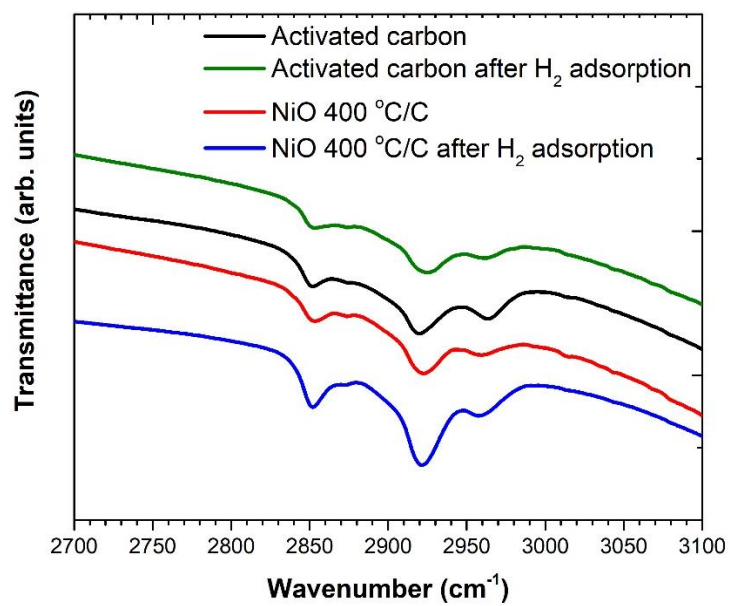


Figure S12 – FTIR measurements of the NiO 400 °C/C nanoparticles and activated C before and after H<sub>2</sub> adsorption measurement.