Liquid-phase exfoliation of violet phosphorus for electronic applications

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Abstract
Large-scale production of two-dimensional (2D) materials still is a crucial point toward its practical applications. Violet phosphorus (VP) with a wide bandgap accelerates and broadens the potential applications of elemental phosphorus in optoelectronics. Here, we demonstrate the scalable production of solution-processable violet phosphorus flakes stably dispersed in several solvents. The exfoliated VP flakes exhibit thickness-dependent visible photoluminescence characteristics, which covers the shortcoming of black phosphorus. Meanwhile, the VP-based field-effect transistor reveals relatively competitive electrical properties to other liquid-phase exfoliated 2D materials. Our study paves the way for a wide range of applications of optical devices, energy storage, catalysis, and sodium batteries based on large-scale VP flakes.

KEYWORDS
field effect transistor, liquid-phase exfoliation, photoluminescence, violet phosphorus

1 | INTRODUCTION

Two-dimensional (2D) materials, such as black phosphorus (BP) and transition metal dichalcogenides (TMDCs), beyond graphene have attracted much attention around the world due to their outstanding potential applications in many fields, including photovoltaic devices,¹-³ sensors,⁴,⁵ optics,⁶-⁸ and catalyst.⁹-¹¹ BP has ultrahigh charge carrier mobility among these materials but exhibits a narrow bandgap (ranges from 1.5 eV in the monolayer to 0.3 eV in bulk), limiting its applications.²-⁴,⁸ Also, atomically layered molybdenum disulfide (MoS₂), one member of the TMDCs family, shows a large bandgap and demonstrates impressive on/off ratios of more than 10⁸ and ultralow standby power dissipation.¹² However, the normal charge carrier mobility¹³-¹⁵ independent of the devices fabricated on

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mechanical exfoliated or chemical vapor deposition-deposited MoS$_2$ films is still not satisfactory. Interestingly, phosphorus has several allotropes. The so-called Hittorf’s (or violet) phosphorus has been predicted$^{16-19}$ to have hole mobility up to 7000 cm$^2$/V·s (monolayer).$^{18}$ Meanwhile, monolayer VP has a desirable direct wide bandgap of 2.5 eV,$^{18}$ which can cover the shortcoming of BP photodetectors. VP is a single-element material and considered as an alternative wide-bandgap 2D semiconductor in many areas of electronics and optoelectronics. Furthermore, VP exhibits high mobility, tunable bandgap, relatively high on/off ratios ($>10^3$), and anisotropic properties.$^{18,20}$ However, most of the current studies on the mechanical exfoliation technique, which is not suitable for the semiconductor industry. To thoroughly investigate and explore the excellent properties of VP material, it is necessary to develop another method for the scalable production of VP nanoflakes. Although there is one paper focusing on liquid-phase exfoliation of VP,$^{21}$ the detailed exfoliation process and hosting solution are not systematic studied. Meanwhile, the electronic properties of pure exfoliated VP are still lack of study. Herein, we utilized the improved liquid-phase exfoliation technique to produce ultrathin highly crystalline VP nanoflakes in different solutions using the ultrasonication technique (see Section 2). The relevant electronic devices are also demonstrated.

# Experimental Section

## Sample preparation

First, bulk VP crystal was synthesized by the method reported in Zhang et al.$^{19}$ Here, we demonstrated that the purity of VP crystal could be improved by tuning the amount of SnI$_4$. Then the obtained bulk VP was ground into powder. After that, $\approx$2 mg VP powder was immersed in a 10 ml solution. The prepared samples were exfoliated into layered flakes by a probe-sonication process with a power of 630 W for 6 h at an ice bath (kept at 24°C). Then the obtained solution was centrifuged for 30 min in 3000 r/min, and the top 70% of the solution was collected for the study.

## Characterizations of VP flakes

Raman and photoluminescence (PL) signals for VP flakes on SiO$_2$/Si substrates were collected using a WITEC alpha 300 Raman microscopic system equipped with a 532 nm laser operating at 1 mW. The spot size of the excitation laser is $\approx$1 $\mu$m. The PL signal and PL lifetime for VP flakes in NMP were recorded using an FLS920P Edinburgh Analytical Instrument apparatus with Xe lamp (400 nm) and a 485 laser as excitation sources, respectively. The AFM measurements were performed in a Veeco Dimension-Icon system with a scanning rate of 0.972 Hz. The structural properties of the VP flakes were characterized by a Jeol JEM-2100F STEM and energy dispersive X-ray (EDX) operated at 200 kV. The morphology of the VP samples was examined by a Hitachi S-4800 SEM system. The UV-vis spectra were recorded at room temperature on a Shimadzu UV-2550 UV-vis-NIR spectrophotometer.

## Fabrication and electrical measurements of VP FET

The samples were transferred onto SiO$_2$/Si substrate using the spin-coated technique with a speed of 500 r/min and were immediately annealed at 100°C for 30 min to remove the excess organic residue. Then the electrode patterns were carried out in an electron-beam lithography (EBL) process and fabricated on Si substrate with 300 nm thick oxide. After developing, the samples were loaded in a metal evaporation vacuum chamber, and 10 nm of titanium followed by 60 nm of gold were deposited. The samples were annealed again in an argon atmosphere at 260°C for 1 h to remove fabrication residues. The electrical performance of the devices was monitored using Keithley 2612A source-meter in a closed-cycle cryogenic probe station with a base pressure of $\approx$10$^{-5}$ Torr.

# Results and Discussion

To deeply understand the fundamental properties of VP in solution form, we introduced several solvents in this study, including iso-propyl alcohol (IPA), methanol, ethanol, acetone, dimethyl sulfoxide (DMSO), N-methyl-2-pyrrolidone (NMP), and deionized (DI) water ($\text{H}_2\text{O}$), which covers a wide range of surface tensions (21.7–72.7 dyne/cm) and Hansen solubility parameters. Generally, 20 mg VP bulk was firstly ground into powder, and then 2 mg of the obtained powder was selected to be separately immersed in different solutions (10 ml) and sonicated for 6 h by a probe-assisted sonication process. The solutions were then centrifuged at 3000 r/min for 30 min. The supernatants were finally collected by a pipette and transferred into another container. The preparation process for liquid-phase exfoliation can be found in Figure 1A, and the final VP solutions look brown in ethanol and NMP solvents. It can be found the exfoliated VP in different solvents exhibit slightly different colors, which are
due to the variation in concentration of VP in the solvents, as shown in Figure S1A. As shown in Figure 1B, VP’s crystal structure consists of tubes with pentagonal cross-sections, which belong to the monoclinic structure with a space group of P2/c (No. 13) and is unlike the orthorhombic structure of BP. These double-tube layers are stacked together and bonded via the weak van der Waals force. Figure 1C shows the optical image of the VP-ethanol solution drop-casted onto SiO2/Si substrate. It can be seen that the bright dots in Figure 1C reveal the thicker VP samples. Figure 1D depicts the Raman spectra for VP flakes and bulk VP crystal, implying three feature peaks located at 360.9 cm⁻¹, 374.8 cm⁻¹, and 475.8 cm⁻¹, which are induced by intratube vibrations of bond bending and bond stretching. Compared with the bulk VP, the Raman peaks for solution-processed VP flakes show a blue shift of 9 cm⁻¹, revealing the few-layer feature of VP flakes. Meanwhile, the frequency gap between 298 cm⁻¹ and 301 cm⁻¹ can be observed, which is a feature characteristic related to VP. On the other hand, the PL spectrum of the few-layer VP flake demonstrates a direct bandgap at around 2 eV (Figure 1E-AS prepared), which is larger than its bulk form (1.87 eV), revealing the characteristics of the layer-dependent bandgap. Then the bandgap of VP can be tuned by reducing its thickness through further centrifugation with different speeds, as shown in Figure 1E. The largest bandgap we obtained in this study can be up to approximately 2.6 eV, which corresponds to the VP flakes with a centrifugation speed of 4500 r/min. In addition, the bandgap value of bulk VP crystal we got here is close to monolayer MoS2’s bandgap (1.85 eV), but few-layer VP shows a larger bandgap value implying the feasibility of VP-based devices operated in the low wavelength blue color range.

Furthermore, we also investigate the stability of VP solutions in the air, as shown in Figure S1. In terms of the studied solvents, the VP powder can be effectively exfoliated and suspended in solution by the probe-assisted sonication process. However, in some solvents such as acetone, DI water, and DMSO, we can find a
noticeable change in the color of the solution, which means VP is not so stable in such solvents, especially in DI water having the same situation as BP exhibited hydrophilic.24 As for acetone, DMSO, and ethanol solvents, some precipitation exists at the bottom of the bottle. We also measured the UV (ultraviolet) –Vis (visible)-NIR (near-infrared) absorption property of VP solutions, as shown in Figure S1C. There exist three typical UV absorption peaks located at approximately 257 nm for VP in IPA, ethanol, and DI water, approximately 270 nm for VP in DMSO and NMP, approximately 273 nm in methanol. The visible and near-infrared absorption bands between 400 and 800 nm are highlighted in blue and pink. It can be found that the intensity of the optical absorbance is very low in the high wavelength regime, which is similar to that of BP solutions.2 Moreover, we also detect the PL signal of the solution-based VP flakes shown in Figure S2. It can be found a feature PL peak located at 605 nm, which indicates the average bandgap of the VP flakes in NMP solvent can be estimated to be approximately 2.05 eV, a little larger than that of VP flakes spin-coated on SiO2/Si substrate. Then the sample was excited at 485 nm, and the PL lifetime can be obtained in Figure S2B. The decay curve can be best fitted with a two-exponential function giving a slow lifetime $\tau_1$ of 6.2 ns and a fast lifetime $\tau_2$ of 1.7 ns, obtaining an average lifetime of approximately 2.2 ns.

We transferred the VP solutions onto silicon (Si) and SiO2/Si substrates to further characterize VP flakes. Figure 2A shows the low magnification scanning electron microscopy (SEM) image of VP flakes drop-casted on Si substrate, revealing the VP flakes with sizes of several microns randomly stacked with each other. It should be noted here that such VP samples are the as-prepared flakes without centrifugation. The thickness of the samples by centrifugation was measured by atomic force microscopy (AFM), and the corresponding profiles are depicted in Figure 2B. The different locations are surveyed and represented by different colored lines marked by A, B, and C, as shown in Figure 2C. The thickness distribution for the studied VP samples in ethanol and NMP (the inset) solvents can be found in Figure 2D, collected from 50 VP flakes.

**FIGURE 2** Morphological characterization of the obtained VP flakes. (A) Low magnification SEM image of VP flakes on Si substrate. It should be noted here that such VP samples are the as-prepared flakes without centrifugation. (B) AFM image of the VP samples after centrifugation at a speed of 3000 r/min. The dashed box shows the shape of the VP flake on SiO2/Si substrate. (C) The height profiles corresponding to the drawn lines marked in (B). (D) After centrifugation, statistical flake thickness distribution was measured by AFM on 50 VP flakes, collected from ethanol and NMP (the inset) solutions. AFM, atomic force microscopy; SEM, scanning electron microscopy; VP, violet phosphorus.
flakes. It can be seen that the thickness of the VP flakes prepared in ethanol and NMP solvents varies from 9 nm to 145 nm (Figure S3), the most frequent thicknesses have a range of 15–30 nm for VP in ethanol and a range of 20–60 nm for VP in NMP. As labeled by the dash boxes in Figures 2B and S3A, VP's rectangle shape is not so clear due to the absorption of organic residue on the surface. Furthermore, scanning transmission electron microscopy (STEM) was employed to explore the structural properties of the VP flakes. The VP flakes were first transferred onto carbon grids by drop-casting and then placed into the STEM system for measurement.

It should be noted here that there are two types of VP flakes with two different morphologies, one is rectangular (majority), and the other possesses a nearly square shape (minority). The low magnification TEM image of a typical individual rectangle VP flake with a size of 700 nm selected from ethanol solution can be found in Figure 3A. The selected area electron diffraction (SAED) pattern from the surface of VP is shown in Figure 3B, which indicates the high crystalline quality of VP with three dash arrows assigned to (010), (110), and (100) planes with a lattice spacing of 0.93, 0.66, and 0.91 nm, respectively (according to ICDD-PDF: No. 75-0577). The other high-resolution TEM (HRTEM) image for the studied individual VP flake can be found in Figure 3C, revealing the VP flake possessing a periodical stacking sequence. The lattice spacing of the sample is \( \approx 0.23 \) nm, which corresponds to (040) crystal plane of monolithic phosphorus by ICDD-PDF: No. 75-0577. The bottom inset in Figure 3C depicts the line

**FIGURE 3** Structural characterization of the studied VP flakes. (A) A typical low magnification TEM image of VP flakes in ethanol. (B) SAED pattern of VP flake corresponding to (A). (C) High-resolution TEM image of the studied VP flake. The inset shows the line scan profile for lattice spacing of VP flake. (D) Cross-sectional STEM-HAADF image of a VP flake with the atomic arrangement represented by the blue ball-stick models. The inset shows the corresponding EDX result. (E) Statistical particle size distribution collected from TEM images (100 flakes) of VP samples in ethanol and NMP solvents, respectively. EDX, energy dispersive X-ray; HAADF, half-angle angular dark field; SAED, selected area electron diffraction; TEM, transmission electron microscopy; VP, violet phosphorus
scan profile of lattice spacing of the studied VP flake. Figure 3D shows the cross-sectional STEM half-angle angular dark field (HAADF) image of a VP flake with the atomic arrangement represented by the blue ball-stick models, which confirms the atomic structure of VP. Besides the microstructure of this material, its composition is also needed to be noted. As confirmed by the EDX spectrum of the sample in the inset in Figure 3D, the studied sample only consists of P elements except for the Cu originated from the copper grid, which further indicates the actual VP sample we got.

In addition to the VP flake selected from ethanol solution, we also investigate the microstructure of the VP samples from NMP solution, as shown in Figure S4. We can see two rectangle VP flakes stacked with each other, and there is some residual NMP absorbed on the flakes, as shown in Figure S4A. Figure S4B reveals the corresponding local magnification of the red-dash circle marked in (A). The inset exhibits the SAED pattern for the studied VP flake, revealing the highly crystalline quality of VP. The corresponding high-resolution TEM image in Figure S4C indicates the lattice spacing for the VP sample is approximately 0.26 nm, which is assigned to (018) crystal plane by ICDD-PDF: No. 75-0577. The line scan profile and the yellow arrow marked in Figure S4C confirm the lattice spacing of the VP sample. The size distribution of the VP flakes in ethanol and NMP is calculated to be in a range of 50–1000 nm as determined by TEM, as shown in Figure 3E,F. Furthermore, we also investigate the morphologies of the VP flakes in other solvents, as shown in Figure S5. It can be found that most of the VP flakes in IPA, acetone, and DI water also possess a rectangular shape.

Next, we further check the electronic properties of the individual VP flake-based back-gate FET. A typical optical image of the fabricated device with a flake thickness of \( \approx 50\text{ nm} \) is shown in Figure 4A. Figure 4B shows the schematic illustration of the VP-based FET structure. Figure 4C reveals the drain current versus back-gate voltage \( (I_{ds} \text{ vs. } V_g) \) characteristics of the VP device in vacuum conditions at a constant source-drain bias \( (V_{ds}) \text{ of } 0.5 \text{ V} \). Then, the field-effect mobility for holes can be extracted from the linear region by \( \mu = \frac{g_m L}{C_g V_{ds} W} \), where \( g_m = \frac{dI_{ds}}{dV_g} \), \( C_g \) is the gate capacitance, and \( L (4 \mu \text{m}) \) and \( W (0.4 \mu \text{m}) \) are the length and width of the device channel, respectively. Using the corresponding linear slope of the \( I_{ds} \text{ vs. } V_g \) characteristics (here \( V_{ds} = 0.5 \text{ V} \)), the calculated value of the mobility can be up to \( \approx 0.05 \text{ cm}^2/(\text{V·s}) \).
drain current modulation (on/off ratio) for our device is estimated to be approximately 10. Figure 4D shows a typical $I-V$ characteristic of the studied devices measured at a back gate voltage of $-40 \text{ V}$, revealing non-Ohmic characteristic, which is due to its Schottky contacts at the metal-VP interfaces. It could be attributed to the possible trap states and residual solvent at the flake interface with the substrate and the metal electrodes. A similar characteristic has also been found in liquid-phase exfoliated MoS$_2$ flakes FET device.25 Meanwhile, the low mobility we got in this study is mainly due to the Schottky barrier and large contact resistance, which induces a large portion of the applied $V_{ds}$ drops across the contact resistances.

4 | CONCLUSION

In summary, we have demonstrated large-scale production of pure and good quality VP flakes via the liquid-phase exfoliation method. Such VP flakes can be well dispersed and protected against degradation in several solvents. The exfoliated VP flakes show thickness-dependent visible PL characteristics. The VP-based FET reveals relatively competitive electrical properties compared to other liquid-phase exfoliated 2D materials. Our work opens up a new pathway for preparing large-scale VP flakes-based thin films and composites for a wide range of applications.

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CONFLICT OF INTERESTS

The authors declare no conflict of interest. [Correction added on 29 June 2021, after first online publication: Conflict of Interest section has been added.]

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**SUPPORTING INFORMATION**
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