

ADVANCED MATERIALS INTERFACES

Supporting Information

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The Role of Water in the Preparation and Stabilization of
High-Quality Phosphorene Flakes

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The role of water in the preparation and stabilization of high quality phosphorene flakes

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Test A) P/H₂O molar ratio equal to 15.0.

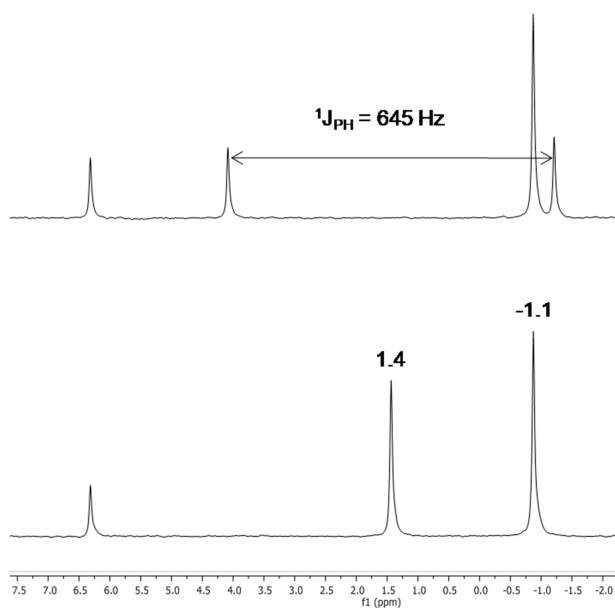


Figure S1. Up: ^{31}P NMR and down: $^{31}\text{P} \{^1\text{H}\}$ NMR (DMSO- d_6 , $T = 25^\circ\text{C}$, 121.49 MHz) spectrum of a sample of Test A after 4 hours of sonication.

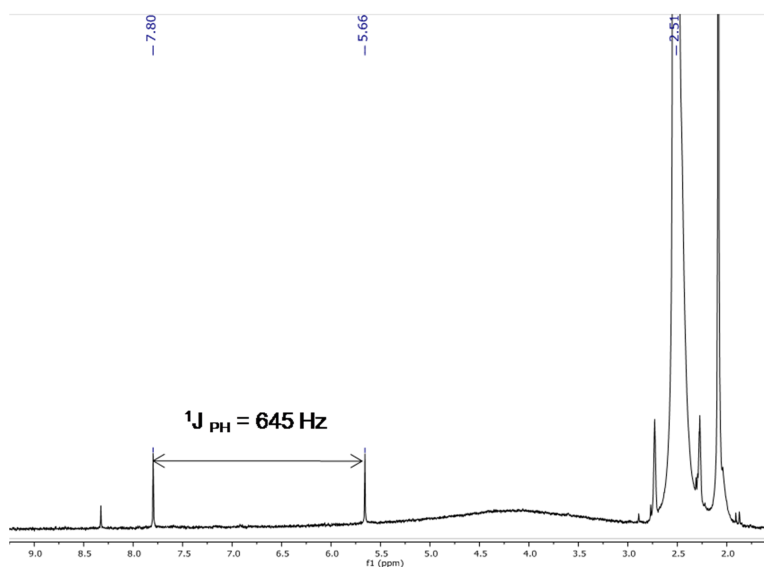


Figure S2. ^1H NMR spectrum (DMSO- d_6 , $T = 25^\circ\text{C}$, 300.13 MHz) after 4 hours of sonication.

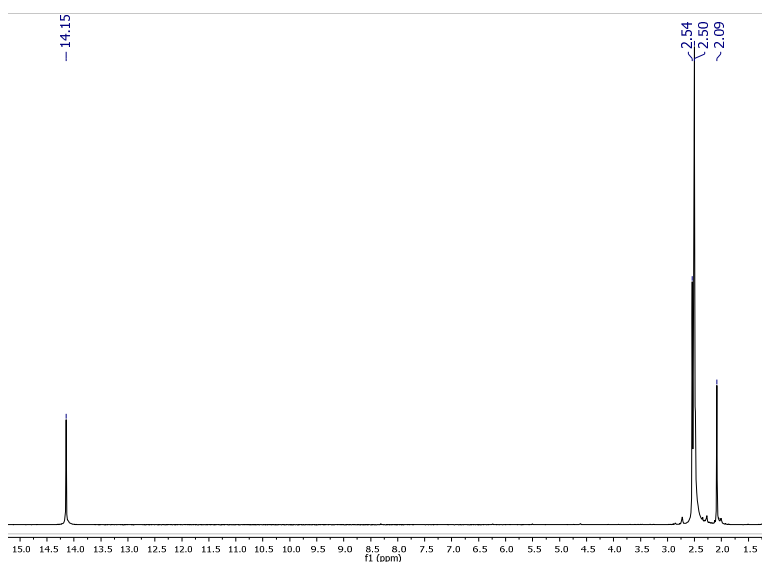


Figure S3. ^1H NMR spectrum (DMSO- d_6 , $T = 25^\circ\text{C}$, 300.13 MHz) after 20 hours of sonication.

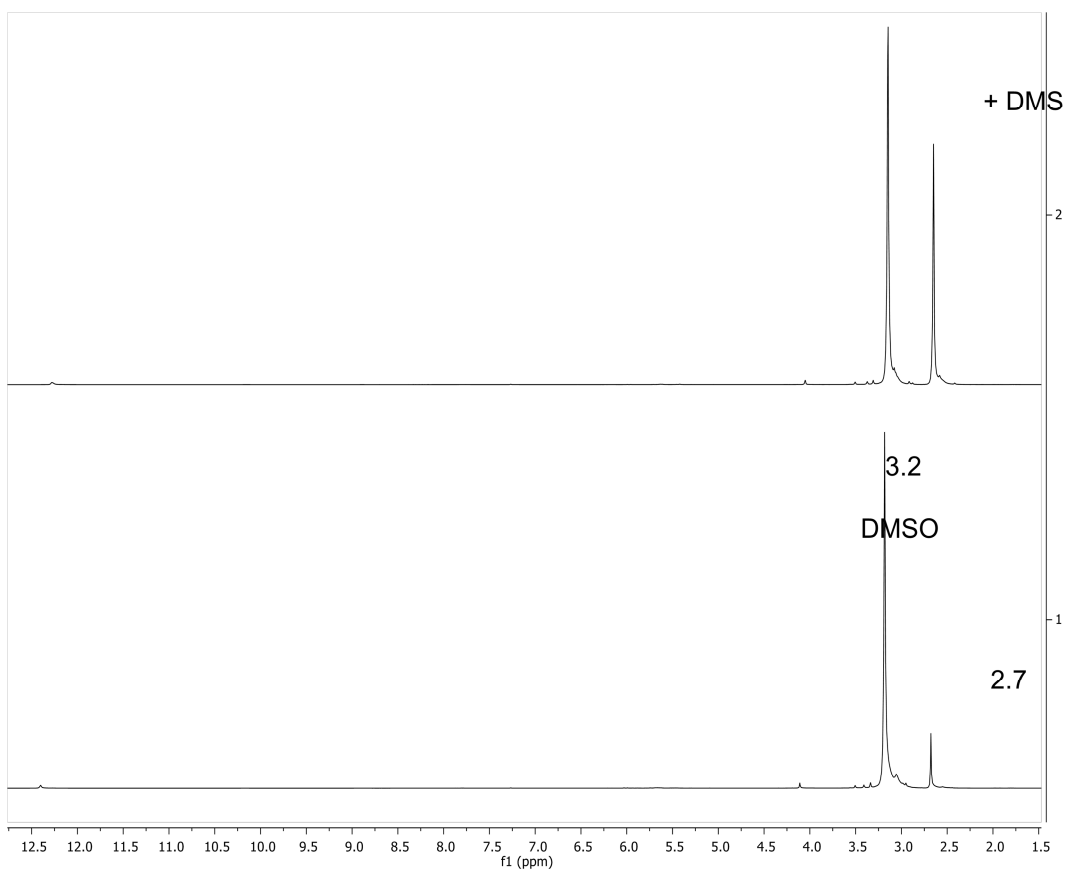


Figure S4. ^1H NMR (DMSO, $T = 25^\circ\text{C}$, 300.13 MHz, capillary C_6D_6). Down: reaction mixture before adding $[\text{S}(\text{CH}_3)_2]$; top: after adding $[\text{S}(\text{CH}_3)_2]$.

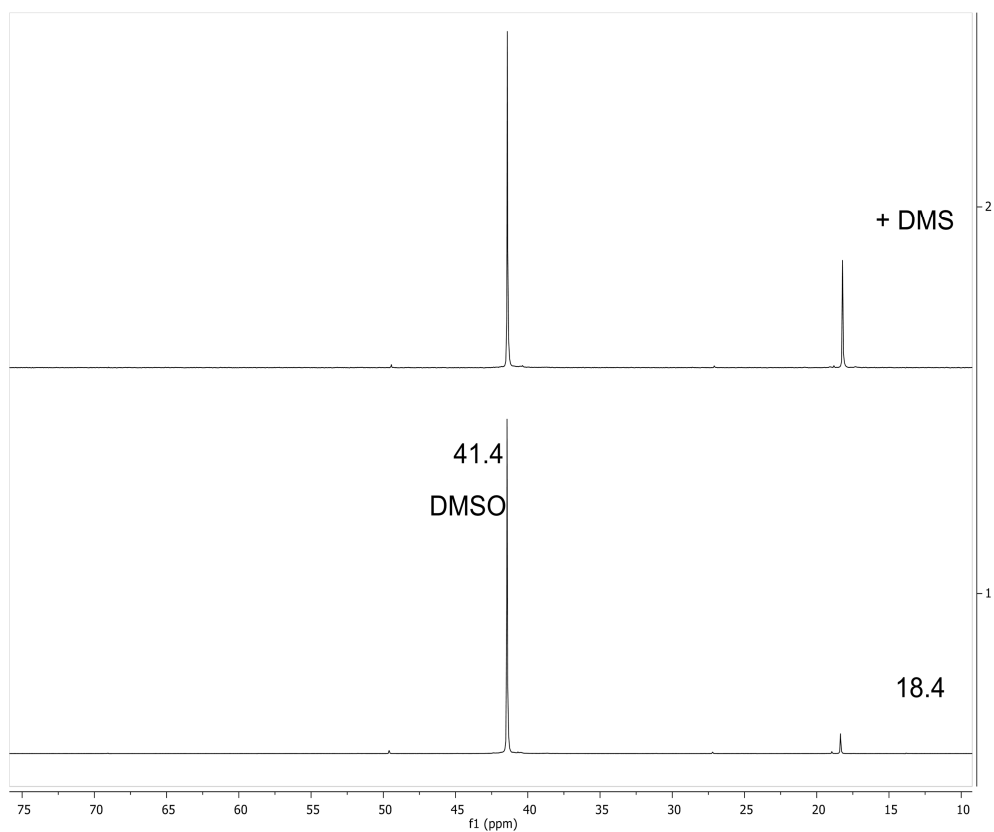


Figure S5. ^{13}C NMR (DMSO, $T = 25^\circ\text{C}$, 75.5 MHz, capillary C_6D_6). Down: reaction mixture before adding $[\text{S}(\text{CH}_3)_2]$; top: after adding $[\text{S}(\text{CH}_3)_2]$.

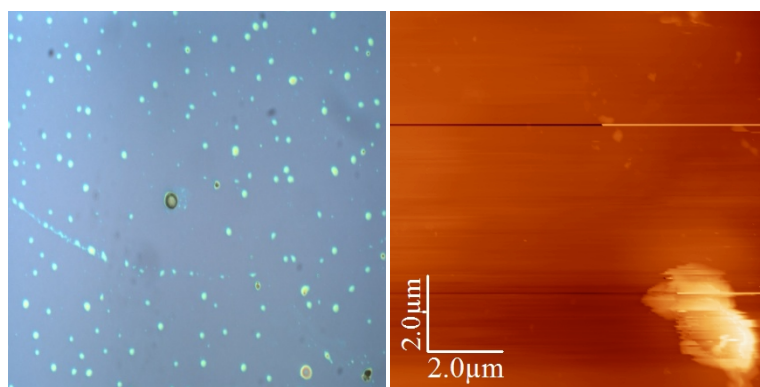


Figure S6. Left: optical microscopy image; Right: AFM of a sample of Test A after 20 hours of sonication, scale bar 2.0 μm .

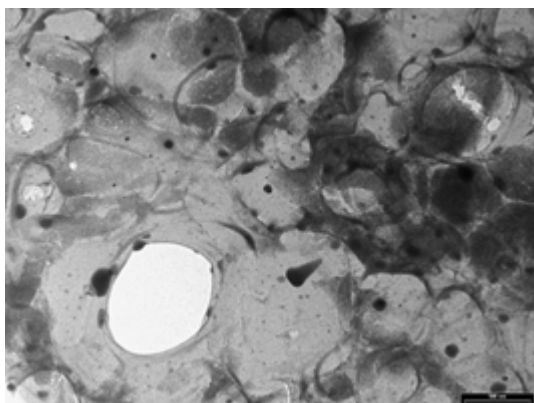


Figure S7. Bright field TEM image on holey carbon Cu grid of the sample in DMSO where P/H₂O molar ratio equal to 15. Scale bar: 500 nm.

Test B) P/H₂O molar ratio equal to 2.0.

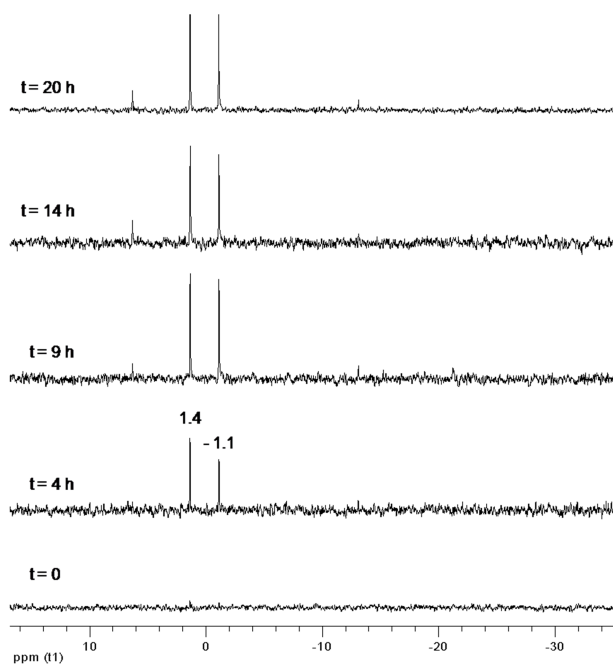


Figure S8. ³¹P{¹H} NMR spectra (DMSO-d₆, T = 25°C, 121.49 MHz) of a sample of Test B, P/H₂O molar ratio equal to 2, during sonication.

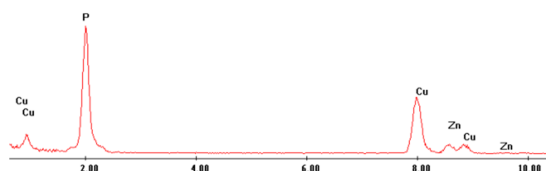


Figure S9. EDX carried out on the sample of Test B, P/H₂O molar ratio equal to 2, after 20 h of sonication.

Test C) P/H₂O molar ratio equal to 0.7.

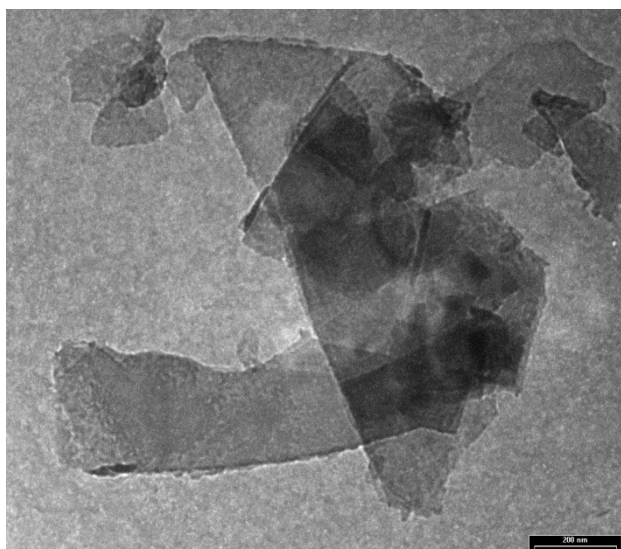


Figure S10. Bright field TEM image of DMSO-exfoliated BP nanosheets. Scale bar: 200 nm.

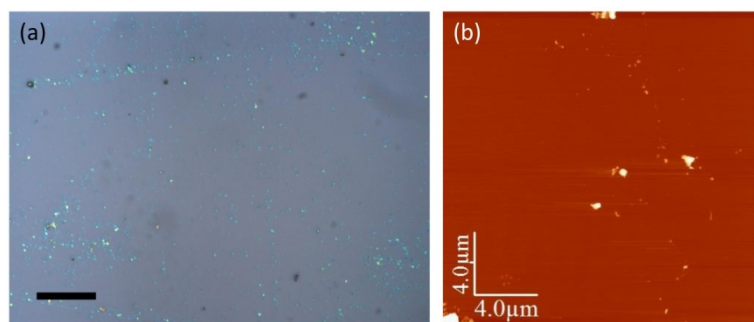


Figure S11. Left: Optical microscopy image, 144 μm x 108 μm, scale bar 30 μm; right: AFM image of a sample with P/H₂O molar ratio equal to 0.7.

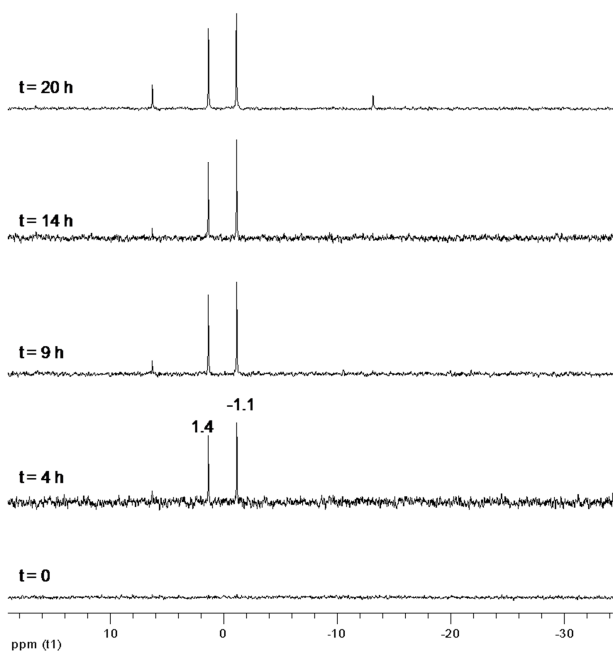


Figure S12. $^{31}\text{P}\{^1\text{H}\}$ NMR spectra (DMSO- d_6 , $T = 25^\circ\text{C}$, 121.49 MHz) measured during the sonication.

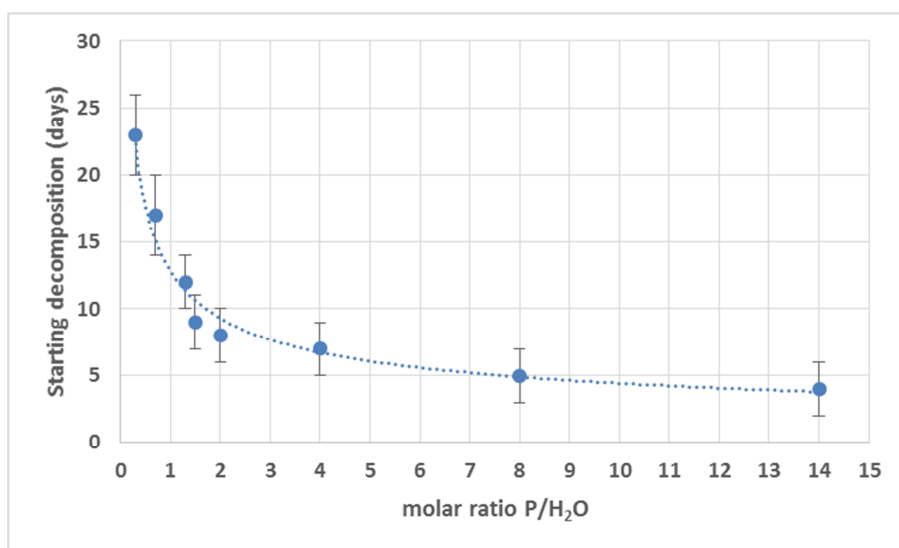


Figure S13. Starting of the exfoliated BP decomposition plotted against the P/ H_2O molar ratio (as shown by the appearance of the -24.9 ppm signal in the $^{31}\text{P}\{^1\text{H}\}$ NMR)

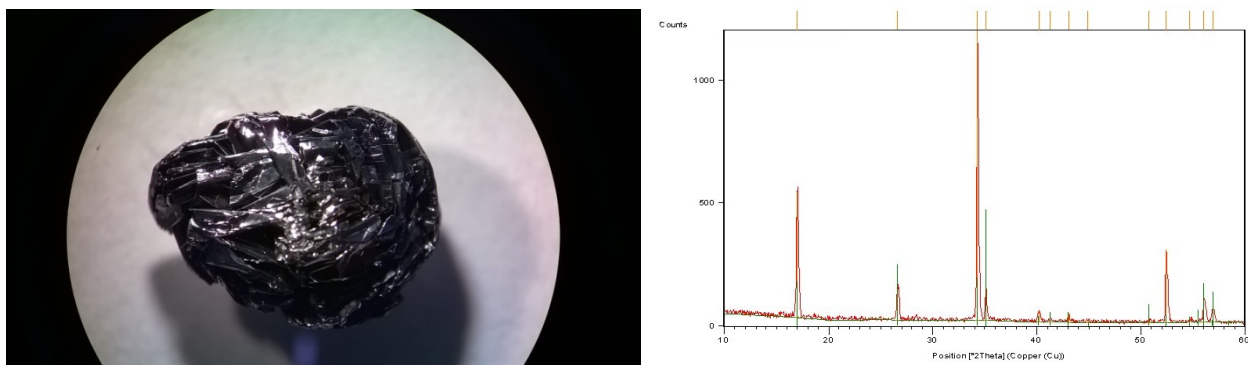


Figure S14. Picture of a BP crystal obtained after the synthesis (left); X-Ray powder diffraction of BP crystals (right).