

Vibrational properties of mass produced graphene monolayer by chemical method

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Abstract. Graphene is a two-dimensional crystal of carbon atoms arranged in a honeycomb lattice. It is a zero band gap semimetal with very unique electronic optical and mechanical properties which make it useful for many applications such as ultra-high-speed field-effect transistors, p-n junction diodes, terahertz oscillators, low-noise electronic, NEMS and optical sensors. When the high quality mass production of this nanomaterial is a big challenge, we have used chemical method which helped to achieve this goal. Raman and FTIR vibrational spectroscopies were investigated to the examination of the production quality.

1. Introduction

Graphene has received much attention recently in the scientific community because of its distinct properties and potentials [1]. The recent emergence of this stable two-dimensional (2-D) carbon, graphene, has initiated a rapid exploration of its potential in a broad range of fields such as nanoelectronics, sensors, catalysis and nanocomposites [2].

A central challenge of rapid graphene implementation is its mass production and researchers are investigating a number of alternative means for producing the nanomaterial. These methods have shown great promise for industrial-scale production of graphene and the chemical exfoliation of graphite through oxidation and then dispersion in water, down to single graphene sheets, is one of the potential methods of achieving this goal [3].

Raman and FTIR spectroscopies have been utilized as a powerful tool for the characterization of graphene, as it can identify the number of layers, the electronic structure, the edge structure, the type of doping and any defects in the graphene [4].

In this work we developed a simple chemical method for producing large quantities of the promising nanomaterial graphene. This method needs little processing, and paves the way for cost-effective mass production of graphene. Raman and FTIR spectroscopies were investigated to study the vibrational properties in the goal to determine structural properties of our prepared graphene powder.

2. Experimental

The graphite used in this work was purchased from Sigma-Aldrich. We synthesized the graphene powder using a modified Hummers method which involved the exfoliation of graphite in the presence of strong acids and oxidants [5]. The obtained solution was reduced with hydrazine at 40 °C with

stirring for 6h followed by the dispersion in 2-Propanol and heat treatment to produce graphene powder (Fig.1).

To analyse our synthesized graphene powder we have used Bruker Sentarra RAMAN spectrometer with an excitation wave length of 785 nm, Bruker Vertex 70 Fourier transform infrared (FTIR) Spectrometer and SEM images were obtained by a Nova NanoSEM 230. The absorption spectrum was recorded at room temperature using a Cary UV-visible-near-infrared spectrometer. Lastly, the Element composition was investigated with an Energy Dispersive X-ray (EDX).



Figure 1. Graphene powder

3. Results and discussion

3.1. Structural properties

To examine the structural properties of the graphene powder UV-VIS spectroscopy, SEM and EDX were investigated.

3.1.1. UV-VIS spectroscopy.

As shown in (Fig.2), the absorption peak at 272 nm indicates that the electronic conjugation within the reduced graphene sheets was revived upon reduction of graphene oxide. This result is in total agreement with the one done in the paper [6].

3.1.2. SEM imaging.

The SEM images obtained using Nova NanoSEM 230 Electron Microscope show typical large flakes of graphene powder (Fig.3). The transparency of these flakes confirms the successful exfoliation of graphene sheets and indicates that each flake is composed of a few individual graphene sheets as well as the large scale production.

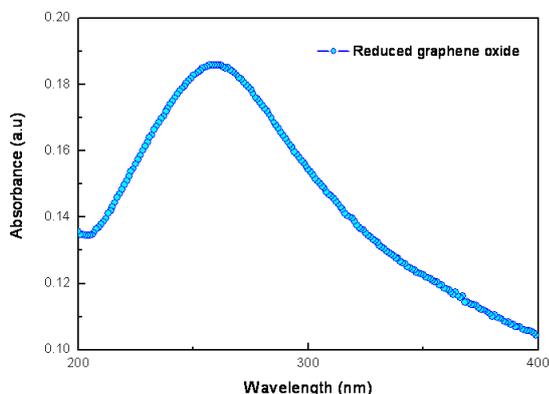


Figure 2. Uv-Vis spectrum shows the reduction of the exfoliated graphene oxide.

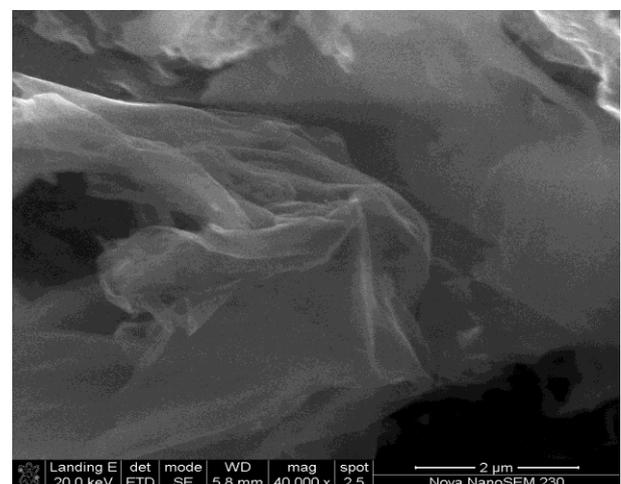


Figure 3. These two figures have been placed side-by-side to save space. Justify the caption.

3.1.3. EDX analysis.

EDX analysis was done for different area of the graphene powder. From the EDX spectrum results in (Fig.4) chemical residues and Oxygen atoms are totally removed and the percentage of carbon atoms in our samples was higher than 99% confirming the purity of this product.

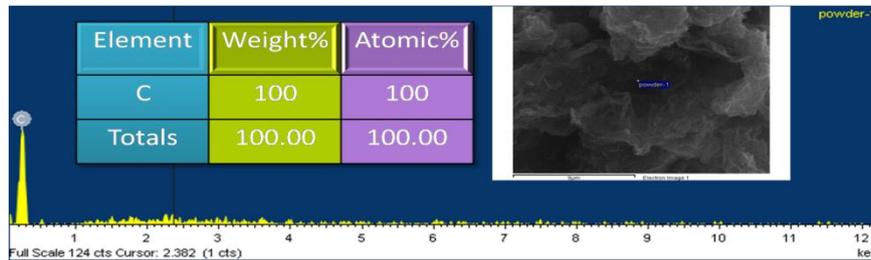


Figure 4. Graphene powder

3.2. Vibrational properties

3.2.1. Raman spectroscopy

During the graphite powder's oxidation process in the presence of concentrated acids using Hummer's method, the original extended conjugated π -orbital system of the graphite was destroyed and oxygen-containing functional groups were inserted into the carbon skeleton. After reduction with hydrazine, oxygen groups were then replaced with nitrogen ones. However, after a purification treatment, these functional groups derived from the intensive oxidation and hydrazine hydrate were eliminated, which can be proved by the existence of D, G and G' bands only, reflecting the skeletal vibration of graphene sheets [7]. The G band that originates from in-plane vibration of sp^2 carbon atoms is doubly degenerate (TO and LO) phonon mode (E_{2g} symmetry) at the Brillouin zone center. The 2D band originates from a two phonon double resonance Raman process (figure 5). The D band is attributed to defects in reduced graphene oxide nanosheets. These results indicate that the graphite oxide has been successfully exfoliated to pure graphene sheets.

3.2.2. FTIR spectroscopy.

FTIR spectra of rGO and GO are shown in figure 6. The absence of graphite and graphene oxide peaks is a strong indication of the purity of our prepared reduced graphene, at 3400 cm^{-1} (O-H stretching vibrations), at 1620 cm^{-1} (skeletal vibrations from unoxidized graphitic domains), at 1220 cm^{-1} (C-OH stretching vibrations), at 1060 cm^{-1} (C-O stretching vibrations) [8], and stretching vibrations from C=O at 1720 cm^{-1} due to the remaining of carboxyl groups even after hydrazine reduction and the vibration modes of epoxide (C-O-C) at ($1230\text{-}1320\text{ cm}^{-1}$, asymmetric stretching at 850 cm^{-1} , bending motion) [9].

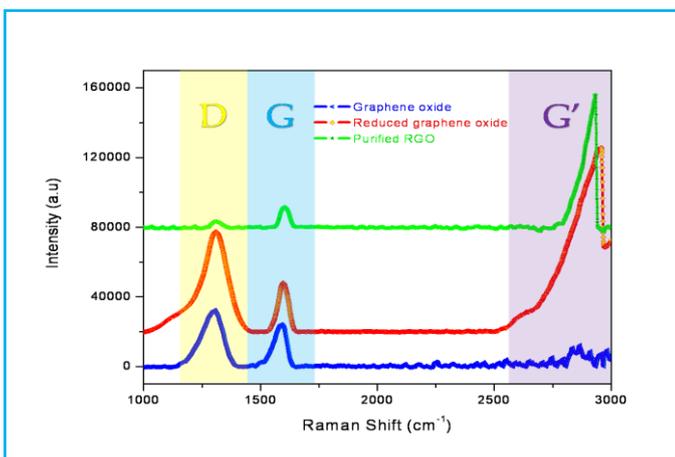


Figure 5. Raman spectra of graphene oxide and graphene.

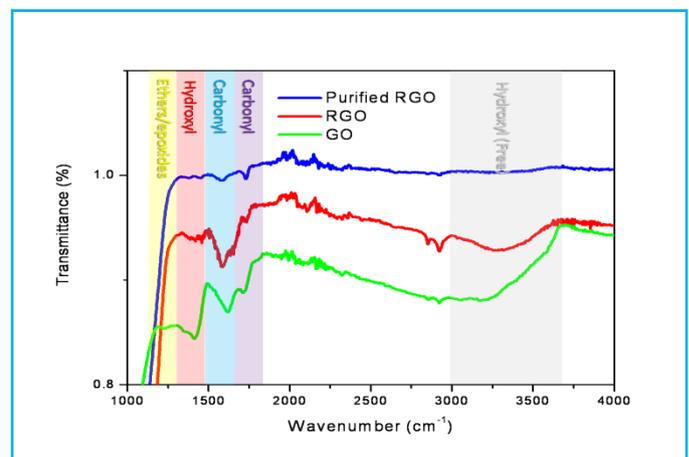


Figure 6. FTIR spectra of graphene oxide and graphene.

Conclusion

The obtained results in this work confirm that graphene sheets are fully exfoliated and the final graphene product is composed of a few pure layers. This work opens the way to combine graphene properties with other nanostructured materials to generate promising nanocomposites for optoelectronic applications as well as its integration in large fields of applications.

References

- [1] Geim A K and Novoselov K S 2007 Nat. Mater. 6 183.
- [2] Robinson J T, Perkins F K, Snow E S, Wei Z Q and Sheehan P E 2008 Nano Lett. 8 3137.
- [3] Jung J, Dikin D, Piner R D and Ruoff R S 2008 Nano Lett. 12 4283.
- [4] Ferrari A C, Meyer J C, Scardaci V, Casiraghi C, Lazzeri M, Mauri F, Piscanec S, Jiang D, Novoselov K S, Roth S and Geim A K 2006 Phys. Rev. Lett. 97 187.
- [5] Hummers W S Jr and Offeman R E 1958 J. Am. Chem. Soc 80 1339.
- [6] Li D. et al. 2008 Nature Nanotechnol. 3 101.
- [7] Guo P, Song H, Chen X 2009 Electrochem. Commun 11 1320.
- [8] Xu Y X, Bai H, Lu G W, Li C and Shi G Q 2008 J. Am. Chem. Soc 130 5856.
- [9] Li D, Muller M B, Gilje S, Kaner R B and Wallace G G 2008 Nature Nanotech. 3 101.