

Communication

Investigating the effect of graphite electrodes structures on the

synthesized grapheneusing electrochemistry method

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✉: H. Sayahi sayahi@ccerci.ac.ir In this study, a simple and efficient approach is reported to product graphene using electrochemical exfoliation of graphite electrodes, in acidic solutions. The electrolyte concentration, applied voltages, electrodes distance and preparation time were taken as control parameters to optimize the produced graphene structures. The optimized graphene sample was prepared in a $H₂SO₄$ (0.5 M) at a bias of +10 V. The interlayer spaces, crystallite size and the average number of layers were determined by XRD. Surface morphology, layer thickness and characteristics of obtained samples were investigated by Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), and FTIR. The obtained results confirmed the graphene structures.

Keywords: Graphene, Graphite Electrode, electrochemical synthesis, electrochemical saving energy

1. Introduction

Graphene, a two-dimensional material formed from $sp²$ bonded carbon atoms packed in a honeycomb crystal lattice, has become one of the most exciting research topics in the last decade because of their extraordinary mechanical, optical, electronic, and electrochemical properties [1,2]. Various methodologies such as scotch-tape isolation, epitaxial growth, bottom-up synthesis from aromatic precursors, and chemical vapor deposition of gaseous reagents do not seem readily scalable because of high cost, process complexity and/or low yield. Beyond that, wet chemical approaches, including reduction of graphene oxide (GO)] and liquid-phase exfoliation of graphite, may present plausible alternatives for manufacturing graphene on a large scale. Electrochemical exfoliation of graphite has drawn increasing attention over the last few years as a potentially scalable method. Generally, it involves the use of an electrolyte (e.g., aqueous or non-aqueous solution) and an electrical current to drive structural expansion at a graphite electrode [3]. Electrochemical exfoliation of graphite has drawn increasing attention over the last few years as a potentially scalable method. Generally, it involves the use of an electrolyte (e.g., aqueous or non-aqueous solution)and an electrical current to drive structural expansion at a graphite electrode. According to the charge of the intercalated ions, the graphite electrode works as an anode or cathode and representing oxidation or reduction reactions, respectively. In contrast to other exfoliation processes, this method is not equipment- intensive and is typically performed under ambient conditions [4].

 In this study, we report a simple, cost-effective electrochemical approach to produce graphene by electrochemical exfoliation of graphite electrode, in acidic electrolytes. X-ray powder diffraction (XRD) was used to determinate the number of graphene layers, the distance interlayers, and the size of graphene crystallites. The obtained materials were also investigated by FT-IR Spectroscopy, Scanning Electron Microscopy (SEM), and Atomic Force Microscopy (AFM).

2. Experimental

 All reagents were of analytical grade and used without further purification. All solutions were prepared using double distilled water. Sulfuric acid was purchased from Merck chemicals. graphiteelectrode with high purity (99.99%) were used.

3. Results and discussion

 The electrochemical exfoliation process of graphite electrodes to product graphene (Fig. 1) was shown that it is significantly dependent on a couple of parameters which affects the quality of the final products: the electrolyte concentration and the applied bias voltages. The influence of the applied bias potential and electrolyte concentration, was studied in the 1–10 V of potentials and in the 0.2–1 M concentration, Respectively.

Fig. 1. Schematic of an electrochemical cell.

 The XRD analysis was used to characterize the crystalline nature and phase purity of the synthesized materials (Fig. 2). The mean crystallites size (D) for each sample was calculated from full width at half maximum (FWHM) of the XRD pattern, using the Debye–Scherrer equation, as previously reported by other research groups. The interlayer distance (d) was found using Bragg equation [1,5]. The average number of graphene layers (n) was calculated by "Eq. 1":

$$
n=\frac{D}{d} \qquad (1)
$$

Fig. 2. The XRD patterns of the samples

Fig. 3 (a,b) reveals a graphene Flake with an average thickness, corresponding to multi-layer graphene.

Fig.3. The typical AFM image of the sample and SEM images

4. Conclusions

 In conclusion, a one-step method of the synthesis of high quality graphene by electrochemical exfoliation of graphite electrode using acidic electrolyte. Different electrochemical parameters (e.g. applied bias, electrolyte concentration) were studied in details. In all of the as-prepared materials a mixture of few-layer and multi-layer graphene was present. The experimental conditions for the optimum sample were as follows: 0.5 M electrolyte concentration, 10 V applied bias and 30 min exfoliation time. This method, like other methods, has some disadvantages and advantages, but showed that it is a simple, costeffective and efficient way to produce high quality graphene sheets. Obviously, the reported method can be optimized to produce less graphene layers.

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