Review article

Graphene Synthesis, Properties and Applications

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ARTICLE INFO	
Corresponding Email. <u>m.badra@zu.edu.ly</u>	ABSTRACT
Received : 11-11-2023 Accepted : 07-12-2023 Published : 09-12-2023	Scientists and engineers have been very interested in graphene since 2004. Due to its exceptional properties, such as its large specific surface area, physicochemical properties, mechanical strength,
Keywords . Synthesis of Graphene, Nanomaterials, Chemical Vapor Deposition.	and exceptional inernal and electronic conductivity, graphene, a two-dimensional monolayer planar sheet of sp ² -bonded carbon atom, has seen a revolution in applications in recent years. To produce large amounts of high-
This work is licensed under the Creative Commons Attribution International License (CC BY 4.0). <u>http://creativecommons.org/licenses/by/4.0/</u>	quality graphene, various methods are employed. The synthesis of graphene using chemical, mechanical, and chemical vapor deposition processes is summed up in this study. Furthermore, the techniques for characterizing
	graphene and its applications in several fields of study have been covered. This article concludes by providing a brief overview and highlighting the issues of graphen and some of the recommendations that would push towards conducting scientific studies in the field of nanomaterials in more depth.

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INTRODUCTION

Numerous studies on the unusual physical, chemical, and mechanical properties of graphene have been investigated [1]. Graphene sheets exhibit nearly all of the remarkable characteristics of carbon nanotubes. Graphene is a single atomic thick layer of two-dimensional closely packed hexagonal carbon lattice, and its derivatives have attracted much attention because of flexibility of its bonding [2].

Graphene exhibit an unlimited number of different structures with an equally large variety of physical properties. These physical properties are mostly a result of the dimensionality of structures. currently, Graphene is a trending nanomaterial which is replacing silicon in different applications among numerous scientific area. This is according to their nano scale physical, mechanical, thermal, and chemical properties. Graphene has a thickness of only 0.334 nm, making it the world's thinnest materials [3].

Due to its unique properties graphene has numerous distinguishing qualities, such as a high specific surface area, strong electrical conductivity, excellent thermal conductivity, extreme optical transparency ,strong mechanical strength, high electron mobility, and ease of functionalization as shown in table1 [4-6].

Physical properties	Estimated value	Reference
Surface area	2600cm ² /g	[4]
Electrical conductivity	1738 Siemens/m	[4]
Thermal conductivity	$Wm^{-1}k^{-1}5000$	[5]
Optical transparency	97.4%	[5]
Mechanical strength	Young' Modulus ~1100 GPa	[5]
	Fracture strength ~125 GPa	[5]

Table 1	. Physical	properties	of	graphene
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Ease of functionalization	π - π stacking interaction Electrostatic interaction	[6]
Electron mobility	200,000 cm ² /Vs	[6]

Structure and properties of Graphene

Graphene is a single 2-dimensional (2D) layer of a hexagonal structure consisting of sp^2 -bonded two carbon atoms packed tightly in a honeycomb lattice crystal. and its derivatives have received increasing attention in several fields, due to its unique physical, chemical and thermal properties. Graphene is made out of carbon atoms arranged on a honeycomb structure made out of hexagonal, and can be thought as composed of benzene rings jointed where hydrogen atoms are replaced by the carbon atoms as shown in figure 1.

Figure 1. A schematic illustration of the hexagonal lattice structure of graphene [7]

The valence and conduction bands slightly overlap as a result of the fusing of these benzene rings, allowing electrons from the top valence band to move to the bottom conduction band without producing heat. The specific surface area is determined by the combination of the graphene layers. When other substances are added to pure graphene, the aggregation is reduced and the effective surface area is increased. Researchers have been very interested in graphene recently because of its remarkable mechanical, electrical, optical, magnetic, thermal, and optical properties as well as its huge surface area [7].

Synthesis

Several techniques have been employed recently to synthesize graphene .Graphene extraction is the term for this synthesis method, depending on the intended product's purity. Following the discovery of graphene in 2004, several methods were developed to create graphene layers and thin films. Although Top-Down and Bottom-Up approaches produce graphene of different quality. Some of the frequently utilized techniques for graphene synthesis are chemical vapor deposition (CVD), chemical exfoliation], chemical synthesis, and mechanical cleaving [8]. The synthesis methods of top-down and bottom-up are based on the average size, thickness, number of layers, and kind of graphene materials. Graphite and its derivatives, such as graphite oxide, are exfoliated and separated to create graphene sheets in a top-down growth mechanism process [9]. Some of graphene synthesis are illustrated in figure 2.



Figure 2. Schematic representation of the graphene synthesis. a) mechanical exfoliation. b) liquid phase exfoliation. C) thermal decomposition. d) CVD [10].

1. Mechanical exfoliation

The peel-off procedure or Scotch tape method are other names for mechanical exfoliation. Using an adhesive tape to press the graphene layers apart, Novoselov and Geim employed this method for the first time to produce graphene. With this technique, the graphene peels off the tape in layers, but as it peels off again, it cracks open into a handful of graphene flakes. In order to separate the tape, it is affixed to a specific substrate (acetone), and then it is peeled again using a new tape to produce flakes that are distinct in size and thickness and visible on surfaces under a light microscope. Due to the lengthy and inaccurate nature of this technique, graphene is most frequently studied for its properties rather than being used for commercial purposes [11].

2. Electrochemical exfoliation

In order to produce graphene in large quantities, a straightforward but highly productive method called graphite exfoliation by electrochemical techniques has emerged recently. This method uses graphite in a variety of forms, including foils, plates, rods, and powders, as electrodes in an aqueous or non-aqueous electrolyte with an electric current to cause the electrodes to expand. Depending on the amount of power provided, the electrodes could be either cathodic (negative) or anodic (positive) [12]. In their investigation, Wang et al, employed Pure Graphite as the electrode material and PSS (Polysodium-4-styrenesulfonate) dissolved in deionized water as the electrolyte. They were positioned alongside the graphite rods in an electrolyte-filled electrochemical cell. A constant 5 V current was used. After a few minutes of electrolysis, a black substance accumulated at the anode. For four hours, the exfoliation procedure was done in order to separate the product from the cell. After centrifuging it for a full minute at 1000 rpm, the result was slowly poured out. It was discovered that the obtained dispersion was remarkably steady. Dry graphene powder was produced by vacuum-drying the dispersion after it had been cleaned with alcohol and deionized water. Next, the dry powder and sediment were weighed in order to determine the yield. Keshri et al, have demonstrated the chemical- and solvent-free graphite exfoliation using plasma spraying with high conductivity and very low defect density as shown in fig (3) [13].



Figure 3. (a) Graphite plasma spray exfoliation, (b–d) Raman spectra of the synthesized graphene, the basal plane and the edge of graphene flakes [14].

3. Chemical vapor deposition

A bottom-up synthesis method called chemical vapor deposition (CVD) is employed to produce high-quality graphene on a massive scale. Since the method was first reported in 2008/2009, chemical vapor deposition (CVD) has become a significant method for producing graphene for a variety of applications [15]. In this account, we review graphene CVD on a variety of metal substrates, with a focus on Ni and Cu.In this process, a gas molecule and a surface substrate are combined inside a reaction chamber with certain gas flow rate, pressure, and temperature parameters. A quartz reaction chamber, a mass flow controller, a pump, thermocouples for temperature measurement, a gas delivery system, a vacuum system, an energy system, and a computer for auto-control are all common components of a CVD equipment [16]. For the formation of graphene films, CVD uses a variety of substrates, including Nickel (Ni), Copper (Cu), Iron (Fe), and stainless steel. Common carbon sources include acetylene (C_2H_2) and methane (CH₄). The carbon source is activated using two CVD processes: thermal CVD and plasma-enhanced CVD (PECV).

Vacuum tubes, high-temperature heating furnaces, vacuum pumps, vacuum gauges for controlling pressure, and mass flow controllers for controlling the carbon and career gas used in the synthesis of graphene are the components of thermal CVD. In PECVD, the gas source decomposes due to plasma, which subsequently combines with the metal substrate to produce the formation of graphene films. As a plasma source, power sources like radio frequency, microwave, and direct current (DC) have been employed. The ability of PECVD to produce graphene at low pressure and temperature is a benefit over thermal CVD [17]. Carbon source breaks down into hydrogen and carbon atoms at a high temperature.

In order to eliminate and clean undesirable oxides from the metal catalyst surface, this CVD process uses argon and hydrogen gas as carrier gases. Graphene has traditionally grown by CVD on transition metal substrates like Cu and Ni. This growth process consists of two basic steps: (i) pyrolysis of the gas precursor to create carbon, and (ii) use of the segregated carbon on the metal catalyst surface to form the carbon structure of graphene . To produce graphene, for example, polycrystalline nickel is first annealed in an H₂ environment at a temperature of 900–1000 °C with a grain size of that desired. The substrate is exposed to a combination of H₂/CH₄ gases, whereby CH₄ serves as the carbon source.[18] The carbon atom dissolves in the Ni film where the solid solution forms as a result of the hydrocarbon's breakdown. Because nickel is highly soluble at high temperatures, its solid solution is cooled in argon gas to produce a precipitate called Ni-C, which etches graphene. Ni is a suitable substrate for graphene synthesis, but the amount and size of monolayer graphene can vary depending on the quality of the Ni film. The thickness and quality of graphene are influenced by the cooling rate, and the development of graphene shape can also be influenced by the Ni microstructure. fig(4) [19].



Figure 4. a) CVD furnace system, (b) Raman spectra and mapping of the produced graphene, (c) CVD apparatus, (d) raman spectra[20]

Characterization of graphene

An essential component of graphene study and research is its characterization. Graphene shape, characteristics, flaws, and layers are investigated by spectroscopic and microscopic studies in the characterization process. Characterization techniques include X-ray diffraction (XRD), Raman spectroscopy, Transmission Electron Microscope (TEM), Scanning Electron Microscope [21]. Through Raman spectroscopy can be used to investigate graphene layers and structural quality. The molecular vibration of graphene interacts with the monochromatic light of Raman spectroscopy, causing a scattering-related change in radiation . In graphene, three primary peaks are recognized, namely the D, G, and 2D peaks. peak is seen at 1350 cm⁻¹, suggesting that sp2 hybridization is disordered. Lattice vibration is represented by the G peak, which is positioned at 1580 cm⁻¹, and second-order Raman scattering at the Dirac point is the source of 2D, which is located at 2700 cm⁻¹ Fig (5).

The ratio I_D/I_G rises when graphene disorder develops because of elastic scattering brought on by greater defect intensities. Nonetheless, the I_D/I_G ratio falls as the carbon structure gets more amorphous. Raman analysis of N-layer graphene's ion-induced flaws. The stretching of the C–C bond causes graphene's Raman spectrum, sometimes referred to as the G band, to display the G mode. It is distinguished by a prominent peak at 1580 cm⁻¹, the first-order permitted feature in the Raman spectra, which originates from the zone center (photon wave vector q = 0). The most popular method for examining the number of layers and structural quality of graphene is the Transmission Electron Microscope (TEM). When the electron beam interacts with the substance being studied, TEM images are created [22].



Figure 5. Raman spectra of graphene deposited on alumina substrate with Ni electrode [23]

Based on cell dimension units, the X-Ray Diffraction technique is primarily used to determine the material's phase. The XRD of graphene, graphite oxide, and graphite is shown in fig (6). Graphite exhibits a high and severe diffraction peak at 26.6 degrees. The existence of an oxygen molecule is then indicated by the peak shifting to 13.3 degrees. There is no rise after fabrication, suggesting that graphene was produced artificially. Graphene oxide and pure graphene are two types and layers of graphene that can be characterized using UV–visible spectroscopy. Monolayer graphene oxide has an absorption peak at around 230 nm due to the uV-visible absorbance, which is caused by the transition of electrons at carbon pi bonds (π - π *transition), while pristine graphene exhibits an absorption peak between 250 and 270 nm. UV transmittance can be used to study the thickness and number of layers in graphene. AFM, or atomic force microscopy, is used to measure the thickness and surface structure of graphene.

Using a tiny cantilever to scan a sample's surface, an AFM creates images. The cantilever is bent and the amount of laser light reflected into the photo-diode is altered when the sharp nanoscale tip at the end of the cantilever makes contact with the surface. After that, the cantilever height is changed to bring back the response signal that was obtained by measuring the cantilever height as it traced the surface [24].

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Figure 6. XRD spectra of graphene, graphite oxide and pristine graphite [23]

Applications of graphene

Graphene is widely used in many various applications because of its exceptional qualities, which include its light weight, electrical conductivity, great mechanical strength, and thermal strength. the most important applications are energy storage, water filtration, coating, transistor and diagnosis of viruses.

Batteries and capacitors

Because of its large surface area, graphene is used in energy storage devices like batteries and supercapacitors. Graphene is a common anode material in Li-ion batteries, with a capacity of approximately 1000 mAh g⁻¹, three times greater than graphite electrode. Moreover, graphene provides speedier recharge times in seconds and longer-lasting batteries (fig.7a). Moreover, graphene is employed as a printed solid-state supercapacitor in textiles for wearable electronics because of its flexibility. As seen in Fig. 7b, the theoretical specific energy density rises as the supercapacitor's graphene content rises [23].



Figure 7. Schematic diagram of (a) graphene supercapacitor structure (b) theoretical specific energy density of graphene supercapacitor at different graphene's density content [25]

Transistors

Because of its incredibly high carrier mobility at room temperature, graphene is used in transistors. One of the promising applications of the graphene its use in field effect transistors [26]. Lemme et al, introduced that since graphene is a conductive substance, transistor applications cannot directly use it. Graphene, however, functions as a semiconductor when it takes the form of nanoribbons. This implies that it can have a band gap and be switched on and off, making it a potentially important part of nanotransistors. The band gap can be obtained by patterning designed with values as high as 400 meV. Vicarelli et al, employed antenna-coupled field-effect graphene transistors to detect

terahertz radiation. Since then, field-effect graphene transistors have been used to sense proteins, biomolecules, cells, gas, and DNA [27].

Biomedicine

The significant applications of graphene derivatives in biology and medicine are made possible by their biocompatibility [28]. The early diagnosis of viruses is one of the most important uses. for instance, Miranda et al, created an electrical biosensor based on graphene for the early identification of malaria parasites (Fig. 8a,b), Likewise, Walters et al, created a graphene sensor platform for rapid hepatitis virus detection. Moreover, even with lesser volume samples, such as 5 µL, reported a detection limit down in less than 4 minutes. The sensor may be simply modified to detect markers of various viral diseases, including HIV, SARS-CoV-2, hepatitis B virus, and many more. The use of face masks in the fight against the COVID-19 epidemic has become essential. Huang et al, found that in antibacterial masks modified with laser-induced graphene, the bacterial inhibition rate could be enhanced by up to 80% when paired with the photothermal action of the graphene layer, resulting in 99.998% bacterial killing effectiveness within 10 min. In less than five minutes, a graphene-coated filter paper sensor could identify COVID-19. Gold nanoparticles encapsulated with single-stranded nucleic acid (ssDNA) probes unique to the SARSCoV-2 RNA were placed on this sensor surface. The ssDNA interacted with these probes when SARS-CoV-2 RNA was present. The electrical response of the sensor changes as a result of the viral RNA hybridizing with the ssDNA sequence, which raises the charges at the graphene-solution interface. When comparing positive samples to negative samples, the sensor found a noticeable rise in voltage, and it was almost 100% accurate in confirming the presence of the virus (fig. 8c) [29].



Figure 8. (a) The 2DBioFET, (b) the results of PfLDH detection in human serum by the 2DBioFET, (c) schematic of the electrochemical sensing platform for COVID-19 [30].

Tissue engineering and drug delivery systems

Graphene's unique properties, including conductivity, mechanical stability, and biocompatibility, have made it an effective material to utilize in drug delivery systems. Hydrophobic molecules are adsorbed by a mixture of graphene oxide and polymers such as polyethylene glycol (PEG) and poly (vinyl alcohol) (PVA). Hydrogels have efficiency in biological drug delivery systems. One instance is the application of multiwalled carbon nanotubes in methacrylic acid hydrogels to improve the electrical response [31].

CONCLUSION

This review covers the structure, characteristics, synthesis techniques, characterizations, and applications of graphene along with a brief history. As a result of its exceptional and distinctive qualities, including its high tensile strength, high electrical conductivity, high carrier mobility, high elasticity, high thermal conductivity, and approximately 97% optical transparency, graphene is regarded as one of the most attractive functional nanomaterials in existence. Following its discovery in 2004, graphene has drawn significant interest from scientists and engineers worldwide for its potential applications in a variety of fields, including sensors, batteries, biomedicine, transistors, filtration technologies, and supercapacitors.

For electrochemical energy storage devices, including lithium batteries and supercapacitors, its high surface area and nonflammable nature make it an ideal electrode material that enhances device performance in comparison to conventional carbon electrode materials. Graphene is used as a sensor in medical equipment because of its great

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sensitivity, especially for the detection of specific viruses like COVID-19 and malaria. Currently, the large-scale synthesis method for producing high-quality graphene is chemical vapor deposition. But in order to fully investigate graphene's economic potential, a few important issues like toxicity, long-term stability, and environmental consequences must be resolved.

Recommendations

We recommend supplying the funds required for scientific research, developing scientific knowledge, and establishing a connection with the developed world through visits, experiences, and resource exchanges. Moreover, establishing a center specialized to nanotechnology and providing it with modern equipment and devices as well as qualified staff members.

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تركيب الجرافين وخصائصه وتطبيقاته مريم بدره¹* , سمية السطي² ¹قسم الفيزياء، كلية العلوم العجيلات، جامعة الزاوية، ليبيا ²قسم صحة الاسرة والمجتمع، كلية العلوم الصحية, جامعة الزاوية, ليبيا

المستخلص

لقد أبدى العلماء والمهندسون اهتمامًا كبيرًا بالجرافين منذ عام 2004. ونظرًا لخصائصه الاستثنائية، مثل مساحة سطحه النوعية الكبيرة، وخصائصه الفيزيائية والكيميائية، وقوته الميكانيكية، والتوصيل الحراري والإلكتروني الاستثنائي، فإن الجرافين عبارة عن صفيحة مستوية أحادية الطبقة ثنائية الأبعاد من - sp² ذرة الكربون المترابطة، شهدت ثورة في التطبيقات في السنوات الأخيرة. لإنتاج كميات كبيرة من الجرافين عالي الجودة، يتم استخدام طرق مختلفة. يتم تلخيص انتاج الجرافين باستخدام عمليات ترسيب البخار الكيميائية والميكانيكية والكيميائية في هذه الدراسة. تمت تغطية تقنيات توصيف الجرافين وتطبيقاته في العديد من الجرافين عالي والكيميائية في هذه الدراسة. وإلقاء الحرو في هذه الدراسة. عليات ترسيب البخار الكيميائية والميكانيكية والكيميائية في هذه الدراسة. وإلقاء الحرو على قضايات ترسيب البخار الكيميائية والميكانيكية والكيميائية في هذه الدراسة. وإلقاء الضوء على قضايا الجرافين وتطبيقاته في العديد من المجالات المختلفة . ويختتم هذا المقال بتقديم لمحة موجزة النانوية بشكل أكثر تعمقا.