



Hydrothermal reaction induced phase transition of vanadium oxide towards high-performance zinc ion batteries cathode

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Abstract

Aqueous zinc ion batteries (ZIBs) are widely researched due to the low-cost and intrinsic safety. However, the rate capability and specific capacity of ZIBs is limited due to the dissolution and structural collapse of cathode materials. It is crucial to construct stable cathode materials to promote rate capability and cycle stability of ZIBs. In this paper, The V_3O_7 was tightly attached to the surface of graphene oxide (GO) by a hydrothermal reaction and a V_3O_7 /GO heterostructure was successfully achieved. The GO could increase structural stability, enhance electrical conductivity, and expand specific surface area of V_3O_7 . Therefore, the V_3O_7 /GO with a heterostructure exhibits an improved cycle stability and rate capability compared with V_3O_7 . It displayed a specific capacity of 275.6 mA h g^{-1} at a current density of 1.0 A g^{-1} . The study paves the way for promoting the Zn^{2+} storage performance of vanadium oxide and developing stable cathode materials of ZIBs.

Keywords Zinc ion batteries · Heterostructure · Vanadium oxide · Zinc ions storage

Introduction

Aqueous zinc ion batteries (ZIBs) are becoming an ideal candidate in new energy storage equipment areas due to the excellent safety, non-toxic electrolyte, and environment friendly [1–3]. However, the development of ZIBs has been hindered because of lack of suitable cathode materials that extend cycle life and high-rate capability [4–6]. Despite manganese-based oxides [7, 8], Quinone analogs [9] and Prussian blue analogs [10], as well as sustainable Chevrel phase compounds [11, 12], have been widely researched in the field of ZIBs. The development of stable cathode materials remains a huge challenge due to the low specific capacity and poor cycle stability.

Among the reported cathode materials, vanadium-based oxide possesses outstanding framework structures that have

been widely studied due to various redox reactions and the layered structure provides space for the insertion of Zn^{2+} [13–20]. However, most of them are still plagued by the dissolution of vanadium oxide and the formed of electrochemically byproduct during cycling, leading to capacity fading and limited cycle stability. Several methods have been mentioned to increase the capacity and improve cycle stability of vanadium oxide. Wang et al. [21] confirmed that barium ions inserted into the interlayer of vanadium oxide can promote the electrochemical performance of $Ba_{1.2}V_6O_{16} \cdot 3H_2O$ by enhancing the stability of vanadium oxide and accelerating the diffusion rate of Zn^{2+} . Liang et al. [22] presented the mixed vanadium valence states (V^{4+}/V^{5+}) of V_6O_{13} with open framework structure, which exhibits an excellent rate capability and outstanding specific capacity of 206 mA h g^{-1} at a current density of 10 A g^{-1} . Tamilselvan et al. [23] reported that the $(NH_4)_{0.37}V_2O_5 \cdot 0.15(H_2O)$ possesses highly crystalline, which displays the initial specific capacity of 400 mA h g^{-1} at the current density of 0.5 A g^{-1} . Besides, V_2O_5 with pre-embedded water molecules and/or metal/non-metal ions [24, 25], vanadate complex [26, 27], and other vanadium oxide with layered/tunneled structures [28–31] can also be employed as cathode materials of ZIBs. These reports have demonstrated the excellent Zn^{2+} storage performance by employing the pre-embedded metal/nonmetal

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ions or change the structure of vanadium oxide. However, volume expansion of vanadium oxide during Zn^{2+} insertion/extraction and low conductivity continues to impede the development of ZIBs. How to effectively alleviate the volume expansion and improve the conductivity possesses important significance to the performance improvement of the cathode materials.

In this study, the GO-supported $\text{V}_3\text{O}_7/\text{GO}$ heterostructures with mixed valence states are prepared by a hydrothermal method. The mixed valence states can provide more possibilities for redox reaction, which is beneficial to the reaction. The V_3O_7 nanosheets were attached on the surface of GO, which increase specific surface area of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures. The stability was improved due to the hydrogen bonds between V_3O_7 and the functional groups on the surface of GO, which increases the specific capacity, cycle stability, and rate capability. The $\text{V}_3\text{O}_7/\text{GO}$ heterostructures show an outstanding cycle stability, rate capability, and impart fast ion storage kinetics to electrode material due to rapid ion/electron diffusion and stable structure. The $\text{Zn}/\text{V}_3\text{O}_7/\text{GO}$ heterostructures battery possesses an excellent practicability and high safety in application devices. The results can provide a guidance for the design of high cycle stability zinc ion batteries.

Experimental section

Preparation of sample

A total of 20 mL 1.5 mM citric acid solution was slowly added into 30 mL 2 mM NH_4VO_3 solution at 65 °C and stirring for 2 h. Twenty milliliters of graphene oxide dispersion (1 mg/mL) was added to the solution and stirring for 3 h. Then the obtained solution was poured autoclave lined and heated at 200 °C for 24 h to conduct a hydrothermal reaction. Finally, $\text{V}_3\text{O}_7/\text{GO}$ heterostructures were obtained after washing the product with deionized water and acetone five times and dried in oven at 65 °C for 10 h. For comparison, V_2O_5 nanosheets were synthesized without adding GO.

Characterization

The phase of the samples is analyzed using a D/max-2110 PC X-ray diffraction (XRD, Japan). The elemental composition was confirmed through X-ray photoelectron spectroscopy (XPS, PHI 5700). The microstructures were examined employing a JSM-IT100 field-emission scanning electron microscope (SEM; JEOL, Japan) and a JEM-2100 Plus transmission electron microscope (TEM; JEOL, Japan). The Brunauere-Emmette-Teller (BET) surface area was tested employing the specific surface area analyzer (JW-BK200, JWGB SCI. & TECH).

Electrochemical measurements

The electrochemical performance of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures was research as cathode, zinc foil as anode with 3 M ZnSO_4 as electrolyte by assembled CR-2032 type coin batteries. The cathodes on stainless steel foil were synthesized by smearing slurry, containing 70 wt% of active material, 10 wt% of polyvinylidene fluoride (PVDF), and 20 wt% acetylene black together with the appropriate quantity of N-methyl pyrrolidinone (NMP) dried at 60 °C for 12 h. Then the electrode was placed in a vacuum oven and heated at 80 °C for 12 h. Finally, the electrodes were cut into a disk with 7 mm radius after 5 min at 12 MPa. The mass loading amount of active material for $\text{V}_3\text{O}_7/\text{GO}$ heterostructures electrodes in the electrochemical characterization is 0.7 mg cm^{-2} . The galvanostatic charge/discharge (GCD) was tested by the land battery testing system with 0.2–1.6 V voltage. The cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) were studied by the CHI660E electrochemical workstation.

Results and discussion

The preparation procedures of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures are showed in Fig. 1a, and the uniform $\text{V}_3\text{O}_7/\text{GO}$ heterostructures were produced with hydrothermal reaction. The X-ray diffraction (XRD) pattern of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures is shown in Fig. 1b, and the peaks located at 24.9° and 49.5° correspond to the (−111) and (020) plane of V_3O_7 (JCPDS No.27–0940), while the sharp peak at 8.5° indicates the (001) plane of GO. Differently, vanadium oxide without adding GO corresponds to the standard card of V_2O_5 (JCPDS No.72–0433) in Figure S1. The TG curve of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures is displayed in Figure S2. Through calculation, the content of GO is 9.3271%. There is no obvious water content missing, which proves that the cathode material is $\text{V}_3\text{O}_7/\text{GO}$. The valence of vanadium was changed due to the enhanced reducibility of citrate acid after the addition of GO, which resulted in conversion of V^{5+} to V^{4+} . The valence state and element composition were researched by the X-ray photoelectron spectroscopy (XPS) measurement and the XPS survey spectrum of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures displays characteristic peaks of C 1s, O 1s, and V 2p, which explains that the $\text{V}_3\text{O}_7/\text{GO}$ heterostructures are composed of C, O, and V elements (Fig. 1c), comparing with the V_2O_5 only exists V and O elements (Figure S3a). The high-resolution XPS spectra of V 2p for $\text{V}_3\text{O}_7/\text{GO}$ heterostructures are demonstrated in Fig. 1f, where the peaks of V 2p_{3/2} and V 2p_{1/2} correspond to 517.2 and 524.4 eV, respectively [32]. The V 2p_{3/2} peak

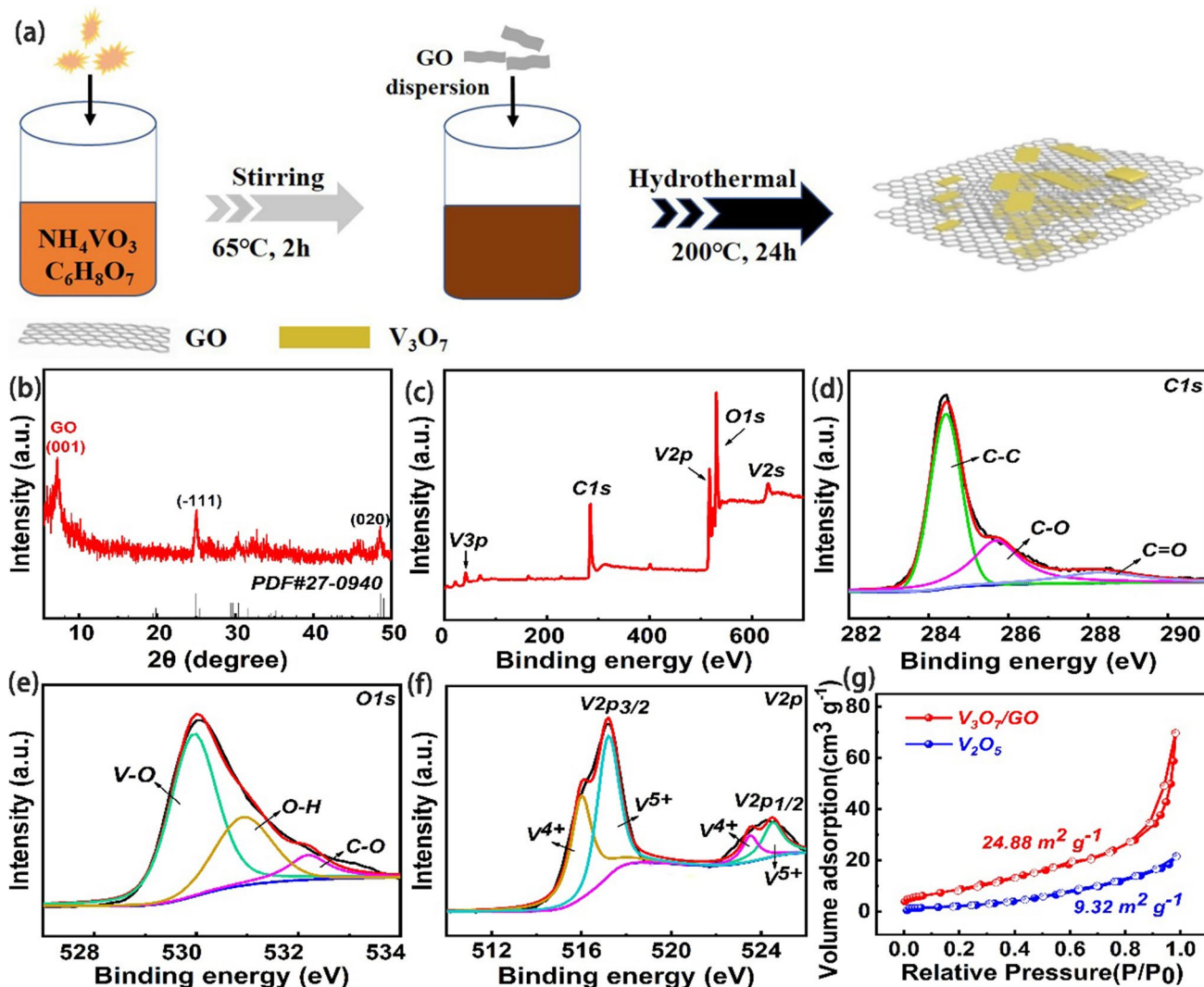


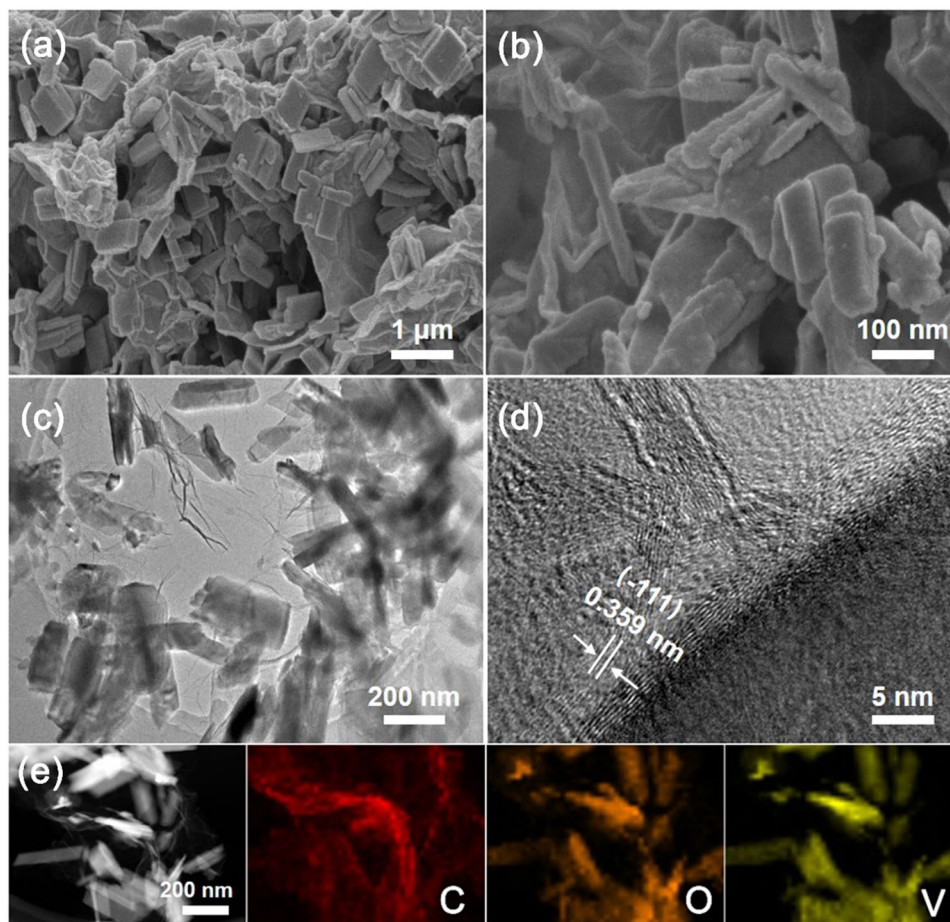
Fig. 1 a Schematic diagram of preparation procedures of V_3O_7/GO heterostructures, b XRD patterns, c XPS survey spectrum, d C 1 s, e O 1 s, f V 2 p core-level spectra of V_3O_7/GO heterostructures, g nitrogen adsorption/desorption curves of V_3O_7/GO heterostructures and V_2O_5

is divided into two peaks at binding energies of 515.9 and 517.2 eV, which corresponded to V^{4+} and V^{5+} , respectively [33]. Compared with V_2O_5 (Figure S3c), the appearance of V^{4+} in V_3O_7/GO heterostructures is due to the reduction of V^{5+} to V^{4+} when the GO is present. The C 1 s core levels of V_3O_7/GO heterostructures are exhibited in Fig. 1d. The peaks at 288.4, 285.8, and 284.3 eV are attributed to the C=O, C–O, and C–C bonds of GO, respectively [34]. The O 1 s core levels of V_3O_7/GO heterostructures (Fig. 1e) exhibit three peaks at 529.8, 531.0, and 532.5 eV, respectively, the peak of V–O bonds is assigned at 529.8 eV, the peak at 531.0 eV is assigned to the attached residual water at the surface, and while the peak at 532.5 eV corresponds to –OH/–COOH groups of GO. The hydrogen bond between V_3O_7 nanosheets and –OH/–COOH groups on the surface of GO can buffer the expansion and contraction

of V_3O_7/GO during the Zn^{2+} insertion/extraction process. Therefore, the O 1 s core levels of V_2O_5 are inconsistent with V_3O_7/GO heterostructures (Figure S3b). The specific surface areas of V_3O_7/GO heterostructures and V_2O_5 (Fig. 1g) are obtained by nitrogen adsorption/desorption measurement as $24.88\text{ m}^2\text{ g}^{-1}$ and $9.32\text{ m}^2\text{ g}^{-1}$. The results proved that the importing of GO can increase the specific surface area of V_3O_7 , which provides more active sites and is promising to obtain a high capacity. Moreover, the GO can also serve as electron-conductive frameworks that could improve the conductivity and structural stability of V_3O_7 .

The scanning electron microscope (SEM) and transmission electron microscopy (TEM) images of V_3O_7/GO are exhibited in Fig. 2. The images of SEM present a heterostructures between V_3O_7 nanosheets and GO

Fig. 2 **a** and **b** SEM images of the V_3O_7 /GO heterostructures, **c** TEM image and **d** HRTEM image of V_3O_7 /GO heterostructures, **e** TEM elemental mappings of C, O, and V elements in V_3O_7 /GO heterostructures



layers (Fig. 2a–b), which is composed of interlaced V_3O_7 nanosheets. The heterostructures are further confirmed by TEM image (Fig. 2c), and V_3O_7 nanosheets were tightly attached on the surface of GO to form an ultrathin flat heterostructures which provide rapid diffusion channels for Zn^{2+} . By contrast, V_2O_5 is shown as a blocky structure in Figure S4, and the long pathway of Zn^{2+} ion diffusion in blocky structure is unsuitable to achieve a high-rate capability. The high-resolution transmission electron microscope (HRTEM) image of V_3O_7 /GO heterostructures is shown in Fig. 2d, the interlayer spacing of 0.359 nm corresponding to the (-111) planes of V_3O_7 . The element distributions of V_3O_7 /GO heterostructures are displayed in the TEM mapping (Fig. 2e), and the V, O, and C elements are uniformly distributed, which confirms that the V_3O_7 nanosheets were uniformly distributed on the surface of GO in the heterostructures. It can be explained that V_3O_7 nanosheets are connected with GO by hydrogen bonds between V–O and functional groups on the surface of GO. The GO can prevent the volume expansion of V_3O_7 and improve the conductivity of V_3O_7 due to its high mechanical strength and inherent conductivity, improving the cycle stability and rate capability.

The zinc storage performance of V_3O_7 /GO heterostructures was evaluated employing Zn foil as anode and 3 M $ZnSO_4$ (saturated vanadium oxide) as electrolyte. The cyclic voltammetry (CV) curves at a scan rate of 0.1 mV s^{-1} are displayed in Fig. 3a. The second and third cycles are mildly different from the first cycle, which is attributed to activated process of electrode in the first cycle. The redox peaks at 0.74/0.60 V and 1.03/0.92 V can be found in the second cycle, which explains a mechanism of multistep zinc insertion/extraction reaction during the discharge/charge process. Compare with V_3O_7 /GO, the CV curves of V_2O_5 exhibit an inferior consistency (Figure S5a), which confirms that an irreversible phase change occurred in the cyclic processes. The CV curves of V_3O_7 are exhibited in Figure S6a, which shows a similar shape to V_3O_7 /GO heterostructures. The galvanostatic charge/discharge (GCD) curves of first three cycles of V_3O_7 /GO heterostructures at a current density of 1.0 A g^{-1} are presented in Fig. 3b. The discharge capacity of first cycle is $275.6 \text{ mA h g}^{-1}$ and the first three charge/discharge curves are almost overlapped, suggesting the outstanding cycle stability of V_3O_7 /GO heterostructures. The GCD curves of V_2O_5 are shown in Figure S5b, which has the same voltage plateaus compare to V_3O_7 /GO

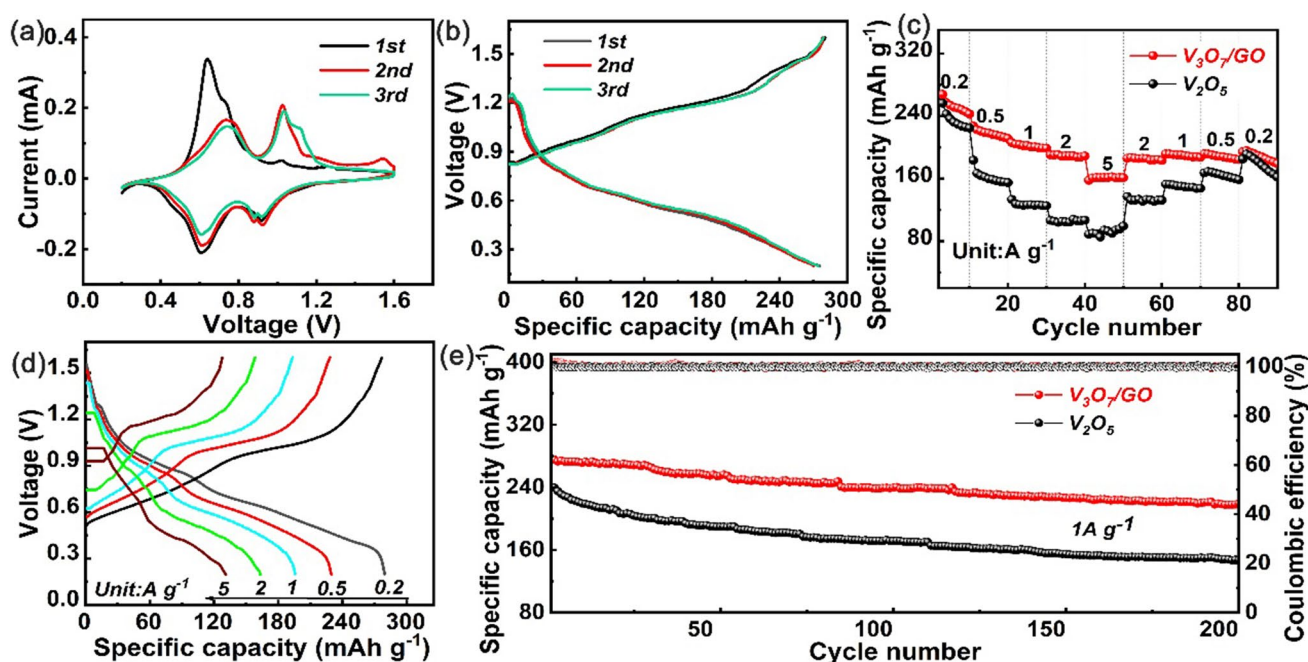


Fig. 3 **a** The cyclic voltammetry curves of the first three cycles at 0.1 mV s^{-1} , **b** galvanostatic charge/discharge curves at 0.1 A g^{-1} , **c** rate capability at the different current densities, **d** galvanostatic

charge/discharge curves at the different current densities, **e** the cycle stability at a current density of 1.0 A g^{-1}

heterostructures. The rate capability of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures at the different current densities is displayed in Fig. 3c and d, and the specific capacities are 268.0, 227.3, 206.1, 190.6, and 161.5 mA h g^{-1} at the current densities of 0.2, 0.5, 1.0, 2.0, and 5.0 A g^{-1} , respectively. The current density is 5 A g^{-1} , the specific capacity is 161.5 mA h g^{-1} , and the capacity retention rate is 60.3%. The specific capacity is restored to 195.9 mA h g^{-1} and the restoration ratio is 73.1% when the current density is recovered to 0.2 A g^{-1} . In comparison, the specific capacities of V_2O_5 are 236.5, 159.7, 126.5, 104.7, and 89.0 mA h g^{-1} at the same current densities (Figure S5c). The specific capacities of V_3O_7 are 185.8, 145.0, 125.0, 108.4, and 85.5 mA h g^{-1} at the same current densities (Figure S6b). The specific capacity is restored to 131.3 mA h g^{-1} and the restoration ratio is 68.3% when the current density is recovered to 0.2 A g^{-1} . The specific capacity and capacity retention of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures are superior to that of V_2O_5 , which explains $\text{V}_3\text{O}_7/\text{GO}$ heterostructures possess outstanding rate capability (Fig. 3c). The cycle stability of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures and V_2O_5 at the current density of 1.0 A g^{-1} is displayed in Fig. 3e, and a remarkable discharge capacity of 275.6 mA h g^{-1} is obtained for $\text{V}_3\text{O}_7/\text{GO}$ heterostructures. The cycle stability of V_3O_7 at the current density of 1.0 A g^{-1} is displayed in Figure S6a, the specific capacity reaches 217.7 mA h g^{-1} , and the capacity retention is only 44.5% after 200 cycles. The long cycle performance of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures at the current density of 2.0 A g^{-1} is displayed in Figure S7,

the specific capacity reaches 279.9 mA h g^{-1} , and remarkable capacity retention of about 76% after 200 cycles. It is obvious that the specific capacity of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures is higher than V_2O_5 , which confirms that $\text{V}_3\text{O}_7/\text{GO}$ heterostructures possess more active sites to storage Zn^{2+} . The cycle stability, rate capability, and specific capacity of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures are superior than V_2O_5 due to GO can improve conductivity, buffer expansion of volume, and increase specific surface area.

To further explore the storage kinetics behavior of Zn^{2+} , the CV curves were investigated that the scan rates from 0.2 to 1 mV s^{-1} as shown in Fig. 4a. The CV curves maintained the same shapes with the increased of scan rates from 0.2 to 1 mV s^{-1} , which demonstrates the outstanding rate capability and reversibility of reaction reversibility of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures. Compared with V_2O_5 (Figure S8a), the CV curves of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures show sharper redox peak, which suggests the faster reaction kinetics. To determine the capacity behaviors of Zn^{2+} storage in $\text{V}_3\text{O}_7/\text{GO}$ heterostructures, the relationship between the peak current (i) and the scan rate (ν) of CV curves be expressed by the following formula [35]:

$$i = a\nu^b$$

$$\log i = b \log \nu + \log a$$

where ν is the scan rate, i is the current response, and a and b are adjustable constants. The slope of $\log(i)$ versus

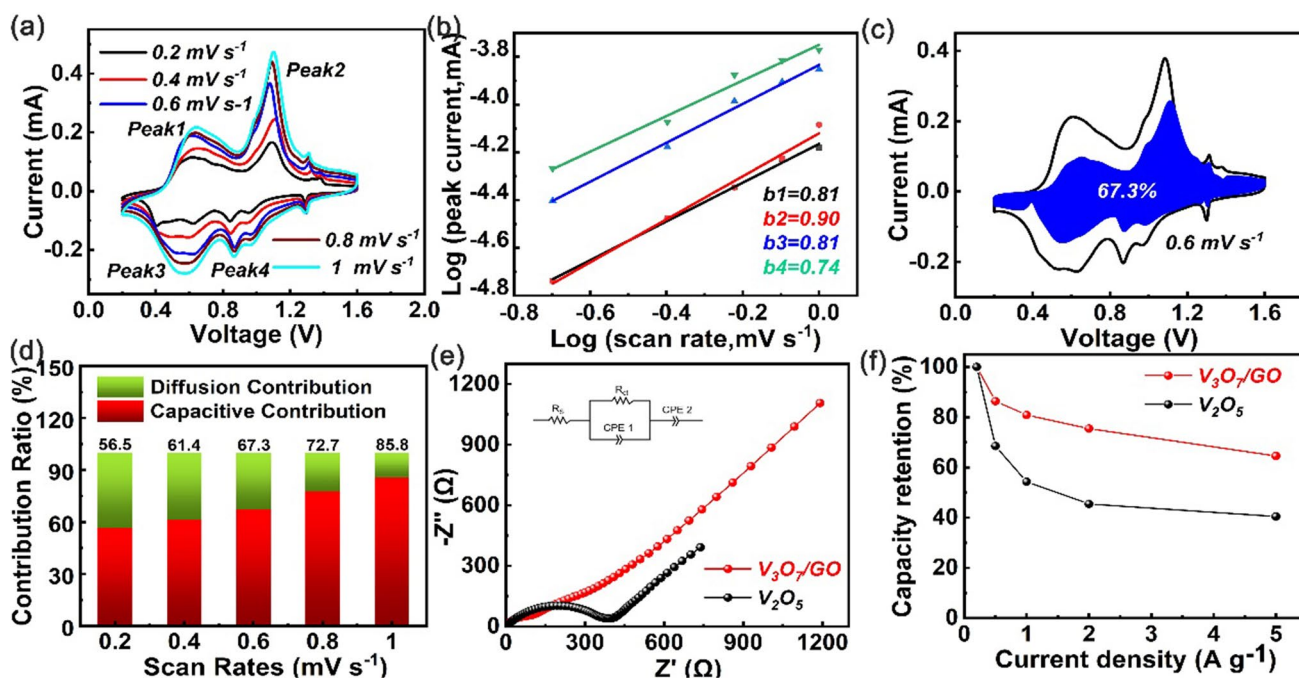


Fig. 4 **a** Cyclic voltammograms at different scan rates, **b** $\log(i)$ – $\log(v)$ plots for specific peak currents, **c** Surface-controlled capacity contribution at 0.6 mV s^{-1} , **d** surface- and diffusion-controlled capac-

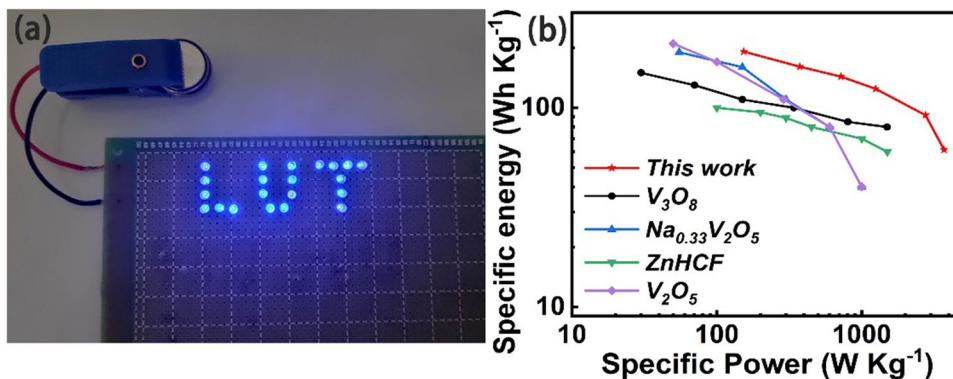
ity contributions at different scan rates of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures, **e** Nyquist plots and **f** capacity retention of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures and V_2O_5 at different current densities

$\log(v)$ plot is decided by the b value, which ranges from 0.5 to 1. The charging/discharging process is dominated by ion diffusion when $b=0.5$, and $b=1.0$ for a surface-controlled capacity dominates [36]. The b values of peaks 1, 2, 3, and 4 of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures are 0.81, 0.90, 0.81, and 0.74, respectively (Fig. 5b), while that of V_2O_5 are 0.82, 0.88, 0.76, and 0.70, respectively (Figure S8b), proving the mainly contributed by the surface-controlled process and the slight contributed by diffusion-controlled process in storage processes of Zn^{2+} . The surface-controlled process is crucial to achieve a high-rate capability compared with diffusion-controlled process. The capacitance contribution at a certain scan rate can be determined by employing the equation by [37]:

$$i = k_1 v + k_2 v^{1/2}$$

where k_1 and k_2 are constants, and the surface- and diffusion-controlled capacity contributions can be determined by considering the ratio of k_1 and k_2 . To clarify this case, the surface-controlled capacity contribution of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures and V_2O_5 can be displayed by the CV curves at 0.6 mV s^{-1} is displayed in Fig. 4c and Figure S8c, respectively. The distribution of surface-controlled capacity contributions and diffusion-controlled capacity contributions at the different scan rates of $\text{V}_3\text{O}_7/\text{GO}$ heterostructures as shown in Fig. 4d, where the surface-controlled capacity contribution enhances from 57.7 to 87.0% with the increased scan rate from 0.2 to 5 mV s^{-1} . The increased surface-controlled

Fig. 5 **a** The $\text{Zn}||\text{V}_3\text{O}_7/\text{GO}$ batteries were employed to light the LED bulb. **b** The Ragone plot of the $\text{Zn}||\text{V}_3\text{O}_7/\text{GO}$ battery compared with other aqueous ZIBs



capacity contribution indicates that V_3O_7/GO possesses a rapid reaction rate, which caused by the increased specific surface area and promoted conductivity by forming heterostructures. The surface-controlled capacity contribution of V_2O_5 enhances from 39.3 to 76.9% with the same scan rate (Figure S8d). It is clear that surface-controlled capacity contribution of V_3O_7/GO heterostructures is higher than that of V_2O_5 , which proved the outstanding kinetic performance of V_3O_7/GO . The reaction kinetic analysis of V_3O_7/GO heterostructures and V_2O_5 was conducted by the electrochemical impedance spectroscopy (EIS). The corresponding Nyquist plots of V_3O_7/GO heterostructures and V_2O_5 were tested from 0.01 Hz to 100 kHz. The semicircle is the high frequency area, which represents the charge transfer resistance (R_{ct}) at the electrode/electrolyte interface. The R_{ct} of V_3O_7/GO heterostructures is smaller than that of V_2O_5 in Fig. 4e, confirming that the storage reaction of Zn^{2+} in V_3O_7/GO heterostructures is easier than that in V_2O_5 because of the promoted conductivity and increased specific surface area of V_3O_7/GO heterostructures. The straight line is the low frequency area, which represents the Zn^{2+} diffusion resistance within the electrodes. The slope of the V_3O_7/GO heterostructures is much steeper than V_2O_5 , indicating faster Zn^{2+} diffusion in V_3O_7/GO heterostructures/electrolyte interface than that of V_2O_5 . The rate capability is significantly improved due to the rapid diffusion rate of Zn^{2+} and the result can be further confirmed by the capacity retention rate in Fig. 4f. The tight attachment of V_3O_7 and GO with enlarged surface area provides a large interface of electrolyte/electrode and rapid charge transfer for the storage of Zn^{2+} , which improves the reaction kinetic performance.

To illustrate the practicality of V_3O_7/GO heterostructures, the $Zn||V_3O_7/GO$ ZIBs were assembled employed Zn foil as anode and V_3O_7/GO heterostructures as cathode. The working voltage can reach 3.0 V by two batteries in series and the LED lights can be driven in Fig. 5a. The Ragone plot of ZIBs is displayed in Fig. 5b, and the $Zn||V_3O_7/GO$ ZIBs deliver a high energy density of 191.8 Wh kg^{-1} at the power density of 153.4 W kg^{-1} and the energy density of 61.6 Wh kg^{-1} at the power density of 3696 W kg^{-1} . The result is higher than the currently reported V_3O_8 [27], $Na_{0.33}V_2O_5$ [38], $ZnHCF$ [39], and V_2O_5 [40], which confirms the V_3O_7/GO heterostructures electrode possesses a favorable practicability.

Conclusions

In summary, V_3O_7/GO heterostructures were successfully synthesized by a hydrothermal reaction, and the heterostructures were formed due to the V_3O_7 nanosheets were tightly attached on GO surface. The V_3O_7/GO heterostructures possess a high structural stability and large specific surface area,

which displays high specific capacity and outstanding cycle stability. The specific capacity of V_3O_7/GO heterostructures is 275.6 mA h g^{-1} at the current density of 1.0 A g^{-1} and the capacity retention is 79.6% after 200 cycles. The excellent Zn^{2+} storage performance of V_3O_7/GO heterostructures can be attributed to the following advantages: (i) The enlarged specific surface area can provide abundant active sites for the storage of Zn^{2+} , (ii) V_3O_7/GO heterostructures can remain an outstanding structural stability, and (iii) the electrical conductivity is enhanced due to the addition of GO. The study paves the way for promoting the Zn^{2+} storage performance of vanadium oxide and developing stable cathode materials of ZIBs.

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s11581-021-04255-y>.

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