Polypyrrole Wrapped V$_2$O$_5$ Nanowires Composite for Advanced Aqueous Zinc-Ion Batteries

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Aqueous zinc-ion batteries (ZIBs) have obtained increasing attention owing to the high safety, material abundance, and environmental benignity. However, the development of cathode materials with high capacity and stable cyclability is still a challenge. Herein, the polypyrrole (PPy)-wrapped V$_2$O$_5$ nanowire (V$_2$O$_5$/PPy) composite was synthesized by a surface-initiated polymerization strategy, ascribing to the redox reaction between V$_2$O$_5$ and pyrrole. The introduction of PPy on the surface of V$_2$O$_5$ nanowires not only enhanced the electronic conductivity of the active materials but also reduced the V$_2$O$_5$ dissolution. As a result, the V$_2$O$_5$/PPy composite cathode exhibits a high specific capacity of 466 mAh g$^{-1}$ at 0.1 A g$^{-1}$ and a superior cycling stability with 95% capacity retention after 1000 cycles at a high current density of 5 A g$^{-1}$. The superior electrochemical performance is ascribed to the large ratio of capacitive contribution (92% at 1 mV s$^{-1}$) and a fast Zn$^{2+}$ diffusion rate. This work presents a simple method for fabricating V$_2$O$_5$/PPy composite toward advanced ZIBs.

Keywords: V$_2$O$_5$ nanowires, surface-initiated polymerization, polypyrrole, cathode material, aqueous zinc-ion battery

INTRODUCTION

The ever-increasing energy consumption, and limited fossil fuels, necessitates effective utilization of renewable energy resources (Xu F. et al., 2020). For that purpose, large-scale efficient energy storage systems are desired (Shao et al., 2021). Although lithium-ion battery has found widespread applicability, it suffers from safety issues caused by flammable organic electrolytes as well as the availability of Li source (Dong et al., 2020; Lu et al., 2020). Aqueous zinc-ion batteries (ZIBs) are regarded as a suitable alternative for scalable energy storage systems, due to the usage of zinc metal anode which, apart from high abundance and environmental friendliness, has a large theoretical capacity (820 mAh g$^{-1}$) and a low redox potential [−0.76 V vs. SHE (Wang F. et al., 2018; Wang et al., 2020b; Zhang et al., 2019a). Furthermore, the possibility of an aqueous electrolyte endows an intrinsic non-flammability and high ionic conductivity (Wang et al., 2020a). However, corresponding ZIB cathode materials with high capacity and stable cyclability need to be further explored (Zhang et al., 2019b).

Manganese oxides (Khamsanga et al., 2019; Wang J. et al., 2019), Prussian blue analogs (Liu et al., 2020; Zampardi and La Mantia, 2020), vanadium-based compounds (Yang et al., 2020), and some organic materials (Wang et al., 2020c) have been investigated as cathode materials for aqueous...
Among those, vanadium-based materials, particularly vanadium oxides, are very attractive because of the advantage of high theoretical capacities due to multiple oxidation states of vanadium. Unfortunately, the electrochemical performance of vanadium oxides in ZIBs is hindered by their poor electronic conductivity and noticeable solubility in the electrolyte (Zhang et al., 2020). To address these issues, various strategies have been applied, such as using pre-insertion materials (V₂O₅·H₂O) (Wang X. et al., 2019), integration with carbon materials (Yan et al., 2018), as well as electrolyte modifications (Wan et al., 2018). Another viable approach is to incorporate conducting polymers along with V₂O₅ (Du et al., 2020). Polypyrrole (PPy) is a widely used conductive polymer, and V₂O₅/PPy composites have been shown to exhibit enhanced performance in supercapacitors and LIBs (Wang J.G. et al., 2018). Therefore, with regard to aqueous ZIBs, an effective PPy coating can aid in enhancing the electronic conductivity of V₂O₅ as well as help to reduce the solubility in the electrolyte.

Herein, V₂O₅ nanowires were synthesized by a facile hydrothermal method, and a surface-initiated polymerization method was utilized to fabricate a PPy-wrapped V₂O₅ nanowire composite. V₂O₅ served as the initiator to induce the polymerization reaction of pyrrole monomer at room temperature due to the strong oxidizing property of V⁵⁺. Benefiting from the improved electronic conductivity and restricted V₂O₅ dissolution due to the PPy layer, V₂O₅/PPy cathode delivered a higher specific capacity and rate performance in comparison to the pristine V₂O₅ nanowire cathode. Therefore, the V₂O₅/PPy composite is a promising high-performance cathode material for aqueous ZIBs toward large-scale energy storage applications.

**EXPERIMENTAL SECTION**

V₂O₅ nanowires were synthesized by a facile hydrothermal method according to previously reported literature (Wang J.G. et al., 2018). 200 mg of obtained V₂O₅ nanowires was dispersed into deionized water. Then, pyrrole (0.1 ml) dissolved in DMF (4 ml) was slowly added to the above V₂O₅ nanowire suspended solution and stirred for 24 h. The obtained V₂O₅/PPy was washed carefully and dried in a vacuum oven.

More detailed synthesis and characterization processes are available in electronic Supplementary Information.

**RESULTS AND DISCUSSION**

V₂O₅ nanowires were synthesized by the hydrothermal method. The as-obtained V₂O₅ nanowires show a diameter of approximately 15 nm with a cable-like nanostructure (Supplementary Figure S1A). The V₂O₅/PPy composites were prepared using a surface-initiated polymerization strategy, as shown in Figure 1A. Owing to the strong oxidizing property of V₂O₅, the pyrrole monomer can be polymerized with V₂O₅ initiation, resulting in the surface coating of V₂O₅ with PPy. The morphology of V₂O₅ nanowires was well-maintained after PPy coating, indicating that the wrapping procedure has no significant influence on the V₂O₅ morphology (Figure 1B). The TEM image also confirms the nanowire morphology of the V₂O₅/PPy composite (Figure 1D). EDS elemental mappings show the homogeneous distribution of C, O, V, and N throughout the entire V₂O₅/PPy composite, indicating the presence of PPy (Figure 1C and Supplementary Figure S1B).

The XRD data of the V₂O₅ nanowires mainly fit with the layered orthorhombic structure (JCPDS no. 40-1296), and typical (001) and (003) reflection peaks are present (Figure 1E). A little amount of V₄O₇ was also indexed and may be assigned to the reduction of P123. The interlayer distance is estimated to be 0.96 nm by Bragg’s law from the (001) peak. This large distance is beneficial for Zn⁡²⁺ insertion/extraction during the electrochemical process. After the PPy coating, no significant change is observed in the XRD data, indicating that the layered structure was well maintained after the polymerization process. In order to confirm the PPy coating and identify the valence state of vanadium in the V₂O₅/PPy composite, XPS was carried out. Figure 1F shows the survey spectrum with the clear presence of N 1s and C 1s, confirming the polymeric coating (see also Supplementary Figure S2). Figure 1G shows the V 2p spectrum, with strong V 2p3/2 and V 2p1/2 peaks of V⁵⁺ located at 517.6 eV and 525 eV, along with shoulder peaks at 516 eV and 523.8 eV, corresponding to V⁴⁺ (Liu et al., 2019). The presence of a small amount of V⁴⁺ (9.3 at.%) corresponds to the oxygen vacancies generated in the V₂O₅ surface due to the redox reaction between V₂O₅ and pyrrole. Previous studies on V₂O₅ demonstrated that such vacancies enhance the electrochemical performance (Liao et al., 2020).

The electrochemical performance of pristine V₂O₅ and V₂O₅/PPy composites is evaluated in aqueous ZIBs. Figure 2A presents the rate capability of the pristine V₂O₅ cathode and V₂O₅/PPy composite cathode. The V₂O₅/PPy composite cathode delivers a high initial capacity of 466 mAh g⁻¹ at 0.1 A g⁻¹, as compared to the V₂O₅ nanowire electrodes (425 mAh g⁻¹). Even at a very high current density of 5.0 A g⁻¹, the V₂O₅/PPy composite cathode still possesses a higher discharge capacity of 174 mAh g⁻¹ than that observed for the V₂O₅ nanowire cathode (142 mAh g⁻¹). The results point to the better rate performance of V₂O₅/PPy composite in comparison to non-modified V₂O₅ nanowire electrodes. The voltage-capacity plots for the V₂O₅/PPy composite at different current rates demonstrate that the redox plateaus are well maintained even at a high current density of 5.0 A g⁻¹ (Figure 2B). In comparing to voltage-capacity plots for pristine V₂O₅, the overpotentials are slightly lower suggesting improved kinetics due to the higher electrical conductivity of the composite (Supplementary Figure S3).

Based on the voltage profiles, the energy/power densities of the batteries were calculated and are shown in the Ragone plot (Figure 2C). Impressively, the batteries base on the V₂O₅/PPy composite cathode display a high energy density of 235 Wh kg⁻¹ at a power density of 56 W kg⁻¹ and exhibit a relatively high energy density of 100 Wh kg⁻¹ even at a high power density of 2335 W kg⁻¹. Moreover, the V₂O₅/PPy composite cathodes are highly competitive among the aqueous ZIBs based on the different cathodes: V₂O₅ (Hu et al., 2017),
FIGURE 1 | (A) Schematic illustrating the preparation of V$_2$O$_5$/PPy composite. Characterizations of the V$_2$O$_5$/PPy composite. (B) SEM image and (C) Corresponding elemental mappings, (D) TEM image, (E) XRD patterns, (F) XPS survey spectrum, and (G) V 2p spectrum with fitting showing mixed valence of V after PPy coating.

FIGURE 2 | Electrochemical performance of V$_2$O$_5$/PPy composites cathode in aqueous ZIBs. (A) Rate performance in comparison to pristine V$_2$O$_5$. (B) Voltage profile plots at different current rates. (C) Ragone plot. (D) Cycling performance at 1A g$^{-1}$ and (E) Long-term cycling performance at 5A g$^{-1}$, in comparison to pristine V$_2$O$_5$. 
TABLE 1 | Comparison of the initial capacity and cycling stability of \( \text{V}_2\text{O}_5/\text{PPy} \) composite with recent literature data on vanadium oxide-based cathodes in aqueous ZIBs.

| Electrodes       | Rate (mA g\(^{-1}\)) | Initial capacity (mAh g\(^{-1}\)) | Capacity Retention | References                  |
|------------------|------------------------|-----------------------------------|--------------------|-----------------------------|
| \( \text{V}_2\text{O}_5/\text{PPy} \) | 1000                   | 329                               | 94% (100 cycles)   | This work                   |
|                  | 5000                   | 174                               | 96% (1000 cycles)  |                             |
| \( \text{K}_0.25\text{V}_2\text{O}_5 \) | 1000                   | 205                               | 83% (50 cycles)    | Li S. et al., 2019          |
| \( \text{Na}_1.25\text{V}_2\text{O}_5 \) | 1000                   | 280                               | 75% (50 cycles)    | Xie et al., 2020            |
| \( \alpha\text{-Zn}_2\text{V}_2\text{O}_7 \) | 4000                   | 163                               | 85% (1000 cycles)  | Sambandam et al., 2016      |
| \( \text{V}_2\text{O}_5\cdot\text{nH}_2\text{O} \) | 6000                   | 281.7                             | 71% (600 cycles)   | Yan et al., 2018            |
| \( \text{H}_2\text{V}_3\text{O}_8 \) | 5000                   | 173.6                             | 94.3% (1000 cycles)| He et al., 2017a            |
| \( \text{Li}_4\text{V}_2\text{O}_5\cdot\text{nH}_2\text{O} \) | 5000                   | 252                               | 92.1% (500 cycles) | Yang Y. et al., 2018        |
| \( (\text{NH}_4)_2\text{V}_6\text{O}_{16} \) | 1000                   | 361.6                             | 76.1% (100 cycles) | Xu L. et al., 2020          |

NH\(_4\)V\(_4\)O\(_10\) (Yang G. et al., 2018), \( \text{Na}_3\text{V}_2(\text{PO}_4)_3 \) (Li et al., 2016), heterogeneous vanadium oxide nanowire with \( \text{V}_2\text{O}_5\cdot\text{nH}_2\text{O} \) shell and \( \text{V}_3\text{O}_7\cdot\text{H}_2\text{O} \) core (h-VOW) (Li X. et al., 2019), VS\(_2\) (He et al., 2017b), \( \text{Zn}_3\text{V}_2(\text{OH})_2 \) (Chao et al., 2018), and \( \text{NaV}_6\text{O}_{15}/\text{V}_2\text{O}_5 \) (Lanlan et al., 2020). In addition, as shown in Figure 2D, the \( \text{V}_2\text{O}_5/\text{PPy} \) composite cathode exhibits a high initial capacity of 329 mAh g\(^{-1}\) at 1 A g\(^{-1}\) and a capacity retention of 94% after 100 cycles, which is much higher than that of pristine \( \text{V}_2\text{O}_5 \) cathodes (234 mAh g\(^{-1}\), 82%). Furthermore, long-term cycling performance of the cathodes was evaluated, because it is a key feature for practical applications. Even after 1000 cycles, the batteries based on the \( \text{V}_2\text{O}_5/\text{PPy} \) composite cathode show a reversible capacity of 174 mAh g\(^{-1}\) with a capacity retention of 95%. In contract, pristine \( \text{V}_2\text{O}_5 \) cathodes exhibit a poor cycling stability, with a specific capacity of only 93 mAh g\(^{-1}\) after 1000 cycles corresponding to a capacity retention of 62% (Figure 2E). The strong capacity fading observed for pristine \( \text{V}_2\text{O}_5 \) cathode is most probably be a result of \( \text{V}_2\text{O}_5 \) dissolution during cycling, which is minimized with the PPy coating for the \( \text{V}_2\text{O}_5/\text{PPy} \) composite. Moreover, such a high cycling stability for the \( \text{V}_2\text{O}_5/\text{PPy} \) composite is better compared to the recently reported literature on aqueous ZIBs with vanadium oxide-based cathodes (Table 1). The high rate performance and stable long cycle life of the \( \text{V}_2\text{O}_5/\text{PPy} \) composite cathode are ascribed to the introduction of a conductive polymer PPy layer, which not only increases the electronic conductivity but also reduces the dissolution of \( \text{V}_2\text{O}_5 \) in the electrolyte.

The electrochemical kinetics of the \( \text{V}_2\text{O}_5/\text{PPy} \) composite cathode was further investigated to understand the impressive performance. Cyclic voltammetry (CV) was performed at various scan rates from 0.1 to 1.0 mV s\(^{-1}\) (Figure 3A). The CV curves show similar redox peaks in the voltage window of 0.3–1.6 V. The characteristic peaks appeared at 0.5/0.7 V as well as 0.8/1.0 V,
reflecting the redox processes in V2O5 that is consistent with reported literature (Yang Y. et al., 2018; Zhang et al., 2018). The capacity is regarded to be originated from two contributed parts: a surface-controlled capacitive part and a diffusion-induced part, as described in the literature (Ming et al., 2018):

\[ i = a v^b \] (1)

In this equation, \( v \) is the scan rate, and \( a \) and \( b \) refer to adjustable parameters. The \( b \) values range from 0.5 to 1.0. Corresponding to \( b = 0.5 \), the observed capacity is fully diffusion-induced. When the capacity is completely determined by a surface-controlled capacitive part, the \( b \) value is close to 1.0. The peak currents at different scan rates are plotted and fitted with a linear function (Figure 3B). The \( b \) values are 0.51, 0.75, 0.61, and 0.64, which implies that the capacity of the V2O5/PPy composite cathode is simultaneously influenced by both the capacitive and diffusion processes. Furthermore, the capacity is divided as a capacitive-controlled part (\( k_1v \)) and diffusion-induced part (\( k_2v^{1/2} \)) as described by the following equations:

\[ i = k_1v + k_2v^{1/2} \] (2)

or

\[ i/v^{1/2} = k_1v^{1/2} + k_2 \] (3)

The ratios of surface-controlled capacitive and diffusion-induced parts with various scan rates are displayed in Figure 3C. The surface-controlled capacitive contribution ratio increases from 57% (0.1 mV s\(^{-1}\)) to 92% (1.0 mV s\(^{-1}\)), indicating that the batteries possess fast charge-transfer kinetics. The kinetics of the V2O5/PPy composite cathode is further evaluated by galvanostatic intermittent titration technique (GITT). The profiles in GITT curves of V2O5/PPy electrode are well in coincidence with the galvanostatic charge–discharge profiles (Figure 3D). The zinc-ion diffusion coefficient during discharging–charging procedures for V2O5/PPy is \( 3.03 \times 10^{-9} – 1.46 \times 10^{-10} \) cm\(^2\) s\(^{-1}\) (Figure 3E), which is comparable to that of the reported aqueous ZIBs based on the V2O5@CNT composite and porous V2O5 nanofiber cathodes (Chen et al., 2019, 2020).

CONCLUSION

In this work, a surface-initiated polymerization strategy was utilized to synthesize PPy-wrapped V2O5 nanowires. Owing to the strong oxidizing property of V\(^{5+}\), the polymerization of the pyrrole monomer could be initiated at room temperature. Due to the introduction of the conductive PPy layer, the V2O5/PPy cathode displayed a superior specific capacity and excellent cycling stability. The outstanding electrochemical properties are explained by the large ratio of a capacitive-controlled process (92% at 1 mV s\(^{-1}\)) and a fast zinc ion diffusion coefficient. Considering the excellent electrochemical performance, coupled with the safe and simple operation process of aqueous ZIBs, the V2O5/PPy composite cathode holds great promise for practical grid-level storage applications.

DATA AVAILABILITY STATEMENT

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

AUTHOR CONTRIBUTIONS

All authors listed have made a substantial, direct and intellectual contribution to the work, and approved it for publication.

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SUPPLEMENTARY MATERIAL

The Supplementary Material for this article can be found online at: https://www.frontiersin.org/articles/10.3389/fenrg.2020.00199/full#supplementary-material

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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