

Preparation of multilayer carbon microspheres by using graphitic carbon nitride as precursor

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ABSTRACT

Multilayer carbon microspheres were synthesized by using graphitic Carbon Nitride as precursor. The result of SEM and TEM showed that micrometer-sized multilayer spherical carbon structure had been prepared without the designed addition of any catalyst or template. The composition of the sample indicated that the sample was pure carbon materials. The result of XRD and Raman revealed low graphitization degree of carbon microspheres. FTIR and the analysis of theoretical spectra calculated by using GAUSSIAN 03 program indicated carbon microspheres structure model consisted of many five-member carbon rings. It also can be the cause of the low graphitized degree.

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1. Introduction

Novel carbon materials, considering the nature of bonding and the arrangement of atoms in the lattice network of a solid, candidate carbon architecture can be distinguished as C₆₀ (fullerene), CNT (Carbon Nano-tubes), OLFS (onion-like fullerenes), GR (graphene) et al. [1–5]. They are regarded as a valuable extension to complement classic carbon material in applications [6]. According to the morphology of these materials, GR and CNT are 2D structure consisting of sp²-hybridized carbon, then, C₆₀ and OLFS have 3D spherical-like structures. GR and CNT have potential applications in many technological fields, particular in the area of electricity [7–9], which come from their semi-metallic nature and 2D structure [10]. However, comparison with excellent physical (esp. electrical) properties of GR and CNT, C₆₀ and OLFS, with a spherical structure consisting of both six-member carbon rings and five-member carbon rings, has continuously attracted tremendous amount of attention due to their remarkable chemical reactivity, which holds great promise for potential applications in many technological fields [11,12]. Then, we must be clear at least one aspect of mechanism coming from their highly chemical reactivity if we want to get a new fullerene-like structure whose chemical reactivity can surpass them. Taking the arrangement of atoms into consideration, an important factor affecting the property should be their unique spherical structure or layered spherical structure which is mainly attributed to existence of five-member carbon rings. Compared with six-member carbon ring, five-member carbon rings which possesses higher chemical

reactivity, and is apt to react with other molecules provide possibility for the shift from 2D planar structure to 3D curved structure. Taking the size into consideration, the size effect of C₆₀ and OLFS should also be an important factor affecting these properties. So five-member carbon ring and size are two key points. But the structure of conventional Fullerene is in accordance with IPR (isolated pentagon rule) [13], it is impossible to get more five-member rings. As for unconventional Fullerene, more five-member rings can be obtained. But the products arising from synthesis of unconventional Fullerenes are mainly unsteady. If we want to get steady unconventional Fullerenes, we must make use of the metal particle embedment [14–16]. However, due to the complicated process of metal particle embedment, we may solve the problem from another aspect: increasing the size of structure is a simple but effective method to obtain the structure with more five-member carbon ring defects. But would big-sized multilayer spherical structure hold different unique potential applications in contrast with small-sized ones? Would multilayer spherical structure with more five-member carbon rings possess higher chemical reactivity? They're interesting questions. Then, synthesis is the first step.

In this paper, we intend to synthesize big-sized multilayer spherical carbon structure. In our previous work, we have successfully synthesized g-C₃N₄ nanobelts and sp³-hybridized BCN compound through using melamine (C₃N₆H₆) as raw materials [17,18]. The g-C₃N₄ nanobelts possess graphite-like turbostratic structure, and is uniform in width and thickness along its typical width ranging from 600 nm to 1.5 μm, which is appropriate for synthesizing multilayer structure and big-sized sphere. Carbon nitride materials show better thermal stability [19–21]. So we selected g-C₃N₄ nanobelts as precursor.

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Table 1

Element	C (wt%)	H (wt%)	N (wt%)
Content	99.85	Not detected	Not detected

2. Experimental details

2.1. Feasibility analysis

According to the research, the nanobelts of $g\text{-C}_3\text{N}_4$ have been obtained. The belt has NH_2 edge in character. For the intensity of single bond ($\text{NH}_2\text{-C}_3\text{N}_4$) is weaker than the conjugated bond of C_3N_4 , the NH_2 would be dissociated first when the compound is heated. When the disfigurement occurred, the conjugated structure would be broken. Due to great amount of intra-molecular and intermolecular reactions, lots of intermolecular reactions occur in the system, which makes the controlled step into reaction. For the description above, the adjacent unsaturated bond would reconstruct. Thus, the unsaturated nanobelt could be curled. These defects transfer from the edge to the interior of C_3N_4 . This method would be a novel way to fabricate Bucky onions [22,23].

2.2. Synthesis

In a typical synthesis process, a quartz tube, 25 mm in inner diameter and 1000 mm in length, was used as the reaction chamber. Fine powders of graphitic Carbon Nitride were prepared in the original work, serving as the precursor. 3 g of the precursor was pressed into a quartz boat and placed in the central region of the quartz tube. First, the reaction chamber was introduced into pure Argon for about 10 min to eliminate the oxygen in it completely. Then the reaction chamber was heated to 1000 °C within 5 h, and this temperature was maintained for 2 h under the autogenous pressure due to the pyrolysis of the precursors. Then, the quartz tube was cooled to ambient temperature naturally. Acicular powders with black colors were collected from the inner wall of the quartz boat.

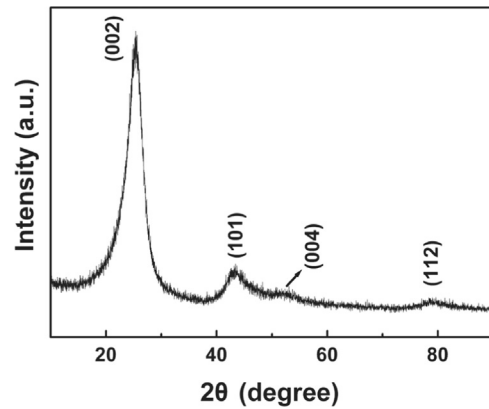


Fig. 2. The XRD pattern of the prepared sample.

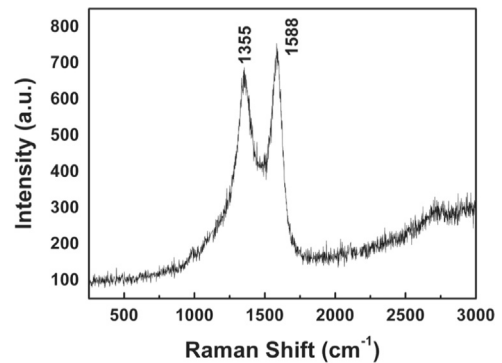


Fig. 3. The Raman pattern of the prepared sample.

2.3. Characterization

The characterizations of the samples were carried out via chemical elemental analysis (CEA), X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron

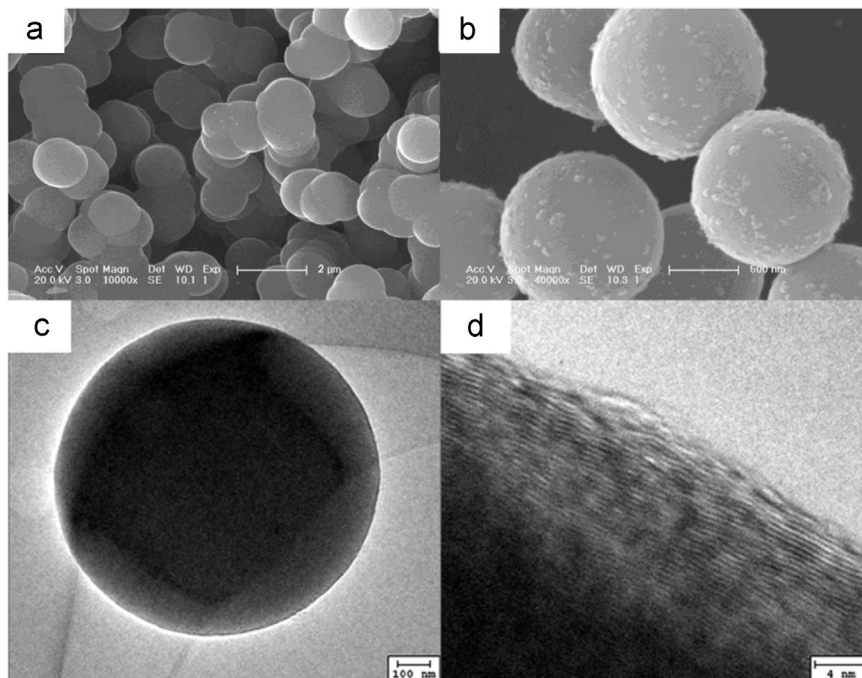


Fig. 1. (a) and (b) SEM images of the sample. (c) and (d) TEM image of spherical structure.

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