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Synthetic routes to phosphorus-doped graphene nanoflakes

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Methods of analysis

High resolution transmission electron microscopy (TEM) images were recorded on a JEOL 2100 F/Cs (Jeol, Japan) microscope operated at 200 kV and equipped with a UHR pole tip and a spherical aberration corrector (CEOS, Heidelberg, Germany).

Scanning electron microscopy (SEM) images were measured on a JEOL JSM-6390LA microscope (Jeol, Japan) operated at 20 kV.

Low temperature nitrogen physisorption isotherms were recorded on an AUTOSORB-1C/MS/TPR analyzer (Quantachrome, Boynton Beach, FL, USA). Before the measurements, samples were degassed at 300 ºC under vacuum for 3 hours.

XPS spectra were acquired on an Axis Ultra DLD spectrometer (Kratos Analytical, UK) using monochromatic AlKa radiation (1486.7 eV). The pass energies of the analyzer were 160 eV for survey spectra and 40 eV for high resolution scans.

Table S1 Synthesis of P-doped graphene nanoflakes (GNFs).

Entry	Sample	Synthetic procedure	Reference
1	GNFs	Hexane was pyrolytically decomposed in the presence of MgO template at 900 °C for 30 min. MgO was removed by boiling in HCl followed by sample washing with water and drying at 110 °C for 8 h.	[S1]
2	GNFsox	GNFs were refluxed with HNO_3 water solution for 1 h. The product was washed with H_2O until neutral pH and dried at 110 °C for 8 h.	[S2]
3	P-GNFs_H_3PO_4	GNFs, synthesized as described in (1), were dispersed in the water solution of 60 wt. % H_3PO_4, dried at 80 ºC and calcinated at 850 ºC for 15 min under Ar atmosphere. The product was washed with water and dried at 110 ºC for 8 h.	[S3]
4	P-GNFs_ht	The 0.9 wt. % solution of PPh_3 in 40 ml of DMF was mixed with 0.10 g of GNFs and aged at 240 °C for 72 h. The product was washed with DMF and dried at 110 ºC for 48 h.	[S4]
5	P-GNFsox_ht	The 0.9 wt. % solution of PPh_3 in 40 ml of DMF was mixed with 0.10 g of oxidized GNFs_ox and aged at 240 °C for 72 h. The product was washed with DMF and dried at 110 ºC for 48 h.	[S4]
6	P-GNFs_pyr	The 2 wt. % solution of PPh_3 in toluene was pyrolytically decomposed in the presence of MgO template at 900 °C for 30 min. MgO was removed by boiling in HCl followed by washing with water and drying at 110 °C for 8 h.	[S5]

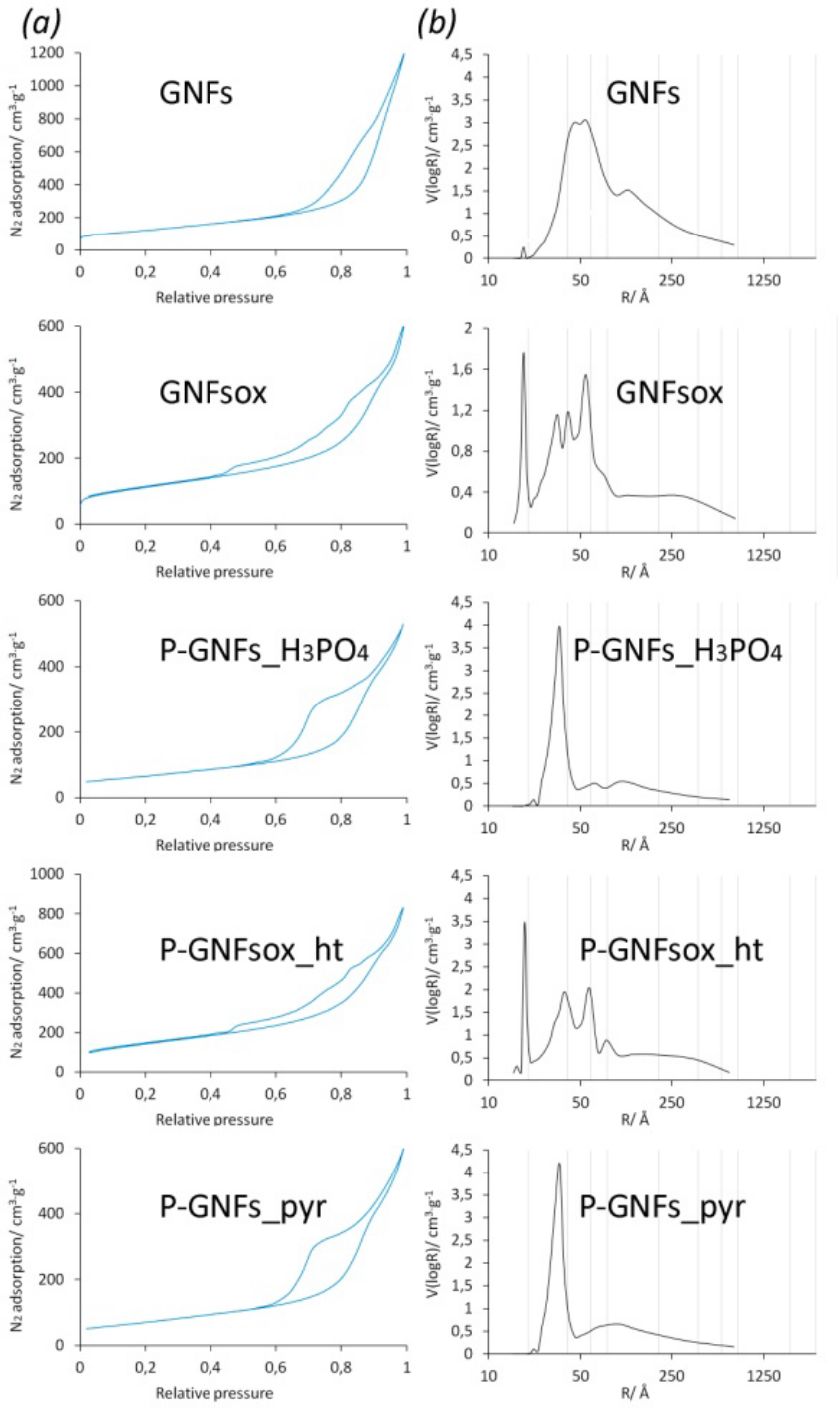

Figure S1 Nitrogen physisorption isotherms of P-GNF materials (a) and corresponding BHJ pore size distributions (b).

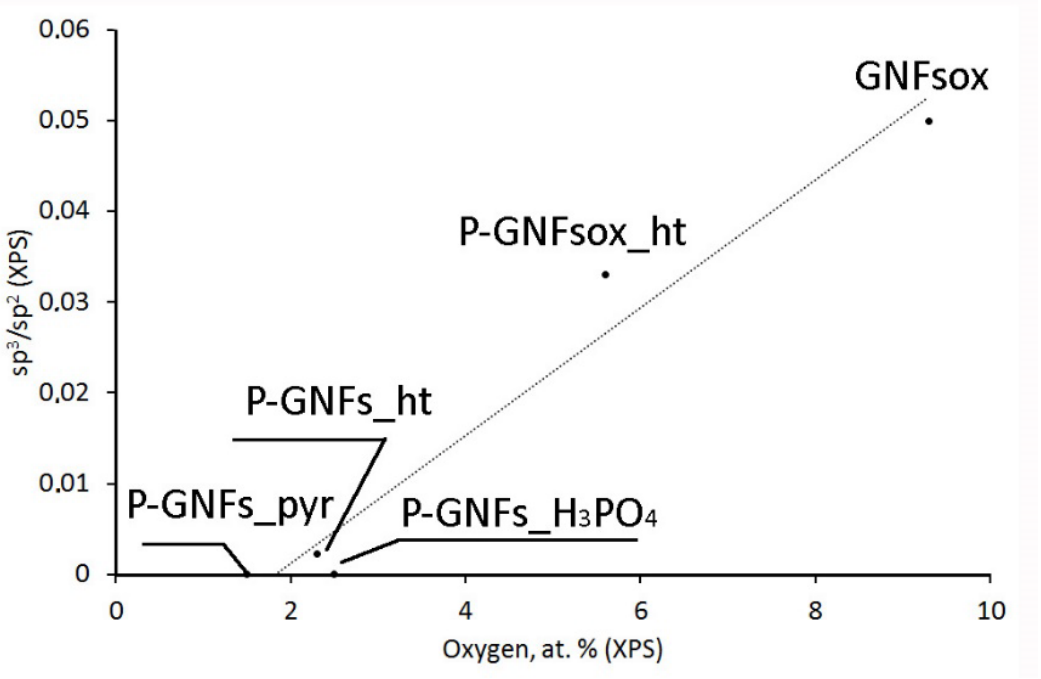

Figure S2 Defectiveness of GNFs (ratio of sp^3 to sp^2 species) calculated from XPS data versus oxygen content.

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