Synthesis of Functionalized Single graphene Sheets by thermal exfoliation of Graphite Oxide

ABDUL JABAR MOHAMMED SALEH AL-EYANI3 and NABIL ABDULLAH NOMAN ALKADASI1,2,3

1Hubei Key Lab of Materials Chemistry & Service Failure, School of Chemistry & Chemical Engineering, Huazhong University of Science and Technology, Wuhan, Hubei, 430074 China .
2Department of Chemistry, Faculty of Education, Al-baida’a , University, Yemen , P.O.Box:39189.
3Mechanical Engineering, Faculty of Engineering, Thamar University, Yemen.
*Corresponding author E-mail: nalkadasi@yahoo.com

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ABSTRACT

Synthesis is described to produce single sheets functionalized graphene through thermal exfoliation of graphite oxide. Synthesis yields a single sheet structure resulting from the reaction sites involved in oxidation and reduction process. Application of graphite used for unelectrical material and used pencil meanwhile main applications of graphene sheets are electrical materials.

Key words: Graphite, Graphene oxide and Graphene sheets, Characterization and application.

INTRODUCTION

Graphene is a single hexagonally flat layer of graphite, which has attracted great interest both for fundamental understanding of its unique structural and electronic properties and for important potential applications in nano-electronics and devices1-6. The unique properties of this two-dimensional (2D) material include the highest intrinsic carrier mobility at room temperature of all known materials and very high mechanical strength and thermal stability7-10. Graphene holds great promise for the development of new composite materials, emissive displays, ultrasensitive detectors and micromechanical resonators11-13. The combination of high mobility, thermal, chemical and mechanical stability with the high surface area offers many interesting applications in a wide range of fields including heterogeneous catalysis where metallic and bimetallic nano-particle catalysts can be efficiently dispersed on the graphene sheets14-16. In many cases, the remarkable properties of single graphene layers extend to bilayers and a few layers of graphene sheets. Several methods have been reported for the production of graphene sheets including micromechanical cleavage and thermal expansion of graphite.
EXPERIMENTAL

Materials
Physical parameters of Graphite 99.95%, Potassium Chlorite, Sulfuric acid $\text{H}_2\text{SO}_4$ and Nitric acid ($\text{HNO}_3$), 99.9% are reported in table 1, 2, 3 and 4 respectively.

Fabrication of graphene sheets
Commercial powdered natural graphite (from Aladdin Industrial Corporation Shanghai, China) was used as our starting material. The commercial graphite has a particle size of 325 mesh with a purity of 99.99%. Graphite was oxidized following modified Method of Staudenmaier method to form graphite oxide (GO). In this method, graphite (2.5 g) was first mixed with sulfuric acid (43.75 mL) and nitric acid (22.5 mL) and stirred. When graphite was uniformly dispersed, potassium chlorate (27.5 g) was added slowly and stirred for over 96 h. After the completion of oxidation reaction, the mixture was added into 4 L deionized water and then filtered. The GO was repeatedly rinsed and redispersed in a 5% solution of HCl. It was then washed continually with deionized water until the pH of the filtrate was neutral. Potassium stayed in the GO even after several times of washing. Therefore multiple washing cycles used in conjunction with bath ultrasonication in fresh ethanol were used for the removal of residual potassium. After this the GO was dried in a vacuum oven at 60°C until used. Finally, the GO was treated with the nitrogen (N$_2$) and then it was heated to 1050°C in the furnace for 30 s to form graphene sheets.

Treatment silicon wafer
Silicon wafer were cut into (3mm x 3mm) used Piranha solution is mixture consisting of sulfuric acid ($\text{H}_2\text{SO}_4$) and hydrogen peroxide ($\text{H}_2\text{O}_2$) is to cleaning silicon wafer. It is typically mixed in concentration ratios of around 3:1 $\text{H}_2\text{SO}_4$: $\text{H}_2\text{O}_2$ for one hour with temperature 60°C. Then followed by triple rinsing in ethanol with ultrasonic cleaning for 30 min then with nitrogen (N$_2$). There are two main applications for piranha in wafer fabrication: it is used to remove organic contaminants from surface of the wafer during cleaning. Silicon wafer were used for SEM.

RESULTS AND DISCUSSION

The GO was reduced to graphene sheet by heating to 1150°C. Plate 1-2 (SEM) and plate 7 (TEM) shows the top-view TEM images of the graphite oxide plate (SEM) and plate 8 (TEM). TEM images of the original graphite plate

Table 1: General Characteristics of Graphite 99.95%

<table>
<thead>
<tr>
<th>Trade Name</th>
<th>Graphite 99.95 %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Black</td>
</tr>
<tr>
<td>Size</td>
<td>325 mesh</td>
</tr>
<tr>
<td>Company</td>
<td>Aladdin Industrial Corporation Shanghai, China</td>
</tr>
</tbody>
</table>

Table 2: General characteristics of Potassium Chlorate

<table>
<thead>
<tr>
<th>Trade Name</th>
<th>KClO$_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>White</td>
</tr>
<tr>
<td>Molecular weight</td>
<td>122.55</td>
</tr>
<tr>
<td>Company</td>
<td>China</td>
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</table>

Table 3: General Characteristics of Sulfuric Acid $\text{H}_2\text{SO}_4$

<table>
<thead>
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<th>Molecular formula</th>
<th>Sulfuric Acid($\text{H}_2\text{SO}_4$), 99.9%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>liquid</td>
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<tr>
<td>Molecular weight</td>
<td>98.08</td>
</tr>
<tr>
<td>Concentration</td>
<td>95 - 98 %</td>
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<td>Company</td>
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</table>

Table 4: General Characteristics of Nitric Acid ($\text{HNO}_3$), 99.9%

<table>
<thead>
<tr>
<th>Molecular formula</th>
<th>Nitric Acid ($\text{HNO}_3$), 99.9%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
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<tr>
<td>Molecular weight</td>
<td>63.01</td>
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<tr>
<td>Concentration</td>
<td>65 – 68 %</td>
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<tr>
<td>Company</td>
<td>Sinopharm chemical reagent Co , Ltd , China</td>
</tr>
</tbody>
</table>
Photo 1: Graphite in water

Photo 2: GO green before dry
GO.dry in 60°C water

Photo 3: Graphene Sheet in water

Plate 1: SEM of Graphene sheets dispersed in water

Plate 2: SEM of Graphene sheets dispersed in ethanol

Plate 3: SEM of Graphene Oxide (Dry) dispersed in water

Plate 4: SEM of Graphene Oxide (Dry) dispersed in ethanol

Plate 5: SEM of Graphite dispersed in water
Plate 6: SEM of Graphite dispersed in ethanol

Plate 7: TEM of Graphene sheets

Plate 8: TEM of Graphene Oxide

Plate 9: TEM of Graphite

(SEM) 5,6 and plate 9 (TEM). A small flake of graphite particle is seen in . The size of the graphene sheet is about 9.35µm from SEM photo. The surface of graphene shows several large meandering wrinkles. The thickness of graphene can be clear in high-magnification TEM image.

CONCLUSIONS

Single sheets functionalized graphene through thermal exfoliation of graphite oxide. Main application of graphene sheets in electrical material. The surface of graphene sheets shows several large meandering wrinkles. The thickness of graphene can be determined from the high-magnification TEM image.

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REFERENCES


