

Supplementary Information

The Birth of Nickel Phosphides Catalysts: Monitoring Phosphorus Insertion into Nickel

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1. Fitting parameters

Spectrum processing was carried out using the CasaXPS software package. Shirley background was used for all spectra. Line shape GL(30) were used, unless stated otherwise.

Species		B.E. (eV)	Doublet splitting	FWHM (eV)	Color
C 1s	Foil native carbide	282.9		0.85	Ochre
	Carbide	283.8		1.0	Ochre
	TOP alkyl chains	285.6**		0.8	Brown
	C-O	288.1		1.2	Yellow
	C-O	286.7		1.2	Yellow
	C-O	289.4		1.2	Yellow
	sp2 carbon/Ni	284.7**		0.8	Green
P 2p	Ni _x P _y	129.3	+0.86	0.9	Brown
	TOP on nickel	132.1	+0.86	1.9	Ochre
	TOP/unsaturated sites	133.0	+0.86	2.0	Yellow
O 1s	Oxide	529.9		1.3	Dark green
	Hydroxyl/carbonates	531.7		1.3	Ochre
	Water	533.0		1.8	Yellow
Ni 3p	Ni	66.0*	+1.6	1.7	Green
	Ni, sat	71.0*		5	Green
	NiO _x	68.9	+1.6	3	Dark green
	NiO _x , sat	72.5		3.4	Dark green
Ni 3s	Ni	110.6		2.5	Green
	Ni, sat	116.0		3.7	Green
	NiO _x	112.5		3.2	Dark green
	NiO _x , sat	119.0		6.5	Dark green

Doublet splitting is in eV. “Color” corresponds to the component color in the article.

*GL(90), **LA(4.2, 9.4).

2. Survey of the cleaned Nickel foil in the range 80-200 eV

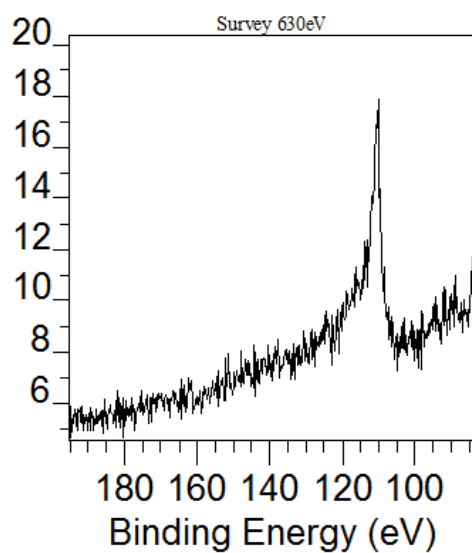


Figure S1: XPS prior to TOP deposition, in the range 80-200 eV, showing the absence of phosphorus species.

3. Transmission electron microscopy (TEM)

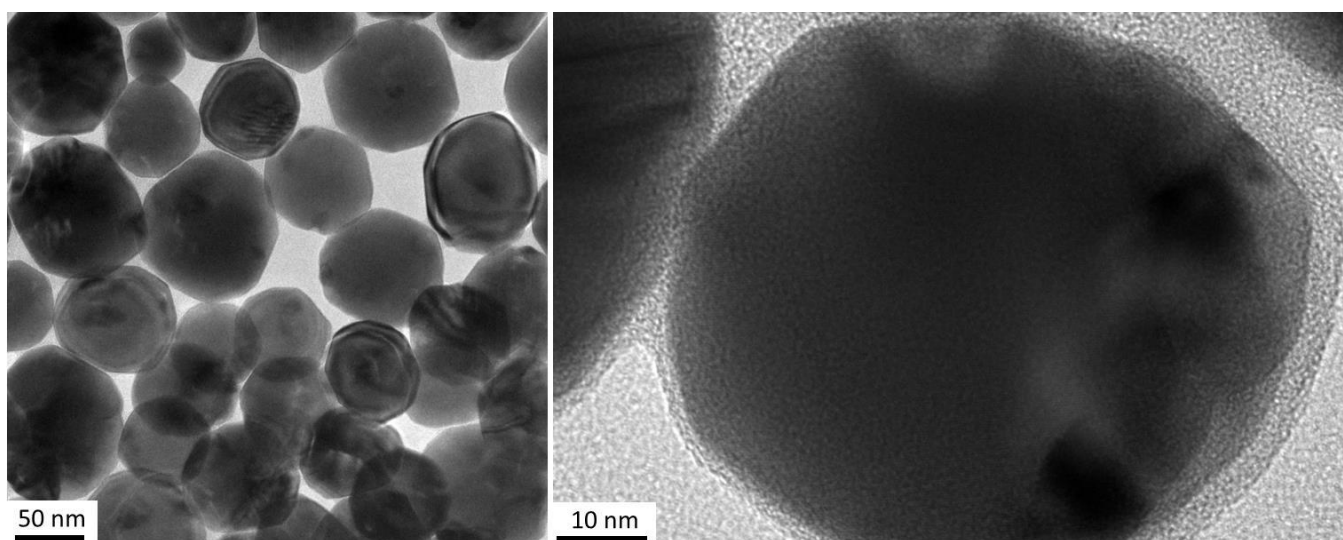


Figure S2: Complementary TEM observations of the nanoparticles after reaction with TOP.