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Research on the Preparation of Graphene Aerogel Materials from Graphene Oxide Reduced by Vitamin C

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Abstract:

This study focuses on the preparation of graphene aerogels with a porous structure using graphene oxide as the precursor and vitamin C as the reducing agent. The reduction of graphene oxide is achieved by heating a solution containing vitamin C, followed by drying to obtain the graphene aerogel. The resulting material exhibits a low density and some compressive strength. Various characterization tests, including oil-water separation and combustion experiments, demonstrate the aerogel's excellent oil absorption and combustion properties. The oil adsorption capacity of the graphene aerogel was found to average 50 mg/mg. These findings suggest that the prepared graphene aerogels possess remarkable oil absorption capabilities, efficient oil-water separation performance, and can be regenerated through combustion, highlighting their promising potential for diverse applications.

Keywords: Ascorbic acid, Graphene oxide, Graphene aerogel, Oil-water separation, Flame resistance

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1. Introduction

Aerogels are materials characterized by a three-dimensional nanoporous structure. When the liquid within the gel's pores is replaced by air, the resulting aerogel maintains the typical pore and network structure. These materials exhibit extremely low density (with up to 95% of their volume composed of air), large open pores, and a high internal surface area. [1] In recent decades, oil spills in marine environments have caused significant pollution, severely impacting coastal ecosystems. Although various methods, such as combustion, chemical treatments, and bio-degradation technologies, have been developed for oil spill remediation, these conventional techniques either have negative ecological impacts or require substantial energy consumption. [9] Recently, the use of oil-absorbing materials has emerged as one of the most effective strategies for oil spill management due to their low production costs, ease of manufacture, environmental sustainability, and excellent recyclability. [10] In the context of global industrial modernization, the release of toxic, non-biodegradable heavy metals from industries like metal processing has become a significant concern. As a result, researchers have developed environmentally friendly aerogels capable of adsorbing heavy metals. [2] Beyond oil spill management, aerogels show considerable potential in other fields. For example, carbon nanotube aerogels, with their high-temperature resistance and superior thermal insulation properties, have found applications in fire-retardant and heat-insulating domains. [3] The successful design of ceramic nanofiber aerogels has also opened new possibilities for the development of next-generation, high-performance sound-absorbing materials. [5]

Most research on aerogel synthesis has focused on silica-based aerogels with large pores and hydrophobic properties. [6] However, with the continued advancement of aerogel fabrication technologies, a variety of other aerogel materials have been successfully synthesized, including inorganic aerogels (e.g., SiO2, TiO2, Al2O3, ZrO2 derived from various alkoxysilanes), organic aerogels (e.g., polyurethane, polyimide, polystyrene), carbon aerogels (e.g., carbon nanotube aerogels with large specific surface areas, oil-absorptive and hydrophobic properties [7], graphene aerogels), and natural polymer-based aerogels (e.g., cellulose, other polysaccharides, and aerogels synthesized from various proteins). [8] Given the remarkable mechanical strength, low density, excellent elasticity, electrical conductivity, and exceptional transparency of carbon nanotubes and graphene, these materials are considered the ideal candidates for the development of ultra-light, elastic, and conductive aerogels. [11]

Kabiri et al. synthesized a graphene-carbon nanotube aerogel with superhydrophobic and oleophilic properties by heating a solution containing graphene oxide, carbon nanotubes, and iron ions. [13] While this aerogel material exhibits high mechanical strength, it is associated with high production costs. [14] Wang et al. developed a fire-retardant, wave-absorbing graphene oxide/phenolic resin (RGO/PR) hybrid aerogel by diluting a graphene oxide solution in ethanol and adding hexamethylenetetramine (HMTA) in specific proportions, followed by aging and drying processes. [15] Chen et al. synthesized a GA/NiFe2O4/ZIF-8 composite aerogel using ascorbic acid as a reducing agent and graphene oxide as the aerogel framework. They employed a cost-effective, simple, and environmentally friendly hydrothermal method, resulting in a composite aerogel with high adsorption capacity and thermal stability. [16]

In this study, graphene oxide (GO) was prepared via the Hummers method [12] and mixed with ascorbic acid to form a GO/VC solution. After water bath heating and freeze-drying, reduced graphene aerogels were obtained and characterized. The oil-water separation mechanism of the graphene aerogel material was investigated, and repeated oil absorption and combustion experiments were conducted to determine its adsorption capacity, recyclability, and thermal stability.

2. Materials and Method

2.1 Experimental Instruments and Reagents

Graphene Oxide, Ascorbic Acid, Paraffin, Kerosene, Perchloroethylene, Methylene Blue

2.2 Materials Preparation

A 100 mL solution of graphene oxide (GO) with a concentration of 2 mg/mL was mixed with 1 g of ascorbic acid (vitamin C) in a mass ratio of 1:5 to prepare the GO/VC solution. The mixture was then heated in a water bath at 80°C for a predetermined period to facilitate the reduction of graphene oxide. The solution was then subjected to freezing to promote gelation. After freezing, the sample was reheated for a total duration of 60 minutes to allow the formation of a graphene hydrogel. The resulting hydrogel was subsequently dried in both a freeze-dryer and an oven at 60°C, yielding a porous, cylindrical graphene aerogel.

Figure 1. Preparation Process for Graphene Hydrogel and Graphene Aerogel

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3 Result and Discussion

3.1 Performance Investigation

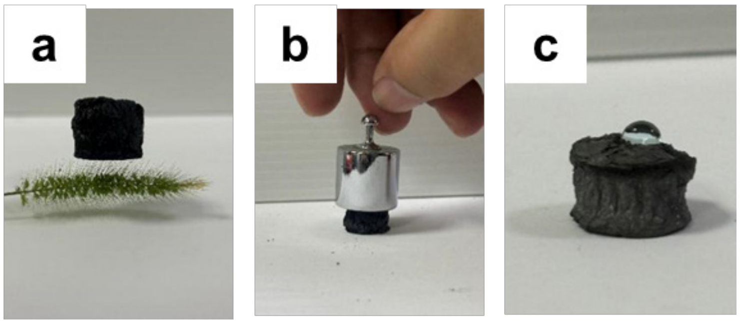

Figure 2. Demonstration of (a) Ultra-light Material Properties (b) Compression Resistance (c) Hydrophobicity

As shown in Figure 2(a), the graphene aerogel material is supported by the dogtail grass fibers, and this support structure demonstrates the exceptional mechanical properties of the graphene aerogel. In the 100 g weight compression test, the aerogel exhibited significant compressive strength, indicating its ability to maintain structural stability under external loads without noticeable deformation (Figure 2(b)). This suggests that the aerogel not only possesses excellent compressive strength but also demonstrates high elasticity, enabling it to quickly return to its original shape after experiencing external forces, ensuring its reliability during long-term use.

Furthermore, the hydrophobicity test (Figure 2(c)) further confirms the superhydrophobic characteristics of the aerogel. Due to its unique microstructure, the aerogel effectively repels water, exhibiting a very low surface energy that prevents water absorption. This hydrophobic property ensures that the aerogel maintains its performance even in humid environments, avoiding a decline in physical properties due to water absorption.

3.2 Thermal Insulation Investigation

The objective of this experiment is to comprehensively assess the thermal insulation performance of graphene aerogel, particularly its behavior under high-temperature conditions. A high-temperature airflow (produced by a heat gun) was applied to one side of the graphene aerogel sample, while an infrared thermometer was used to monitor the temperature variation on both sides in real time.

In the experiment, one side of the graphene aerogel sample was directly exposed to the high-temperature airflow, while the other side remained unheated. Using the infrared thermometer, we were able to observe the temperature difference between the two sides. As the heat gun raised the temperature on the heated side, it eventually reached 800°C, while the unheated side maintained a stable temperature of 30-40°C, with almost no significant change.

Figure 3. Thermal Insulation demonstration

This demonstrates the exceptional thermal insulation properties of the graphene aerogel. Under high-temperature conditions, graphene aerogel effectively blocks the transfer of heat, preventing the high temperature from spreading from one side to the other. Due to the aerogel's unique structure—characterized by its extremely high specific surface area and high porosity—it significantly reduces thermal conductivity, exhibiting strong thermal insulation capabilities. This makes graphene aerogel an ideal material for protecting underlying materials or systems from overheating and potential damage in high-temperature environments.

Furthermore, the excellent thermal insulation performance of graphene aerogel is closely related to its material properties. While graphene itself has good thermal conductivity, the heat transfer within the aerogel structure is greatly reduced due to the presence of bubbles and voids. The aerogel's porosity and low density make it an ideal thermal insulator, a characteristic that holds significant practical value in many applications requiring efficient thermal management.

3.3 Oil-Water Separation Experiment

The core objective of this experiment is to validate the practical application of graphene aerogel in oil-water separation, particularly under simulated marine conditions. To achieve this, heavy oil and perchloroethylene were added to water, with methylene blue used for staining to simulate marine pollution. This setup ensured the visualization of the oil-water mixture, facilitating subsequent observation and analysis (Figure 4(a)). Based on this foundation, the experiment focused on evaluating the oil-water separation efficiency of graphene aerogel under various environmental conditions.

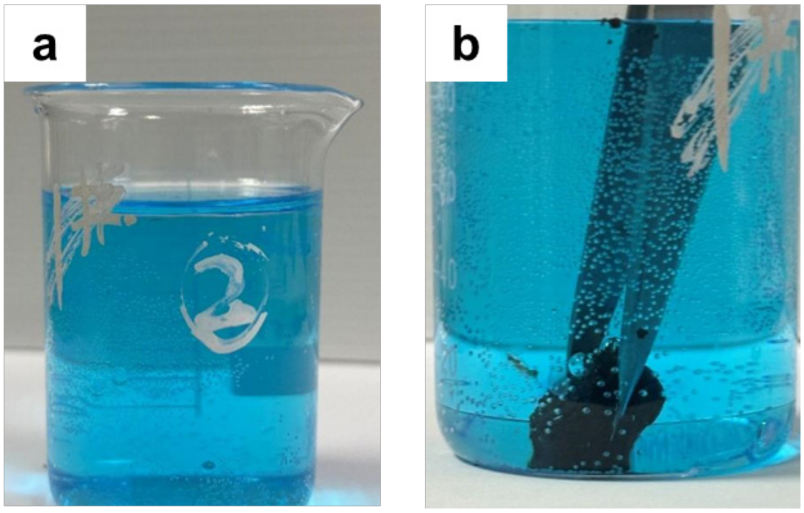

Figure 4. (a) Simulated seawater stained with methylene blue. (b) Aerogel sinking to the bottom of the beaker for oil adsorption.

During the experiment, graphene aerogel demonstrated exceptional oil adsorption performance due to its unique high specific surface area and porous structure. The aerogel rapidly adsorbed heavy oil on the water surface and at the bottom of the beaker. Through repeated oil adsorption and combustion cycles, the heavy oil in the water was effectively removed without causing pollution or damage to the water quality. Notably, the aerogel maintained its high oil adsorption efficiency even after multiple cycles, indicating its strong reusability and stable performance over time.

Moreover, the aerogel's superhydrophobicity and oleophilicity were key factors in its efficient oil-water separation. These surface characteristics enabled the aerogel to selectively adsorb oil while repelling water, ensuring that the water quality remained unaffected and avoiding secondary pollution during the separation process.

The experimental results demonstrated that graphene aerogel is not only highly effective in oil-water separation but also capable of completely removing heavy oil without compromising water quality. This high efficiency, combined with its reusability, highlights the aerogel's significant potential for practical applications, particularly in addressing complex pollution scenarios such as marine oil spills and industrial wastewater treatment.

3.4 Combustion Cycle Experiment

In this experiment, graphene aerogel was fully immersed in kerosene until saturation was achieved. The mass of the aerogel was measured before and after immersion to evaluate its oil adsorption capacity. To simulate the repetitive usage cycles encountered in practical applications, the oil-saturated graphene aerogel was placed on a copper mesh and subjected to combustion. This step aimed to assess its stability and performance recovery under high-temperature conditions.

Following the combustion process, the graphene aerogel was re-immersed in kerosene, and the cycle of adsorption and combustion was repeated ten times. The changes in the aerogel's oil adsorption capacity during these repeated cycles were monitored to determine its durability and reusability in oil-water separation applications.

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Table 1.1 Parameters in the Combustion Cycle Experiment

Mass	23 mg
Radius	0.75 cm
Height	1.1 cm
Volume	1.943 cm^3
Sample Density	11.84 mg/mL
Kerosene Density	800 mg/mL
Absorption Time (until saturation)	7.85 s
Combustion Time	100.75 s

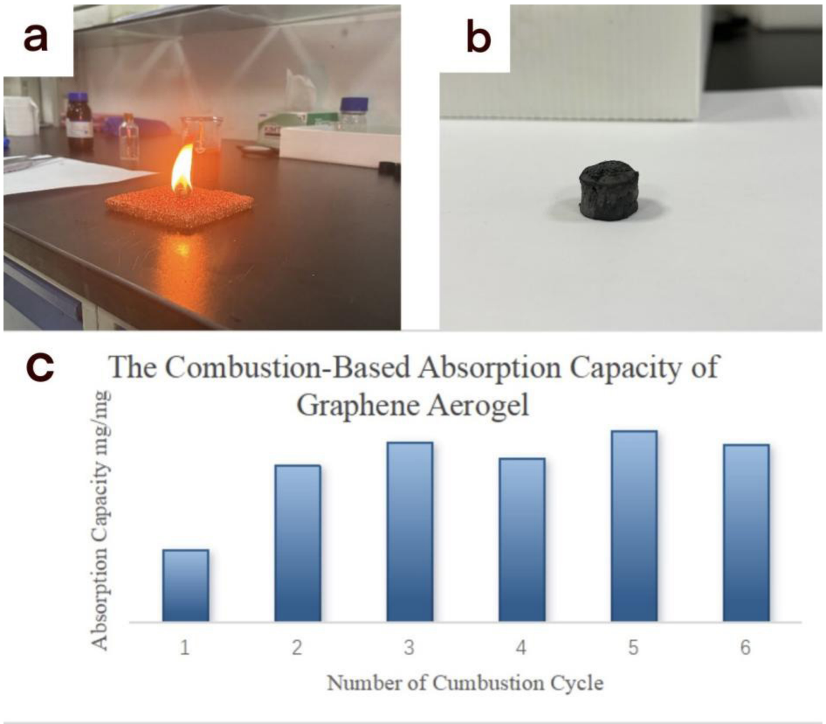

Figure 5. (a) Combustion of graphene aerogel in a saturated state (b) Graphene aerogel after combustion (c) Combustion-based adsorption capacity of graphene aerogel

The experimental results indicate that graphene aerogel exhibits a very high adsorption capacity, and its adsorption performance remains stable during multiple cycles of oil absorption and combustion (Figure 5(c)). This phenomenon suggests that graphene aerogel can effectively maintain its adsorption ability under high-temperature conditions, demonstrating excellent durability and repeatability. Moreover, after several cycles of combustion and oil absorption, the appearance of the graphene aerogel showed no significant changes (Figure 5(b)), further proving its exceptional high-temperature resistance.

During the combustion process, except for the first combustion, where the mass of the graphene aerogel increased compared to the original mass, the mass after each subsequent combustion was lower than the original mass. This change can be attributed to the inclusion of ascorbic acid in the preparation of the graphene aerogel. During combustion, ascorbic acid decomposes, leading to a reduction in mass. However, this mass change did not affect the oil absorption capacity of the graphene aerogel, indicating that it still effectively performs oil-water separation and adsorption tasks.

3.5 Water Retention Rate after Drying of Graphene Hydrogel

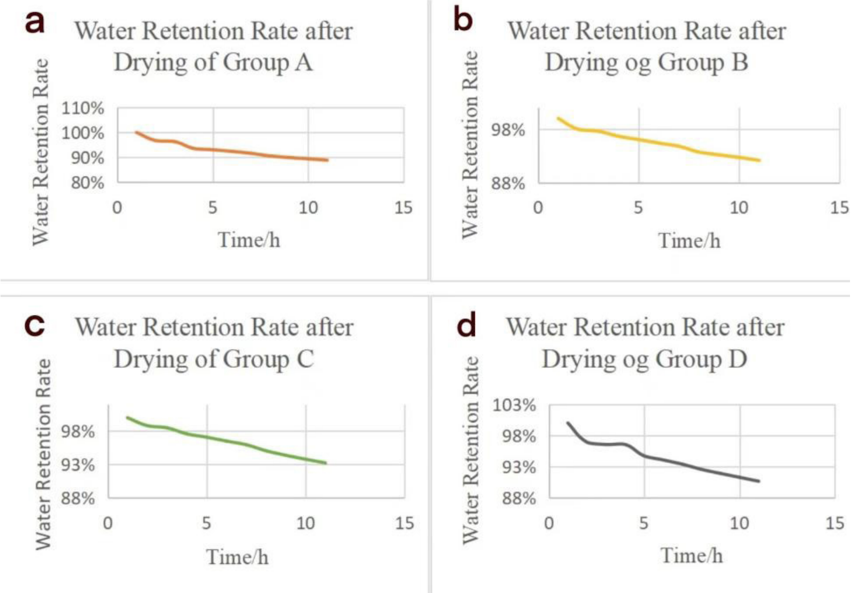

Figure 6. The drying process of graphene hydrogel sample a, b, c, d demonstrates that the graphene hydrogel has a good water retention rate.

The experimental data indicates that during the drying process of graphene hydrogel at 60°C, only approximately 10% of the water evaporates over a duration of 9 hours. This result suggests that the moisture within the porous material does not simply undergo rapid evaporation, but rather remains effectively encapsulated within the material's pore structure, thus being well-protected and preventing rapid volatilization under elevated temperatures.

This phenomenon highlights the unique characteristics of porous materials, such as graphene aerogels, in moisture retention. The porous architecture provides a network of microvoids capable of efficiently capturing and retaining water, thereby slowing its evaporation rate. As a result, even in high-temperature environments, the moisture exhibits prolonged stability.

4. Summary

This study uses graphene oxide as the framework material for aerogels and ascorbic acid as a reducing agent to prepare graphene aerogels. The graphene aerogel material with a porous structure was successfully synthesized through freeze-drying and drying methods. Experimental results show that the prepared graphene aerogel exhibits ultra-lightweight, hydrophobic, compressive, and excellent thermal insulation properties. Furthermore, after over 10 cycles of oil absorption and combustion, the graphene aerogel retains stable adsorption performance, demonstrating significant adsorption capacity and good thermal stability. Overall, graphene aerogel excels in multiple key properties, offering broad application prospects, particularly in fields such as oil-water separation, high-temperature insulation, and environmental protection.

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