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A promising electrochemical sensor based on PVP-induced shape control of hydrothermal synthesizing layered structured vanadium disulfide for the sensitive detection of sulfamethoxazole antibiotic

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The presence of sulfamethoxazole (SMX) in natural waters has become a significant concern recently because of its detrimental effects on human health and ecological environment. To address this issue, it is of utmost urgency to develop a reliable method that can determine SMX at ultra-low levels. In our research, we utilized a PVP-induced shape control of hydrothermal synthesizing method to fabricate layer-like structured VSs, and employed it as an electrode modification material to prepare an electrochemical sensor for the sensitive determination of SMX. Thus, our prepared VS electrodes exhibited linear range of 0.06–10.0 μM and limit of detection (LOD) lower as 47.0 nM (S/N=3) towards SMX detection. Additionally, the electrochemical sensor presented good agreement with HPLC method, and obtained perfect recovery results (97.4–106.8%) in practical analysis. The results validated the detection accuracy of VS electrodes, and demonstrated its successful applicability toward sensitive determination of SMX in natural waters. In conclusion, this research provides a promising approach for the development of electrochemical sensors based on VS composite materials.

Introduction

Sulfamethoxazole (SMX) is a synthetic antibiotic with a broad antibacterial spectrum of activity.1,2 Particularly, it is effectively against Staphylococcus aureus and Escherichia coli. It finds widespread application in the medical, agricultural, and animal husbandry industries, making it one of the most utilized sulfonamide medications.3 However, according to the World Health Organization’s International Agency for Research on Cancer (IARC), Sulfamethoxazole was classified as a Group 3 carcinogen in their 2017 list of carcinogens.4 The reason behind this classification lies in the fact that Sulfamethoxazole is a large molecular organic compound that exhibits limited degradation capabilities. Living organisms in a sulfamethoxazole-contaminated environment might experience compromised growth and reproductive capabilities.5 It is true that these antibiotics can enter the human body through the enrichment effect of the food chain, thereby posing a risk to human health. Therefore, ensuring the sensitive detection of sulfamethoxazole is of utmost importance.

Presently, the detection methods for Sulfamethoxazole mainly consist of spectrophotometry, high-performance liquid chromatography (HPLC),6 7 enzyme-linked immunosorbent assay (ELISA),8 capillary electrophoresis,9,10 and several other techniques. Nevertheless, the application of traditional detection methods in antibiotic detection technology is significantly impeded by the need for costly equipment, intricate sample preparation, and time-intensive detection procedures.11 On the other hand, electrochemical detection techniques have gained significant attention due to their inherent advantages of being fast, straightforward, portable, and cost-effective.12 The careful selection of appropriate electrode modification materials is a critical step in the electrochemical detection process.
In the past few years, the field of inorganic nanomaterials has witnessed a remarkable surge in interest, largely attributed to their exceptional physicochemical properties. Particularly, transition metal dichalcogenides (TMDCs) like MoS$_2$, WS$_2$, and V$_2$S$_3$ have gained considerable attention ascribing to their distinct morphology and graphene-like properties. These materials demonstrate excellent chemical, physical, optical, mechanical, magnetic, and electrical characteristics. Layered transition metal dichalcogenide (TMD) crystals are composed of interconnected layers connected by strong in-plane covalent bonds. Meanwhile, the S-M-S interlayer structure, which consists of sulfur molecules (S) and transition metals (M), is held together by comparatively weaker out-of-plane van der Waals forces. The distinctive structural characteristics of TMD crystals contribute to their extensive range of physicochemical properties, notably a significant specific surface area and impressive conductivity. These attributes have facilitated their exceptional performance in various fields, including electrocatalysis, lithium-ion batteries, optoelectronic devices, and energy storage. Consequently, TMDs have become a focal point of intense research and development in the past few decades. Among different TMDs, vanadium disulfide (V$_2$S$_3$) stands as an exemplary material. Recent advancements in first-principles theoretical calculations and experimental research have demonstrated the remarkable properties of two-dimensional layered V$_2$S$_3$, which include excellent conductivity, a high aspect ratio, ultrathin edges, and favorable mechanical characteristics. Consequently, V$_2$S$_3$ fulfills the essential criteria for an effective electrochemical sensor. Karthik et al developed a promising non-enzymatic electrochemical sensor for detecting hydrogen peroxide based on simple sonochemical synthesis of novel grass-like vanadium disulfide. Villan et al employed a facile hydrothermal method to synthesize electrodes comprised of gold nanoparticles decorating V$_2$S$_3$-reduced graphene oxide sheets, achieving a calibration dynamic range of 10-340 nM and detection limit of detection (LOD) lower as 0.44 nM towards sulfadiazine detection. Therefore, the incorporation of two-dimensional V$_2$S$_3$ nanocrystals holds great potential in the manufacturing of electrochemical sensors. However, to propose a promising V$_2$S$_3$-based electrochemical platform, comprehensive studies focusing on the structural regulation and electrochemical optimization of V$_2$S$_3$ are still in deficiency and necessary.

In this work, we utilized a PVP-induced shape control of hydrothermal synthesizing method to fabricate layered structured V$_2$S$_3$, and firstly employed it as an electrode modification material to prepare an electrochemical sensor for the sensitive SMX detection in natural waters. The features and properties of the synthesized material were studied by various characterization methods. Still, various electrochemical techniques were further applied for evaluating the detection performance of the electrochemical sensor. Particularly, the differential pulse voltammetry (DPV) analysis demonstrated that the sensor exhibited high sensitivity and a wide linear range, indicating its substantial potential for environmental water analysis applications.

Experimental

Chemicals

Phosphate buffer solution (10× PBS, containing 1.37 M Na$_2$HPO$_4$, 26.8 mM KCl, 81.0 mM Na$_2$HPO$_4$, and 17.6 mM KH$_2$PO$_4$) was purchased from Sangon Biotech (Shanghai) Co., Ltd. (Shanghai, China). H$_2$SO$_4$, potassium ferricyanide (K$_3$[Fe(CN)$_6$]), potassium hexacyanoferrate (II) (K$_4$[Fe(CN)$_6$]), and Ammonium hydroxide (NH$_3$·H$_2$O) were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Polyvinylpyrrolidone (PVP), ammonium metavanadate (NH$_4$VO$_3$), and thioacetamide (TAA) were purchased from Sigma Aldrich (Shanghai) Trading Co., Ltd. (Shanghai, China). Sulfamethoxazole, trimethoprim (TMP), furazolidone (FRZ), erythromycin (ERY), chloramphenicol (CPL), and glucose (Glu) were purchased from Shanghai Aladdin Bio-Chem Technology Co., Ltd. (Shanghai, China). All chemical reagents used in our experiments were analytical grade without further purification. Milli-Q deionized water was used throughout our tests.

Fabrication of V$_2$S$_3$ Electrodes

The preparation method of V$_2$S$_3$ nanosheet was presented as follows. 1.0 g PVP was dissolved in a mixture comprising 30 mL of deionized water and 2 mL of NH$_3$·H$_2$O. Then 0.234 g NH$_4$VO$_3$ and 1.5 g TAA were added into the mixture sequentially. After undergoing magnetic stirring at room temperature for 1 h, the mixture was displaced into a Teflon-lined stainless-steel autoclave with a volume of 50 mL and heated at 180 °C for 20 h. The product was collected by employing centrifugation and washed repeatedly with anhydrous ethanol. Subsequently, the washed product was vacuum-dried at 60 °C for 24 h. Finally, V$_2$S$_3$ nanosheets were obtained by annealing 2 h in N$_2$ atmosphere at 300 °C at a rate of 2 °C min$^{-1}$. At last, 1 mg mL$^{-1}$ dispersion of the VS$_3$ nanosheets was prepared and preserved for the following experiments.

Before the electrodes modification, glassy carbon electrodes (GCEs) with a 3 mm diameter were first polished using a 0.05 μm alumina slurry. Subsequently, the electrodes were subjected to ultrasonication in deionized water and ethanol to ensure thorough cleaning. Following the thorough cleaning, GCEs were scanned via repetitive potential range scanning from -1 – 1 V in 0.5 M sulfuric acid at a scan rate of 100 mV s$^{-1}$ to activate the electrodes. Finally, 8 μL droplet of VS$_3$ aqueous dispersion was drop coated on GCE and dried at 60 °C for 10 min to obtain VS$_3$ electrodes.

Analytical tests

For the experiment, 1× PBS obtained after 10 times dilution of 10× PBS was used as the electrolyte. The VS$_3$ electrodes were used as working electrodes, while a saturated calomel electrode (SCE) and a Pt electrode were employed as reference electrode and counter electrode, respectively. Cyclic voltammetry (CV), DPV and electrochemical impedence spectroscopy (EIS) tests were performed to analyse the voltammetric responses of various modified electrodes. CV curves were conducted from -0.2 to 0.6 V in five cycles at a scan rate of 0.2 V s$^{-1}$, while DPV tests were performed from 0.7 to 1.1 V with the parameters as: pulse period of 0.5 s, step potential of 0.004 V and amplitude of 0.005 V. EIS was performed in 0.1 to 100 kHz with 10 mV amplitude of AC voltage. All our electrochemical tests were conducted through CHI660e electrochemical workstation (Shanghai Chenhua Co., LTD., China). Zeta potential of V$_2$S$_3$ dispersion and SMX aqueous solution were determined through dynamic light scattering-zeta (Zeta potential, Zetasizer Ultra, UK). HPLC (e2695-
**Characterizations**

Field emission scanning electron microscopy (FE-SEM QUANTA 250 FEG, FEI, Hills-boro, OR, USA), energy dispersive spectroscopy (EDS), and transmission electron microscopy (TEM, JEM-2100FF, JEOL, Japan) were employed to observe morphology of the modified material. Raman spectroscopy (Renishaw in Via Reflex, Renishaw plc, Wotton-under-Edge, London, UK) with a laser wavelength of 532 nm and X-ray diffraction (XRD, D8 Advance, Germany) with Cu Kα radiation (λ: 1.54 Å) were applied for the elemental analysis and crystalline structure of the synthetic material. X-ray photoelectron spectroscopy (XPS, Axis SUPRA+, Shimadzu, Japan) was used to indicate the surface compositions and chemical states.

**Results and Discussion**

**PVP-induced shape control of preparing layer-by-layer stacked VS<sub>2</sub> nanosheets**

The schematic diagram in Fig. 1 depicts the preparation process of layer-by-layer stacked VS<sub>2</sub> nanosheets by PVP-induced shape control method. After thoroughly combining 0.234 g of NH<sub>4</sub>VO<sub>3</sub> with 1.500 g of TAA, the mixture was dissolved in an alkaline solution of adding 0, 1.000, and 3.000 g PVP, respectively. The resulting mixture underwent a hydrothermal process, where it was kept at 180 °C for 20 h. Following that, annealing was carried out at 300 °C for 2 h to synthesize layer-by-layer stacked VS<sub>2</sub> nanosheets, named as VS<sub>2</sub>–A, VS<sub>2</sub>–B and VS<sub>2</sub>–C. The morphology evolution of VS<sub>2</sub> nanosheets induced by PVP were illustrated in Fig. 2a. The morphology of VS<sub>2</sub>–A prepared without PVP presented elongated and hexagon structure, as seen in Fig. 2b. Interestingly, the morphology of VS<sub>2</sub> transformed from hexagon to circular structure and its stacked layers turned to be exfoliated into a structure like an accordion with the adding of PVP, as presented in Fig. 2c. PVP, as an anionic surfactant, possesses hydroxyl groups at the ends of its molecules and can be adsorbed onto VS<sub>2</sub> layers through electrostatic interactions. With the aid of PVP, the stacked layers of VS<sub>2</sub> were inclined to be exfoliated through sonication in the dispersion. By increasing the amount of PVP to 3.000 g, stacked VS<sub>2</sub> nanosheets were further exfoliated into fewer layers, as shown in Fig. 2d. The purity of VS<sub>2</sub> samples prepared were confirmed by TEM element mapping and element composition, as presented in Fig. S1 and Table S1.

To determine the optimal structure of VS<sub>2</sub> induced by PVP, DPV curves of VS<sub>2</sub>–A, VS<sub>2</sub>–B and VS<sub>2</sub>–C electrodes were conducted with 10 μM SMX, as shown in Fig. 2e. The result demonstrated that VS<sub>2</sub>–B electrodes exhibited the best electrochemical response toward SMX, which was chosen as the optimized VS<sub>2</sub> nanosheets for the following experiments. Compared to stacked layers of VS<sub>2</sub>–A, VS<sub>2</sub>–B layers intended to be exfoliated by the induced regulation of PVP. A higher specific surface area of VS<sub>2</sub>–B electrodes enhanced the adsorption capability of SMX, presenting the better electrochemical response. When regulated with the excessive PVP, VS<sub>2</sub>–C electrodes showed dispersed structure with fewer layers, decreasing its adsorption capability towards SMX. Thus, VS<sub>2</sub>–B indicates the priority towards SMX detection.

**Characterization of layer-by-layer stacked VS<sub>2</sub> nanosheets**

SEM images in Fig. 2f and 2g depicted the morphology of VS<sub>2</sub>. VS<sub>2</sub> was composed of vertically stacked nanosheets, exhibiting a diameter and thickness up to 500 to 1000 nm and 20 to 30 nm approximately. Elemental analysis using an energy-dispersive spectrometer (EDS) confirmed the presence and uniform distribution of V and S elements, as shown in Fig. 2h and 2i. TEM and high-resolution TEM (HRTEM) images revealed the crystal structure of VS<sub>2</sub> as presented in Fig. 2j and 2k. TEM images revealed that the VS<sub>2</sub> nanosheets were stacked in the [001] direction, with an interlayer spacing of approximately 5.79 Å. This value closely matches the normal interplanar spacing (5.76 Å) of the original VS<sub>2</sub> (001) plane. Electron diffraction was...
elemental mapping of (h) V and (i) S. (j) TEM image, (k) HRTEM image and (l) the corresponding SAED patterns of VS$_2$ nanosheets.

Fig. 3. (a) Schematic presentation of crystal structure of VS$_2$ nanosheets. (b) XRD patterns, and (c) Raman spectra of VS$_2$ nanosheets. (d) Vibration bands of VS$_2$ nanosheets. High-resolution XPS spectra at (e) V$_{2p}$ and (f) S$_{2p}$ regions of VS$_2$ nanosheets.

employed to demonstrate the polycrystalline nature of the synthesized sample, as indicated in Fig. 2l.

Fig. 3a presents the crystal information file (CIF) for the unit cell and molecule of VS$_2$, referenced from the PDF card number 89-1640. From the observation in Fig. 3a, each layer of vanadium disulfide consists of a vanadium layer sandwiched between two sulfur layers, and the sandwiched structures connected by van der Waals forces.

From the XRD patterns (Fig. 3b), all the observed diffraction peaks in the graph can be assigned to VS$_2$ (JCPDS#89-1640), with lattice constants of $a = b = 3.22$ Å and $c = 5.76$ Å. These findings provide further confirmation of the successful formation of VS$_2$ nanosheets with regular interlayer spacing. The chemical structure of the synthesized VS$_2$ was investigated using Raman spectroscopy, as shown in Fig. 3c. It displayed characteristic vibration bands at nearly 281 and 405 cm$^{-1}$, corresponding to $E_{1g}$ and $A_{1g}$ modes, respectively. These modes represent the in-plane vibration ($E_{1g}$) and out-of-plane vibration ($A_{1g}$) of the S-V-S bonds, as depicted in Fig. 3d. In fact, the Raman signals of VS$_2$ demonstrate an analogy to other transition metal sulfides within the vanadium group.

In Fig. 3e, the high-resolution XPS spectrum of V 2p unveiled two prominent peaks centered at approximately 525.0 and 517.5 eV. These peaks are assigned to V 2p$_{3/2}$ and V 2p$_{1/2}$, respectively, affirming the existence of the V$^{4+}$ oxidation state. Additionally, the XPS spectrum also exhibits two smaller peaks located at approximately 522.2 and 514.2 eV. These minor peaks suggested the presence of a small quantity of V$^{3+}$ ions, which can be attributed to the reducing properties of organic amines and the strong reducing nature of hydrogen sulfide generated from the decomposition of thioacetamide in the preparation. The XPS spectrum of S 2p in Fig. 3f showed distinct peaks at around 162.7 and 161.5 eV, corresponding to S 2p$^{3/2}$ and S 2p$^{1/2}$ of S$^2-$ species. Peaks located at around 163.7 and 164.5 eV are attributed to S$^2-$ species, while the peak at around 169.6 eV can be assigned to sulfate species. The result indicated that slight oxidation and contamination with elemental sulfur and sulfates had occurred in the VS$_2$ sample due to its exposure to air.

Performance optimization of VS$_2$/GCE electrodes toward SMX detection

To investigate the electrochemical performance of VS$_2$ towards SMX, the electron transferring ability on VS$_2$ electrodes need to be understood. The oxidation peak of potential at 0.90 V was designated as the characteristic peak for SMX electrochemical analysis. The oxidation peak of potential at 0.90 V was designated as the characteristic peak for SMX electrochemical analysis. The oxidation peak of potential at 0.90 V was designated as the characteristic peak for SMX electrochemical analysis. The oxidation peak of potential at 0.90 V was designated as the characteristic peak for SMX electrochemical analysis. The oxidation peak of potential at 0.90 V was designated as the characteristic peak for SMX electrochemical analysis. The oxidation peak of potential at 0.90 V was designated as the characteristic peak for SMX electrochemical analysis. The oxidation peak of potential at 0.90 V was designated as the characteristic peak for SMX electrochemical analysis. The oxidation peak of potential at 0.90 V was designated as the characteristic peak for SMX electrochemical analysis.
VS₂ electrodes in 10 mM [Fe(CN)₆]³⁻/⁴⁻ (Fig. 4a). Through the model simulation according to its Nyquist curves, the equivalent circuit model of VS₂ electrodes is equivalent to R₁(Qₓ(Rₓ(R₁-Zₓ)))). Particularly, R₁ denotes the interfacial electron transfer resistance and determines the electron transferring ability on various electrodes. 38 Rₓ values of VS₂ electrodes and GCE were 567.2 and 5079.0 Ω, respectively. Rₓ value of GCE was higher than VS₂ electrodes, demonstrating that VS₂ enhances the charge transferring ability of electrodes. The fitted values of various parameters in the model are presented in Table S2.

To further enhance the detection performance of VS₂ electrodes, analytical parameters such as the modified mass of VS₂ on electrode, scan rates, and electrolyte pH were treated in optimization. To determine the appropriate mass of VS₂ during electrode preparation, DPV curves of VS₂ electrodes modified with various mass of VS₂ as 2, 4, 6, 8, 10 and 12 μg were carried out in PBS containing 10 μM SMX. As shown in Fig. 4b, 8 μg of VS₂ was selected for further experiments. To evaluate the electrochemical behaviors on VS₂ electrodes surface, CV responses were obtained in 10 mM [Fe(CN)₆]³⁻/⁴⁻ at scan rates ranging from 20–200 mVs⁻¹ (Fig. 4c). The peak current density of Iₓ and Iₓ of VS₂ presented a linear increasement with the square root of scan rate v¹/₂ (Fig. 4d), indicating that the redox reaction on VS₂ electrodes were diffusion-controlled. The oxidation mechanism of SMX is proceeded via pH-dependent reaction. The maximum peak current was achieved at pH = 6 and was selected as the optimal pH. According to the result in Fig. 4f, the linear relationship of potential Epc versus pH is fitted as Epc (V) = 1.088 – 0.032 pH (R²=0.990). The obtained slope value is smaller than Nernstian value (59 mV pH⁻¹), demonstrating the same numbers of protons and electrons involved in the redox reaction.

**Table 1.** Comparison of detection performance on various electrodes toward SMX.

<table>
<thead>
<tr>
<th>Electrodes material</th>
<th>Techniques</th>
<th>Linear range (μM)</th>
<th>LOD (μM)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>VS₂</td>
<td>DPV</td>
<td>0.06–10.0</td>
<td>10.0</td>
<td></td>
</tr>
<tr>
<td>Graphene</td>
<td>DPV</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CNTs</td>
<td>DPV</td>
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**Electrochemical determination of SMX by VS₂ electrode and its sensing mechanism**

The DPV method was employed for the quantitative electrochemical detection of SMX on the VS₂ electrodes, as depicted in Fig. 5a and Fig. S2. The peak current values on VS₂ electrodes exhibited an upward trend as the SMX concentration increased. A calibration curve for SMX on the VS₂ electrodes was acquired within the linear range of 0.06 to 10 μM, as illustrated in Fig. 5b. The calibration equation is Ipc = kpcC₂ + bpc.
dimethylformamide | Graphene | CNTs/N,N-dimethylformamide | CNTs/Ti-3 | CNTs/Prussian blue nanocubes
| DPV | DPV | Amperometry | DPV | DPV |
| 50–10000 | 1.99–10.9 | 0.50–110 | 0.2–100 | 1.0–10.0 |
| 10 | 0.413 | 0.094 | 0.060 | 0.038 |

The sensing mechanism model of VS₂ electrodes is proposed to better illustrate the reason why VS₂ electrodes can be a good candidate of electrochemical platform towards sensitive detection of SMX, as presented in Fig. 5c. Voltammetric technique is applied for the determination of SMX on VS₂ electrodes in river water samples. During the electrochemical oxidation of SMX on VS₂ electrode interface, two electrons and protons are involved in the reaction process. Herein, on behave of carbon-based nanomaterial with good electrical conductivity, CNTs and rGO were used to compare their charge transferring ability with VS₂. DPV curves of VS₂, graphene and CNTs electrodes with 10 μM SMX were conducted in Fig. 5d, demonstrating the superior catalytic response of VS₂ toward SMX. Based on the fitting of Nyquist curves of CNTs, rGO and VS₂ electrodes in Fig. 5e, VS₂ electrodes presented the lowest Rct values. The result demonstrated that the charge transferring ability of VS₂ prior to CNTs and rGO at electrode interface, validating the privilege of VS₂ as a promising base material modifying electrode for sensitive SMX detection. The result is also in consistent with Table 1. Furthermore, CNTs or rGO based nanocomposite modified electrodes present good detection performance towards SMX in Table 1, supporting the great potential of VS₂ based nanocomposite modified electrodes for the sensitive detection of SMX in the future.

### Repeatability, anti-interference, and real sample analysis

To evaluate the reproducibility of VS₂ electrodes for SMX detection (10 μM), DPV tests were conducted on 10 different electrodes in the potential range of 0.7–1.1 V. The DPV curves remained stable at a potential of 0.90 V approximately and maintained good overlapping of current density, as shown in Fig. 6a. The relative standard deviation (RSD) of the peak current was approximately 2.6%, demonstrating good repeatability of VS₂ electrodes. Interference experiments were performed by DPV curves (Fig. 6b), where 10 μM TMP was used as a typical interfering substance within the concentration range of 0.1–10 μM. Notably, to achieve continuous synergistic effect of antibacterial activity, the combination of TMP and SMX is usually utilized in the clinical treatment. The interference of TMP to SMX detection in real samples is necessary.

### Fig. 6.

(a) The reproducibility and (b, c) anti-interference of VS₂ electrodes. (d) HPLC tests for SMX determination in tap samples. (e) DPV responses of VS₂ electrodes. (f) Results comparison within HPLC and DPV tests.

The result demonstrated that TMP did not affect SMX detection, supporting the potential practical applicability of VS₂ electrodes. Besides, i-t technique was performed on VS₂ electrodes in electrolyte containing 10 μM SMX and other interfering substances such as 50 μM CCL₃, 50 μM FRZ, 50 μM ERY, 50 μM Glu, 100 μM Na⁺, 100 μM K⁺, 100 μM Mg²⁺, and 100 μM Ca²⁺, as shown in Fig. 6c and Fig. 5d. The results revealed the exceptional resistance of VS₂ electrodes to interferences from other molecules during electrochemical detection.

To validate the detection accuracy of VS₂ electrodes by DPV method, HPLC was employed to detect SMX in the same real samples. Relative error (RE) is used as the evaluation index and functioned as the equation $RE=|a-b|/b \times 100\%$, wherein a and b present the average value of DPV and HPLC, respectively (μM). Real water samples were prepared by adding certain mass of SMX in tap water and designated as sample A, B, C, and D. HPLC results were obtained by the computation of the calibration equation, which was determined as $A_r = 8.58 \times 10^3 C – 206$ ($R^2=0.999$). Herein, C is the concentration of SMX (μM), and $A_r$ denotes the peak area at a
retention time of 11.3 minutes (μV·s), as presented in Fig. 6d. DPV curves of VS₂ electrodes were performed in the same real samples, as shown in Fig. 6e. Based on data comparison of the two methods in Fig. 6f, RE for samples A, B, C and D are determined as 2.89%, 3.15%, 3.36%, and 2.62%, respectively, validating the good accuracy of our electrodes. To further present the practical applicability of VS₂ electrodes, recovery performance in river water samples were conducted by using the standard addition method. As shown in Table S3, the prepared SMX sensor exhibited good recovery rates (97.4-106.8%) and low RSD values (1.16–1.79%), demonstrating its significant capability for real sample analysis.

Conclusions
This study constructed a sensitive SMX sensor based on layered structured VS₂ nanosheets prepared by a PVP-induced shape control method of hydrothermal synthesizing method. Compared to the bare GCE, the VS₂ electrode exhibited higher sensitivity towards SMX oxidation, demonstrating excellent SMX detection performance within a detection range of 0.06 to 10 μM and LOD lower as 0.047 μM. The interference resistance of the VS₂ electrode was validated using the VS₂ electrode. Furthermore, the interference resistance of the VS₂/GCE electrode was examined by adding other potential interfering substances. In conclusion, this research provides a promising approach for the development of electrochemical sensors based on VS₂ composite materials.

Author Contributions
Mingjiao Shi: Data curation, Methodology, Formal analysis, and Writing—original draft. Peizheng Shi: Software, Investigation, Data curation. Xinxin Yang: Methodology, Data curation, Supervision. Ningbin Zhao: Investigation, Data curation. Mengfan Wu: Software, Data curation. Jing Li: Investigation, Data curation. Chen Ye: Data curation, Methodology. He Li: Methodology, Supervision, Data curation. Nan Jiang: Methodology, Supervision, Writing—review and editing. Xiufen Li: Methodology, Data curation, Writing—review and editing. Guosong Lai: Resources, Validation, and Software. Wan-Feng Xie: Data curation, Supervision, Writing—review and editing. Li Fu: Methodology, Supervision, Data curation, Visualization, Methodology, Supervision. Yangguang Zhu: Methodology, Data curation, Writing—review and editing. Hsu-Sheng Tsai: Visualization, Supervision, Writing—review and editing. Cheng-Te Lin: Conceptualization, Writing—review and editing. Funding acquisition.

Conflicts of interest
There are no conflicts to declare.

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