



Fabrication, characterization, purification and photoluminescence properties of carbon nanomaterials over water-soluble alkali salts



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ABSTRACT

Over water-soluble alkali carbonate particles, coiled carbon nanofibers were produced in large quantities through the pyrolysis of acetylene at 450 °C. By controlling the component of catalyst, coiled carbon nanofibers and carbon nanofibers could be synthesized selectively. And the pyrolysis temperature also had a great impact on the yield and morphology of the obtained samples. By regulating the temperature, carbon nanofibers, carbon nanotubes and bamboo-like carbon nanotubes could be synthesized, respectively. Because the used catalysts were water-soluble alkali salts, the purification was very mild and high purity of carbon nanomaterials could be obtained expediently through a simple water washing process. The photoluminescence investigation indicated that the obtained samples exhibited a blue and two ultraviolet peaks, and the photoluminescence intensity could be enhanced greatly by increasing the excited wavelength. Therefore, the proposed route for the growth and purification of carbon nanomaterials is simple, environmentally friendly and low-cost.

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1. Introduction

In the last two decades, carbon nanostructures essentially carbon nanotubes (CNTs) and carbon nanofibers (CNFs) have attracted intense research interests owing to their outstanding properties and potential applications [1–4]. Apart from the filamentous arrange of graphene sheets conducting to CNF or CNT, carbon atom can also bond in other different ways to create other novel structures with dissimilar properties. For example, the pairing of pentagonal, heptagonal and hexagonal carbon rings can result in the formation of carbon nanocoils or carbon nanospheres [5–7]. These novel carbon nanomaterials (CNMs) have been predicted that possess unique properties and have wide-range potential applications [8–10]. It is thus very desiderative to exploit a simple and reliable process to produce CNMs. Generally, the present methods for CNM synthesis involve electric arc discharge, laser evaporation and catalytic chemical vapor deposition (CCVD) [11–13]. However, the raw CNMs obtained by the mentioned

methods are usually accompanied by metallic impurities, which makes some properties and applications of CNMs have not been realized up to now [14,15]. Therefore, obtaining high purity of CNMs is very important and essential.

In order to obtain high purity of CNMs, considerable progresses in the CNM purification have been made and a number of methods including gas phase oxidation, liquid phase oxidation, filtration, and centrifugation have been developed [16–18]. Generally, the methods of CNM purification can be basically classified into three categories: namely chemical method, physical technique, and a combination of them [14,19]. However, the reported chemical method based on the idea of selective oxidation, which always influences the structure of CNMs [20,21]. And the physical method ground on the differences in their physical size, gravity, and magnetic properties, etc., which is always complicated, time-consuming and less effective [22,23]. Therefore, until now, the simple, low-cost and pollution-free technique for CNM purification without any destruction is still a hot topic and huge challenge.

In the light of the previous work [24–28], in this paper, we report a simple route for the CNM preparation and purification. Moreover, the photoluminescence (PL) investigation indicates that the obtained samples exhibit a blue and two UV PL peaks.

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2. Experimental

2.1. Synthesis of catalyst

In a typical experiment, NaOH, KOH and $C_6H_8O_7$ were dissolved in 200 mL of absolute ethanol with stirring at 60 °C for 6 h. After the obtained solution dried at 80 °C for several hours, the obtained white powder was heated in air at 550 °C for 4 h to produce the corresponding catalyst. To prepare the different components of catalysts (denoted as A, B, C and D hereinafter), the amount of $C_6H_8O_7$ was kept unchanged (0.05 mol), the amounts of NaOH and KOH were 0 and 0.1 mol, 0.025 and 0.075 mol, 0.05 and 0.05 mol, 0.075 and 0.025 mol, respectively.

2.2. Production of CNMs

Unlike the proposed route given by Xu et al. [29], in our designed CNM growth process, 0.1 g of the catalyst powder was dispersed on a ceramic plate which was placed inside a quartz reaction tube. Subsequently, the tube furnace was heated from room temperature (RT) to the predetermined temperature in Ar, and then the pyrolysis of acetylene was carried out at the designed temperature for 6 h under atmospheric pressure. To investigate the effect of catalyst component on the CNM growth, acetylene was decomposed at the same temperature (450 °C) over catalysts A, B, C and D, respectively. The samples obtained over the catalysts A, B, C and D were denoted as A-450, B-450, C-450 and D-450, respectively. In order to study the temperature-dependent growth of CNMs, the decomposition of acetylene was conducted over the same catalyst (C) at 400, 450, 500, 550 and 600 °C, respectively. And the corresponding obtained samples were named as C-400, C-450, C-500, C-550 and C-600, respectively. Finally, each experiment was repeated three times to confirm the obtained experimental results further.

2.3. Characterization of products

The samples were examined on an X-ray powder diffractometer (XRD) at RT for phase identification using $CuK\alpha$ radiation (model D/Max-RA, Rigaku). Raman spectroscopic investigations were performed using a Jobin-Yvon Labram HR800 instrument with 514.5 nm Ar^+ laser excitation. The morphology investigations were examined using a transmission electron microscope (TEM) (model JEM-2000EX, operated at an accelerating voltage of 20 kV) and field emission scanning electron microscope (FE-SEM) (model FEI Sirion 200, operated at accelerating voltages of 5 kV). Fourier transform infrared (FTIR) spectroscopic studies (in KBr pellets) were conducted over a Nicolet 510P spectrometer. The

photoluminescence (PL) spectrum were obtained at RT by a spectrofluorophotometer (Shimadzu RF-5301PC) using a Xe lamp as the light source.

3. Results and discussion

3.1. XRD patterns of catalysts

The typical XRD patterns of the catalysts A and C are shown in Fig. 1. As shown in Fig. 1(a), besides the presence of $K_2CO_3 \cdot 1.5H_2O$ (JCPDS: 73-0470), all the peaks can be assigned to those of K_2CO_3 (JCPDS: 71-1466). The presence of its aquo-compound should be related to the water-absorption property of K_2CO_3 , which was also reported before [30–32]. The result indicates that the obtained catalyst A is K_2CO_3 . Similar to that of the catalyst A, as shown in Fig. 1(b), the phase of $K_2CO_3 \cdot 1.5H_2O$ can also be detected over the obtained catalyst C. And the other peaks can be identical to those of $KNaCO_3$ (JCPDS: 01-1038). On the whole, the as-prepared catalysts (A–D) for the CNM growth are the water-soluble alkali carbonates, which also imply that high purity of CNMs may be obtained easily through the water-washing process.

3.2. Effect of catalyst component on the CNM growth

In order to investigate the effect of catalyst component on the final products, acetylene was decomposed at 450 °C over the catalysts A, B, C and D, respectively. Fig. 2 gives the XRD patterns of the as-prepared samples (A-450, B-450, C-450 and D-450). As shown in Fig. 2(a), all the diffraction peaks of C-450 are ascribable to graphite carbon and the corresponding catalyst $KNaCO_3$ (JCPDS: 01-1038). Similar to that of C-450, the XRD patterns of the samples A-450, B-450 and D-450 also suggest the presence of graphite carbon and the corresponding water-soluble alkali salt, which indicates that all the samples obtained over the different catalysts are the mixture of graphite carbon and the corresponding alkali salt. And the different intensity ratios of carbon and catalyst in the XRD patterns can also be observed clearly over the obtained samples, which indicate the different contents of catalyst in the corresponding sample and also reflect the different catalytic abilities of the as-synthesized catalysts. In the study, the same amount of the catalyst (0.1 g) was used for the catalytic decomposition of acetylene at the same temperature (450 °C). Based on the XRD results, one can conclude that the addition of Na into the catalyst is not favorable to synthesize CNMs in large quantities. As shown in Table 1, the obtained experimental results show that the designed experiments have a good reproducibility and the mass of the obtained sample reduces with the increasing Na

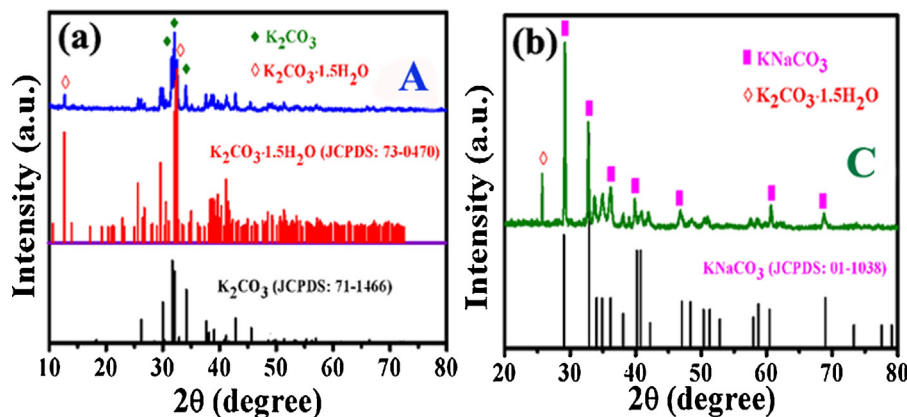


Fig. 1. The XRD patterns of the obtained water-soluble catalysts: (a) A, and (b) C.

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