UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF POLYANILINE-GRAPHENE QUANTUM DOT AND THE POTENTIAL FOR PYRENE DETECTION USING PHOTOLUMINESCENCE SPECTROSCOPY

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By

MAHNOUSH BEYGISANGCHIN

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Chairperson : Professor Suraya binti Abdul Rashid, PhD
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Detection of Pyrene as a toxic material is vital to possess a healthy, non-polluted and well sustainable environment since Pyrene is highly toxic and ubiquitous and is of environmental concern due to its adverse health effects. Several methods currently measure Pyrene concentration, divided into analytical and nanomaterial-based sensors/sensing. Although analytical methods are accurate and give reliable measurements, they are costly, require more extended preparation, heavy equipment, qualified operators, and a large volume of solvent in separation and extraction procedures. Moreover, nanomaterials-based sensors/sensing, particularly semiconductor quantum dots (SQDs), is ultra-sensitive, fast, and easy; however, the most significant issue related to SQDs-based probes is that there is worry regarding cadmium used in the core, which can potentially leach and further contaminate the environment after discarding the probes. Therefore, there is a need to develop a novel method which includes proper materials with a low limit of detection (LOD), cost-effective, easy, fast, simple, and user-friendly to overcome all those challenges. In this research, polyaniline-graphene quantum dot (PANI-GQD) nanocomposite films were prepared in different GQD concentrations (100 - 500) ppm by the chemical methods as a fluorescence nanomaterial, simple, sensitive, low cost and novel sensing element for the detection of Pyrene via photoluminescence (PL) spectroscopy. Before nanocomposite film preparation, PANI film was optimised using different acidic medium/dopant types (PTSA, CSA, Acetic acid, and HCl), PTSA concentrations (0.5% - 6%) selected acidic medium/dopants, and NMP concentrations (0.5% - 6%) as solvent. PANI and PANI-GQD nanocomposite films were characterized and evaluated using FT-IR, UV-vis, XRD, FE-SEM, EDS, TGA, four-point probe, and PL spectroscopy. The 1% toluene-4-sulfonic acid monohydrate and 3% N-Methyl-2-pyrrolidone doped PANI was introduced as optimized PANI film with a high conductivity value of 2.45 $(\Omega\ \mathrm{cm})^{-1}$, high PL intensity (excitation: 77334, emission: 37650), and low bandgap value of 2.54 (eV) due to orderly organized benzenoid and quinoid parts in its structure. In

PANI-GQD nanocomposite films, the carboxylic acid groups of GQD are well-doped optimized PANI films characterized by FT-IR and UV-vis. The morphology of the PANI-GQD nanocomposites exhibited a change from nanoflakes to nonspherical with increasing GQD concentration. The PANI-GQD in 300 ppm of GQD concentration was introduced as the optimized PANI-GQD nanocomposite film with a high conductivity value of 2.28 $(\Omega \text{ cm})^{-1}$, high PL intensity (excitation: 231982, emission: 161435) and low bandgap value of 2.39 (eV). The PL results revealed the interaction of optimized PANI and PANI-GQD nanocomposite films with Pyrene. The LOD for Pyrene was calculated at 6.61 and 0.40×10^{-9} mol L^{-1} (S/N = 5) in the linear range of $(0.001 - 10) \times 10^{-9}$ mol L^{-1} based on optimized PANI and optimized PANI-GQD nanocomposite films, respectively. Furthermore, the PANI-GQD nanocomposite film showed the lowest LOD of Pyrene. The obtained LOD was comparable with WHO standards and specifications for Pyrene, which is 3.461×10^{-9} mol L^{-1} (0.7 μg/l) in the environment. Thus, this study proposes PANI-GQD nanocomposite film as a novel sensing element for detecting Pyrene.

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sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SINTESIS DAN PENCIRIAN POLYANALINE-GRAPHENE QUANTOM DOT DAN KEBOLEHAN UNTUK MENGESAN PYRENE MENGUNAKAN SPEKTROSKOPI FOTO-PENCAHAYAAN

Oleh

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Pengesanan Pyrene sebagai bahan bertoksik adalah penting untuk memiliki persekitaran yang sihat, tidak tercemar dan mampan kerana Pyrene sangat toksik dan terdapat di mana-mana serta membimbangkan alam sekitar kerana kesan terhadap kesihatan. Kini, terdapat beberapa kaedah untuk menyukat kepekatan pyrene, dibahagikan kepada secara kaedah analitik dan sensor berasaskan bahan nano. Walaupun kaedah analisis adalah tepat dan memberikan ukuran yang boleh dipercayai, ia mahal, memerlukan penyediaan yang lebih lanjut, peralatan berat, pengendali yang berkelayakan, dan jumlah pelarut yang besar dalam prosedur pengasingan dan pengekstrakan. Selain itu, sensor berasaskan bahan nano, terutamanya titik kuantum semikonduktor (SQD), adalah ultra sensitif, pantas dan mudah; walau bagaimanapun, isu paling ketara yang berkaitan dengan probe berasaskan SQD ialah terdapat kebimbangan mengenai kadmium yang digunakan dalam teras, yang berpotensi boleh larut lesap dan seterusnya mencemari alam sekitar selepas pelupusan probe. Oleh itu, terdapat keperluan untuk membangunkan kaedah baru yang merangkumi bahan yang sesuai serta mempunyai, had pengesanan (LOD) yang rendah, kos efektif, mudah, cepat, ringkas dan mesra pengguna untuk mengatasi semua cabaran tersebut. Dalam penyelidikan ini, filem nanokomposit polyaniline-graphene quantum dot (PANI-GQD) telah disediakan dalam kepekatan GQD yang berbeza (100 - 500 ppm) dengan kaedah kimia sebagai bahan nano berpendarfluor, mudah, sensitif, kos rendah dan elemen penderiaan baru untuk pengesanan Pyrene melalui foto-pencahayaan (PL) spektroskopi. Sebelum penyediaan filem komposit nano, filem PANI telah dioptimumkan menggunakan medium berasid/dopan yang berbeza (PTSA, CSA, Asid asetik, dan HCl), kepekatan PTSA (0.5% - 6%) medium berasid/dopan terpilih, dan kepekatan NMP (0.5% - 6%) sebagai pelarut. Filem nanokomposit PANI dan PANI-GQD telah dicirikan dan dinilai menggunakan FT-IR, UV-vis, XRD, FE-SEM, EDS, TGA, prob-4 mata, dan spektroskopi PL. Asid sulfonik p-toluena 1% dan PANI doped N-Methyl-2-pyrrolidone 3% telah diperkenalkan sebagai filem

PANI yang dioptimumkan dengan nilai kekonduksian tinggi 2.45 (Ω cm) $^{-1}$, keamatan PL tinggi (pengujaan: 77334, pelepasan: 37650), dan nilai jurang jalur yang rendah iaitu 2.54 (eV) disebabkan bahagian benzenoid dan quinoid yang teratur dalam strukturnya. Dalam filem nanokomposit PANI-GQD, kumpulan asid karboksilik GQD ialah filem PANI yang dioptimumkan dengan dop dengan baik yang dicirikan oleh FT-IR dan UV-vis. Morfologi nanokomposit PANI-GQD mempamerkan perubahan daripada nanoflakes kepada bukan sfera dengan peningkatan kepekatan GQD. PANI-GQD dalam 300 ppm kepekatan GQD diperkenalkan sebagai filem nanokomposit PANI-GQD yang dioptimumkan dengan nilai kekonduksian tinggi 2.28 (Ω cm) $^{-1}$, keamatan PL tinggi (pengujaan: 231982, pelepasan: 161435) dan nilai jurang jalur yang rendah daripada 2.39 (eV). Keputusan PL mendedahkan interaksi filem nanokomposit PANI dan PANI-GQD yang dioptimumkan dengan Pyrene. LOD untuk Pyrene dikira pada 6.61 dan 0.40 × 10^{-9} mol L^{-1} (S/N = 5) dalam julat linear (0.001 - 10) × 10^{-9} mol L^{-1} berdasarkan PANI yang dioptimumkan dan PANI yang dioptimumkan -Filem komposit nano GQD, masing-masing. Tambahan pula, filem nanokomposit PANI-GQD menunjukkan LOD Pyrene yang paling rendah. LOD yang diperolehi adalah setanding dengan piawaian dan spesifikasi WHO untuk Pyrene, iaitu 3.461 × 10^{-9} mol L^{-1} (0.7 µg/l) dalam persekitaran. Oleh itu, kajian ini mencadangkan filem nanokomposit PANI-GQD sebagai elemen penderiaan novel untuk mengesan Pyrene.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

0D	Zero-dimensional
ACS	Advanced Chemicals Supplier
APS	Ammonium persulfate
AuNPs	Gold Nanoparticles
CNT	Carbon Nanotube
CPs	Conducting Polymers
CSA	Camphor Sulphonic Acid
Cu_2O	Copper (I) oxide
EB	Emeraldine Base
EDS	Energy-Dispersive X-ray
EEFM	Excitation-Emission Fluorescence Matrix
EPA	Environmental Protection Agency
ES	Emeraldine Salt
FE-SEM	Field Emission Scanning Electron Microscopy
FT-IR	Fourier Transform Infrared
G	Graphene
GC	Gas Chromatography
GNR	Graphene Nano- Ribbon
GO	Graphene Oxide
GQD	Graphene Quantum Dot
H_2O_2	Hydrogen peroxide
H_2SO_4	Sulfuric acid
HCl	Hydrochloric acid

HPLC	High-Performance Liquid Chromatography
ICPs	Intrinsically Conducting Polymers
IR	Infrared Spectroscopy
LbL	Layer by Layer
LOD	Limit Of Detection
MS	Mass Spectrometry
MWCNT	Multiwall Carbon Nanotube
NMP	N-Methyl-2-pyrrolidone
OMC	Ordered Mesoporous Carbon
PAHs	Polycyclic Aromatic Hydrocarbons
PANI	Polyaniline
PARAFAC	Parallel Factor
PCR	Polymerase Chain Reaction
PL	Photoluminescence
POPs	Persistent Organic Pollutants
PPM	Parts Per Million
PPP	Polyparaphenylene
PPV	Polyphenylvinylenes
PPy	Polypyrrole
PT	Polythiophene
PTSA	Toluene-4-sulfonic acid monohydrate
QDs	Quantum Dots
R	Resistance
RCF	Resistivity Correction Factor

RGO	Reduced Graphene Oxide
Rs	Surface Resistivity
Rv	Volume Resistivity
SERS	Surface-Enhance Raman Scattering
SPR	Surface Plasmon Resonance
SQDs	Semiconductor Quantum Dots
TEM	Transmission Electron Microscopy
TGA	Thermogravimetry Analysis
TiO_2	Titanium dioxide
US	United States
UV–vis	Ultraviolet-Visible
XRD	X-Ray Diffraction
ZnO	Zinc oxide

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Polycyclic aromatic hydrocarbons (PAHs) are organic pollutants in the environment. They can be found in the air, water, soil, and food (Balmer et al., 2019a). PAHs contain more than one fused benzene ring, and the crucial sources of PAHs are human activities, including the combustion of fossil fuels in the burning form of natural gas and coal oil, petroleum spills, industrial effluents, runoffs from agriculture, and so on (Baali & Yahyaoui, 2020).

Among the 16 PAHs recorded as significant compounds by the environmental protection agency (EPA), Pyrene is selected due to its ubiquitous environment and high toxicity (Qazi et al., 2021). It is also present at thousands of sites throughout the United States (US) and is involved in the bacterial co-metabolism of the potent carcinogen benzo[a]pyrene (Nzila & Musa, 2021). World Health Organization (WHO) and the United States Environmental Protection Agency (USEPA) have measured the maximum contamination level (MCL) of benzo[a]pyrene in drinking water to be 0.7 µg/L and 0.2 µg/L, respectively (Cotruvo, 2017; Organization, 2017).

Pyrene is strong electron donor material and can be combined with several materials to make electron donor-acceptor systems. In addition, Pyrene can be used in sensors, biosensors energy conversion, light-harvesting and production dyes applications owing to high fluorescence emission in the range of 375–405 nm (Bains et al., 2012; Mohapatra et al., 2022). Pyrene with high concentration can enter the human body using food webs and have devasting effects on human health such as lung cancer, bladder cancer, shrinkage of kidneys, and increased size and weight of the liver (Hussain et al., 2018; Mohapatra et al., 2022).

Conducting polymers (CPs) study dates back to the 1960s, when Pohl, Katon, and their co-workers, first prepared and characterized semiconducting polymers (Shirakawa et al., 1977). The investigation of the high conductivity of polysulfurnitride was a step in advancing research in CPs. Intrinsically conducting polymers (ICPs), also called ‘Synthetic Metals’, are new materials that include electrical conductivity properties like metals and semiconductors through a combination of the electrical, magnetic, optical, and environmental stability, mechanical and processability of the CPs.

Polyaniline (PANI), a type of ICP belonging to the family of semi-flexible rod polymers, was discovered about 150 years ago but has only gained widespread scientific attention since the early 1980s (Beygisangchin, et al., 2021). PANI has been more extensively studied than other ICPs owing to its tunable optical and electrical conductivity, easy synthesis, reversible doping/dead doping, extraordinary thermal and environmental stabilities, low cost, and excellent performance anti-corrosion properties. In addition, the electrical and optical features are highly reliant on the nature of the acid medium/dopant, dopant concentration, and other synthetic conditions (temperature, time, aniline monomer, oxidant monomer etc.). Owing to these properties, PANI shows an extensive variety of applications like electrochromic glasses (Lyu, 2020), solar cells (Zarrintaj et al., 2019), electroluminescence machines (Z. Li & Gong, 2020), sensors (X. Zhu et al., 2015), biosensor (Ramanavicius & Ramanavicius, 2021), supercapacitors (Iqbal et al., 2020), neural prosthesis/biotic–abiotic interfaces (Beygisangchin, et al., 2021), scaffolds (Ghorbani et al., 2020), delivery systems (Zare et al., 2020b), anti-corrosion material (Caldona et al., 2017), photovoltaic cells (Mohseni et al., 2021), and gas separation membranes (Elakkiya & Arthanareeswaran, 2022).

The study of carbon nanomaterials originated with the work on fullerenes and correlated compounds in the 1970s, and consequently, a remarkable increase in the study activity in the field has developed. Since then, graphene's appearance in 2010, discovered by Nobel laureates, fueled by an understanding of the properties of chemistry and the development of reliable production methods. This paves the way for new applications of carbon nanomaterials in general and graphene and their products in particular (Pirnat et al., 2015).

Recently, graphene quantum dot (GQD) can be introduced as a zero-dimensional (0D) material (P. Tian et al., 2018), and they can be observed as tiny pieces of graphene; these pieces have new exceptional phenomena owing to edge effects and quantum confinement (Ghaffarkhah et al., 2022). GQD is premier in its unique chemical, optical, and electrical properties, including low toxicity, biocompatibility, and high photostability against photobleaching and blinking. The most significant feature of GQD is the photoluminescence (PL) characteristics which can be obtained using PL (P. Tian et al., 2018). GQD has been considered an essential candidate for application in the sensor (Ghaffarkhah et al., 2021), optoelectrical detectors (Lei et al., 2022), biological imaging (P.-C. Wu et al., 2018), solar cells (Gao et al., 2021), fluorescent agent (Mehrzad-Samarin et al., 2017), light-emitting diodes (LEDs) (L. Yin et al., 2022), photocatalysis (Z. Zeng et al., 2018), the drug carries, and lithium-ion battery (Ji et al., 2017).

Moreover, PANI with carboneous materials such as graphene (G), reduced graphene oxide (RGO), and semiconductor quantum dots (SQDs) such as ordered mesoporous carbon (OMC) are ideal PANI-based composite as sensing materials owing to their excellent chemical, physical, optical, and electrical features in addition to excellent chemical stability (Bruchez et al., 1998; Nakashima et al., 2020; Roy et al., 2018; Ruecha et al., 2015b; Lin Tang et al., 2017; Yanmin Wang et al., 2020b; Dongzhi Zhang et al., 2019a). Nevertheless, processing an extraordinary performance based on PANI with several carboneous materials is still challenging regarding carboneous materials functionalization, processing method, matrix-nanofiller compatibility, and optimized processing parameters. Therefore, looking for eco-friendly replacements is vital and urgent.

Ruecha et al. (2015) prepared a sensitive sensor based on PANI-G composite via electrochemical for the detection of heavy metals such as Zn (II), Cd (II), and Pb (II) in the linear range of (1–300) µg L^{-1}, and LOD were reported at 1.0, 0.1 and 0.1µg L^{-1}, respectively. However, PANI-GO suffers from low solubility. Dongzhi Zhang et al.(2019a) established a sensitive and stable sensor based on PANI-SnO_2-RGO composite via a chemical method to detect glucose in the linear range of 50 ppb to 50 ppm, and LOD were reported at 0.05 ppm. Lin Tang et al. (2017) provided ordered mesoporous carbon (OMC)/self-doped PANI (SPAN) nanofiber composite for the detection of Hg^{2+} using the electrochemical method. The proposed biosensor demonstrated good conductivity and stability, a sensitive and excellent linear range of 10 fM^{-1} µM, and LOD of 0.6 fM (S/N = 3). However, the SQDs have suffered from intrinsic limitations like environmental hazards and heavy metal's potential toxicity (Schwitzguébel & Wang, 2007; Valizadeh et al., 2012).

1.2 Problem Statement

Detection of Pyrene as a toxic material is vital to possess a healthy, non-polluted and well-sustainable environment since Pyrene is highly toxic and ubiquitous and is of environmental concern owing to its adverse health effects. Furthermore, Pyrene commonly occurs as mixtures of congeners, and its toxicity has increased compared to that of Pyrene owing to synergistic effects. There are two methods to detect Pyrene: analytical and nanomaterial-based sensors/sensing. Analytical methods like surface-enhance Raman scattering (SERS) spectroscopy, Fourier transforms infrared (FT-IR), gas chromatography (GC), high-performance liquid chromatography (HPLC), and mass spectrometry (MS) (Rockwood et al., 2018; Schmidt et al., 2004; Soares et al., 2015; Varma-Basil & Bose, 2019; Ling Yang & Watts, 2005; Dong Zhang et al., 2018). Although these methods are accurate and give reliable measurements, most require a longer time for sample preparation (non-real-time), require qualified operators, are costly, are very bulky, and suffer from low detection limits. Moreover, a large sample volume and solvent are required in the separation and extraction processes. The most significant analytical challenge with detecting Pyrene is that it can occur at very low environmental concentrations. Despite such low concentrations, continuous exposure to these levels can adversely affect organisms. Therefore, sensitive methods for detecting Pyrene are needed to overcome these issues.

Researchers have been working on using several nanomaterials as sensing materials to replace approaches for the sensitive detection of Pyrene using fluorescence spectrophotometry, SERS, electrochemistry and surface plasmon resonance (SPR) methods in environmental water samples which will be detailed in chapter 2. Nanomaterial-based sensors/sensing also has advantages and disadvantages; for example, nanomaterials-based fluorescence, particularly SQDs, is ultra-sensitive, fast, and easy; however, the most significant issue related to SQDs-based probes is that there is worry regarding cadmium used in the core, which can potentially leach and further contaminate the environment after discarding the probes. Over-coating the cadmium core with fewer pollutants shells, has reduced this leaching risk (Kuzyniak et al., 2014). The SERS and nanomaterials-based SERS methods also suffer from decreasing sensitivity and poor reproducibility and repeatability since overuse and exposure of the SERS substrates to the incident laser beam can damage the substrate and reduce signal improvement by extension. This issue needs serious consideration when planning re-useable sensing platforms (P. Mosier-Boss, 2017). Nanomaterials-based electrochemistry is sensitive and cost-effective; however almost suffers from high operational temperature and poor reliability due to interference from sensing materials (C. Zhu et al., 2015b). The biggest challenge related to SPR for detecting Pyrene is mass transport limits and the cost of sensor chips and equipment. Therefore, the sensor/sensing system needs to replace proper materials to overcome mentioned issues, which are cost-effective, easy, fast, simple, user-friendly, and highly sensitive.

Furthermore, we are looking for the proper nanocomposite with high stability, high electrical conductivity, high optical properties, high PL properties, and good reaction and interaction with toxic materials. Several attempts have been made to prepare doped forms or composites to improve chemical, optical, electrical, and structural properties since PANI has poor electrical conductivity and processibility in its pure structure. The conductivity of PANI directly depends on the type of acid, which can be determined by measuring the bandgap and crystallite size of the acid. The conductivity increases with increasing the crystallite size and decreasing bandgap (Hatchett et al., 1999; Kulkarni et al., 2004; Sinha et al., 2009). Therefore selecting the proper acid medium and dopant concentration is vital since acid has a dual role in PANI synthesis.

PANI was primarily used in the field of sensors/sensing for its low limit of detection, low cost, high sensitivity, and stability (Sha et al., 2017). However, PANI-based sensors have potential advantages, but most suffer from poor processability (Kenry & Liu, 2018). Therefore, selecting the proper PANI composite-based sensors is vital to overcome these drawbacks. PANI-carboneous materials are ideal PANI-based composites as sensing materials owing to their excellent chemical, physical, optical, and electrical features in addition to excellent chemical stability (Bruchez et al., 1998; Nakashima et al., 2020; Roy et al., 2018; Ruecha et al., 2015b; Lin Tang et al., 2017; Yanmin Wang et al., 2020b; Dongzhi Zhang et al., 2019a). However, some PANI-carboneous materials, like SQDs, have suffered from intrinsic limitations like environmental hazards and heavy metal's potential toxicity (Schwitzguébel & Wang, 2007; Valizadeh et al., 2012). Moreover, processing an extraordinary performance based on PANI- carboneous materials is still challenging regarding carboneous materials functionalization, processing method, matrix-nanofiller compatibility, and optimized processing parameters. Therefore, looking for eco-friendly replacements is vital and urgent.

Recently, GQD, a new-style carboneous materials system, offers strong potential for suitable candidates to replace traditional SQDs. GQD is premier in its unique chemical, optical, and electrical features like non-toxic, biocompatibility, high photostability against photobleaching and blinking, high solubility in different solutions, and stable PL. The most significant feature of GQD is revealed in the PL properties, which can be obtained using the PL method (Ghaffarkhah et al., 2022; P. Tian et al., 2018). GQD-based composites were reported using fluorescence, electrochemical, SPR-based sensors, Förster Resonance Energy Transfer (FRET), and electrochemiluminescent. Nevertheless, GQDs issue related to sensitivity is still a primary challenge in the sensor/sensing application.

Recent research has shown that combining PANI and GQD is capable of electrical modification and the polymer matrix's mechanical features, mainly utilized in supercapacitors (M. Dinari et al., 2016; Gebreegziabher et al., 2020b; Jin et al., 2018; Luk et al., 2014; Mondal et al., 2015) and electrochemical biosensor (J. Cai et al., 2018; Hsu & Wu, 2019; Punrat et al., 2016; Dongzhi Zhang et al., 2019b).

Since there is still a gap in the literature on the fluorescence-based PANI-GQD nanocomposite film regarding studies reported, the present effort has focused on synthesising and characterising PANI-GQD nanocomposite film using PL spectroscopy.

1.3 Research Hypothesis

The PANI-GQD is a flexible nanocomposite to detect and interacts with toxic chemicals (Saisree et al., 2021). The PANI-GQD has a high potential to interact with Pyrene because it contains a carboxyl functional group and aromatic rings, which can interact with the PANI-GQD nanocomposite (Sadrolhosseini et al., 2020). Moreover, the GQD has high PL properties and PANI were attached to GQD within Van der Waals interactions at the edge of the molecules, as that is where electrons are made available to the polymer (Kausar, 2020). Therefore, Pyrene can interact with PANI and GQD. PANI has two roles in detecting Pyrene, including the absorber of Pyrene and providing the electron for enhancing the PL properties of GQD (Facure et al., 2020; Ghaffarkhah et al., 2021). In addition, GQD can also interact with the Pyrene and the optical properties of GQD change due to capturing the Pyrene (S. A. & P. B. C., 2020). By investigating the interaction of PANI-GQD nanocomposite with Pyrene, it is believed that a fundamental understanding and application of PANI-GQD nanocomposite in sensors and biosensors can be achieved.

1.4 Research Objective

The study aims to develop a novel sensing element for detecting Pyrene based on optimized PANI and PANI-GQD nanocomposite films via PL spectroscopy. The specific goals of this study are as stated below:

1. To investigate the effect of acidic medium type (Toluene-4-sulfonic acid monohydrate (PTSA), Camphor sulphonic acid (CSA), Acetic acid, and Hydrochloric acid (HCl)), dopant (PTSA) concentration, and solvent (N-Methyl-2-pyrrolidone (NMP)) concentration on the properties of PANI film.

2. To investigate the effect of GQD concentration on the properties of PANI-GQD nanocomposite film.

3. To demonstrate the successful detection of Pyrene using PANI-GQD nanocomposite film by PL spectroscopy.

1.5 Research Scope

This study's challenges refer to the synthesis, characterization, fabrication and testing of PANI-GQD nanocomposite film for detecting Pyrene in the solution via PL spectroscopy. Since the body of knowledge is quite uncertain in this area, it is hard to determine the outcome at the beginning; especially considering that there has not been an agreement on the mechanism of optimized PANI-GQD nanocomposite formation. The scope of work is listed below to achieve the research objectives which were stated earlier. The scope of work covers:

To investigate the effect of acidic medium type (PTSA, CSA, Acetic acid, and HCl), dopant (PTSA) concentration, and solvent (NMP) concentration on the properties of PANI film;

1. The effect of acidic medium type, i.e. PTSA, CSA, Acetic acid, and HCl, on the chemical, optical, structural, morphological, thermal, and electrical properties of PANI films.

2. The influence of PTSA concentration in the range of (0.5 - 6) % on the chemical, optical, morphological, and electrical properties of PANI-PTSA films.

3. The effect of NMP concentration as a solvent in the range of (0.5 - 6) % on the 1% PTSA doped PANI films.

To investigate the effect of GQD concentration on the properties of PANI-GQD nanocomposite film;

1. The effect of GQD concentration in the 100-500 ppm range optimises PANI film's chemical, optical, morphological, and electrical properties.

2. Comparison of achieved chemical, optical, reconfirming, structural, morphological, chemical compositional, thermal and electrical conductivity properties related to optimized PANI and PANI-GQD nanocomposite film; using FT-IR, Ultraviolet-Visible (UV–vis), Raman spectroscopy, X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FE-SEM), Energy-Dispersive X-ray (EDS), Thermogravimetry Analysis (TGA), and four-point probe, respectively.

To demonstrate the successful detection of Pyrene using PANI-GQD nanocomposite film by PL spectroscopy;

1. The interaction and mechanism of optimized PANI and PANI-GQD nanocomposite films when interacting with different concentrations of Pyrene in the range of (0.001-10) × 10^{-9} mol L^{-1}. GQD can strongly cap the optimized PANI with hydroxyl and carboxyl groups at the edge of the GQD molecule, and optimized PANI have high optical properties so that they can donate their free electron to the GQD molecule.

2. Detection of Pyrene in the (0.001–10) × 10^{-9} mol L^{-1} range using optimized PANI and PANI-GQD nanocomposite films via PL spectroscopy. The mechanism of fluorescence property of optimized PANI and PANI-GQD nanocomposite films correspond to the surface-related defective sites, which generally refer to any sites with nonperfect sp^2 domains that will result in surface energy traps.

1.6 Significance of the Study

The sensitive, accurate, fast, and novel sensing element is a fascinating subject to measure the low concentration of Pyrene using the PL method. Two significant reasons drive the motivation to remark in this study. Firstly, because of no communication has been reported by the scientific community on functionalized PANI-GQD nanocomposite using the PL method. The second motivation is that PANI-carboneous materials are superior for tremendous morphological, sensing, conducting, and capacitance features (Gómez et al., 2021). Among carbon nanomaterials, graphene nanomaterials, including graphene oxide (GO), graphene nano- ribbon (GNR), and GQD have gained interest recently (Ghaffarkhah et al., 2022; P. Tian et al., 2018). PANI is a class of aromatic conjugated polymers with a rigid, planar π-π electronic conjugated system, showing absorption in the region of ultraviolet and visible light (Molaei, 2020). In addition, GQD with PL properties includes functional groups such as hydroxyl and carboxyl groups, and Pyrene has aromatic rings in its structures. So the interaction between PANI and GQD at the edge of the molecules then a mechanism for detecting Pyrene might provide exciting results that will be discussed extensively among the scientific community.

1.7 Outline of the Thesis

This study is organized into five chapters as follows;

Chapter 1 discusses the background of Pyrene, PANI, GQD, and PANI-based carbon nanomaterials. In addition, the chapter contains the problem statement, research hypothesis, research objective, research scope, and finally significance of the study.

Chapter 2 reviews CPs, synthesis and characterization of PANI, PANI-based composite, particularly PANI-based sensing, GQD-based composite, mainly GQD-based sensing, the background and identification of PAHs and Pyrene, and finally, the background of PL method for detection of Pyrene.

Chapter 3 presents the experimental techniques applied to this study and explains the experimental's framework in detail. Then, it describes the synthesis procedures of nanocomposite films used for detecting Pyrene. This chapter also discusses the laboratory procedures for analyzing and characterizing the synthesized nanocomposite films.

Chapter 4 describes the results and discussion of this study which consists of the characterization of PANI and PANI-GQD nanocomposite films. Moreover, the results for interacting and detecting Pyrene will be presented using optical methods, including FT-IR and PL, based on the optimized PANI and PANI-GQD nanocomposite films.

Chapter 5 concludes the research findings. It shows the overall summary of this thesis. It highlights the significant findings and recommends further enhancing the sensor's performance.

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