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The Preparation of Compressible and Fire-Resistant Sponge-Supported Reduced Graphene Oxide Aerogel for Electromagnetic Interference Shielding

Changzhen Liu, Shibing Ye, Jiachun Feng*

Abstract: We here report a facile method to fabricate sponge-supported reduced graphene oxide aerogel (S-RGOA) using commercial melamine sponge and graphene oxide (GO). Firstly, GO sheets were self-assembled within the melamine sponge by the assistance of chemical cross-linking agent; and then, freezing-drying and thermal treatment were adopted to prepare S-RGOA, in which continuous porous reduced graphene oxide (RGO) network formed between the skeleton. The resulting S-RGOA exhibits a high electromagnetic interference shielding effectiveness (EMI SE) of 20.4-27.3 dB in 8-12 GHz and the specific EMI SE could reach 1437 dB-cm²/g. Mechanical test suggests that the lightweight S-RGOA is compressible and possesses low energy dissipation. Burning and TGA measurements indicate that S-RGOA is fire-resistant and has excellent thermal stability. Our work provides an economical and environmentally friendly method to fabricate RGO aerogels for using as electromagnetic interference materials.

Introduction

Graphene aerogels, which are typical three-dimensional (3D) porous materials constructed by graphene sheets, possess fascinating features including low density, high porosity, large internal surface area, and high electrical conductivity. Those unique properties lead to their great potential for various practical applications, such as energy storage, supercapacitors, absorptions and microwave shielding materials. Due to the promising applications, many researchers have paid attention to exploring the methods of fabricating graphene aerogels. Approaches such as chemical vapor deposition (CVD) over a porous template, one-step hydrothermal method, and chemically converted technique were miraculously created to prepare graphene aerogels with well-defined and interconnected 3D porous network. These approaches open the door of fabricating graphene aerogels, but some crucial obstacles with the prepared graphene aerogels are still remained to be solved. For instance, CVD-grown graphene aerogel is usually fragile and will prone to collapse when removing the thicker metal template while aerogels prepared by hydrothermal and chemically converted methods are easy to crack due to that different cooling rates and freezing sequences of external and internal part may damage the integrated structure. Therefore, developing more simple methods to prepare 3D porous graphene aerogels, which keep good strength to avoid collapsing in preparation or application, is still a challenging topic.

Polymer-based sponges, which have good strength and porous structure, are usually used as cleaning, soundproofing and packaging materials. Recently, graphene composites based on polyurethane (PU) sponge have drawn wide attention. With the introduction of sponge, the 3D network characteristic of sponge and the advantages of graphene sheets are combined together. For examples, Jiang et al. and Yu et al. creatively coated graphene sheets around the skeleton of sponge by infiltration and drying, realizing the preparation of mechanically flexible graphene composites. Similarly, Yang et al. successively synthesized a graphene-silicon network for lithium storage, utilizing a dip-coated method based on commercial sponge. Inspired by these studies that graphene sheets were tightly coated “around” the skeleton of sponge to obtain composites with excellent special properties, we expect that if reduced graphene oxide (RGO) aerogel was formed “between” the skeleton of sponge, the composites may inherit the advantages of commercial sponge and the unique properties of RGO aerogel. Correspondingly, the collapsing problems may be partially solved. However, to the best of our knowledge, there are still no attentions on this subject in the open literatures. With the rapid development of electronic devices, lightweight electromagnetic interference (EMI) shielding materials have drawn continuous attention. Polymer foaming and formation of porous network are commonly used to lower the weight of EMI shielding materials. Recently, lightweight graphene based composites has been intensively explored in EMI shielding areas for its large aspect ratio and good electrical conductivity. For example, the preparation of ultra-light graphene foam and aerogel with high EMI shielding performance have been reported. Enlightened by these studies, preparation of RGO aerogel and sponge composites may endow EMI shielding materials with lightweight and good mechanical properties.

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In this work, we report a facile method to fabricate sponge-supported reduced graphene oxide aerogel (S-RGOA) with good EMI shielding performance, by utilizing the unique structure and property of sponge. Melamine sponge, which owns good fire-resistant performance and mechanical strength, was selected as the skeleton. Graphene oxide (GO) sheets were self-assembled into a 3D RGO network between the skeleton of melamine sponge by freeze-drying and thermal treatment. The resultant S-RGOA demonstrates a high electromagnetic interference shielding effectiveness (EMI SE) of 27.3 dB and the specific EMI SE could reach 1437 dB·cm²/g. Moreover, S-RGOA is lightweight, compressible, fire-resistant, and thermal stable, which has great potential for its practical application.

Results and Discussion

Fabrication and structural analysis of S-RGOA. The synthesis process of S-RGOA is illustrated in Figure 1. Firstly, melamine sponge was infiltrated in graphene oxide (GO, TEM image of a piece of GO is shown in Figure S1) mixed solution with polyetheramine; secondly, the product was hermetically reacted and freeze-dried to prepare precursory sponge-supported reduced graphene oxide aerogel (P-S-RGOA); finally, after thermal treatment, sponge-supported reduced graphene oxide aerogel (S-RGOA) with porous RGO aerogel between the skeleton was prepared. Melamine sponge was white while P-S-RGOA is black. The color change indicated the introduction and primary reduction of GO sheets (Figure S2). The overall framework of resultant S-RGOA showed a slight shrinkage compared with P-S-RGOA after thermal treatment. Melamine sponge helped the composites keep good integrity during freeze-drying and thermal treatment. Since sponge acting as the role of skeleton, the dimension of S-RGOA is adjustable based on the shape of melamine sponge.

In order to explore the mechanism of forming S-RGOA, the morphologies and structures of sponge, P-S-RGOA and S-RGOA are investigated by microscopic observation. As shown in Figure 2a, melamine sponge shows an interconnected and porous structure with macropores about several hundred micrometers. After infiltration, reaction, and freeze-drying, P-S-RGOA was prepared by assembling GO sheets into 3D networks among the skeleton of sponge (Figure 2b). Thermal treatment of P-S-RGOA conducted the preparation of S-RGOA, in which RGO aerogel was formed between the skeleton of melamine sponge (Figure 2c). As shown in Figure 2d, the RGO aerogel with micropores was formed in the macropores of melamine sponge. The skeleton of S-RGOA is coarse and wrinkling with RGO sheets well packaged (Figure 2e); in contrast, the surface of melamine sponge after thermal treatment (T-Sponge) is smooth and flat (Figure 2f). The SEM observations fully demonstrate that the RGO sheets assemble into RGO aerogel between the skeleton of the sponge. In the final S-RGOA, the RGO aerogel and the skeleton form an integrated composite (The SEM images of T-Sponge and RGO aerogel is shown in Figure S3).

The elemental compositions of P-S-RGOA and S-RGOA are exhibited by Figure 3a. The percentages of N in P-S-RGOA and S-RGOA are 12.3 and 11.4 %, originating from the constituents...
of melamine sponge. The C/O ratio of P-S-RGOA is 3.5 while the C/O ratio of S-RGOA is 9.0. In the C 1s spectra of P-S-RGOA (Figure 3b), there are four configurations centering at 283.3 eV (C=O), 284.6 eV (O=C=N), 285.2 eV (C–O), and 286.8 eV (C=C). As comparison, for S-RGOA (Figure 3c), the peak intensity ratio of C-C increased while the oxidation groups (C=O, O=C=C, O–C=O) decreased. The high C/O ratio and low intensities of the oxygen-contained peaks further confirm the removal of oxygen groups during reduction. And the increase of sp² carbon peak reveals a high degree reduction of GO. The Raman spectra of P-S-RGOA and S-RGOA is shown in Figure 3d, there are two peaks at 1341 and 1586 cm⁻¹, which should be assigned to D band and G band of graphene. Compared with the lower ratio of D and G bands (I_D/I_G) of P-S-RGOA, the value of S-RGOA is increased to some extent, indicating that more sp² carbon domains are recovered and the chemical reduction and thermal treatment. X-ray diffraction (XRD) patterns of P-S-RGOA and S-RGOA are shown in Figure 3e. No diffraction peak of GO at about 11° was found in the patterns of P-S-RGOA, indicating the primary reduction of GO sheets. Unlike P-S-RGOA, the pattern of S-RGOA exhibits an obvious peak at about 26°, demonstrating the formation of graphitic structures and confirming that GO is transformed into RGO in S-RGOA. FT-IR test of RGO, which was prepared with identical chemical reduction and thermal treatment as S-RGOA, also demonstrate that reduction of GO (Figure S4). In order to investigate the carbonization degree of melamine sponge, TGA curves of pristine melamine sponge and T-Sponge in N₂ atmosphere are shown in Figure 3f. For pristine melamine sponge, it starts to lose weight with a high speed from 350.7 °C, indicating that, at the 500 °C thermal treatment step during our preparation of S-RGOA, the carbonization of melamine sponge is already occurred. In comparison, for T-Sponge, the weight loss is rather slight at the temperature lower than 500 °C. However, it still keeps losing weight with a high speed from 514.4 °C to 800 °C, indicating that melamine sponge after thermal treated at 500 °C may be further carbonized. Combining the changes of appearance (Figure S5) and TGA curves of melamine sponge and T-Sponge, we can conclude that the melamine sponge in our S-RGOA is partly carbonized after thermal treated at 500 °C in Ar for 0.5 h. The part of melamine sponge which is not carbonized may be important for the strength of melamine sponge after thermal treatment.

![Figure 3.](image_url)

**Figure 3.** (a) XPS survey spectra of P-S-RGOA and S-RGOA. (b) C 1s spectra of P-S-RGOA. (c) C 1s spectra of S-RGOA. (d) Raman spectra of P-S-RGOA and S-RGOA. (e) XRD patterns of P-S-RGOA and S-RGOA. (f) TGA curves of pristine melamine sponge and melamine sponge after thermal treatment (T-Sponge) in N₂ atmosphere.

**EMI shielding performance of S-RGOA.** According to previous researches, materials with 3D porous RGO network structure usually show a good electromagnetic interference shielding effectiveness (EMI SE). [50, 29, 11d, 13a, 15] For our sponge-supported reduced graphene oxide aerogel, EMI shielding performances were investigated in the frequency of 8-12 GHz. As shown in Figure 4a, melamine sponge after thermal treatment (12 mm thickness) exhibits hardly any EMI SE, which indicates that the paraffin and the skeleton of melamine sponge are transparent to electromagnetic waves. In contrast, with the special structure formed by the synergistic effect of RGO aerogel and melamine sponge, the average EMI SE of S-RGOA (12 mm thickness) is found to be about 23.2 dB, higher than the requirement of 20 dB for commercial EMI shielding materials. [16]

Generally, for EMI shielding composites, the types and content of filler, sample thickness, filler architecture, and electrical conductivity are important factors to influence the performance of EMI SE. [17] For our S-RGOA, inner RGO sheets...
with large aspect ratio, defects and high polarity offer more opportunity to induce the polarization of electromagnetic waves. We also prepared the reduced graphene oxide coated sponge (S-RGOC, Figure S6) using same GO concentration with the preparation of S-RGOA. Compared with the EMI SE of 23.2 dB for S-RGOA, the value is only 17.0 dB for S-RGOC with same thickness (12 mm). The improved EMI SE of S-RGOA may be due to that more porous structure based the inner RGO aerogel brings more interfaces to scatter waves. When changing the thickness of S-RGOA (prepared with 2 mg/mL GO solution) from 3 to 12 mm, the average EMI SE is increased from 2.4 to 13.3 dB (Figure 4b). This may be attributed to that more interfaces and propagation paths in thicker sample induced more attenuation of electromagnetic waves. Similarly, as for RGO content, the EMI SE of S-RGOA (prepared with 2 and 4 mg/mL GO solution, 12 mm thickness) is increased from 13.3 dB to 19.5 dB (Figure 4c). With 3D RGO aerogel formed in S-RGOA, oscillatory current consumes substantial electromagnetic wave energy and conductivity (3.59 S/m for S-RGOA) plays the main role towards electromagnetic wave attenuation. As a consequence of these factors, our S-RGOA exhibits a commercial available EMI performance.

Figure 4. (a) EMI SE of Sponge after thermal treatment (T-Sponge), S-RGOC, and S-RGOA in the frequency of 8-12 GHz (12 mm thickness). (b) EMI SE of S-RGOA (prepared by infiltrating melamine sponge in 2 mg/mL GO solution) with different thickness (3, 6, and 12 mm thickness). (c) EMI SE of S-RGOA (prepared by infiltrating melamine sponge in 2 and 4 mg/mL GO solution, 12 mm thickness) (d) SE, SE, and SE of S-RGOA in the frequency of 8-12 GHz. (e) Schematic description of electromagnetic wave transfer across S-RGOA. (f) Comparison of specific EMI shielding effectiveness of S-RGOA with other reported results.

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Considering eco-friendly issues, an EMI shielding material with strong absorption and weak reflection is important for ideal lightweight EMI shielding materials. Usually, the incident power of electromagnetic wave is divided into absorbed wave, reflected wave, and transmitted wave. The total EMI SE (SE T) is the sum of all attenuating mechanisms, consisting of absorption (SE A), reflection (SE R), and the multiple reflection (SE M). It can be expressed by the equation: SE T = SE A + SE R + SE M. The SE M is usually neglected when SE T > 10 dB. Based on this point, SE A and SE R from 8 GHz to 12 GHz are calculated from the measured scattering parameters (S 11 and S 21). For the S-RGOA, the absorption mainly contributes to the shielding effect while the reflection nearly can be neglected (Figure 4d). For example, the SE T, SE A, and SE R are 27.3, 27.0, and 0.3 dB for S-RGOA at the frequency of 9.4 GHz. The absorption could be explained by two aspects. On one hand, for RGO aerogels, the functional groups and defects enhance the polarization loss. Since the dielectric loss induced by polarization loss, the adsorption correspondingly increased. On the other hand, as Shen et al. proposed, the porous structure of RGO aerogel in S-RGOA provided a large interface area (Figure 4e). Electromagnetic waves entering S-GA could be scattered in the hole. Two parallel RGO sheets may reflect and scatter the incident waves many times, increasing their propagation paths, which could further increase the absorbing ability. Therefore, most of the incident microwaves are dissipated as heat through the porous S-RGOA.

Notably, the S-RGOA has a very low density, which is measured to be 19.0 mg/cm 3. The good EMI SE combined with low density endows S-RGOA a superior specific EMI SE of 1437 dB·cm 3/g in the X-band frequency range (Figure 4f). The value is higher than typical metals,[20] graphene foam-based materials,[13a, 11d, 21] and recent sponge composites coated with Ag nanoparticles.[2f] The high EMI SE and absorption-dominant feature of lightweight S-RGOA make it have great potential to be used in aircraft and building materials areas.

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RGOA at $\varepsilon = 50$ and 70% is also investigated (Figure 5c and d), relatively small residual strains of 10 and 15% are found to appear respectively, exhibiting that S-RGOA endows a good elastic recovery even at relatively large strain (Compression stress-strain curve with a strain further increased to 90% is shown in Figure S7). The S-RGOA even did not collapse even with a strain of 95% (Figure S8). In Figure 5e, a set of digital images show the recovering process of S-RGOA with a strain of 70%. The good recovery property of the flexible S-RGOA may be attributed to the partial carbonization of melamine sponge skeleton after thermal treatment.

To better understand the mechanical stability of S-RGOA, the hysteresis loops are also investigated by cyclic tests as depicted in Figure 5. The hysteresis loop area of S-RGOA with strains of 10, 30, 50, and 70% is big during the first cycles. The deformation in first cycles involves more buckling and bending of RGO aerogel and melamine sponge skeleton, which may cause mutual contact, collision, and zipping between the 3D networks. After the first cycles, the hysteresis loop area drastically reduce. The reduced loop area indicates that the related energy dissipation processes are substantially inhibited. This low energy dissipation characteristic of S-RGOA, which may originates from the special 3D structure, makes it different from other 3D networks which show large stress-strain hysteresis. The compression curves demonstrate that S-RGOA holds not only good elasticity but also wonderful mechanical stability.

Fire-resistance and thermal stability of S-RGOA. In some harsh conditions, electromagnetic interference shielding materials may need to be fire-resistant. Alcohol burner burning tests were used to study the fire-resistance of melamine sponge (Figure 6a) and S-RGOA (Figure 6b). Melamine sponge and S-RGOA were put on the fire of alcohol burner for 60 seconds and then moved away. The flame did not self-propagate for both melamine sponge and S-RGOA. Compared with melamine sponge, after alcohol burner burning tests, S-RGOA better maintained its pristine shape. In order to further investigate the fire-resistance of S-RGOA, vacuum oil absorption/burning tests were adopted and shown in Figure 6c (Considering the porous structure of S-RGOA, its adsorption capacity for different solvents was investigated as depicted in Figure S9). After absorbing enough vacuum oil, S-RGOA was easily ignited and the fire was fierce. The fire spontaneously extinguished when vacuum oil was burned away. As shown in Figure 6c, after 5 cycles of vacuum oil absorption/burning tests, S-RGOA retains about 80% of its first absorption capacity, and still maintains the 3D structure with only a little shrinkage. Alcohol burner burning and vacuum oil burning tests clearly demonstrate that S-RGOA has high fire-resistance. This may be attributed to the special structure which forms RGO aerogel between the skeleton of melamine sponge. The fire-resistance feature of S-RGOA is vital for its practical use in daily life considering that some petroleum-based polymeric foams are easily ignitable or the addition of flame retardants may bring negative environmental and health impacts.

Thermal stability is also important for electromagnetic interference shielding materials. To study the thermal stability of our samples, TGA measurements were carried out in air atmosphere (Figure 7). As shown in Figure 7a, the temperature of 5% weight loss for melamine sponge is 307 °C, indicating that melamine sponge is relatively thermal stable. In contrast, this temperature for S-RGOA is 479 °C, 172 °C higher than that of melamine sponge. Similarly, the temperature of maximum weight loss rate for S-RGOA is 568 °C, 186 °C higher than melamine sponge (Figure 7b). These results clearly demonstrate that S-RGOA possesses excellent thermal stability, which is vital for its use in practical applications.

Conclusions

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In summary, we have demonstrated a facile method to fabricate melamine sponge-supported reduced graphene oxide aerogel with special structure by directly forming RGO aerogel between the skeleton of melamine sponge. Porous structure of melamine sponge creates space for the formation of inner RGO aerogel. By wrapping some RGO sheets connected with RGO aerogel around skeleton of melamine sponge, the inner RGO aerogel and sponge form the integral S-RGOA. Notably, S-RGOA exhibits a high EMI SE of 20.4 dB at a frequency range of 8-12 GHz and the specific EMI SE of S-RGOA could reach 1437 dB-cm$^2$/g. The results of compression tests demonstrate that lightweight S-RGOA with low energy dissipation holds good elasticity and wonderful mechanical stability. Also, S-RGOA possesses good fire-resistance ability and excellent thermal stability. These special properties of S-RGOA make it suitable for using as lightweight electromagnetic interference shielding materials. Considering melamine sponge is a cheap and commercial sponge with adjustable size, our method developed an economical and environmentally friendly approach for the fabrication of RGO aerogels.

**Experimental Section**

**Synthesis of S-RGOA.** GO was synthesized from graphite powder (Shengtai Graphite Company, China) based on the method reported by Marcano et al.[25] Melamine sponge (Shenzhen Lianda Company, China) with a density of 9.4 kg/m$^3$ was cut into pieces and ultrasonically clean in ethanol-water solution. After dried, melamine sponge was immersed in mixed solution of GO and polyetheramine (Mx = 230 g/mol, Aladdin) to a completely infiltration (GO concentration is 6 mg/mL, Mx/polyetheramine = 1:0.7). Then the resultant product was consecutively sealed in a crisper, treated in an oven at 95 $^\circ$C for 5.5 h, and frozen in refrigerator for 12 h. Subsequently, freeze-drying was performed for a week under vacuum to form the precursory sponge-supported reduced graphene oxide aerogel (P-S-RGOA). Finally, P-S-RGOA was annealed at 500 $^\circ$C for 0.5 h in a tube furnace (Shanghai Guangshu Electrical Co. Ltd., China) with a flowing stream of Ar. Before heating, the tube was purged with Ar for 0.5 h, and the heating rate was set at 15 $^\circ$C/min. S-RGOA was collected from the furnace after cooled to room temperature with the flowing of Ar.

**Characterization and tests.** The fracture surface morphologies of samples was observed by 5136MM scanning electron microscopy (TESCAN, Czech) at an operating voltage of 20 kV. Thermal gravimetric analysis was undertaken by a Pyris-1 thermogravimetric analyzer (Perkin-Elmer, Massachusetts). Samples were placed into alumina pans, and then were heated from 80 to 800 $^\circ$C at a heating rate of 20 $^\circ$C/min with a gas flow of 40 mL/min. X-ray diffraction patterns were recorded by X'pert PRO (PAAnalytical, Netherlands) with Cu Kα radiation at a current of 25 mA. X-ray photoelectron spectroscopy (XPS) spectra were recorded on a Thermo ESCALAB 250XI electron spectrometer with monochromatic 150 W Al Kα radiation. Transmission electron microscopy (TEM) image of a piece of graphene oxide was collected using a Tecnai G2 20 TWIN TEM operated at 200 kV. Fourier transform infrared (FT-IR) spectra were recorded on a Nicollet 6700 FTIR spectrophotometer using the ATR method. Raman spectra were recorded on a HORIBA XploRA Laser Raman spectrometer with 532 nm laser excitation. The electrical conductivity was measured by a 5T263 four-probe instrument (Suchou Jingge Company, China) at room temperature. Mechanical tests were carried out by a CMT-6503 universal testing machine (SANS, China) with a 50 N load cell, equipped with two flat-surface compression stage. All samples were cut into cuboid with a dimension of 24 × 13 × 15 mm$^3$ using razor blades. Samples with flat top and bottom surfaces were compressed at a loading rate of 2 mm min$^{-1}$ for strain cycles. Loading and unloading curves with strains of 10, 30, 50, 70, and 90 % were recorded. The electromagnetic interference shielding effectiveness was measured with a WILTRON 54196A scalar measurement system in the frequency of 8-12 GHz at room temperature. The samples for EMI measurement were filled with paraffin, whose minor complex electromagnetic parameters is close to those of air, as the supporting matrix.[26] The mass ratios of paraffin are 97.7 and 98.6 wt % for T-Sponge and S-RGO; and the mass ratios of paraffin are 98.0 - 98.7 wt % for S-RGOA samples. To fit the sample holder for the measurement, the samples were cut into rectangular pieces with the size of 22.5 × 10.0 mm$^2$. The fire-resistance was investigated by alcohol burner burning and vacuum oil absorption/burning tests. The integrity and self-propagated property of samples after placing on the fire of alcohol burner for 60 seconds, as well as the final residual capacity and shrinkage after 5 times of vacuum oil absorption/burning circles were used to evaluate the fire-resistance of S-RGOA.

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**Keywords:** graphene aerogels electromagnetic interference shielding mechanical properties fire-resistant


Entry for the Table of Contents

FULL PAPER

This work proposed a facile method to fabricate sponge-supported reduced graphene oxide aerogel (S-RGOA). The mechanical stable and fire-resistant S-RGOA is demonstrated to be an excellent electromagnetic interference shielding material.

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