Letter to the editor

One-step room-temperature preparation of expanded graphite

**Abstract**

Here, we propose a simple, effective method to prepare expanded graphite in which the intercalation and expansion of graphite are realized by only one step under ambient conditions, not involving any heating or any sophisticated devices. We demonstrate that the expanded graphite prepared by such an one-step room-temperature method exhibits an expansion volume of up to 225 times. Most importantly, the used amount of concentrated H₂SO₄, which acts as the intercalant, can be dramatically decreased by about 85% in comparison to that used in the conventional methods, which will greatly reduces the effluent and alleviate the environmentally detrimental effect caused by the excessive usage of H₂SO₄.

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In the past decades, expanded graphite (EG), as a new kind of functional carbon-based material, has attracted enormous attention due to its great application potential in the fields of gasket, thermal insulator, fire-resistant composite, etc. [1,2]. Recently, the discovery of graphene further evokes the great interest in EG, since it is promising as a precursor to the mass production of graphene by additional exfoliation in organic solvents with ultrasonic treatments [3].

Generally, EG is obtained by rapid heating of graphite intercalation compounds (GICs) synthesized through chemical oxidation or electrochemical method of nature flake graphite (NG) [4,5]. However, there exist some drawbacks for the conventional EG preparation routes. Firstly, the preparation process is relatively complicated, involving too many procedures, such as intercalation of NG, water-washing, drying and thermal expansion, etc. [6]. Secondly, the washing process will not only consume a lot of water resources, but also result in the problem of the effluent discharge. Thirdly, the thermal expansion of GICs must be realized by means of high temperature or irradiation treatment, which will lead to large amounts of energy consumption. Finally, the excessive usage of concentrated H₂SO₄ as the intercalant in the conventional method can also cause serious environmental pollution [7]. Therefore, it is of great value to seek a more simple, more energy-conserving and less-polluted approach to prepare EG.

Herein, we demonstrate a novel and convenient method of mass production of EG, in which EG can be obtained only by one-step preparation at room temperature. As a conventional preparation method, the binary-component system made by a combination of ammonium persulfate ((NH₄)₂S₂O₈) and concentrated H₂SO₄ (98%) at different weight ratios were mixed by sonication for 5 min. Then, 1g of NG (purity of 99%, -50 mesh, produced in Man Country, China) was added into the resultant mixture and stirred for 30s under ambient conditions, acquiring a slurry comprised of (NH₄)₂S₂O₈, H₂SO₄ and NG. After simple standing for certain time at room-temperature, the slurry was transformed into the RITEG with a worm-like structural character. The EV value was determined by measuring the volume of the RITEG prepared from 0.2000 g NG.

Fig. 1a−g displays the NG morphological evolution with the standing time. The as-prepared slurry is comprised of (NH₄)₂S₂O₈ and concentrated H₂SO₄ (98%) at different weight ratios were mixed by sonication for 5 min. Then, 1g of NG (purity of 99%, -50 mesh, produced in Man Country, China) was added into the resultant mixture and stirred for 30s under ambient conditions, acquiring a slurry comprised of (NH₄)₂S₂O₈, H₂SO₄ and NG. After simple standing for certain time at room-temperature, the slurry was transformed into the RITEG with a worm-like structural character. The EV value was determined by measuring the volume of the RITEG prepared from 0.2000 g NG.

To further investigate the morphological characteristics of the samples, FSEM observation was performed, as displayed in Fig. 2a−f. It is clear that NG has been changed from the scale-like structure to the worm-like (Fig. 2a and d), and the surface morphology of RITEG exhibits well exfoliated structure to prove full and regular expansion in direction of c-axis. Furthermore, NG possesses a tightly stacked layered structure (Fig. 2b and c).
whereas RTEG exhibits a homogeneous dilation and a porous disordered structure (Fig. 2e and f). The calculated EV value of the RTEG sample is up to 225 ml/g, comparable to those of the EGs obtained by high temperature or irradiation treatment.

Fig. 3a shows the XRD patterns of the NG and RTEG samples. A (002) diffraction peak can be clearly observed on both the XRD patterns. However, the (002) diffraction signal of RTEG is much weaker compared to that of NG. This is indicative of positional disorder in RTEG caused by the exfoliation of the graphite layers [9]. The significant decrease in the intensity of the (002) peak has also been observed in the EG prepared by the conventional routes [10]. Moreover, relative to that of NG, the (002) peak of the RTEG sample shifts slightly to the left, suggesting the increase in the interlayer spacing of the graphite due to the intercalation. We also did Raman detection to further characterize the structure of the RTEG sample (Fig. 3b). Comparatively, the D peak of RTEG becomes a little more obvious, and, the calculated ID/IG value for RTEG and NG is 0.16 and 0.06, respectively, indicating more structural disordering of RTEG, which is in well agreement with the result of XRD measurement (Fig. 3a).

In attempt to disclose the possible preparation mechanism of RTEG by the one-step approach at room temperature, we further probed into the effect of the amount of concentrated H$_2$SO$_4$ in the binary-component system on the RTEG formation, as displayed in Fig. 4a. It can be clearly seen that the amount of the concentrated H$_2$SO$_4$ has great impact on the EV value. As the H$_2$SO$_4$ amount increases, the EV value first increases and then decreases. The maximal EV value can be obtained when the ratio of H$_2$SO$_4$ and
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