

Preparation and supercapacitive performance of three-dimensional

Reduced Graphene oxide/polyaniline Composite

Wangjian-de¹, Pengtong-jiang², Xianhai-yang³, Sun-juan³

¹ school of materials Science and Engineering, Southwest University of science and Technology, Mianyang 621000, Sichuan Province, P. R.

² Center of forecasting and analysis, Southwest University of science and Technology, Mianyang 621010, Sichuan province, P.R.China;

³ institute of mineral materials & application, Southwest University of science and technology, Mianyang 621000, Sichuan province, P.R.China

Abstract: three-dimensional reduced graphene oxide (RGO)/polyaniline (PANI) composite has been prepared in a single step by the ultrasonic irradiation of a suspension of graphite oxide and gels using a PANI method. Scanning electronic microscopy (SEM), Transmission electron microscope (TEM), X-ray diffraction (XRD), Fourier transform Infrared Spectra (FT-ir), X-ray photoelectron spectra (XPS), and electrochemical measurements were performed. The morphology, structure, and supercapacitive performance of the composite. The result showed this composite maintained the basic morphology of RGO, and that PANI was inlaid inside network. An outstanding supercapacitive performance is obtained when the mass ratio of graphite oxide and PANI was 1:1. Furthermore, the capacities reached 758 and 300 $\text{C}\cdot\text{g}^{-1}$ at 0.5 and 30 $\text{A}\cdot\text{g}^{-1}$, respectively. The retention rate is found to be 86% after 1000 cycles.

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corresponding author. Email: tjpeng@swust.edu.cn; Tel: +86-816-2419276.

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1 $\text{A}\cdot\text{g}^{-1}$. These results therefore indicate that this new composite possesses the rate and capability of the cycle. Supercapacitive performance is better than that of pure RGO. The excellent supercapacitive performance of this composite can be attributed to the mutual synergy of RGO and PANI.

Keywords: Graphite oxide gel; Polyaniline; Hydrothermal method;

three-dimensional reduced graphene oxide/polyaniline; Supercapacitive Performance

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1. Introduction

Polyaniline (PANI)¹ as the ideal electrode for supercapacitors material, with process simple, low cost, reversible good, ratio measure high, can perform a quick doping and doping process such as a series of excellent point, is widely watched;² but due to swelling and contraction behavior during along charge/discharge cycle, causes its loop poor stability, restricting its further application. With carbon-based materials Composite is one of the best ways to mitigate this defect.^{3,4} and as a new carbon material graphene⁵ with good structural stability, strong conductivity and large specific surface area, is considered to be used to overcome PANI structurally unstable. One of the best carbon materials.⁶ recently, Use reduction graphite allyl (RGO) vs. PANI to promote the electrochemical properties of materials quite compelling.⁷⁻⁹

because the PANI The main chain of has a series of conjugate groups, can be passed static, conjugate and hydrogen bonding forces and RGO knot close⁺¹. so, preparing reductive oxidation graphene/pani (RGP) The main methods for composite materials are dispersion blending method,^{11,12} electrochemical deposition Method, Only, In-situ oxidative polymerization, covalent grafting, and so on. Xu, and so on in acidic solution with RGO to base in-situ oxidative polymerization of aniline single The body gets vertical growth in RGO surface PANI nanofibers, in composites RGO and PANI shows good Synergy with, better than capacitor and cyclic stability than simple RGO and PANI. Liu, and so on using the principle of electrostatic absorption, to bring negatively charged oxygen graphite Graphene (GO) wrapped in positive PANI Hollow Ball surface, and then by electrochemical restoring the RGP Composite, This composite material on a current density of more than a capacitor up to 614 F_g⁻¹ (8) after the second cycle than the capacitance retention rate is 90%, shows a better power Chemical Properties. Liu, and so on The presents another type of GO surface covalent connection branches PANI methods, first with SOCl₂ and GO perform acyl chloride reaction, Then and PANI The amino reaction on the forms the amide key, again Restore implemented PANI nanofibers in RGO on a covalent fix decoration, modified electrode material maximum than capacitance up to 623 F_g⁻¹. Looking at previous research findings,¹⁵⁻¹⁸ Although there are many different method to prepare RGP Composite, But the main idea is in the GO surface oxidative polymerization of aniline monomer, again to implement, make aniline in GO Nano sheets suppress GO when face grows or GOA group of slices and slices Clustering problem. Although this idea has some advantages, But aniline is in suppress GO when the patch is reunited, also blocks the of its slices joins, so it's hard to fully reflect RGO on composite material great Advantage, and the structural stability of composite materials is also difficult to significant elevation.^{15,9}

Recent three-dimensional with hydrothermal synthesis RGO has a porous network-like structure and excellent electrochemical performance; so, This job first in better conditions for the preparation of graphite oxide and PANI nanowires, will two by quality to 1:1 and 1:3 Mixed-ultrasound dispersion, gets the GO vs PANI Mixed Dispersions, and a mixture of the two. to use a one-step hydrothermal method for the precursor a three-dimensional structure of the RGP composite. analyzes the morphology of composite materials, Structure and super capacitance performance, Explore PANI in RGO in the mesh structure Existing status, to achieve structural stability, electrochemical performance more OK RGP composite Purpose.

2. Experiment

2.1 reagents and instruments

Natural Flake Graphite (Qingdao Shen Shu Graphite Products Factory, carbon content 90%-99.9 %, 2 (8) Mesh); potassium permanganate and concentrated sulfuric acid (Chemical reagent, parsing pure); aniline and ammonium persulfate (Chengdu Kelon Chemical reagent, Sub Analysis Pure; 5 % H₂O₂ solution and 0.05 mol·L⁻¹ for HCl Solution (Chengdu Kingsoft Chemical reagent); Experimental water is deionized water (>10 MΩ·cm). DF-101 Stype thermostatic water bath electromagnetic stirrer (Gongyi to

Hewindcompany), JT2003type electronic balances(on Hai Sunyu Instrumentscompany), Freezonetype freeze dryer(United Stateslabconcocompany).

2.2 Preparation of materials

Preparation of graphite oxide gels: in 98% Strong sulfuric acid for, slow join 1g Flake Graphite, Stir in, make the two fully mixed evenly; To call 4g KMnO₄ slow join to above mixtures, Continue stirring 2h; reaction End after using steam feed reaction (temperature control in 60-80 °C) dilution to 5(8) mL around, and then add the appropriate 5% for H₂O₂ to Solution no longer Take bubbles; then add Ten mL 5% for HCl Solution, And a large amount of deionized water full dialysis to neutral, filter, get oxide stone gel; take a certain amount of oxidized graphite gel on the? C conditions under dry out, determine the quality of graphite oxide gel before and after drying

Than.

PANI Preparation of nanowires: First distill the aniline monomer preprocess, with a concentration of 1 mol L⁻¹ HCl Compounding concentration 0.05 mol L⁻¹ aniline monomer solution, Mixing This solution ultrasound Ten min after transfer to ice bath keep min, and then stir quickly under conditions of slow drops dissolve in 1 mol L⁻¹ HCl solution Ammonium persulfate Solution, in which ammonium persulfate and aniline monomer molar ratio To 1:1, "Stir reaction under ice bath conditions" 7h; Filters When the reaction is complete, Wash several times with anhydrous ethanol and distilled water, Freeze Dry again dry, get the PANI nanowires.

three-dimensional RGP Preparation of composite materials: will PANI Add to strong degree 2 mg mL⁻¹ Graphite oxide suspension, where PANI and Graphite quality ratio 1:1 and 1:3, Mixed Ultrasound min get the PANI and G mixed dispersions of, and then transfer it to the water hot inner lining of the reactor, 160 °C Conditional Launch Heat response 5h, get the three-RGP composite hydrogels, Freeze-Dry again, get three-dimensional RGP Composites, marks the sample as, respectively RGP-1 and RGP-3; in the G does not add in the dispersion PANI, also to prepare the RGO.

Preparation of working electrodes: Freeze Dry complete sample cut 1 cm x 1 cm square slice, quality is about 3 mg, stainless steel net (180) to set fluid, on 8 MPa Press the composite under pressure To get the working electrode on the set fluid, then work electrode in electrolysis Soak in the liquid 2h Alternate.

2.3 characterization of the material

XX-ray diffraction (XRD) The analysis is based on the Netherlands Barnard company born product X ' pert [J] MPD Prototype XX-ray diffraction, Cu Target, scan scope 3 -80 . Fourier transform infrared FT-(IR) Spectral analysis use American Hi-Power Instrument co., Ltd production Nicolet-57 (8) Type infrared light spectrometer, Scan Range is 4000-4 (8) cm⁻¹, KBr Tablet-like method. XX-ray photoelectron spectroscopy (XPS) profiling using the UK Kratos Company, xsami Multifunctional Surface Analysis Electronic spectrometer, Al Target (1486.6 eV), X light gun power [kv x MA, take the FAT square style, data with contaminated carbon C1s (284.8 eV) correction. Micro-topography scanning electron microscope (SEM) adopt German Zeiss instrument Company's Ultra, Type field emission scanning electron microscope. field emission transmission electron display Mini (TEM) analysis using German Zeiss Company production Libra FE e-microscope Analyzer.

electrochemical testing using Shanghai Chen Hua Instrument Co., Ltd. production, CHI660 Electrochemical workstation, Platinum chip Electrodes (1.5 cm x 1.5 cm) to electrodes, Select three electrodes test system. RGO electrolyte for 6 mol L⁻¹ for KOH Solution, Hg/HgO The electrode is a reference pole, cyclic voltammetry (CV) and constant current charging and discharging (GCD) test power

the bit range is -1.0V, RGP The electrolyte for is 1 mol L⁻¹ for H₂SO₄ Solution, The saturated mercury electrode is a reference electrode, CV and The range of test potentials for GCD is "-0.2-0.8" V and -0.2-0.7v; RGO RGP, CV Scan rate 5, ten, and 50 mV s⁻¹; current density is 0.5-30 A Metallurgy⁻¹; electrochemical Communication Impedance (EIS) test frequency 0.01-1 (8) kHz, amplitude is 5 MV.

3. Results and discussions

3.1 Profiling of materials

diagram1 is RGO, PANI, RGP-1 and RGP-3 for SEM

diagram. to see, with hydrothermal method RGO internal appearance is multiple hole mesh structure (1(a)), the hole wall consists of a few layers of RGO overlay set to; PANI. This is interleaved with different lengths of nanowires. To (1(b)). when graphite oxide and PANI. The quality ratio of is 1:1, prepared RGP-1 Composite SEM diagram1 (c, D-[], ", " and "@" ()) Show, to see RGP-1. There is still a mutual intersection in the low magnification network structure, The folds that perform well in high numbers, but no obvious PANI existence; when graphite oxide and PANI. Quality of better to 1:3 when, prepared RGP-3 Composite SEM diagram as shown 1 (e, F) show, visible in higher magnification, to clearly show PANI embedded in RGO in the mesh structure of. but contrast 1 (c, D, e, F) discovery in low magnification factor, RGP-3 mesh knot construct less than RGP-1 is evenly distributed. indicates that the method can be used to prepare a net structure RGP Composite, and PANI Nano Line self-RGO Mesh Structure.

RGO, PANI, RGP-1 and RGP-3 TEM2 diagram shows. RGO still displays the interconnected network structure (Chart2(a)), and PANI nanowires are intertwined through each other (Chart2 (b)). after the composite, Composite RGP-1 still behaves as cross-linked mesh structure (Chart2 (c)), But you can see a small amount of PANI tessellation in RGO in the mesh structure of, and PANI spread more even (Chart2 (D)); and when the quality ratio is raised, in RGP-3 Mesh The structure is studded with a large number of PANI, but in laid PANI displays the more serious reunions (Chart2 (e, F)).

Union diagram1, diagram2 to see, Simple RGO behaves as a multi-hole mesh structure, and simple PANI is a different length of nanowires intertwined with each other is more serious. After the two are combined, in RGP-1 low-quality ratio. Although from SEM figure see less than significant PANI mosaic in RGO NET structure, but its mesh structure changed, fold up more, edge rolled heavily, and the from its TEM Chart shows a small amount of PANI nanowires mosaic in RGO mesh structure, and spread more evenly. so, can be thought of as PANI less content, go in hot water also

The same time for dispersed PANI implements the wrapping, causes the SEM Network structure changes in, and cannot see the obvious PANI Nano Line exists. But the quality is greater than the, a prepared RGP-3 can be significantly see PANI nanowire mosaic to RGO in the mesh structure of, but the PANI is easy to reunite itself when the content is large, resulting in the setting of this embedded in RGP-3: PANI reunion more severe.

to explore three-dimensional RGP Composite Formation process. diagram3(a) is Go, PANI, Go/PANI(1:1) and Go/PANI(1:3)

(a) Dispersion of samples sedimentary conditions, (b) RGP-1 and RGP-3 hydrogels, (c) the preparation process of the sample; Go: graphene oxide

deposition after one month of dispersion, because the Goknot The construct contains a large number of hydrophilic groups, and thus evenly dispersed across in aqueous solution with bright yellow, and PANI with water compatibility poor, mostly deposited at the bottom of the reagent bottle. But will be oxidized graphite with PANI, solution with deposition of dark green and no polyaniline occurs. This is mainly due to the PANI positively charged, and Go surface is negatively charged, Go blending helps the PANI uniformly dispersed, where Go acts as a surfactant for with. to go and PANI mixed dispersions for hydrothermal reaction when, due to Go implements the while the restore is in progress PANI in the network mosaic in structure, with three-dimensional structure RGP condensate glue, and with PANI has more content than, three-dimensional RGP hydrogels three Dimension structure size is also increasing (as shown 3 (b)). Its entire response has been process mechanism diagram3 (c) show.

3.2 Structural analysis of materials

diagram4 is Go, RGO, PANI, RGP-1 and RGP-3 for

XRD diagram. to see $d = 9.5$ a peak appears near the sharp feature diffraction peak, $d = 0.95$ nm, corresponds to (001) face net, after hydrothermal reaction, characteristic diffraction peaks of disappear, RGO in 25 Å characteristic diffraction peak with a larger peak width near the, $d = 0.37$ nm, boils down to RGO for (002) face Network, indicates a hot-water anti-should implement go Restore. for a pure PANI for, its primary The feature diffraction peaks are located in the 9.31, 14.72, 20.12 and 25.31 near, respectively corresponds to PANI (001) , (011) , (020) and $(+)$ face Net Strict, and after compounding RGP-1 compared to pure RGO and PANI for 25 Å a weak peak is found near, Peak width large, and RGO for (002) and PANI for similar feature Yanshoot peak, no PANI Other characteristic diffraction peaks of appear; But with the complex

in composite material PANI more than content, in RGP-3 in the spectrogram of the Now it's a feature diffraction peak with PANI. indicates that the composite when PANI less than, Has no obvious PANI feature diffraction for Peak appears, and RGO and PANI The interaction of the makes the composite feature diffraction peak strength weakened.

Diagram 5 is Go, RGO, PANI and RGP-1 for IR Spectrum diagram. you can see from the diagram, in Go The structure contains more oxygen-functional Regime, in 1730, 1631, 1400, 1220 and 1055 cm^{-1} disposition not corresponding to $\text{C}=\text{O}$ Key Flex Peak, curved oscillation of water molecules dynamic peaks, carboxyl groups OH bending vibration peaks of, C-O-C Telescopic Oscillation Peak and C-OH Telescopic vibration peaks of; and after hydrothermal reaction, in RGO The vibration peaks of carboxyl groups in the structure disappear basically., C-O-C and C-OH the strength of the flex peak is also significantly reduced weak. The indicates that the was removed by the hydrothermal reaction Go Most of the structure contains Oxygen Functional Group.,

in a pure PANI in an IR absorption chart of, 3235 cm^{-1} to should be N-H scale vibration Peak, 2910 cm^{-1} corresponds to aromatic ring Last year² Miscellaneous C-H Stretch Vibration Peak, 1587 and 1498 cm^{-1} Place Corresponding to quinone rings and benzene rings respectively C=C Telescopic Vibration Peak, 13 (8) cm^{-1} attributed to secondary aromatic amine C-N Flex Peak, 1120 cm^{-1} corresponds to the benzene ring C-H bending vibration peaks of., then compound RGP-1 contains a pure PANI and RGO individual feature Peaks, Has both PANI of quinone rings and benzene rings in C=C Flex Vibration Peak, and RGO C-O-C and C-OH Flex Vibration peak to

to further identify changes in functional groups in the material, to Go, RGO and RGP-1 on XPS spectrogram analysis. from Diagram 6 (a) A full spectrum of the sample shows the, Go and RGO is only in ??? and 534 eV There are two feature peaks at, corresponds to C and O, spectrogram, and after a hot-water reduction reaction, C/O Atomic ratio (same as) consists of the 2.10 elevated to 5.62 and PANI after is combined, Material RGP-1 in EVA new appears at Non Chart, and C/O margin continue elevated to 7.17. indicates that there is a in the composite PANI The presence of.

will Go, RGO and RGP-1 for C (spectrogram to sub-peak to Close, As shown in figure 6 (b) shows. to see Go consists of the 5a He Feng composition, where 284.5, 285.2, 286.1, 287.5, 288.9 eV at for C=C, C-C, C-OH, C-O-C, O-C=O characteristic peaks of, Triple after hydrothermal restoration in RGO in the structure of O-C=O Peak disappears, C-OH and C-O-C peaks significantly minus weak, and FT-IR token result consistent; but with PANI after composite RGP-1 relative to RGO in 286.1 eV A new feature appears at peak, This boils down to PANI structure C-N/C=N features Peak. other, to RGP-1 for N (spectrogram for peaks fitting places rationale., as shown, 6 (c) show, where 399.3, 4 (8) . 2 and 401.6 eV the corresponds to the quinone-type amine structure (=, N-), linear amine structure (-NH-) and proton nitrogen structure (N+). based on TEM, XRD and FT-IR The characterization analysis of further indicates that the PANI successfully in laid to RGO NET structure.

3.3 Super Capacitance performance analysis for materials

Diagram 7 (a) to RGO, PANI, RGP-1 and RGP-3 on scan rate 5 mV_s^{-1} under conditions of CV Curve. to see RGO CV the curve does not have an obvious redox peak, has a similar Rectangle shape, show ideal double layer capacitance. Sand Pure PANI two to redox peaks (Restore State PANI and oxidize State PANI), because PANI with oxidation-Restore Status, compared to low Electric double layer capacitance of carbon-based material, PANI Show more large Faraday

pseudo capacitor. To compound the two by different quality after RGP-1 and RGP-3 for CV curve with a pure PANI redox peaks for and RGO. The rectangle attribute of the, shows a good

electrochemical Performance; But the redox peak position of the composite is compared to a pure PANI slightly changed, This could be due to the RGO with PANI collaboration effect changed PANI. The corresponding voltage of the When a redox reaction occurs cause.⁹

diagram 7 (b) to RGO, PANI, RGP-1 and RGP-3 on 0.5 A

Under current density GCD Curve. to see RGO for GCD Curved lines are triangular symmetric distribution, belong to typical double electric layer capacitor special ex; Pure PANI for GCD The curve has a significant voltage drop, Complex RGP-3 after Although it has the longest charge/discharge time, but also a certain voltage drop exists, and RGP-1 has a triangular symmetry and no voltage drop present shows a fast electrochemical reaction and good Good electrochemical reversibility. By contrast RGP-1 has better electrochemical Performance. Is based on the GCD The curve consists of a formula (1) to calculate the sheet material capacity

$$C_0 = \frac{Q}{\Delta V} \quad (1)$$

where Q is the charge/discharge current, t is the discharge time, m is active substance quality, ΔV for potential window.

According to the formula (1) calculates the RGO, PANI, RGP-1 and RGP-3 in 0.5 A/g. The specific capacitance of the current density is 315, 303,

758 and 868 F/g. To see that the composite is larger than the size of the Pure RGO and PANI, and with composites PANI content up multiple, It's increasing larger than capacitance; and RGP-3 has the largest more than capacitor, but its GCD curves have less symmetry than RGP-1 for Good, and a certain voltage drop. This is mainly due to the RGO for conductivity is better than pure PANI, and in composites PANI content causes excessive internal resistance to be caused by the

diagram 7 (c) for samples RGO, PANI, RGP-1 and RGP-3 on 0.5-30 A/g. Under current density (1) calculates the ratio of capacitance curve. The shows that the specific capacitance of all samples varies with current density Increase and decrease, but PANI and RGP-3 lower than capacitance of Faster, and PANI in the current density of Ten A/g is more than already Capacitor appears; and RGO, RGP-1 and RGP-3 Even in large current density A/g under conditions with a capacitance of up to 109 and 283 F/g, shows good magnification characteristics, but RGO for lower than capacitance. To indicate that, Composite RGP-1 and RGP-3 better electrochemical performance than pure PANI and RGO, This maintain to RGO Excellent mesh structure and PANI greater than capacity association results with effect.

diagram 7 (D) to PANI, RGO, RGP-1 and RGP-3 EIS Spectrum

diagram, where the illustration is an enlarged image of the sample in the High-frequency and intermediate frequency regions. To see, Four samples have high frequency regions, Intermediate and low frequency, where the diameter of the half arc in the High-frequency region reflects the electrode/electrolyte interface mass-transfer resistance R_t ; If area is one and x-axis to 45 in the corners slashes, infiltration of ions in electrolyte into electrode materials/spread over threads with; The Low-frequency area is one and x-axis vertical line, reflect material material capacitance performance.⁹²⁵ an enlarged view of the high frequency area clearly shows that, complex composite material RGP-1 and RGP-3 where T values are less than pure PANI and RGO values, This is attributable to PANI and RGO Interoperability of Action, and RGP-1 has a bar value that is less than RGP-3 The pinning value of the, this This is mainly because the Addition of the RGO promotes the conductivity of the composite rate, but when PANI The content of is much more than the resistance change of composite materials causes its conductivity to decrease again; Four samples in intermediate frequency area warbury Impedance zone not large, (But in a low frequency area, RGO show the best capacitance characteristics, Composite RGP-1 and RGP-3 The capacitance characteristics of are slightly better than the pure PANI, This is mainly due to the compound material RGO The formation of a loose open pore structure in favor of the Power of the the transfer of ions in solution. To indicate that, Composite RGP-1 and RGP-3 all exhibit good super capacitance performance, and better than Plain PANI, But by contrast RGP-1 has better super capacitance to.

diagram 8 is RGO, PANI, RGP-1 and RGP-3 in 1A·G⁻

cyclic stability curve under current density passes 10 (8) Secondary Charge and discharge Loop after RGO, PANI, RGP-1 and RGP-3 greater than capacity rate is 91 %, 45 %, 86 % and 65 %, carbon material RGO with highest loop stability. This is its specific structure and storage mechanism about and composite RGP-1. The circular stability of is close to the carbon material RGO, significantly higher than RGP-3 and Pure PANI, where pure PANI has the worst cycle stability. can assume that the composite RGP-1 has better cycling stability due mainly to the following two aspects: one, RGO has the added conductivity of the composite can be increased, and PANI embedded in RGO in the network structure of, is fast charging and discharging process to effectively shorten the diffusion and migration of ions in the electrolyte path, to increase active materials electrochemical utilization during circulation rate; second, when PANI even in RGO network structure when, effectively blocks PANI long charge/discharge cycle. The destruction of its structure due to volume expansion, and when PANI has more content, due to PANI itself is easy to reunite and is hard to disperse evenly. Long cycles make it structurally unstable, to result in a composite material. The Loop stability for RGP-3 is also not ideal. Summary, combines diagram 7 and diagram 8 to discover Composites RGP-1 shows the best electrochemical performance.

4. Conclusion

(1) with GO and PANI. The mixed dispersions of are precursors, Mining using one-step hydrothermal method RGP composite, is good for PANI evenly spread of, maintains three-dimensional RGO excellent mesh structure, This is used as an electrode material to provide good ion conduction in the electrolyte good transport channel and larger storage area.

(2) because of the RGO. The addition of can provide a good for composite materials conductivity and excellent mesh structure, PANI add to composite material material provides higher Faraday pseudo capacitor, and PANI embedded in RGO. There is also a certain amount of space blocking in the network structure of, the Interoperability makes composites RGP show Better Superlevel capacitance performance. and when the composite is PANI has an extra amount of overtime, PANI in RGO aggregation is more severe in the network structure of, has a larger specific capacitance, But its cyclic performance is not ideal; phase below, RGP-1 than RGP-3 and Pure PANI and RGO has more Good super capacitance performance.

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