بیستمین کنفرانس اپتیک و فوتونیک ایران
و ششمین کنفرانس مهندسی و فناوری فوتونیک ایران
۸ تا ۱۰ بهمن ماه۱۳۹۲ - دانشگاه صنعتی شیراز

ساخت و مشخصه‌یابی گرافن با بازده بالا به روش لایه‌برداری گرافیت در حلال زیستی

معصومه اسدی[۱]، فاطمه زهرایی[۱]، کوروش رحیمی[۲]، مهدی غضنفری[۲]، احمد یزدانی[۲] و رضا پور صالحی[۲]

[۱]گروه فیزیک، دانشگاه آزاداسلامی، واحد قم، قم، ایران

[۲]گروه فیزیک، دانشگاه تربیت مدرس، تهران

چکیده - خواص منحصر به فرد گرافن، آن را به نامزد خوبی برای طراحی نسل بعدی دستگاه‌های الکترونیکی و نوری تبدیل کرده است. گرافن مانند دیگر مواد نوین از روشی مناسب برای تولید، با بازده بالا رنج می‌برد. در این مطالعه گرافن چند لایه با کیفیت بالا از طریق لایه برداری فاز محلول گرافیت در متیل پیرولیدن سنتز شد. وجود گرافن چند لایه را توسط طیف‌سنجی رامان ثابت شد و طیف سنجی فرابنفش-مرئی پیک جذب گرافن سنتز شده در ۳۱۰ نانومتر نشان داد که خود اثباتی بر وجود تشکیل گرافن می باشد.

کلید واژه- گرافن، متیل پیرولیدن، طیف‌سنجی رامان، طیف‌سنجی فرابنفش-مرئی

Synthesis and characterization of Graphene in high yield with exfoliation of graphite in organic solvent

Masoomeh Asadi[1], Fatemeh Zahraei[1], kourosh rahimi[2], Mahdi ghazanfari[2], Ahmad Yazdani[2] and Reza Poursalehi[2]

[1] Department of physics, Qom Islamic Azad University, Qom, Iran

[2]Department of physics, Tarbiyat Modares University, Tehran

Abstract- Graphene's unique properties make it a good candidate for the design of next-generation electronic and optical devices. Graphene suffers from a problem that is common to many novel Materials, the lack of a method for producing it at high yields. In this study high-quality few layer graphene was synthesized via solution-phase exfoliation of graphite in N-Methyle-pyrrolidone (NMP). The presence of multi-layer graphene confirmed by Raman spectroscopy and UV-visible spectroscopy showed the absorbance peak of synthesized graphene in 310 nm and it was a proof that graphene was composed.

Keywords: Graphene, NMP, Raman spectroscopy, uv-visible spectroscopy

[Downloaded from www.opsi.ir on 2026-11-05]

1 Introduction

The electronic and optical properties of graphene, a single atomic layer of sp^2-hybridized carbon forming a honeycomb crystal lattice, can be described in terms of massless Dirac fermions with linear dispersion near the Fermi energy [1]. high electron mobility at room temperature (250,000 cm2/Vs), exceptional thermal conductivity ($5000 Wm^{-1} K^{-1}$), superior mechanical properties with Young's modulus of 1 TPa[2] and novel effects such as an ambipolar field effect, a room-temperature quantum Hall effect and the breakdown of the Born–Oppenheimer approximation[3], have all been observed in graphene.

According to graphene properties, it can be used in optoelectronic device [4]. The tunability, provided by the electrical gating and charge injection in graphene-based optoelectronics devices, have been predicted to develop tunable IR detectors, modulators, and emitters[2].

Another usage of graphene is in optically transparent electrodes (OTEs). Graphene - based OTEs (G-OTEs), which exhibit optical transparency over a broad frequency range, low sheet resistance, robust chemical stability, and a wide electrochemical potential window [5].

Also it is shown that these G-OTEs offer great promise as electrodes for UV–Vis spectroelectrochemical studies due to their amazing properties [5].

However, reliable production of single- and few-layers graphene samples with a lot of unique properties, in high quality and yield is still a challenge [6, 7].

The standard procedure used to make graphene is micromechanical cleavage because in this method the product has good quality but such approach is limited by its low production [2, 3 and 8]. This gives the best samples to date, but the size of graphene that produced in this method is small [8] and it is difficult to envisage how to scale up this process to mass production [9]. Some works have also reported graphene growth on metal substrates [10], but this would require transfer of the sample to insulating substrates in order to make useful devices, by mechanical transfer or through solution processing. Recently, a large number of papers have described the dispersion and exfoliation of graphene oxide (GO) [11]. In fact GO is an insulator [12] rather than a semi-metal and is conceptually different from graphene. We can change the GO to graphene but it leaves a significant number of defects, which continue to disrupt the electronic properties of graphene [11].Thus, a non-covalent, solution-phase method to produce significant quantities of defect free, unoxidized graphene is urgently required. Liquid-phase exfoliation is a mass-scalable approach for the production of graphene [3, 7].

In this paper, we synthesized few layer graphene by exfoliating of graphite in organic solvent, N-methyl pyrrolidone (NMP). This method is a facile rout for producing graphene and the product has high quality and it can be used to produce graphene-based composites or films, a key requirement for many applications, such as thin-film transistors, conductive transparent electrodes for indium tin oxide replacement or for photovoltaic [11].

2 Materials and method

Graphite and NMP (98%) were supplied by the merck Company. In this study, 0.1 g of graphite dispersed in 100 ml of NMP were introduced in a 250 mL flask, the mixture was sonicated in an ultrasonic bath at room temperature for 24 h, Then, the dispersion was centrifuged for 30 min at 8000 rpm, and the residual solid graphite was removed. The solution including graphene and NMP, was filtered through PVDF filter (0.22nm) and then was heated in 180°C in 550mm Hg vacuum for 10 hours.

[Downloaded from www.opsi.ir on 2026-11-05]

3 **Results and Discussion**

The point group symmetry of ideal single grapheme layer is D_{6h}. There are two equivalent atoms per unit cell, giving six normal modes for the zone center. The irreducible representations for the zone center modes of a single layer are given by:

$$\Gamma^{graphene} = A_{2u} + B_{2g} + E_{1u} + E_{2g}$$

The A_{2u} represents translation perpendicular to the graphene plane and E_{1u} represents the translation in the grapheme plane and E_{1u} represents the translation in the graphene plane. The B_{2g} mode is an inactive optical phonon where the carbon atoms move perpendicularly to the grapheme plane. The E_{2g} mode is doubly degenerate Raman active optical vibration, where the carbon atoms move in graphene plane. In the first-order optical spectrum of graphene we expect only one Raman active E_{2g} optical mode at ≈ 1587 cm^{-1}.

If number of layers increases from graphene (n = 1) to graphite (n = ∞), frequency of G-band decreases, while G-band intensity increases.

Fig-1 shows the raman spectroscopy of graphene obtained after filtration of its dispersion in NMP by PVDF filter (pore size: 200 nm), to evaporate NMP, the filter paper placed in 550 mmHg vacuum at 180 ℃ for 10 h. The most prominent Raman features for graphitic materials are: a defect-induced D band at~1350 cm^{-1}, an in-plane vibration of sp^2 carbon at~1580 cm^{-1} (G band), and a two phonon double resonance process at about 2700 cm^{-1} (2D band) [11] The 2D peak can be used to identify graphene: the 2D peak is a single and sharp peak in the case of monolayer graphene, while in AB-stacked bilayer the 2D peak is composed by four bands. Graphite shows a broad and up-shifted 2D peak, which in first approximation can be fitted with two peaks. The 2D peak shape quickly evolves with the number of layers, so that the 2D band of a sample containing more than 8–10 layers is hardly distinguishable from that of bulk graphite [12].

The presence of graphene is clearly visible in figure 1 because the spectra showes clearly visible common features of graphene, The G line (1,591 cm^{-1}) and 2D lines (2,665, 2,719 cm^{-1}). However, the D peak (1,371 cm^{-1}) is only visible in the spectrum of the very small flake, as expected due to edge effects. Also the shape of 2D peak shows the few layer graphene has been composed.

Figure 2 shows the optical absorbance from 240 nm to 340 nm of graphene that synthesized with solution method. The absorption peak is in 310 nm and it shows that with increasing the wavelength absorption deacreases as it is expected for graphene.

So we showed the synthesized graphene with exfoliation of graphite in NMP has the good quality.

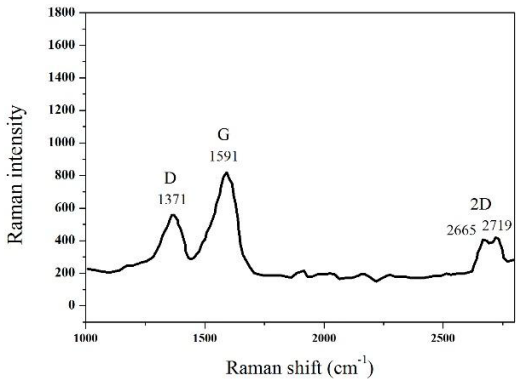


Fig.1: Raman spectra of graphene obtained after filtration of NMP and drying in 550 mmHg vacuum at 180 ◦C

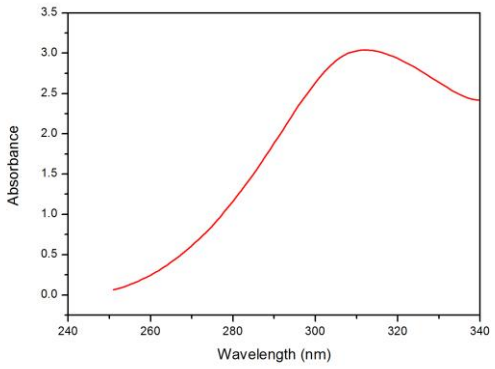


Fig 2: the UV-Vis absorption spectra of graphene

4. Conlusion

Results of room-temperature Raman scattering studies of graphene was reported.

Graphene electronic structure and electron–phonon interaction that evolves with the number of layers can be viewed in their Raman spectra. This established the Raman spectroscopy as a very helpful nondestructive tool in identification of graphene layers and studies of their properties.

References

[1] Q. Bao and H. Zhang, "Atomic-Layer Graphene as a Saturable Absorber for Ultrafast Pulsed Lasers", Adv. Funct. Mater, 19, 2009, 3077–3083

[Downloaded from www.opsi.ir on 2026-11-05]

[2] V. Singh and D. Joung, "Graphene based materials: Past, present and future, Progress in Materials" Science, 56, 2011, 1178–1271
[3] Y. HERNANDEZ and V. NICOLOSI, "High-yield production of graphene by liquid-phase exfoliation of graphite", nature nanotechnology, 3, 2008, 563-568
[4] V. V.Cheianov and V. Fal'ko, "The Focusing of Electron Flow and a Veselago Lens in Graphene p-n junctions", science, 315, 2007, 1252-1255
[5] C. M.Weber and D. M.Eisele, "Graphene-Based Optically Transparent Electrodes forSpectroelectrochemistry in the UV–Vis Region", 6, 2010, 184-189
[6] K. S.Novoselov and D. Ziang, "Two-dimensional atomic crystals", Proc. Natl Acad. Sci. USA, 102, 2005, 10451–10453
[7] H. Yang and Y. Hernandez, "A simple method for graphene production based on exfoliation of graphite in water using 1-pyrenesulfonic acid sodium salt", 53, 2013, 357–365
[8] Du. X.Skachko and A .Barker. "Approaching ballistic transport in suspended graphene" Nature Nanotech, 3, 2008, 491–495
[9] S. Y.Zhou and D. Siegel, "Origin of the energy bandgap in epitaxial graphene—Reply" Nature Mater, 7, 2008, 259–260
[10] P. W.Sutter, J.–I.Flege, "Epitaxial graphene on ruthenium" Nature Mater, 7, 2008, 406–411
[11] S. Stankovich and D. A.Dikin, "Graphene-based composite materials" Nature, 442, 2006, 282–286
[12] S. Stankovich and D. A.Dikin, "Synthesis of graphene-based nanosheets via chemical reduction of exfoliated graphite oxide" Carbon, 45, 2007, 1558–1565

[Downloaded from www.opsi.ir on 2026-11-05]