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Polyaniline/graphene/carbon nanotubes nanocomposites for sensing environmentally hazardous 4-aminophenol

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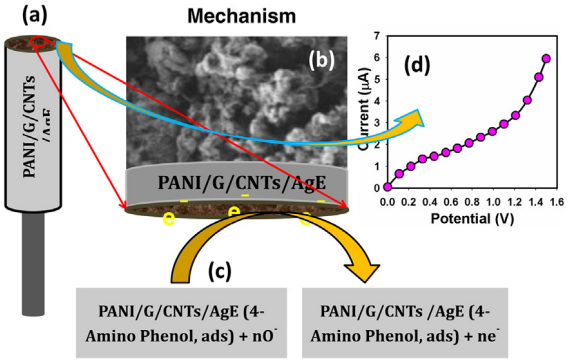
HIGHLIGHTS

- PANI/G/CNTs nanocomposites were synthesized by *in-situ* polymerization process.
- 4-aminophenol sensor based on PANI/G/CNTs/AgE is developed by reliable *I–V* technique.
- It is displayed the highest sensitivity (2.1873 $\mu A\ cm^{-2}\ \mu M^{-1}$) and lowest detection limit (63.4 pM) ever published in the literature.
- Real environmental effluents are analyzed.
- Environmental safety.

GRAPHICAL ABSTRACT



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ABSTRACT

New formulations of polyaniline/graphene/carbon nanotubes nanocomposites (PANI/G/CNTs) were synthesized *in-situ* polymerization process in acidic solution. Initially, the suggested nanocomposites were prepared with fixed weight from aniline monomer and different mixed ratio from graphene nanosheets and carbon nanotubes assembly (GNS/CNTs). The structure of the new formulation was confirmed by variable characterization techniques, including FT-IR, XRD, SEM, TEM and electrical conductivity etc. PANI/G/CNT_3 was considered as the optimized combination in materials composition. The PANI/G/CNTs nanocomposites were deposited on flat-silver electrode to result in a phenolic chemical sensor that has a fast response to selective 4-aminophenol (4-AP) in buffer system. Features including sensitivity, detection limit, reproducibility, selectivity, and electrochemical performances were investigated in details with the PANI/G/CNT_3 nanocomposites fabricated electrodes. The calibration plot (at +0.5 V) is linear over the large 4-aminophenol concentration range (0.1 nM to 0.01 M). The sensitivity and detection limit of target 4-AP is calculated as ~2.1873 $\mu A\ cm^{-2}\ \mu M^{-1}$ and 63.4 pM (at a signal-to-noise-ratio, SNR of 3) respectively. With such excellent features as wide linear dynamic range, higher sensitivity, good stability, lowest detection limit, excellent selectivity, the sensor provides a new strategy for determination of 4-AP in environmental real sample with satisfactory results.

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1. Introduction

Several conducting polymers are known in the past few decades, but polyaniline (PANI) is taken into account as the most important member owing to its singular conductivity behavior, electrochemical properties, excellent environmental steadiness, low-cost, facile polymerization consolidated with high standards of thermoelectric behavior and electrical conductance as well as optical properties [1]. It can be prepared either chemically or electrochemical method. The most reasonable method for the mass production is through oxidative polymerization using ammonium per-oxodisulfate as an oxidant [2]. Recently, considerable attentions were focused to develop the PANI properties through the fabrication of nano-composites by incorporating inorganic nanoparticles in the PANI structure [3–6]. Using this process, combination features that are complicated to obtain with the single components can be attained.

Determination of small toxic organic chemicals is a very significant task for environmental protection and human health. Usually, 4-aminophenol is mostly utilized for the production of lauryl aldehyde, lauric acid, light-green; furthermore, it can also be utilized as a common solvent, plasticizer, low temperature lubricant, as a flavoring and fragrance in food, cosmetics, pharmaceuticals, soap and for tobacco flavor. The existence of phenol above the authorized limits in food will not only critically affect the people's health, but also can badly contaminate the environment [7]. In many countries, it is listed as one of the first control toxic chemicals [8]. The improvement of techniques for the determination of 4-aminophenol is critical in terms of environmental and safety considerations. Up to now, various analytical techniques to detect the quantity of phenol are based on the use of gravimetric and ultraviolet spectroscopic methods and polaro-graphic analysis [9,10]. Luminescence quenching based chemical determination by luminescent materials offers an alternative that has been confirmed to be a much simpler, sensitive and convenient technique [11–14]; it mostly relies on the monitoring of transmission signals formed by the structural or electronic interactions between sensing nanomaterials and nanocomposite substrates. The sensor signal is significantly enhanced in presence of nanocomposites or functional materials whether doping or un-doping fabrication onto the sensor surfaces in chemical or biological systems. For the reason of environmental aspect by reliable chemical routes, various efforts have been made to establish simple, easy, economical, consistent, and reliable sensors utilizing composites [15–18]. To the best of our knowledge, no electrochemical technique has been reported for the sensitive detection of 4-aminophenol using doped nanocomposites in buffer phase till to date. Use of capillary zone electrophoresis, the determination of phenol has been also reported elsewhere. No stability demonstrating assay of phenol in bulk and solid dosage form could be outlined in the literature. The aim of the present study is the development of a simple, reliable, accurate and sensitive electrochemical technique for the detection of 4-aminophenol on the basis of nanostructure fabricated sensor. Nanocomposite material is broadly demonstrated for the detection of toxic molecules in electrochemical control process due to their several benefits over conventional chemical techniques in term of large-surface area for monitoring in healthcare and environmental fields. In conventional method, it exhibits the slower response, surface fouling, noise, unstable signals, and lower dynamic range as well as lower sensitivity with the nanocomposite electrodes towards phenolic detection. Hence, the modification of the sensor surface with PANI/G/CNTs nanocomposites is very urgent to attain higher sensitive, repeatable, and stable responses. Therefore, a simple and reliable I–V electrochemical approach is immediately required for relatively easy, convenient, and inexpensive instrumentation which displays higher sensitivity and lower detection limits compared to conventional methods.

Basically, nanocomposites based sensors owing to their many meticulous advantages over the conventional methods, such as high response, low charge, and portability, are widely employed for the detection of contaminated or toxic pollutants, chemical process control, and monitoring of air/water contamination in the environment [19–21]. Removal of phenolic compound ions from industrial wastewater is one of the most important issues in environmental and health science. Various methods were reported for the removal of carcinogenic compounds from industrial water effluents. There are some issues still remaining unsolved such as facile and low-cost green preparation of the nanocomposites material, removal efficiency in hazardous phenolic compounds, and facile reusability of the stable nanocomposite materials. In addition, the mesoporous nature of the nanocomposite material allows its facile recycling without significant loss of sensor efficiency. The excellent adsorption/absorption capacity of the nanocomposites, together with other advantages such as its reusability, easy separation, and environmentally friendly composition, makes it a suitable sensor for removal of target 4-aminophenol from environmental and industrial wastes.

Here, it is employed a simple method to prepare PANI/G/CNTs nanocomposites aggregation with virtually controlled morphology, which revealed a steady growth development in composites and their prospective applications. For PANI/G/CNTs nanocomposites, it is an excellent chemical sensing application to confirm the electrical properties as well as improve the development of frequent electronic and optoelectronic materials [22–25]. PANI/G/CNTs nanocomposites permit very sensitive transduction of the liquid/surface interactions to modify in the chemical properties. The significant prospect is to appearance a variety of morphologies offer different vision of modification of the toxic chemical sensing possessions. A low cost, portable and reliable phenolic sensor with high sensitivite, selective and fast response features is needed for the current network of sensor monitors, especially the pollution control in liquid phase and timely detecting of high local concentrations. PANI/G/CNTs nanocomposites have been used to fabricate a simple and efficient chemical sensor and assessed the chemical sensing performance selectively considering 4-aminophenol at room conditions. To the best of our knowledge, this is the first report for detection of target 4-aminophenol with prepared PANI/G/CNTs nanocomposites using simple, convenient, and reliable I–V technique in short response time.

2. Experimental

2.1. Materials and methods

Aniline analytical grade (BDH, 99%) was freshly purified before use using simple distillation method as mentioned previously in the literature [26]. Phosphoric acid (Merck, 97%), ammonium persulfate (BDH, 99%) were used without purification. Multi-walled carbon nanotubes (CNTs) with an average diameter of (110.0–170.0) nm was purchased from Sigma-Aldrich and used as received without any purification. Graphene nanosheet (GNS) was also purchased from Sigma-Aldrich and used as extradited. Target 4-aminophenol, monosodium phosphate, benzaldehyde, butyl carbitol acetate, disodium phosphate, ethyl acetate, dichloromethane, acetone, chloroform, ethanol, pyridine, *n*-hexane, isopropyl alcohol, and all other chemicals were in analytical grade and purchased from Sigma-Aldrich Company.

2.2. Instrumentation

FT-IR spectra were examined by using ATR smart part technique in the wave number range 4000–400 cm^{-1} using Thermo-Nicolet-6700 FT-IR spectrophotometer. Powder X-ray diffractograms were

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