

C54. In Situ Synthesis Process of Nanographene and Its Characteristic.

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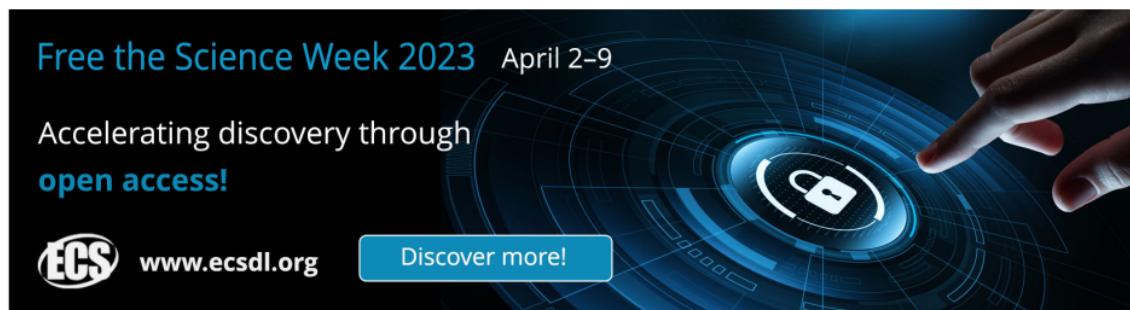
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In situ synthesis process of nanographene and its characteristic

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Abstract. The results of the study of synthesis nano graphene from pyrolysis of tartaric acid compound obtained 29.8% yield. Crystallinity of nano graphene was analyzed by X-ray diffractometer and showed peak 2 θ at 23.15° from the diffractogram. Nano graphene from tartaric acid has a particle size of 1 nm by the result of atomic force microscopy analysis. The photoluminescent properties were measured using spectrofluorometer with a 405 nm Diode Laser producing a peak at wavelength of 403.1 nm (blue region). Nano graphene maintain its stability in the pH range of 3-12 as well as on the addition of salt with concentration of 0.15 M; 0.30 M; and 0.50 M for 24 hours.

1. Introduction

Graphenes are flat monolayer of atom carbons tightly packed into two dimensional (2D) honeycomb lattice [1], which showed interesting properties to be applied in many fields. Graphene arranged one-atom thick layer of extended sp² carbon show extremely fast electron mobility and high charge carrier density which is interesting object to be studied [2]. However, single-layer graphene with nearly perfect structure lack of electronic band gap which cause this material not photoluminescence. Therefore, creating energy band gap can be a strategy to make graphene photoluminescent. Morphology of graphene, including size, shape, and thickness, effect the properties of graphene [3]. Cutting graphene into small pieces or directly producing small pieces of graphene can be one of strategy to make fluorescence emission in graphene [4]. The small pieces of graphene called nanographene which varying size from 1 to 100 nm. Nanographene shows intrinsic luminescence as a result of quantum confinement, surface defects and edge structure [2].

Nanographene can be synthesized through top down or bottom up method. In top down method, nanographene generally obtained by cutting carbon resources such as graphite powder, carbon nanotubes, and carbon fibers [5]. However, this process cannot control the structure and size distribution of nanographene [6]. Besides, the method produces low yield products and require special equipment [7]. Meanwhile, bottom up method provide one step preparation and high yield percentage, which are efficient route to produce nanographene in large scale. Nanographene that synthesized using bottom up method can be obtained by self-assembly of polycyclic aromatic hydrocarbons (PAHs), organic synthesis from small molecules, carbonization of some organic precursors [5].



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Carbonization process by thermal treatment allows precise control over morphologies and size distribution of products. Besides, this method can use common organic precursor. Our previous research used citric acid as organic precursor and selectively prepares GQD and GO by tuning the carbonization degree [8-10]. However, GQD obtained in this research contain of some incompletely carbonized citric acid. This probably due to citric acid structure, which consist of carboxylic substituent. When citric acid was heating in high temperature, its molecules will experience self-assembly to form six rings and leaving carboxylic substituent unreacted.

Citric acid can be replaced by tartaric acid, which has similarity application, such as dental filling and food preservatives. In this two application, tartaric acid and citric acid can be replaced for each other due to its similar properties and structure. However, tartaric acid structure are not contain of carboxylic substituent, which has probability to react completely forming nanographene.

Nanographene, which possess photoluminescence properties could be applied in biomedical field, such as diagnostic, near-infrared (NIR) light induced photothermal therapy and bioimaging. Therefore, in living organism, stability of material such as various acidity and salt solution, should be a concern for further application.

In this work, nanographene was synthesized from tartaric acid and characterized. Furthermore, stability of nanographene was also tested in various acidity and NaCl solution to discover whether it can be applied in biomedical field.

2. Experimental section

2.1. Materials and Chemicals

Tartaric acid was purchased from Sigma-Aldrich and sodium hydroxide are technical grade. Sodium chloride and hydrochloric acid were used to test the colloidal stability of nanographene.

2.2. Synthesis of nanographene

Nanographene was synthesized by direct pyrolysis method. 200 g of tartaric acid was placed on porcelain cup then is heated for 2 hours. The product of pyrolysis then cooled and dissolved in 10 ml of 2% NaOH. The obtained solutions filtered using membrane 0.22 μm to separate the bigger molecules of nanographene and unreacted tartaric acid.

2.3. Characterization of nanographene

The steady-state photoluminescence spectra were measured using fluorescence spectrophotometer. The nano-morphology of nanographene was studied by atomic force microscopy (AFM). FTIR spectra and X-ray Diffraction pattern of nanographene were also studied using FTIR spectrometer and X-ray diffraction instrument, respectively.

2.4. Nanographene stability analysis

Nanographene stability were tested by observing two parameters, stability at various acidity and addition of salt.

2.4.1. Stability of nanographene at various pH. Acidity of nanographene solution were set with addition of HCl and NaOH. The solutions made in 10 variation ranged of pH from 3 to 12. Each solution were observed whether any color degradation or precipitation.

2.4.2. Stability of nanographene with addition of NaCl. Sodium chloride with concentration 0.15 M, 0.3 M, and 0.5 M were added in nanographene solutions. Stability of solutions observed in 6 and 24 h.

3. Results and Discussion

3.1. XRD studies

Nanographene were synthesized by pyrolysis method. Tartaric acid were heated at 270°C for two hours. In high temperature, the molecules will decompose when temperature reach its melting point (168°C to 170°C). Furthermore, heating will cause self-assembly of atomic carbon to form its stable structure, which is 6-ring carbon. This process was growing to form graphitic structure, which called nanographene. The yield of nanographene obtained from this research was 29.8%.

Graphitic structure of nanographene were confirmed by XRD patterns (based on JCPDS26-1076) which showed broad peak at 2θ range 20° to 30°. This diffractogram pattern (Figure 1) showed d-spacing at 0.38 nm.

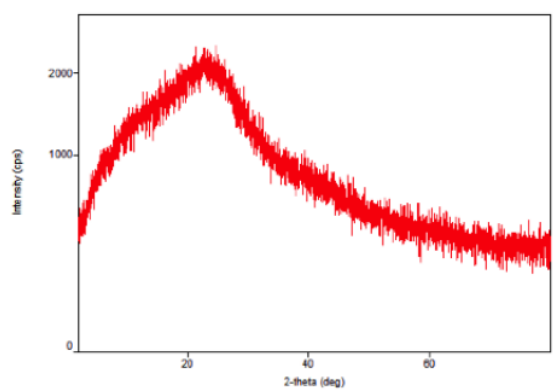


Figure 1. XRD Pattern of nanographene.

3.2. FTIR studies

FTIR spectra of nanographene exhibit absorption of OH group at 3630.03 cm^{-1} and carboxyl group at 1718.58 cm^{-1} , indicating that nanographene contain of $-\text{COOH}$ group. Compared to broadness of OH peak in tartaric acid, nanographene shows smaller broadness which indicating that OH group in nanographene has been much reduced. The intensity of carboxylic peak for nanographene also lessen compared to tartaric acid peak. FTIR spectra indicated no absorption of C-H, which showed complete carbonized of tartaric acid. FTIR spectra shows on Figure 2.

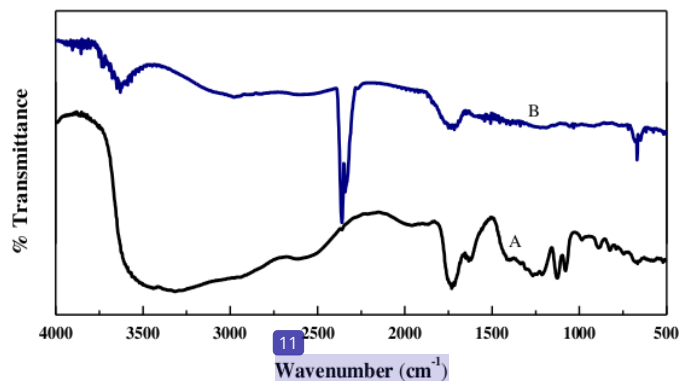


Figure 2. FTIR spectra of tartaric acid (A) and nanographene (B).

3.3. Atomic Force Microscopy analysis and Size measurement

Nanographene morphologies investigated using Atom Force Microscopy (AFM). Distribution of nanographene shows in Figure 3, which exhibit diverse size and shape.

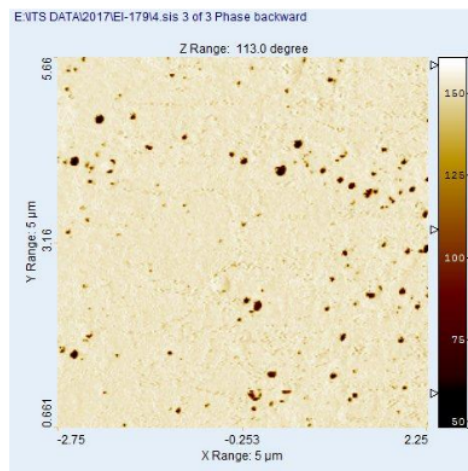


Figure 3. AFM image of nanographene.

Size distribution of nanographene analyzed using image j computer program. Size of nanographene ranged 1 to 8 nm with average sizes are 1 and 2 nm. The result shows in Figure 4.

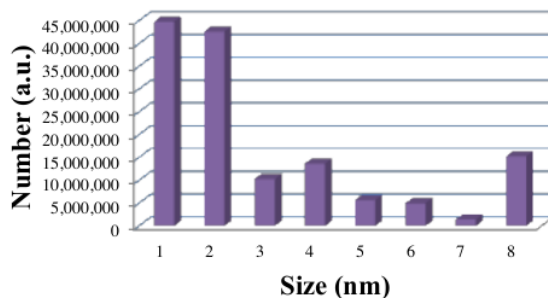


Figure 4. Size distribution of nanographene.

3.4. Photoluminescent analysis

Photoluminescence properties of nanographene analyzed by PL spectrophotometer. Nanographene solutions were excited using diode laser at wavelength 405 nm and the measured wavelength were 403.1 nm. Wavelength emitted from materials showed color spectra of photoluminescence quantitatively. Meanwhile, photoluminescence of nanographene were range around blue light (from 450 to 500 nm). Therefore, blue light emitted by nanographene show the size of nanographene relatively small around 1 to 6 nm [5].

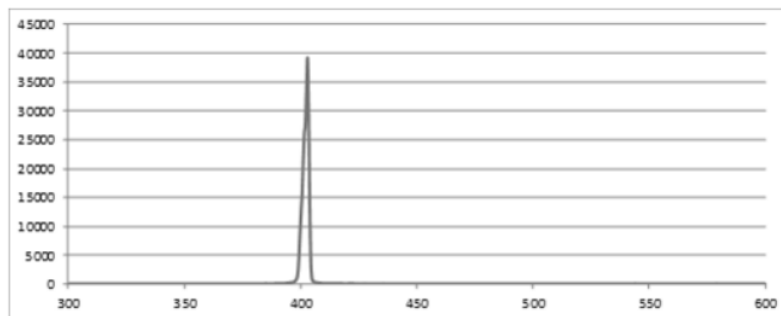


Figure 5. Photoluminescence of nanographene

3.5. Nanographene stability analysis

Stability of materials is one of condition of material to be used in biological system. Therefore, stability test is important parameter to be tested before being used in further application. Stability of nanographene was studied by setting two parameters, stability at various acid value (range pH 3 to 12) and stability at various salt concentrations. Stability of nanographenes at various acidity shows in Table 1 and stability at various salt concentration shows in Table 2.

4. Conclusions

Nanographene has been synthesized through pyrolysis method by heating tartaric acid as precursor at 270°C for two hours. Characterizations of nanographene showed that XRD pattern exhibit a broad peak range 20° to 30° which indicating graphitic structure. Size of nanographene was obtained on range 1 to 8 nm and emitting blue light photoluminescence. Nanographene considered as stable material, which can be applied further, in biological system.

Table 1. Stability of nanographene at various acidity

Duration (h)	pH Value										
	3	4	5	6	7	8	9	10	11	12	13
Control	-	-	-	-	-	-	-	-	-	-	-
6 h	-	-	-	-	-	-	-	-	-	-	-
24 h	-	-	-	-	-	-	-	-	-	-	-

Table 2. Stability of nanographene at various salt concentration

Duration (h)	NaCl concentration			
	0 M	0.15 M	0.3 M	0.5 M
Control	-	-	-	-
6 h	-	-	-	-
24 h	-	-	-	-

- No color degradation and no precipitation
- + Color degradation occurred and form precipitation

Stability test of nanographene at various acidity or salt concentration show no color degradation or precipitation. This results indicated that nanographene are tend to be stable.

9 Acknowledgment

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