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Electroexfoliated graphene for electro-responsive gel preparation

Zhifeng Wang[1,*], Zhu Yang[2], Pinzhang Chen[1]

[1]Beijing University of Chemical Technology, Beijing 102299, China
[2]Tongji University, Shanghai 201804, China
**Corresponding author: 2020020069@buct.edu.cn*

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Abstract: The existing graphene preparation methods are complicated, and the prepared graphene has poor conductivity and mechanical properties. In this experiment, electrochemical methods will be used to mechanically exfoliate graphene with good electrical conductivity and flexibility in a green and efficient way. There are hydrogel preparation methods, and the prepared hydrogels have poor electrical response and mechanical properties. In this experiment, we will use photoinitiated polymerization to prepare graphene composite hydrogels with excellent electrical responsiveness and mechanical strength.

1. Introduction

Graphene is a single layer of carbon atomic surface material exfoliated from graphite material, which has a broad development prospect in various fields. In particular, it is widely used as a conductive material in the field of electronics. This requires good electrical and mechanical properties of graphene itself.

Commonly used methods for the preparation of graphene include: graphite oxide reduction, epitaxial growth, chemical vapor deposition CVD, and micromechanical exfoliation. However, these methods suffer from the problems of introducing more oxide groups, difficulty in reducing the production cost and scaling up, small monolithic size, and long reaction time. Therefore, it is an important research direction in the field of graphene to prepare electrically exfoliated graphene with good electrical conductivity and flexibility in an efficient way.

Gels are hydrophilic polymers with reticulated cross-linked structure, which are soft in nature, can maintain a certain shape and can adsorb a large amount of water, and have a good prospect for future development. The existing gel applications are mainly focused on biology pharmacology and other aspects. The electro-responsive properties and mechanical properties of the gels have not been widely concerned, and it is worthwhile to study them in depth and develop their applications.

The existing thermal polymerization preparation method has a long reaction time, and the prepared hydrogels also have the problems of poor electrical response and mechanical strength. Therefore, it is very important to change the gel and improve the preparation method to improve the electro-responsiveness and mechanical properties of the gel.

2. Experimental part

2.1 Reagents and instruments

2.1.1 Experimental reagents

Tetramethylammonium bisulfate; Deionized water; Graphite paper.

2.1.2 Experimental instruments

Electrochemical workstation; Infrared spectrometer; TU-1910 UV-V is spect-rophotometer; Raman spectrometer; Scanning electron microscope; Electrophoresi pool.

2.2 Experimental procedure

2.2.1 Preparation of electroexfoliated graphene (EG)

Weigh 1.7122 g of tetramethylammonium bisulfate solid, dissolve, and transfer to a 100 mL volumetric flask for volume determination. Cut two strips of carbon paper about 1 cm wide by 7 cm long, weigh them and record them as m1. Turn on the electrochemical workstation and set the initial potential to 10 V using the Amperometrici-t Curve, the experiment time to 900 s, and the sensitivity to 1.e-001 A/V. Clamp one end of each strip of carbon paper to the counter electrode and the working electrode, and submerge the other end in the solution. , energized. Observe the surface morphology of the electrode and the change of the solution. After the reaction, the carbon paper strips were removed and placed in a Petri dish. The solution was filtered through a 500 nm microporous membrane, and the filter cake was washed with deionized water two to three times, and the filter cake was transferred to a Petri dish. The carbon paper strips and the filter cake were dried in an infrared oven for about 20 min. The mass m2 of the dried carbon paper strips was weighed, and the mass change $\Delta m1=m1-m2$ was calculated as a difference from the initial value. samples were taken for the characterization of Raman spectra.

2.2.2 Preparation of EG composite gel (10 mL)

In a 50 mL beaker, 1.45075 g 2-acrylamido-2-methylpropanesulfonic acid (AMPS, 0.7 mol/L), 0.2132 g acrylamide (AAm, 0.3 mol/L), 0.0771 g N,N'-methylenebisacrylamide (MBA, 0.05 mol/L) and 0.0449 g photoinitiator 2959 (2 % monomer molar ratio). Add 10 mL of N, N-dimethylformamide (DMF) to fully dissolve the solid, add 10 mg of the prepared electroexfoliated graphene, and sonicate the sample for 30 min to fully disperse it. The liquid was poured into a mold (medium-sized sample tube) and irradiated using a UV mercury lamp for 5-8 min to obtain the EG composite gel. The gels were washed 4-6 times with deionized water to remove as much DMF as possible. samples were taken for characterization by infrared spectroscopy, electron scanning microscopy, etc.

2.2.3 Validation of the electro-responsive properties of EG complex gels

Prepare 0.5 mol/L sodium sulfate (or sodium chloride) solution, and fix the gel in the solution along the direction perpendicular to the electric field and parallel to the ground. Using an electrophoresis power supply, etc., access a DC voltage of 30 V to 60 V and observe the deflection direction and deflection rate of the gel, etc.

3. Results and Discussion

3.1 Characterization of graphene

3.1.1 Characterization of electrically exfoliated graphene

Raman spectroscopy is a non-destructive and relatively effective means of characterizing the number of layers of graphene. The shape, width and position of the spectral lines correlate with the number of layers. Graphene has more distinct absorption peaks at about 1580 cm^{-1} (G peak) and 2700 cm^{-1} (2D peak) as the graphite body. Compared with the graphite body, the absorption peak at 1580 cm-1 is broadened and less intense, while the absorption peak at 2700 cm^{-1} is more intense, and the width and position of the absorption peak at 2700 cm^{-1} are slightly shifted for different layers of graphene. Figure 1 shows the Raman spectra of graphene and graphite.

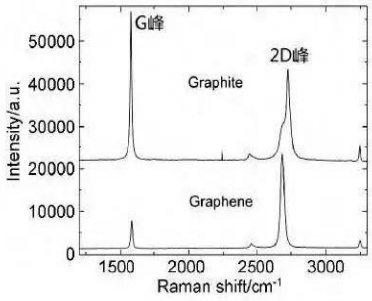


Figure 1 Raman spectrogram of graphene

3.1.2 SEM image of graphene

The number of graphene layers is related to the color and surface folds of the SEM image. The greater the degree of folds, the less the number of graphene layers. In the SEM image of our prepared electrically exfoliated graphene, it is possible to reach obvious uneven lamellae with folds and the number of lamellae is small. The diameter of single lamellae is about 1 μm.

3.2 Characterization of gels

3.2.1 Infrared spectra of gels

The wave numbers from 3500 to 3300 in the infrared spectrum represent the stretching vibrations of the N-H bond in the amide, and the wave numbers from 1700 to 1680 and 1660 to 1640 are the stretching vibrations of the carbonyl group in the free and bonded amide, respectively. Figure 2 shows the infrared spectra of the gels.

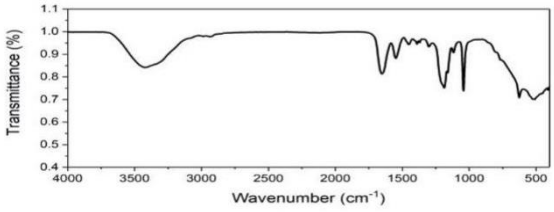


Figure 2 Infrared spectra of gels

3.2.2 SEM images of the gel

A distinct porous structure is observed in the SEM image of the graphene composite gel. As an

example, a porous structure with a length of 26.22 μm and a width of 15.44 μm was selected in Figure 3.

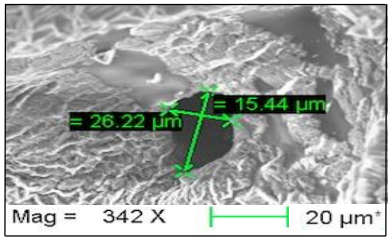


Figure 3 SEM image of the gel

3.3 Electroresponsive properties of electrically exfoliated graphene gels

3.3.1 Electrical response rate of electrically exfoliated graphene gels

Tests were performed in an electric cell containing 0.01 mol/L NaCl solution with an electric field strength of 3 V/cm. It was observed that the deflection angle of the gel loaded with EG (15 °) was 3 times higher than that of the gel not loaded with EG (5 °) for the same time (10 s). Deflection of the gel is shown in Figure 4.

Figure 4 Deflection of the gel

3.3.2 High pressure resistance of electroexfoliated graphene gel

Tests were performed in an electric cell containing 0.01 mol/L NaCl solution with an electric field strength of 13.5 V/cm. Immediate disintegration of the gel without EG loading was observed, while the gel loaded with EG deflected normally. Deflection of gel under high pressure is shown in Figure 5.

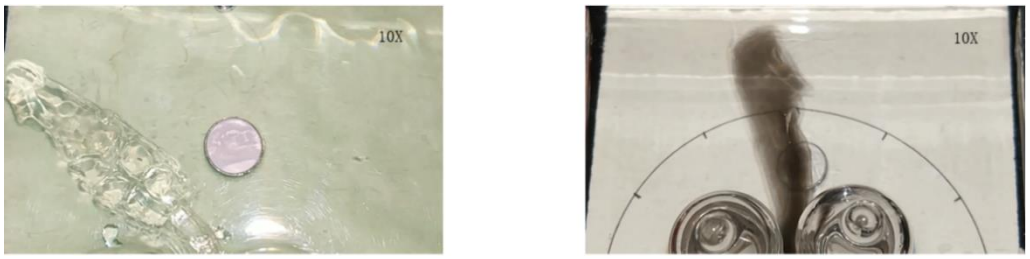
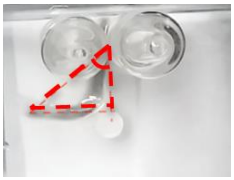
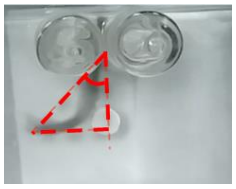
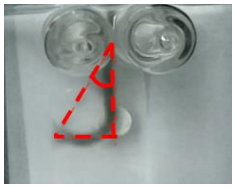
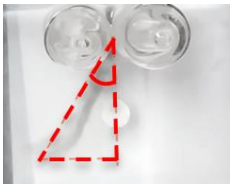


Figure 5 Deflection of gel under high pressure

3.3.3 Electrical exfoliation of graphene gel deflection angle versus voltage

We conducted a test of the deflection angle in 8s at different field strengths. It was observed that the bending angle increased with the field strength in a certain range. The relationship between the external field strength and the deflection angle of the gel is shown in Table 1.

Table 1 The relationship between the external field strength and the deflection angle of the gel

Field Strength (V/cm)	13.5	12.0	11.5	9.0
tan	1.5000	0.9529	0.6275	0.6241
Bending angle ( )	56.31	43.62	32.11	31.97
Schematic				

3.4 Application of electroexfoliated graphene gel

Through our experiments we found that the electroexfoliated graphene gel can be used as an electrically responsive switch. Figure 6 shows our test in a 0.01 mol/L NaCl solution at an electric field strength of 3 V/cm. It was found experimentally that the electroexfoliated graphene gel can push a metal cube with an area of about 1 cm2 and weighing 1 g.

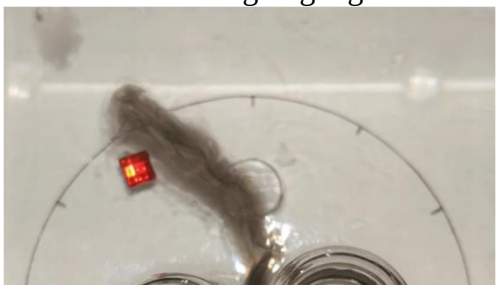


Figure 6 Gel pushes obstacles

4. Conclusion

In this experiment, graphene was prepared in a green and efficient way by mechanical exfoliation through electrochemical method. The graphene composite hydrogels with excellent properties were prepared by photoinitiated polymerization method. The prepared gels possess the electrical response property of directional bending in the electric field. Through comparative experiments, our prepared electroexfoliated graphene gels have better electrical response performance. The electrical response rate is fast, and the bending angle increases with the increase of voltage in a certain range. The mechanical strength of the electroexfoliated graphene composite gels was also improved, and the high voltage resistance was stronger than that of the ordinary gels. The prepared electrically exfoliated graphene gels can be applied to make electrically responsive switches.

5. Innovation points

5.1 Improved preparation conditions, simple, fast and safe operation

Green and efficient graphene exfoliation by electrochemical workstation and rapid polymerization of hydrogels by photoinitiation

5.2 Strong hydrogel performance

The mechanical strength and electrical response performance of electroexfoliated graphene composite gels are enhanced

5.3 Interesting, both basic and comprehensive

The experiments are simple and fast, and exercise fundamental operations. The experimental principle is based on several courses such as inorganic chemistry, physical chemistry, analytical chemistry and polymer chemistry. The experimental effect is interesting and obvious.

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