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Graphene Deposited Conducting Polymers: Synthesis and Sensing Applications

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Abstract

Graphene and its derivatives, with outstanding characteristics corresponding to smart electrical and mechanical properties, received greater attention of researchers to explore novel materials with potential applications. Recently, graphene based novel nanocomposite materials and conducting polymers such as polyaniline (PANI), polypyrrole (PPY), polyethyldioxythiophene (PEDOT), polythiophene (PTH) and their derivatives are widely utilized as active materials in gas sensing because of their distinctive electrical conductivity, redox property, and optimal working capacity at room temperature. The sensing performance is enhanced by combining these two materials, namely, pure graphene and conducting polymers. This may be attributed to the enhanced specific area of the nanocomposites. The general methods of preparation of nanocomposite preparation involves in situ polymerization, electropolymerization, resolution mixing, self-assembly approach, etc., The effective gas sensing performance of those prepared nanocomposites has been established by various researchers. In this point of view, recent efforts and applications of graphene and conducting polymer nanocomposites for sensing are discussed in this review paper.

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Graphene, conducting polymer, nanocomposite, electropolymerisation, sensing applications.

Introduction

Graphene consists of uncombine thin two-dimensional layer of sp^2 carbon atoms in a hexagonal structure. It has been shown to have many preferable properties, such as high mechanical strength (1), electrical conductivity (2), molecular barrier capacity (3) and other remarkable characteristic properties. For these reasons, the target of countless studies is to incorporate graphene into polymers to produce polymer-based nanocomposites (4-7). However, because of the original graphene has complex bottom –up synthesis (8), poor solubility and solution agglomeration due to vander Waal’s interaction (2)proved difficult. When using the original graphene alternatively, the top-down method can be used to synthesize graphene like compounds from graphite or other carbon sources, thereby obtaining more benefits of graphene when the surface is impregnated with functionalized oxygen blocks. The graphite oxide produced by the solvent is formed by stacking multiple layers of graphene on top of every single graphene oxide (GO).

Graphene oxide (GO) is a thick two dimensional material and it is a graphene derivative with various oxygen–containing functions(such as epoxide, hydroxyl and carboxyl) (9). Contrary to hydrophobic graphene, GO is hydrophilic and hence, it is water soluble (10). GO has a honeycomb carbon structure associated with many local defects in the interior, surface and edges which are caused by oxidation (11). Graphene oxide possess medicinal applications due to its solubility in aqueous media, greater colloidal stability, cost efficiency, scalability, large surface area and thermal properties(12-14). Also, graphene oxide has an extraordinary ability to immobilize a large number of substances, including metals, drugs, biomolecules, florescent cells and probes (15). It is reported that it has inherent absorption in the near- infrared region, making it useful as a photothermal agent for cancer treatment (9,13,14,16).

Graphene derivatives (Graphene Oxide (GO), reduced Graphene Oxide(rGO), Graphene Quantum Dots (GQD)) have been proven to be effective fillers in polymer nanocomposites as a result of their ideal material properties with good dispersing ability in the polymer matrices (17,18) which has led to more applications. They are almost ideal barriers for gas molecules (19,20) which illustrate its use in packing materials (17), protection of sensitive electronic device (20)and even corrosion –resistant materials (21-22). The fine–tuning of the filler content of the nanocomposite can be used to adjust the selectivity of certain–sized molecules to generate outstanding membrane technology (23,24). In addition, GO possess unique hydrophilic, thermal and electrical properties which can be used for materials that respond to stimuli-responsive nature. (25,26). This review aims to summarize the synthesis methods, unique material properties (mechanical, electrical, thermal etc.) of functional GO/rGO nanocomposites, and some typical applications (coating, film, corrosion protection and stimulation).

Conducting polymers synthesized through a typical oxidative polymerization method(27-29) have been widely used in many fields such as energy storage (30-34),electronic (35-37) and biological (38-43) due to their interesting properties like distinctive redox behaviour, adjustable electrical conductivity, inherent biocompatibility and high flexibility (30-43).However, despite its many advantages, there are some disadvantages of conducting polymers such as poor electrochemical stability, poor mechanical strength etc., often makes it difficult to handle practical application. In order to overcome this limitation, there is growing interest in mixing conducting polymer with other additives (38,44,45,46).

Conducting polymer with outstanding electrical conductivity, high electronic affinity, and low ionization capability play a vital role in biosensor performance. They are simply polymerized at the electrode surface through a noble salt or a dopant to enhance conductivity, catalytic activity and high surface area. The high conductivity is constantly a preferred characteristic to reduce the interface resistance among the electrolytes, ensuing in high signal response. In extensive sense, conducting polymers encompass- conjugated polymers and polymers whose backbone has varied single and double covalent bonds. Since their discovery, conducting polymers have been considerably used in the biomedical discipline for biosensor fabrication and tissue engineering. Conducting polymer have proven extraordinarily strong immobilization of biomolecules on their surface with whole retention in their activity. The most conventional methods used are cross-linking, covalent binding, physical entrapment and adsorption.

Among the conducting polymers, polypyrrole (PPY) and its derivatives were used significantly in biosensor construction. They are biocompatible and protect electrodes from fouling. In a few cases, they form selective films to exclude endogenous electrochemically high active interference. Polyaniline (PANI), another common conducting polymer, loses its conductivity at pH ranges above 4 and is normally used with any other conducting polymer, nanoparticles, or carbon nanomaterials, especially carbon nanotubes (CNT) and graphene.

Electrochemical polymerization (EP) involves the following three steps: (i) solubilisation of oligomers with inside the diffusion layer after monomer oxidation (ii) their deposition by nucleation which is observed by growth and (iii) chain propagation by solid –state polymerization (46). However, a standard mechanism for every polymer cannot be easily established as the technique is governed by working parameters, besides for the preliminary oxidation step (46,47). The properties of eletropolymerized films are based upon electrode materials, dopants, electrolyte, pH, and electrochemical deposition methods. Comprehension of nucleation and increase kinetics permits for the tailoring of polymer properties like density, morphology and crystallinity, consider with requirement. Conducting polymers with carboxyl or amino groups serve as immobilization matrices for covalent attachment of reputation molecules (48-51). In few cases, a composite of two polymers, PPY and PANI are used together to anchor the enzyme at the electrode (52,53). Composite materials are materials fashioned through combination of two or more materials with distinctive properties to provide an end material with specific characteristics. They are aggregation of two or more materials in composites results in choosing the benefit of the excellent properties and characteristics of each. Pristine graphene has a large surface area, outstanding electron mobility and conductivity and a extensive electrochemical window. However, many researchers have proven that pristine graphene may not exhibit significant benefits as an electrode material on its own and to enhance electrochemical activity with preferred functionality and electronic property, it is essential to introduce impurities (54).

Preparation methods of graphene polymer composites

Electrochemical in situ polymerization

The conducting polymer –Graphene composite is synthesized by the chemical or electrochemical insitu polymerization of monomers in the presence of graphene. Graphene-polymer composites are synthesized by combining a wide range of π- conjugated conductive polymer system with graphene (55).Incorporating nanofiller such as graphene into basic polymer has potential application in biosensor, energy storage devices, photocatalysts and drug delivery.

Recently a range of process routes have been reported for dispersing both graphene –polymer and GO-derived fillers into polymer matrices. The fabrication of polymer-graphene composite has been expedited by the utilization of ultra-sonication process for the dispersion of nanofiller, but the controlled quantity of weight percentage and size of the nanomaterial is rigorously taken into consideration (56). A majority of graphene-based polymer composites are investigated using graphene oxide, chemically reduced graphene oxide, or thermally reduced graphene oxide as fillers. Recently, three-dimensional graphene-based polymer nanocomposite are studied as a new generation material for numerous multi-functional applications.

In recent years, many research teams have carried out extensive investigation on graphene-polymer nanocomposites. Salimika *et al.*, (57) prepared a solid –phase microextraction fibre by polyaniline –graphene oxide nanocomposite as sorbent materials on the surface of a platinized stainless-steel wire using electrospinning technique. The nanocomposite structure was characterised by SEM and FTIR and used for determination of nicotine, the limit of detection of nicotine, linear dynamic range, intraday and inter-days precision were found to be 0.01mg/g, 0.05-700µg/g, at 6.9and 8.1 % respectively

Graphene based polymer composites possess significant enhancement in properties that cannot usually be performed by using conventional polymers (58).However, the remaining properties of graphene –based polymer nanocomposites are significantly based on their processing conditions (59). The effectiveness of these upgrades is related directly to the degree of dispersion of graphene within the polymer matrix interfacial strength, affinity of components, and spatial organization, identifying the ultimate stiffness, strength, toughness and elongation of polymer nanocomposite (59). The desire of the preparation method of graphene –based nanocomposites depends on polarity, molecular weight, hydrophobicity, surface functionalization of graphene and reactive groups of the polymer, graphene and solvent (58,59).As it is evident from the works referred in the remaining section, the three most composite are solution mixing (60-71) melt blending (71-73) and insitu polymerisation (74,75) other techniques have been occasionally mentioned for producing graphene- based composites such as the layer-by-layer technique(59).

Solution mixing/ solution casting

Solution mixing/ solution casting is a typically useful technique for developing graphene-based composites, particularly for biomedical applications, because it enables the separation of graphene sheets (76). It maximizes filler dispersion in the polymer matrix via using pre-suspended single –layered graphene sheets (59).The technique consists in three steps step:1 dispersion of fillers in an appropriate solvent using, for example ultrasonication, step:2 incorporation of the polymer and step:3 removal of the solvent by using distillation or evaporation (77). During this process, polymer coats graphene sheets, and when the solvent is evaporated, the graphene sheets reassemble, becoming interconnected with the polymer and forming the nanocomposites. The solvent compatibility of the polymer and the filler performs a crucial role in attaining good dispersion. Common solvents are water acetone, chloroform, tetrahydrofuran, dimethyl formamide or toluene. (58,77). The most important advantage of this approach is its simplicity, facile and fast fabrication step, high level of control on component behaviour and effectiveness for dispersing nanofillers in the polymer matrix. Besides, solution mixing permits the synthesis of intercalated nanocomposites based on polymers with low or even no polarity (58,59).Regarding the disadvantages currently reported about this method, the challenge in finding frequent solvents, toxic solvent utilization, thin –film limitation, difficulties in solvent removal and common aggregation troubles during mixing and solvent evaporation ranges are the most important ones (59,78,79) various polymer composites such as graphene or GO-poly(vinyl alcohol)(PVA), graphene-(Poly vinyl chloride) (PVC) thermally reduced graphene (TRG)have organized using this method(80).

Melt mixing /melt blending

Melt mixing /melt blending is identified as a sensible approach that can be used to produce graphene –based polymer nanocomposites. This is a solvent –free method in which a polymer melt and the graphene or modified graphene (filler in a dried powder form) are mixed under high mechanical shear conditions, through a screw extruder or a blending mixer (59,81). In this method, the polymers segment liquefies at high temperature and allows the dispersion or intercalation of GO and reduced graphene oxidesheets. This method is relevant to both polar and non-polar polymers (80). Comparing to solution mixing, the production of graphene-based composites by melt mixing can be used in massive scale and is more economical. However, melt mixing is less effective in dispersing graphene sheets due to the greater viscosity of the composite at increased sheets loading (80,81).

In situ polymerization

In situ polymerization requires the utilization of monomers, an initiator and a high temperature reactor (82). The method initiates with the dispersion of graphene or modified graphene in a liquid state monomer. An applicable initiator is then diffused and polymerization is initiated either by heat or radiation. It permits the homogeneous dispersion of graphene nanofiller within the polymer matrix and permits the covalent bonding between them through various condensation reactions. However, the high costs of monomer and high temperature vessel are the essential limitations of this process (58).

Biosensor applications

Graphene combined with polymer progresses the execution of biosensors as far as selectivity, sensitivity and reaction time of biosensor for clinical diagnostics, metal ion speciation, arrangements of batteries and capacitors. In recent pat, few investigators have focussed their attention on the manufacture of graphene based polymeric composites for biosensor application.

Electrochemical biosensors

Metal grafted functionalized graphene nanosheets with nanostructured polyaniline (PANI) hybrid nanocomposite is utilised for the detection of ascorbic acid(AA). The flexibility of the composite execution was corroborated by modifying size and density of electrodeposited gold and silver nanoparticles on the graphene oxide (GO), thermally reduced graphene oxide (rGO) and nitrogen functionalized graphene (NFG).The multilayers (PANI/AgNP/NFG) and (PANI/AuNP/NFG) on fluorine doped tin oxidecoated glass, graphite foil and graphene bar electrodes were tested that appear increased electrical conductivity and decreased charge transfer resistance.

It is reported that ultrasensitive identification of AA analyte over full extent with direct sensitivity $10mAmM^{-1}cm^{-2}$ and detection limit less than 1pM having signal –to-noise ratio in the order PANI/AuNP/NFG≤ PANI/AgNP/NFG (83).

Biomedical applications

Biomedical disciplines include a multidisciplinary scope which entails various paramount applications such as gene therapy, drug delivery, bioimaging, toxicology and regenerative medicine. The research on Graphene based polymer composites is an emerging active research area in the biomedical field (84-86).In fact, the rationale behind the use of graphene-based compound has been extensively carried out (87).Moreover, the encapsulation of particle with Graphene based molecule is opening up the way to novel multifunctional materials, imaging contrasts agent, gene/drug delivery systems, tumour treatment systems and stem cell differentiation monitoring systems. However, greater efforts are needed to study their biocompatibility, toxicity and attainable long-term consequences on living organism.

Immunosensor

The electrochemical immunosensor moderately becomes a preferred technique to find chemicals and biomolecules for its sensitive and selective response, because of their massive surface area, high conductivity and biocompatibility. Graphene and conducting polymers have attracted interest within the construction of nanomaterial-based amplification. In some cases, enzymes are utilized in immunosensors as labels to enhance the catalytic activity (89). The most important role of graphene family, particularly GO and RGO, used in biosensors to covalently bind antibody (Ab) and enzymes.

A novel estradiol immunosensor based on PANI/GE composites and carboxylated GO was developed by Li *et al.,* (89).PANI/GE, poly (amidoamine)-gold nanoparticles composites and antibody are in turn coated onGCE to fabricate a modified electrode.HRP –GO-Ab conjugates are prepared by covalent immobilization of HRP and Ab to the carboxylated GO. Finally, further ß-estradiol and HRP-GO-Ab are applied to the modified electrode to facilitate the competitive immunoassay. The immunosensor shows a large linear response to estradiol within the range 0.04-7.00ngml^{-1} and a LOD of 0.02ngml^{-1} during this design. PANI/GE composites are accustomed enhance the electroactivity and stability of the electrode and to enhance the present response of the immunosensor. The carboxylated GO is employed because of the carrier of the antibody and HRP is as the label improved the catalytic activity for hydrogen reduction of the electrode.

Biosensors for hydrogen peroxide and glucose

CP/GE composite could be a promising matrix of enzymatic sensor for the superior conductivity, very good stability, high surface area and good biocompatibility. Hydrogen peroxide is not only the product of the many oxidase- based enzymatic reactions, however, additionally an important intermediary in food, pharmaceutical, clinical industrial, and environmental analyses. For illustration, HRP is extensively utilized in hydrogen peroxide detection (90)is fabricated using a hydrogen peroxide biosensor supported PANI/GE composite film onto that HRP was entrapped. The sensor reacted and approximately about 98% of its consistent state current under 5sdue to large specific area, good conductivity, biocompatibility and quick redox properties of the modified electrode. The linear range convergence of H_2O_2 from 1.0 x10-6 to 1.6x10-4 M, with a slope of 294.9µA.mM^{-1} and a LOD of 0.8x10^{-7}M.PANI is utilized for the catalysis of HRP in presence of H_2O_2.To enhance the conductivity and improve the electrocatalytic action of the modified electrode, carbon nanotube(CNT) and metal nanoparticles(NPs) may be consolidated into the enzyme sensor CP/GE composites, similar to a H_2O_2 sensor built by HRP enzymatically initiated deposition of PANI on the GE-CNT-Nafion/AuPt NPs changed GCE(91).The synergistic impact of GE-CNT hybrid materials, AuPt NPs, and the enzymatically initiated testimony of PANI give a electrochemical biosensor with an identification breaking point of 1.7x10-7M and linear range from 5.0x10-7 to 1.0x10-4 M. Comparable work was additionally revealed by Xia *et al.*, ().Utilizing HRP, Au NPS, graphene, and utilization PPY subordinate to build a hydrogen peroxide biosensor(92). The metal nanoparticles are utilized for the improvement of electrocatalytic action and signal magnification.

Biosensor for oligomers

Apatamers are linking chains of oligonucleotides or peptides, especially for specific goals with an electromagnetic aptamer sensor system developed (93). PPY nitrogen –doped multi-layer graphene (PPY-NDFLG) thin film polymerisation /chemical vapour deposition method used as a signal converter detection of vascular endothelial growth factor RNA aptamers. Apatamers are covalently linked to PPY-NDFLG. The high sensitivity in the field ultimately led to recognition of the target molecule at an very low concentration. The aptasensor has a fast durable response time, partially ultra-sensitive and efficient reusability. Due to its low cost, large surface area and excellent conductivity and conjugation properties, CP/GE nanocomposite it can be applied to bind DNA probes via non-covalent bonds for determination of DNA assembly. (94) developed two PANI/ERGO based DNA sensor changes Electrodes by voltmeter and EIS(95,96) respectively. Increased electrochemical reaction according to PXa. Many carboxyl groups of PXa-ERGNO membranes. Suitable for stable immobilization of probe DNA with amino groups at the 5th end via a covalent bond. EIS(Fe(CN)6)3-/4- and the dynamic detection range is 1.0x10-14 to 1.0x10-8 M with a detection limit of 4.2x 10-15M synthetic effect of integrated PXa-RGO nanocomposite(98).

Gas sensor

Materials with approximate temperature sensitive and selective gas detection at room temperature are effective in gas sensing. In recent years, environmental monitoring and prevention of health risks in industry attracted many research groups on the investigation and utilisation of grapheme and conducting polymer composites. Composites nano sensor (99) exploited, low cost, one step and GO-CuFe2O4 nanocomposites mediated by combusting pathways combine NH_3 gas sensor with outstanding electrical performance ability to characterize GO and detect CuFe2O4. The sensor designed forNH_3concentrations as low as 5ppm can be found at room temperature. (100) Polyaniline @Graphene /Nickel oxide /(Pani@G/NiO) polyaniline /Graphene (PANI/GO and polyaniline) materials are used to detect ammonia.

Fig.1

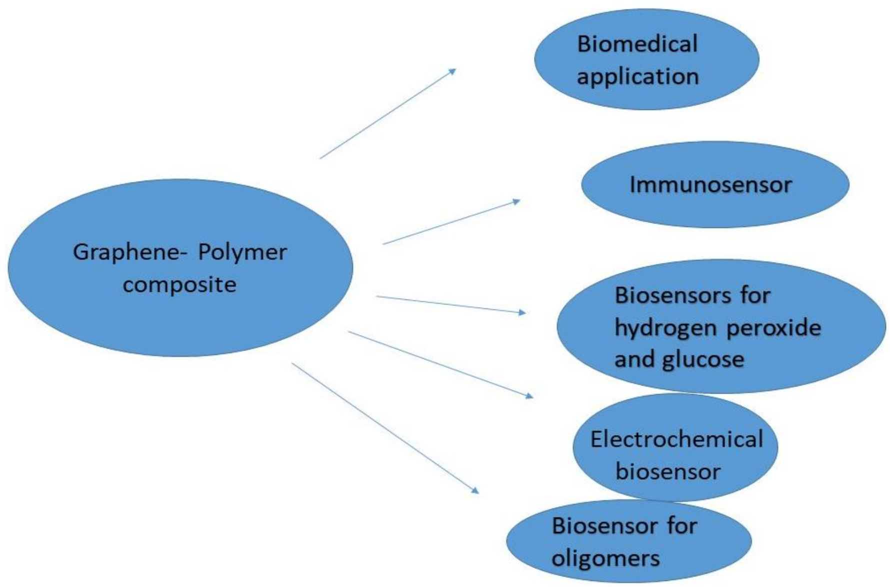
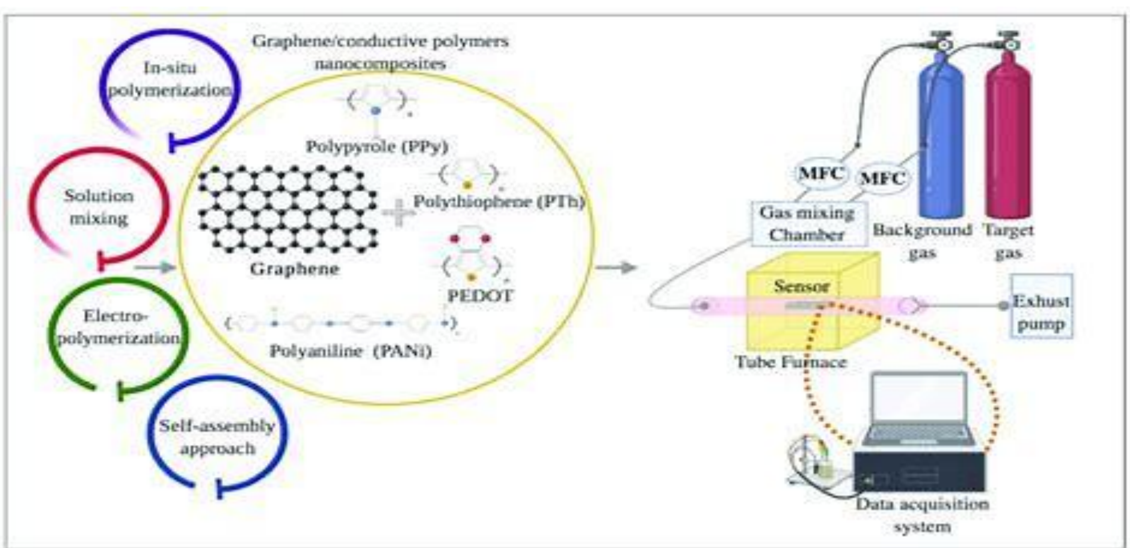


Fig.2 Preparation methods for graphene/conductive polymers composites and their application for gas sensing from Golnoush Zamiri *et al.*, (88)



This review article discussed on the importance of graphene and its polymer composites, in sensing and bio sensing applications. Various research organizations have mentioned the incorporation of graphene with conducting polymer composite and the consequent improvements observed in the mechanical strength as well as the electrical conductivity of the composites. Also, these Graphene -polymer composites exhibit enhanced thermal stability and electrochemical activity, as well as gas barrier properties. The presence of graphene in those composites improves the mechanical/electrical properties of biomaterials and increases cellular attachment and hence, increases the surface of biomaterials. Apart from these, the main benefit of these composites is that the effective changes arise even with a small quantity of graphene filler and in the presence of a number of polymeric compounds. Both graphene oxide and conducting polymer composites are used in many applications, including biosensor, electrochemical sensor and gas sensor.

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