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Synthesis of Hierarchical V₂O₅ Hydrate Composite Micronanostructures as Cathode Materials for Aqueous Zincion Batteries with Good Performance

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It is important to discover novel electrode materials and optimize the performance of existing cathode materials for aqueous zinc-ion batteries. Here, two $V_2O_5 \cdot 3H_2O$ composites are achieved via a facile hydrothermal route. One is flake-like $V_2O_5 \cdot 3H_2O$ built up with multiple platelets with surface defects in parallel or normal positions, and the other is $V_2O_5 \cdot 3H_2O$ composite microflowers built up with multipleyered microplatelets in a radial position without surface defects. Both can be used as cathode materials for aqueous zinc-ion batteries. The former showed higher discharge capacity and better rate performance. The electrode of $V_2O_5 \cdot 3H_2O$ flakes exhibits stable discharge capacity at current densities of 50, 100, 250, 500, 750, 1000, 1500 and 50 mAg⁻¹. The capacity retention is 90.3% after 80 cycles from 237.2 mAhg⁻¹ to 214.1 mAhg⁻¹. While $V_2O_5 \cdot 3H_2O$ microflowers do not exhibit stable discharge capacity at current densities of 50, 100, 250, 600, 750, 100, 250, 500, 750, 100, 250, 500, 750, 100, 250, 500, 750, 100, 250, 500, 750, 100, 250, 500, 750, 100, 250, 500, 750, 100, 250, 500, 750, 100, 250, 500, 750, 1000, 1500 and 50 mAg⁻¹. The capacity retention is 47.96% after 80 cycles from 223.9 mAhg⁻¹ to 107.4 mAhg⁻¹. The difference in cycling performance can be ascribed to surface defects and different compositions.

Keywords: Paramontroseite VO2; Hollow microspheres; Anode; Lithium-ion battery; Cathode

1. INTRODUCTION

Currently, great interest is being shown in zinc-ion batteries for their large-scale energy storage and portable electronic applications due to their low cost, material abundance, high safety, acceptable energy density and environmental friendliness [1]. The concern mainly focuses on the design and synthesis of various electrode materials for aqueous zinc-ion batteries with improved properties in capacities, cycling stability, voltage and electrolyte properties [2], such as manganese oxide [3], and polypyrrole [4].

It is important to determine a suitable cathode with high energy density and voltage for batteries. Vanadium-based materials are the most fascinating materials for energy storage in aqueous zinc-ion batteries due to their high capacity and excellent cycling stability at high discharge rates, which are even better than lithium-ion batteries. A series of promising cathode materials have been developed in in recent years. $Zn_{0.25}V_2O_5 \cdot nH_2O$ showed a reversible Zn^{2+} ion (de) intercalation storage process at fast rates, resulting in a capacity up to 300 mAhg⁻¹ and a capacity retention of more than 80% over 1,000 cycles [5]. VS₄@rGO electrodes deliver a capacity of 180 mAhg⁻¹ with a capacity retention of 93.3% after 165 cycles at a current density of 1 Ag⁻¹[6]. (NH₄)₂V₁₀O₂₅·8H₂O nanobelts have a discharge capacity of 123.6 mAhg⁻¹ with a capacity retention of 94.2% after 5000 cycles at a current density of 5 A g⁻¹, ascribed to large layer distance [7]. The discharge capacity of $Li_xV_2O_5 \cdot nH_2O_5$ is 232 mAhg⁻¹ at a current density of 5A g⁻¹ after 500 cycles due to the expansion of the V_2O_5 nH₂O layer distance [8]. CaV₆O₁₆·3H₂O microwires have a discharge capacity between 160 and 170 mAhg⁻¹ at a current density of 5 A g⁻¹ after 300 cycles [9]. $K_2V_8O_{21}$ has a discharge capacity of 185 mAhg⁻¹ at a current density of 5 A g⁻¹ after 100 cycles [10]. However, V₁₀O₂₄·12H₂O had a capacity of 164.5 mAhg⁻¹ at a high current density of 10 Ag⁻¹ [11]. Sodium vanadate hydrate (NaV₃O₈·1.5H₂O) nanobelts serve as positive electrodes and zinc sulfate aqueous solution with sodium sulfate additive is used as the electrolyte and shows a high reversible capacity of 380 mAhg⁻¹ and capacity retention of 82% over 1000 cycles. The excellent performance is ascribed to simultaneous proton, and zinc-ion insertion/extraction processes [12]. The discharge capacity of NH₄V₄O₁₀ reaches 255.5 mAhg⁻¹ at a current density of 10 Ag⁻¹ after 1000 cycles [13]. The H₂V₃O₈ NW/graphene composite exhibits superior zinc ion storage performance including a high capacity of 394 mAhg⁻¹ at 1/3 C, a high rate capability of 270 mAhg⁻¹ at 20 C and excellent cycling stability of up to 2000 cycles with a capacity retention of 87% [14]. H₂V₃O₈ nanowires exhibit a capacity of 423.8 mAhg⁻¹ at 0.1 Ag⁻¹, and good cycling stability with a capacity retention of 94.3% over 1000 cycles, which is attributed to the larger spacing of $H_2V_3O_8$ [15]. We also first tested $H_6V_4O_{10}$ microspheres as cathode materials for aqueous zinc-ion batteries, which had a capacity retention of 238.8 mAhg⁻¹ and 87.8% after 26 cycles [16]. It can be found that vanadium oxides and vanadates with hydrate, valence between V^{4+} and V^{5+} and a large interlayer distance above 0.9 nm are ideal high-energy cathode materials for aqueous zinc-ion batteries except their lower operation voltage. A suitable vanadium-based cathode with high energy density and voltage for aqueous zinc-ion batteries is being pursued.

Organic pillar preintercalated $V_2O_5 \cdot 3H_2O$ nanocomposites maintain a considerable a capacity of 133 after 1000 cycles at a high current density of 10 Ag⁻¹ owing to the interlayer expansion of organic molecules and the enlarged interlayer and mixed valence [17]. However, in the reference, there is no comparison of $V_2O_5 \cdot 3H_2O$ with the original interlayer distance cathode performance for aqueous zinc-ion batteries. Na- and K-ion intercalated $V_2O_5 \cdot 3H_2O$ nanostructures could be high-performance symmetric electrochemical capacitors [18]. There are few reports on the rare material of $V_2O_5 \cdot 3H_2O$ [19]. Herein, we have developed a straightforward way to prepare two $V_2O_5 \cdot 3H_2O$ microstructure composites. One is Navajoite $V_2O_5 \cdot 3H_2O$ with impurities of $V_2O_2(OH)_3$ microflowers and the other is Navajoite $V_2O_5 \cdot 3H_2O \cdot V_4O_7 - H_{0.37}VO_2$ composite microplates. The electrochemical properties of $V_2O_5 \cdot 3H_2O$ for aqueous zinc-ion batteries were improved via the control of surface defects and composition. $V_2O_5 \cdot 3H_2O \cdot V_4O_7 - H_{0.37}VO_2$ composite microplates showed better cycling stability and excellent rate performance as the cathode material for aqueous zinc-ion batteries.

2. EXPERIMENTAL PROCEDURE

In a typical procedure for the preparation of flake-like $V_2O_5 \cdot 3H_2O$ composites, 30 ml 0.5 mol/L ammonium vanadate, 0.4 mol/L g oxalic acid solution and 0.1 mol/L LiNO₃ were mixed and stirred for 2 h in a beaker at room temperature. After that, the pH of the solution was adjusted to 9, transferred to a 50 ml autoclave, sealed, and heated at 180 °C for 24 h, Then the autoclave was cooled to room temperature. The black-green precipitation was centrifuged and dried at 110 °C for 3 h. For comparison, the pH of the solution was adjusted to 8 under the identical conditions, and a flower-like V₂O₅·3H₂O composite was achieved.

Scanning electron microscopy (SEM) was performed with a Hitachi S-4800 field emission scanning electron microscope. Transmission electron microscopy (TEM) was performed with USA FEI-Tecnai G2. X-ray diffraction (XRD) were collected with a PANalytical, and X'Pert diffractometer. A Land CT2001A battery tester controlled the cycling performance of the battery. Cyclic voltammetry (CV) was performed with a Chi660E electrochemical workstation. Electrochemical measurements were performed with a DH7001 electrochemical test system (Jiangsu Donghua Analytical Instruments Co., Ltd.).

Both flake-like and flower-like $V_2O_5 \cdot 3H_2O$ were tested as cathode materials for aqueous zincion batteries. The active material, a conductive material (superpure carbon) and binder polyvinylidene difluoride (PVDF) were pasted onto the graphite paper in a weight ratio of 7/2/1. The Zn foil was the counter electrode. The electrolyte was 1 M ZnSO₄ aqueous solution.



3. RESULTS AND DISCUSSION

Figure 1. Wide-angle powder XRD patterns of samples (a) V₂O₅·3H₂O flakes, (b) V₂O₅·3H₂O microflowers

X-ray diffraction (XRD) patterns confirm that the crystalline structure of samples prepared at a pH value of 9 was Navajoite V₂O₅·3H₂O with impurities of V₄O₇ and H_{0.37}VO₂ (JCPDS 07-0332), as

shown in Fig. 1a. The sample prepared at a lower pH of 8 is also ascribed to Navajoite V_2O_5 ·3H₂O with impurities of $V_2O_2(OH)_3$ (JCPDS 07-0332) in Fig. 1b. Obviously, the latter has a weaker crystalline structure for fewer diffraction peaks than the former.

SEM images show that the sample prepared at a pH of 9 has a morphology of flakes (Fig. 2), which are between 8 and 14 um long (Fig. 2a), several hundred nm thick. The surfaces of some flakes are not smooth and covered with thin parallel plates in in the vertical position. This unique morphology is rare for vanadium-based materials. Other flakes are completely built up with platelets in the parallel position. Fig. 2b clearly shows the border of these flakes. TEM was further performed to observe the surface characteristics of the flakes, as shown in Fig. 2c. Certain platelets are composed of three layers of thin platelets. The surfaces of platelets have macropores, a long gap and many crystalline finger-like intervals, as shown in Fig. 2d. Therefore, V4O7 microflakes were built up with multiple platelets with surface defects in the parallel parallel or normal position.



Figure 2. SEM images of samples prepared at a pH value of 9 (a, b) and the corresponding TEM images (c, d).

Fig. 3 shows SEM and TEM images of Navajoite $V_2O_5 \cdot 3H_2O$ with impurities of $V_2O_2(OH)_3$. It has a morphology of microflowers composed of microplatelets as shown in Fig. 3a. The large microflower can be up to 16.5 µm long and 8 µm wide. The magnified SEM image shows that these

microplatelets are so thin that some microplatelets become curved, as shown in Fig. 3b. The thickness of these microplatelets is approximately 30 - 40 nm thick. Fig. 3c is the TEM image of certain small microflowers. It can be clearly found that this microflower is built up with multilayered microplatelets in a radial position. The magnified SEM image confirms that the surface of microplatelets is smooth without any defects in Fig. 3d.



Figure 3. SEM images of samples prepared at a pH value of 8 (a, b) and the corresponding TEM images (c, d).

Both $V_2O_5 \cdot 3H_2O$ flakes and $V_2O_5 \cdot 3H_2O$ microflowers are found to be potential electrode materials for aqueous zinc-ion batteries. $V_2O_5 \cdot 3H_2O$ microflowers show higher discharge capacity and better cycling stability. Cyclic voltammetry (CV) was further performed to study the zinc-ion intercalation for $V_2O_5 \cdot 3H_2O$ flakes and microflowers. The first CV curve of the $V_2O_5 \cdot 3H_2O$ flake presents one catholic peak at 0.396 V in Fig. 4a, which shifts to 0.435, 0.449 and 0.482 V after three sequent cycles. Zinc ions can be reversibly intercalated at approximately 0.5 V for $V_2O_5 \cdot 3H_2O$ flakes. In $V_2O_5 \cdot 3H_2O$ microflowers, no extra cathodic peaks appear which implies that zinc-ions cannot be reversibly intercalated to impurities of V_4O_7 and $H_{0.37}VO_2$ More peaks appear in the $V_2O_5 \cdot 3H_2O$ composite microflower CV curves in Fig. 4b. Similar catholic peaks to V_2O_5 ·3H₂O flakes are at 0.373, 0.429, 0.439 and 0.445 for the first, second, third and fourth cycles, respectively. However, two pairs of new peaks, (0.599, 0.800) and (0.792, 0.991), appear in the second cycle.



Figure 4. Cyclic voltammograms of samples (a) V₂O₅·3H₂O flakes, (b) V₂O₅·3H₂O microflowers.



Figure 5. The 1st, 2nd and 20th charge-discharge profiles of V₂O₅·3H₂O flakes /Zn cell at a current density of 50 mAg⁻¹ (a), the cyclic performance at current densities of 750, 1000 and 2000 mAg⁻¹(b) and its corresponding evolution of the reversible capacity at current densities of 50, 100, 250, 500, 750 1000, 2000 and 50 mAg⁻¹ using 0.05-1.3 V potential window(c).

The (0.792, 0.991) pair of peaks disappeared, while (0.599, 0.800) was intact in the third and fourth cycles. Zinc-ions can be reversibly intercalated to impurity phase $V_2O_2(OH)_3$ in V_2O_5 ·3H₂O microflowers. It is clear that the number and sites of zinc-ion intercalation can be controlled by selecting suitable impurity phases.

Fig 5a shows the charge-discharge curves of $V_2O_5 \cdot 3H_2O$ flakes at a current density of 50 mAg⁻¹. The charge curves are nearly the same. The 1st, 2nd and 20th discharge curves have similar shapes implying reversible Zn²⁺ intercalation and deintercalation. The first, second and twentieth discharge capacities are 209.0, 219.7 and 217.1 mAhg⁻¹, respectively. The electrode of the V₂O₅·3H₂O flakes can cycle 500 cycles at current densities of 750, 1000 and 2000 mAg⁻¹ with second discharge capacities of 130.3, 87.9 and 40 mAhg⁻¹, respectively. After 500 cycles, the discharge capacities are up to 120.0, 101.8 and 50.1 mAhg⁻¹. The capacity retentions are 92.1%, 115.8% and 125.2% for current densities of 750, 1000 and 2000 mAhg⁻¹, respectively. The corresponding discharge rate capability of V₂O₅·3H₂O flakes is also tested at current densities of 50, 100, 250, 500, 750, 1000, 1500 and 50 mAg⁻¹, which exhibits stable discharge capacity in Fig. 5c. The capacity retention is 90.3% after 80 cycles from 237.2 mAhg⁻¹ to 214.1 mAhg⁻¹.



Figure 6. (a) The 1st, 2nd and 20th charge-discharge profiles of V₂O₅·3H₂O microflowers / Zn cell at a current density of 50 mAg⁻¹ (a), the cyclic performance at current densities of 750, 1000 and 2000 mAg⁻¹(b) and its corresponding evolution of the reversible capacity at current densities of 50, 100, 250, 500, 50 1000, 2000 and 50 mAg⁻¹ using 0.05-1.3 V potential window(c)

The same electrochemical performance was also obtained for the electrode of $V_2O_5 \cdot 3H_2O$ composite microflowers. Fig. 6a shows the charge-discharge curves at a current density of 50 mAg⁻¹. There is a large big bias between the charge curves because of polarization. The 20th discharge curves are different from the 1st and 2nd discharge curves. The first, second and twentieth discharge capacities

are 196.4, 197.9 and 112.3 mAhg⁻¹, respectively. The electrode of the V₂O₅·3H₂O microflowers cannot stably cycle between 500 cycles at current densities of 750, 1000 and 2000 mAg⁻¹, in addition, the discharge capacity is lower in Fig. 6b. The second discharge capacities at current densities of 750, 1000, and 2000 mAg⁻¹ are 130.3, 87.9 and 40 mAhg⁻¹, respectively. The corresponding discharge rate capability of V₂O₅·3H₂O microflowers was also tested at current densities of 50, 100, 250, 500, 750, 1000, 1500 and 50 mAg⁻¹, which does not exhibit stable discharge capacity in Fig. 6c. The capacity retention is 47.96% after 80 cycles from 223.9 mAhg-1 to 107.4 mAhg⁻¹. The acceptable electrochemical performance of the two V2O5.3H2O composites is ascribed to their novel micronanostructures [20-24] and the presence of 3H₂O. UltrathinV₂O₅·4 VO₂·2.72H₂O nanobelts can effectively shorten the Zn^{2+} diffusion path, which contributes to a good contribution to good cycling performance for aqueous rechargeable zinc-ion batteries [25]. 3D Ti₃C₂T_x@MnO₂ microflowers delivered a high capacity of 301.2 mA h g⁻¹ at 100 mA g⁻¹ as the cathode for an aqueous zinc-ion batteries with a high tapping density (1.52 g cm⁻³), fast charge-transfer kinetics and superior structural integrity [26]. Under our experimental conditions, we found that the V₂O₅ electrode showed a very low discharge capacity and poor cycling stability. Both electrolyte and material structures play an important role in the cycling stability and rate performance of aqueous zinc-ion batteries. Most V₂O₅ based cathode materials showed a higher discharge capacity at a high discharge rate in the presence of a high concentration and expensive Zn(CH₃F₃SO₃)₂ electrolyte[27-40], as listed in Table 1. In this paper, we achieved good cycling and rate performance with a low concentration of ZnSO₄ electrolyte.



Figure 7. The fitted and original Nyquist-diagram of (a) $V_2O_5 \cdot 3H_2O$ flakes, (b) $V_2O_5 \cdot 3H_2O$ microflowers (The top is the fitting impedance circuit model).

Ref.	Materials	Voltage (vs.	Electrolyte	Discharge	Current
		Zn^{2+}/Zn) (V)		capacity	density
				(mAhg ⁻¹)	Ag ⁻¹
27	V ₂ O ₅ hollow spheres	0.2-1.6	3.65 mol L ⁻¹	132	10
			ZnSO ₄		
			aqueous		
			solution		
28	skin-core	0.2-1.8	2M	191.7	0.3
	heterostructured		Zn(CH ₃ F ₃ SO		
	NaV ₆ O ₁₅ /V ₂ O ₅		3)2		
			aqueous		
			solution		
29	3D porous architectures	0.4-1.6	1 M	336	0.05
	V ₂ O ₅		Zn(ClO ₄) ₂ in		
			propylene		
			carbonate		
30	V ₂ O ₅ nanopaper	0.2-1.6	2 M ZnSO ₄	335.1(100)	1.0
			aqueous		
			solution		
31	Cu ²⁺ -intercalated V ₂ O ₅	0.3-1.4	2 M ZnSO ₄	180	10
			aqueous		
			solution		
32	Vanadium pentoxide	0.01-1.4	3 M Zn	503.1	0.1
	nanosheets grown on		(CF ₃ SO ₃) ₂		

Table 1. The reported V_2O_5 based cathode materials for zinc-ion batteries

	titanium (Ti)		aqueous		
			solution		
33	NH ⁴⁺ Preintercalated V2O5•nH2O Nanobelts	0.4-1.4	1 M ZnSO ₄ aqueous solution	276	0.5
8	Li ⁺ intercalated V ₂ O ₅ nH ₂ O	0.4-1.4	2 M ZnSO ₄ electrolyte	232	0.5
34	V ₂ O ₅ nH ₂ O/MXene composite	0.2-1.6	3 M Zn (CF ₃ SO ₃) ₂ aqueous solution	223	0.1
35	Sulfite modified and ammonium ion intercalated vanadium hydrate	0.4-1.6	3 M zinc trifluorometh yl mesylate (Zn(TfO) ₂) solution	108	2
36	polyaniline intercalated−exfoliated V ₂ O ₅ ·nH ₂ O	0.4-1.6	3 M zinc trifluorometh yl mesylate (Zn(TfO) ₂) + 6 M trifluorometh yl sulfimide lithium (LiTFSI)	323	1
37	$Mn_{1.4}V_{10}O_{24}{\cdot}12H_{2}O$	0.4-1.6	3M zinc trifluorometh anesulfonate	456	0.2
38	hydrated zinc vanadium oxide/carbon clot	0.2-1.4	3 M Zn(CF ₃ SO ₃) ₂ electrolyte	135	20
39	Ca ²⁺ - intercalated V ₂ O ₅ ·nH ₂ O	0.3-1.5	3 M Zn(CF ₃ SO ₃) ₂ aqueous solution	409	0.05
40	(NH4) _{0.37} V ₂ O ₅ .0.15H ₂ O	0.2-1.4	2.0 M ZnSO ₄ . 7H ₂ O and 0.25 M MnSO ₄	398	0.5

The electrochemical impedance measurements elucidate the electrochemical difference in V_2O_5 ·3H₂O composite flakes and V_2O_5 ·3H₂O composite microflower electrode as shown in Fig. 7. The fitted equivalent electrical circuit model is on the top of Fig. 7a. R is an active electrolyte resistance and CPE is the double-layer capacitance. The impedance of a faradaic reaction is made up of an active

charge transfer resistance Rct and a specific electrochemical element of diffusion ZW called the Warburg element.

The fitted values of R, Rct and Z_W are 4.701, 108.9, and 8.364 x 10⁻³ Ω for V₂O₅·3H₂O composite flakes, while 4.694, 90.27 and 7.114 x 10⁻³ Ω for V₂O₅·3H₂O composite microflowers, respectively. The V₂O₅·3H₂O composite microflowers have smaller charge transfer and Warburg resistance than V₂O₅·3H₂O composite flakes. However, V₂O₅·3H₂O composite flakes are more stable than the V₂O₅·3H₂O composite microflowers during the process of inserting/deinserting zinc ions. The low Rct shows a fast zinc-ion intercalation kinetics and the small Warburg impedance favors the fast diffusion of zinc ions on the interface between the active material and electrode [41-44]. The fast zinc ion intercalation and diffusion will destroy the crystalline structure of the V₂O₅·3H₂O composite flakes have better electrochemical properties than V₂O₅·3H₂O composite flakes have

4. CONCLUSIONS

Navajoite V₂O₅·3H₂O with impurities of V₂O₂(OH)₃ microflowers and Navajoite V₂O₅·3H₂O-V₄O₇- H_{0.37}VO₂ composite microplates were selectively prepared via a facile hydrothermal route. The latter showed higher discharge capacity and better rate performance ascribed to surface defects and different compositions. This research sheds some light on the design and application of electrode materials with surface defects.

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