Research on Preparation and Plication of Superhydrophobic rGO Composite

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Abstract: rGO is widely used in aerospace, petrochemical, marine and other fields because of its high conductivity, high thermal conductivity, high specific surface area, high light transmittance and excellent mechanical properties. At the same time, due to its good hydrophobicity, it is not only easier to synthesize superhydrophobic materials, but also improve the mechanical stability and electrothermal properties of the composites, prolong the service life and ensure the service effect. In this paper, a modified Hummer method was constructed to prepare a superhydrophobic reduced graphene oxide (rGO) composite material. The synthesized rGO material was characterized by using an X-ray powder diffractometer, and the results proved the successful preparation of rGO. Scanning electron microscope (SEM) was used to test and analyze the prepared rGO composite material to further verify the successful preparation of superhydrophobic rGO. Finally, the influence of the amount of rGO on the wettability of the material was explored, and it was concluded that when the amount of rGO added was 7.5 mg/mL, the hydrophobic performance was the best.

Keywords: hummer method, rGO, superhydrophobic

1. Introduction

In recent years, the superhydrophobic coating has attracted widespread attention due to its excellent anti-icing, anti-fouling, self-cleaning, anti-corrosion, anti-drag and drag reduction properties, and has become a material science and surface material preparation technology in recent years.¹ Etching method, self-assembly method, sol-gel method, electrochemical deposition method, solution casting method, phase separation technology, template method and electrostatic spinning method, etc.² On this basis, it has a wide range of applications in the fields of anti-fouling and water resistance in buildings, improved buoyancy of ships, anti-fogging, anti-icing, fluid drag reduction, oil-water separation, slow drug release, and antibacterial.³

Although the application prospects of superhydrophobic materials are broad and there are many preparation methods, they are still subject to many restrictions in industrial production and real life applications.⁴ The main problems are that the mechanical stability is generally poor, the thermal stability of the surface coating is not high, materials and equipment problems such as high price and complicated process.⁵

In summary, it is very important to develop a super-hydrophobic surface preparation technology with high transparency, good appearance, relatively low cost, strong mechanical stability, simple preparation process, and suitable for industrial production.

rGO has attracted much attention in scientific research and technical application because of its ultra-thin and uniform two-dimensional morphology, excellent compressive strength and other physical and chemical properties, high carrier mobility and superior barrier ability to small molecules. In this paper, rGO was prepared by modified Hummer method, and the synthesized material rGO was characterized. In the experiment, the influence of the amount of rGO on the wettability of the material is explored, so as to obtain the best service conditions.
2. Experimental part

2.1. Experimental materials

2.1.1. Experimental reagents

Natural graphite powder (AR), concentrated sulfuric acid (H$_2$SO$_4$, AR), sodium nitrate (NaNO$_3$, AR), barium nitrate (Ba(NO$_3$)$_2$, (AR)), concentrated nitric acid (HNO$_3$, AR), concentrated hydrochloric acid (HCl, AR), potassium permanganate (KMnO$_4$, AR), hydrogen peroxide (30% H$_2$O$_2$, AR), deionized water (H$_2$O, AR).

2.1.2. Experimental equipment

Ultrasonic cleaner, magnetic stirrer, constant temperature water bath, analytical balance, suction filter device, high-speed centrifuge, multi-function oscillator, vacuum drying oven, X-ray powder diffractometer (XRD), etc.

2.2. Experimental process

2.2.1. Synthesis of rGO

First, the graphite powder and NaNO$_3$ are mixed and then added to the concentrated H$_2$SO$_4$. Then, under ice bath conditions, KMnO$_4$ was slowly added to the reaction solution while stirring. Subsequently, the temperature was controlled, and distilled water was slowly added to the reaction system. After the reaction, deionized water and H$_2$O$_2$ were added to the mixture while stirring, and the precipitate in the mixture was collected by centrifugation. Wash with hydrochloric acid solution and deionized water repeatedly until no sulfate ion is detected in the filtrate. The resulting product was vacuum dried for 24 h and ground into powder.

Take the above powder and prepare it into a solution, and add 0.5 mL NH$_3$·H$_2$O and 0.5 mL N$_2$H$_4$·H$_2$O to the solution according to the volume (mL) of NH$_3$·H$_2$O: the mass (mg) of GO is 1:24 and the volume ratio of NH$_3$·H$_2$O to N$_2$H$_4$·H$_2$O is 1:1. GO was reduced to rGO by reaction in 80 °C water bath, filtered, washed and dried for 12 h.

2.2.2. Material characterization

![Figure 1: XRD image of GO and rGO](image-url)

In order to prove the successful preparation of GO and rGO materials, the synthesized materials GO and rGO were characterized by X-ray powder diffraction (XRD). The phase composition of GO and rGO was studied by XRD. X-ray diffraction characterization and analysis experiments can accurately measure the changes of crystal parameters of samples. The XRD spectra of GO and rGO in the figure show that GO has an obvious and sharp strong diffraction peak at about 10°, while the reduced rGO has a diffraction peak at about 22°. This is consistent with the peak position and shape of rGO reported in the literature, which proves the successful preparation of rGO.
In order to further verify the successful preparation of superhydrophobic rGO composite materials, the rGO composite materials prepared were tested and analyzed by scanning electron microscope (SEM). SEM is also one of the means to characterize graphene morphology, and the number of graphene layers can be roughly observed through the surface wrinkles of the SEM image. It can be clearly seen from the figure that the prepared rGO has a regular sheet-like structure and morphology, indicating that it can provide a larger effective surface area and effectively increase the contact area with water. At the same time, its dense structure is not conducive to the entry of water, thereby enhancing the hydrophobic properties of the prepared rGO.

### 2.3. Analysis of the hydrophobic properties of rGO

In order to investigate the hydrophobic properties of different concentrations of rGO, rGO materials with different contents were prepared by controlling the amount of rGO added and using 0.2% CS (chitosan) as solvent, and coated on the surface of carbon steel to test their hydrophobic properties. The final CA values of the coating hydrophobic properties were 101.3 ° ± 2.7 ° (5 mg / mL), 113.7 ° ± 1.6 ° (10 mg / mL) and 123.4 ° ± 2.9 ° (7.5 mg/mL). When adding rGO, the CA value increases with the increase of the amount of rGO. When the amount of rGO is 7.5 mg / mL, it reaches the peak, and then the CA value decreases to a certain extent. The experiment shows that the effect is the best when the amount of rGO is 7.5 mg / mL.

#### Table 1: Corresponding contact angles of different concentrations of rGO

<table>
<thead>
<tr>
<th>Concentration of rGO</th>
<th>CA</th>
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</thead>
<tbody>
<tr>
<td>5 mg/mL</td>
<td>101.3 ° ± 2.7 °</td>
</tr>
<tr>
<td>7.5 mg/mL</td>
<td>123.4 ° ± 2.9 °</td>
</tr>
<tr>
<td>10 mg/mL</td>
<td>113.7 ° ± 1.6 °</td>
</tr>
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### 3. Experimental conclusion

In this paper, GO was prepared by improved Hummer method, and rGO was prepared by one-step reduction method. The synthesized rGO material was characterized by X-ray powder diffractometer, which proved the successful preparation of rGO. Scanning electron microscope (SEM) was used to test and analyze the prepared rGO composite material to further verify the successful preparation of superhydrophobic graphene oxide composite material. In the experiment, the influence of the amount of graphene oxide on the wettability of the material was explored, and it was concluded that when the amount of rGO added was 50%, the hydrophobic performance was the best.
References