DEVELOPMENT OF SURFACE PLASMON RESONANCE SENSOR USING CARBON-BASED NANOMATERIALS AND CHITOSAN FOR DOPAMINE DETECTION

By

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Dopamine (DA) plays a vital role in the brain and central nervous system. Therefore, there is a great need to develop a sensitive and selective sensor to monitor and determine DA concentrations for diagnostic purposes and diseases prevention. Up to now, employing surface plasmon resonance (SPR) sensors in DA determination is very limited, and its utilization to detect analytes with low concentrations still needs sensitivity enhancement. In this work, the SPR gold chips were modified using carbon quantum dots (CQDs), graphene quantum dots (GQDs), graphene oxide (GO), chitosan (CS), (CS-CQDs), and (CS-GQDs) thin films. The sensor performance for all layers was analyzed in terms of two aspects, sensitivity and accuracy. The surface morphology and roughness of all films were analyzed using AFM, and the existence of the functional groups in the samples was confirmed using FTIR spectroscopy. Experimental data were fitted to theoretical data formula to characterize the optical properties and thickness of the films. SPR sensor showed sensitivity of 0.138°/pM, 0.332°/nM, 0.215°/pM, 0.195°/nM, 0.169°/pM, and 0.195°/fM using Au/CQDs, Au/GQDs, Au/GO, Au/CS, Au/CS-CQDs, and Au/CS-GQDs bilayer films, respectively. The changes in the spectral bands and peaks intensity of FTIR spectra for all sensing films following DA injection verified DA binding to the sensor surface. AFM analysis showed that the surface morphology and roughness of all films changed as well. The thickness changed by 4.42, 2.59, 2.53, 1.25, 1.63, and 2.28 nm for CQDs, GQDs, GO, CS, CS-CQDs, CS-GQDs layers, respectively. By comparing the performance of all sensor films, SPR sensor based on Au/CS-GQDs exhibited excellent performance with ultra-sensitivity 0.195°/fM, lowest detection limit down to 1 fM of DA was obtained for the first time, RI sensitivity of 10.186°/RIU and the strongest binding affinity of 0.430×10^{15} M^{-1}. Interestingly, Au/GO based sensor exhibited competitive performance to Au/CS-GQDs based sensor with high sensitivity 0.215°/pM, RI sensitivity of 12.402°/RIU, strong binding affinity of 3.279×10^{12} M^{-1}. In addition to high sensitivity, good repeatability, reproducibility, and stability demonstrated for these two sensors, they showed good selectivity to low concentration of DA in the presence of higher concentrations of epinephrin, ascorbic acid, and uric acid. This nanomaterials-based SPR sensor represents an advantageous possibility for diagnosing DA deficiency rapidly, inexpensively with high selectivity and sensitivity. Its utilization as a reliable and economic biomedical diagnostic tool of DA-related brain disorders still be a major goal of research in the field of DA sensors.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN PENDERIA RESONANS PLASMON PERMUKAAN MENGGUNAKAN BAHAN NANO BERASASKAN KARBON DAN KITOSAN UNTUK PENGESANAN DOPAMIN

Oleh

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Januari 2023

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Dopamine (DA) memainkan peranan penting dalam otak dan sistem saraf pusat. Oleh itu, terdapat keperluan yang besar untuk membangunkan penderia yang sensitif dan selektif untuk memantau dan menentukan kepekatan DA untuk tujuan diagnostik dan pencegahan penyakit. Sehingga kini, penggunaan penderia resonans plasmon permukaan (SPR) dalam penentuan DA adalah sangat terhad, dan penggunaannya untuk mengesan analit dengan kepekatan rendah masih memerlukan penambahbaikkan sensitiviti. Dalam kerja ini, cip emas SPR telah diubah suai menggunakan titik kuantum karbon (CQDs), titik kuantum grafin (GQDs), grafin oxide (GO), kitosan (CS), (CS-CQDs), dan (CS-GQDs) filem nipis. Prestasi penderia untuk semua lapisan dianalisis dari segi dua aspek, kepekaan dan ketepatan. Morfologi permukaan dan kekasaran semua filem dianalisis menggunakan AFM, dan kewujudan kumpulan berfungsi dalam sampel disahkan menggunakan spektroskopi FTIR. Data eksperimen telah dipadankan pada formula data teori untuk mencirikan sifat optik dan ketebalan filem. Penderia SPR menunjukkan kepekaan 0.138°/pM, 0.332°/nM, 0.215°/pM, 0.195°/nM, 0.169°/pM dan 0.195°/fM menggunakan Au/CQDs, Au/GQDs, Au/GO, Au/CS, Au/CS-CQDs dan Au/CS-GQDs dwilapisan filem, masing-masing. Perubahan dalam jalur spektrum dan keamatan puncak spektrum FTIR untuk semua filem penderiaan selepas menginjeksi DA telah mengesahkan pengikatan DA pada permukaan penderia. Analisis AFM menunjukkan bahawa morfologi permukaan dan kekasaran semua filem turut berubah. Ketebalan berubah sebanyak 4.42, 2.59, 2.53, 1.25, 1.63, dan 2.28 nm untuk lapisan CQDs, GQDs, GO, CS, CS-CQDs, CS-GQDs, masing-masing. Dengan membandingkan prestasi semua filem penderia, penderia SPR berdasarkan Au/CS-GQDs telah mempamerkan prestasi cemerlang dengan kepekaan ultra 0.195°/fM, had pengesanan terendah hingga 1 fM DA diperolehi buat kali pertama, kepekaan RI 10.186°/RIU dan pertalian ikatan terkuat 0.430×10^{15} M^{-1}. Menariknya, penderia berasaskan Au/GO mempamerkan prestasi kompetitif kepada penderia berasaskan Au/CS-GQDs dengan kepekaan tinggi 0.215°/pM, kepekaan RI 12.402°/RIU, pertalian pengikatan kuat 3.279×10^{12} M^{-1}. Sebagai tambahan kepada kepekaan yang tinggi, kebolehulangan yang baik, kebolehulangan dan kestabilan yang ditunjukkan untuk kedua-dua penderia ini, mereka menunjukkan selektiviti yang baik kepada kepekatan DA yang rendah dengan kehadiran kepekatan epinefrin, asid askorbik dan asid urik yang lebih tinggi. Penderia SPR berasaskan bahan nano ini mewakili kemungkinan berfaedah untuk mendiagnosis kekurangan DA dengan cepat, murah dengan selektiviti dan sensitiviti yang tinggi. Penggunaannya sebagai alat diagnostik bioperubatan yang boleh dipercayai dan ekonomi untuk gangguan otak berkaitan DA masih menjadi matlamat utama penyelidikan dalam bidang penderiaan DA.

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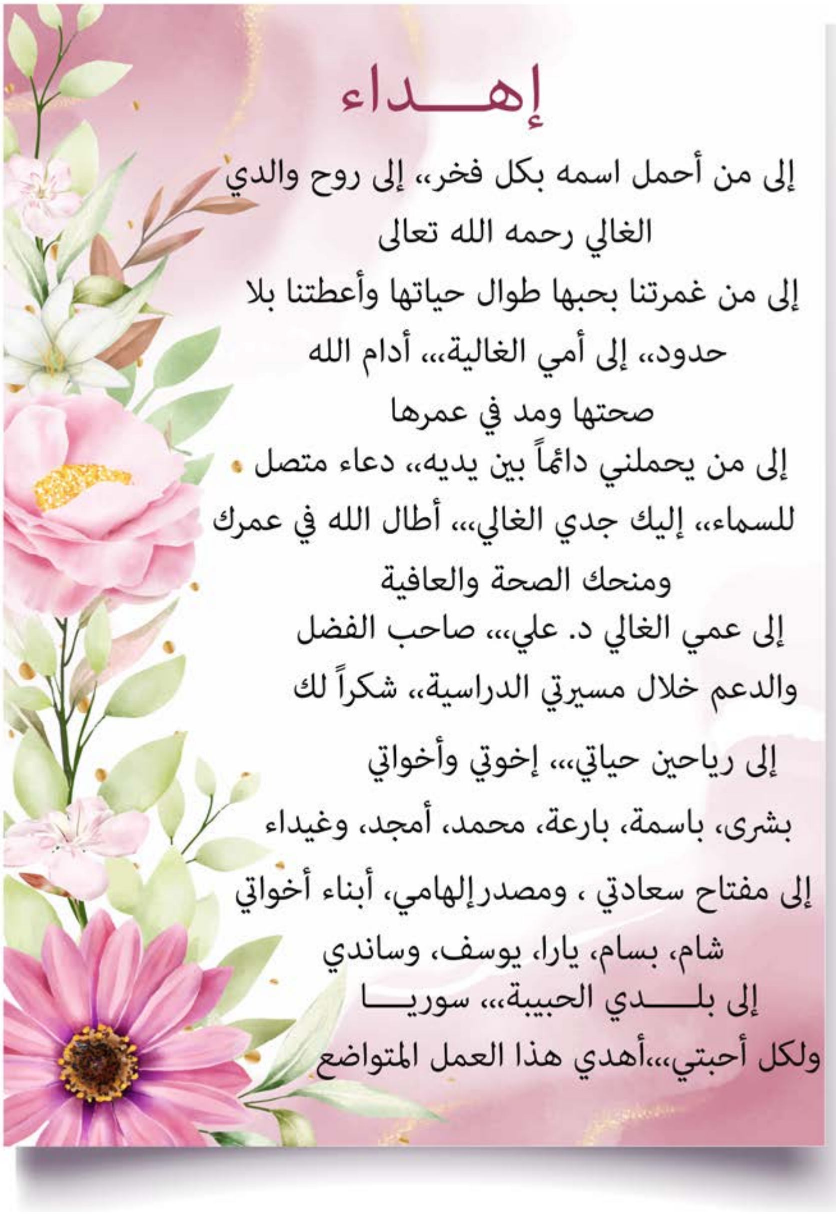

إهـــداء

إلى من أحمل اسمه بكل فخر،، إلى روح والدي
الغالي رحمه الله تعالى
إلى من غمرتنا بحبها طوال حياتها وأعطتنا بلا
حدود،، إلى أمي الغالية،،، أدام الله
صحتها ومد في عمرها
إلى من يحملني دائماً بين يديه،، دعاء متصل
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ومنحك الصحة والعافية
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والدعم خلال مسيرتي الدراسية،، شكراً لك

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إلى بلـــــدي الحبيبة،،، سوريـــا
ولكل أحبتي،،،أهدي هذا العمل المتواضع

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LIST OF ABBREVIATIONS

µM	Micromolar
3D CAG	Three-dimensional carbon aerogel electrode
AA	Ascorbic acid
AEATMS	N-[3-(Trimethoxysilyl)propyl] ethylenediamine
Ag	Silver
Ag Fe (NTA)	Iron-nitrilotriacetic acid linked Ag nanoparticles
ALA	Alpha lipoic acid
APBA	3-aminophenylboronic acid
ATR	Attenuated total reflection
Au	Gold
Au NCs	Au nanoclusters
AuNPs/CA-Fe (III)	Ferric-citrate-functionalized Au nanoparticles
B	Boron
BN-SiCDs	Boron and nitrogen co-doped silicon-carbon dots
BSA	Bovine serum albumin
cDNA	Complementary DNA
CDs	Carbon dots
CF	Carbon fiber
CFMEs	Carbon-fiber microelectrodes
CL	Chemiluminescence
CNC	Cellulose nanocrystals
CNs	Carbon nanospheres
CNTs	Carbon nanotubes
CPE	Carbon paste electrode

CQDs	Carbon quantum dots
CS	Chitosan
CSF	Cerebrospinal fluid
CTAB	Hexadecyl trimethyl ammonium bromide
DA	Dopamine
DAAPT	DA DNA aptamer
DA-RC	D_3-DA receptor
DBA	58-mer DA-binding aptamer
DECDs	Dual-emission CDs
DENV	Dengue virus
DOPAC	3,4-dihydroxyphenyl acetic acid
D-POF	D-shaped plastic optical fiber
DW	Deionized water
EC	Electrochemical
ECL	Electrochemiluminescence
EP	Epinephrine
EPR	Epoxy resin
ERGO	Electrochemically reduced graphene oxide
FCV	Feline calicivirus
fM	Femtomolar
f-MWCNTs	Functionalized multi-walled carbon nanotubes
FRET	Fluorescence resonance energy transfer
FTIR	Fourier transform infrared
FTO	Fluorine doped tin oxide
GCE	Glassy carbon electrode

GCPE	Glassy carbon paste electrode
GE	Graphite electrode
GNBs	Graphene nanobelts
GNP	Graphene nanoplatelets
GNS	Graphene nanosheets
GO	Graphene oxide
GPE	Graphite paste electrode
GQDs	Graphene quantum dots
H-CQDs	Honey-based carbon quantum dots
HPLC	High performance liquid chromatography
HSV	Herpes simplex virus
HVA	Homovanillic acid
IL	Ionic liquid
IL COF-1	Covalent organic framework materials
IR	Infrared
K	Langmuir and Freundlich affinity constant
k_P	Adsorption rate constant
k'	Real part of wave vector
k''	Imaginary part of wave vector
K_A	Equilibrium association constant
K_D	Equilibrium dissociation constant
L-Cys	L-cysteine
L-GSH	L-glutathione
LIG	Laser-induced graphene
LMOFs	Luminous MOFs

LOD	Limit of detection
LSPs	Localized surface plasmons
MA	Melamine
MHA	6-Mercaptohexanoic acid
MIP	Molecularly imprinted polymer
MMW	Medium molecular weight
MOFs	Metal-organic frameworks
MWCNTs	Multi-walled carbon nanotubes
N	Nitrogen
n	Refractive index
n	System heterogeneity index
NG	Nano-graphite
nM	Nanomolar
NPs	Nanoparticles
NTs	Neurotransmitters
NWs	Nanowires
PANI	Polyaniline
PAS	P-aminostyrene
PBS	Phosphate-buffered saline
PD	Parkinson’s disease
PDDA	Poly (diallyldimethylammonium chloride)
PEC	Photoelectrochemical
PEDOT	Poly (3,4-ethylenedioxythiophene)
PFO	Pseudo-first-order
pGO	Porous graphene oxide

PL	Photoluminescence
PLAEMA	Poly (2-lactobionamidoethyl methacrylamide)
PLL	Poly (L-lysine)
pM	Picomolar
POCT	Point-of-care testing
PPy	Polypyrrole
PSPs	Propagating surface plasmons
Pt	Platinum
PVA	Poly vinyl alcohol
R	Reflection coefficient
RF	Ratiometric fluorescent
rGO	Reduced graphene oxide
RI	Refractive index
RMS	Root mean square
RRS	Resonance Rayleigh scattering
SERRS	Surface-enhanced resonance Raman scattering
SERS	Surface-enhanced Raman spectroscopy
SiCDs	Aminosilane-functionalized CDs
SNR	Signal-to-noise ratio
SPCE	Screen-printed carbon electrode
SPE	Screen-printed electrode
SPP	Surface plasmon polariton
SPR	Surface plasmon resonance
SPS	Solid phase spectrophotometry
SPs	Surface plasmons

SWCNTs	Single-walled carbon nanotubes
TM	Teramolar
TMM	Transfer matrix method
Tyr	Tyrosinase
UA	Uric acid
UV-vis	Ultraviolet-visible
θ_{SPR}	Surface plasmon resonance angle
ω	Angular frequency

CHAPTER 1

INTRODUCTION

1.1 Dopamine and Its Critical Role in Human Body

The brain is the most complex organ in mammalian. It is responsible of our actions and interactions; our personality and emotions, regulation of our movements by processing all information received from our sensory organs. Through neurotransmission, the communication between nerve cells occurs where an electrical signal is converted to a chemical signal by the release of neurotransmitters (NTs) as shown in Figure 1.1.

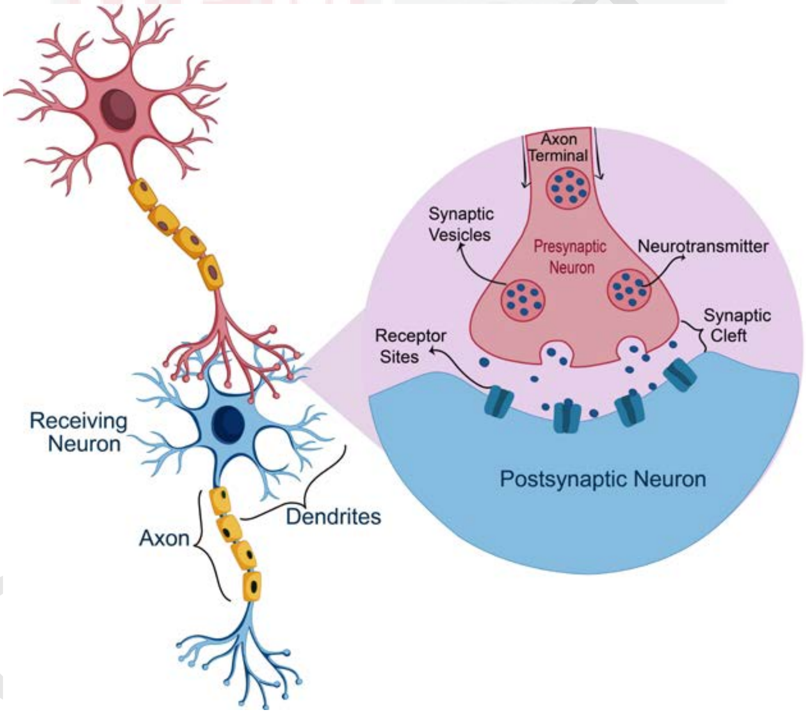

Figure 1.1: Neuron communication process.

Dopamine (DA), with the chemical formula 3,4-dihydroxyphenethylamine, is well known as one of the most essential catecholamine NTs in the central and the peripheral neural systems of mammals, since it conveys brain messages in the form of nerve impulses. This monoamine NT is produced in very specific regions in the brain (Yusoff et al., 2015). The normal concentration of DA in the body affects the function of the central nervous system, supports blood pressure and regulates the physiological processes such as; fine motor activity, stress, mental cognition, attention, inspiration, intuition, learning, motivation, emotions, and memory formation (Leng et al., 2015; Elayappan et al., 2020). It also has a significant influence on the function of hormonal, renal, and cardiovascular systems of the body. The physiological concentrations of DA vary in various body fluids. The concentration of DA in the blood is less than 130 pM according to the Human Metabolome Database, whereas its concentration in human cerebrospinal samples and urine is around 5 nM (Cao & McDermott, 2018). Given the wide range of physiological and pathophysiological effects of DA, it is believed that abnormality of DA concentrations in human blood and brain systems are associated with various diseases. High concentrations of DA may lead to cardiotoxicity and abnormally high pulse and hypertension, as well as heart failure (J. Liu et al., 2013; D. Yuan et al., 2014). Whereas the deficiency of DA is correlated with serious neurodegenerative diseases which including Parkinson's disease (PD) (Kim 2002; Hsu et al., 2012), Alzheimer's disease (Hyman et al., 1984; Zhu et al., 2016), schizophrenia (Jagadeesh. J & Natarajan, 2013; Kesby et al., 2018), and depression (Vázquez-guardado et al., 2019; Fan et al., 2021). As a consequence, simple, sensitive, rapid and accurate analytical methods to determine DA concentrations precisely would be useful for physiological studies and a critical marker for timely diagnostics and therapeutics.

1.2 Methods and Challenges of Dopamine Detection

Up to now, huge efforts deployed and many strategies have been reported for DA detection in biofluids such as high performance liquid chromatography (HPLC) (Yoshitake et al., 2004; Carrera et al., 2007; Muzzi et al., 2008), capillary electrophoresis (Thabano et al., 2009; Zhao et al., 2011; X. Wang et al., 2013), mass spectroscopy (Hows et al., 2004; Syslová et al., 2011), microdialysis techniques (Jamal et al., 2016), Fourier transform infrared (FTIR) spectroscopy (X. Wang et al., 2002), flow injection (Wabaidur et al., 2012), enzymatic methods (Fritzen-Garcia et al., 2013), electrochemical (EC) methods (X. Liu et al., 2012; Sajid et al., 2016; Shin et al., 2017) and other methods. Although these effective strategies are accurate and have their own features, they suffer some drawbacks and limitations. Most of them take time, have limited sensitivity and need expensive equipment, and are not long-term stable. Furthermore, the sensor's surface functionalization is challenging.

DA is an easily oxidizable compound. Due to its electroactive nature, DA detection has been generally developed by EC methods which has received increasing attention because of its several notable characteristics such like fast response, good sensitivity, repeatability and reproducibility, stability, and cheap costing. However, there are some challenges that hinder DA determination under physiological conditions using EC technique. These challenges include the extremely low DA concentrations and the interfering species such as the electroactive ascorbic acid (AA), uric acid (UA), and epinephrine (EP). As an antioxidant, AA is essential in human metabolism. It mostly coexists with DA in relatively high concentrations in the central nervous system, resulting in poor selectivity during DA detection. Furthermore, because AA oxidizes at almost the same potential as DA, the oxidation peaks of a combination of DA and AA overlap (Atta et al., 2010). UA is an antioxidant found in significant amounts in the blood and brain tissue. It can prevent DNA damage results from free radical. It also controls and inhibits iron-mediated oxidation processes that are harmful to biological systems by forming strong complexes with iron ions (Church & Ward, 1994). EP, commonly known as adrenaline, is a hormone as well as a NT found at nanomolar concentrations in brain fluids, urine, and human blood (Dong et al., 2018). Thus, it still remains a great challenge to develop an inexpensive, reliable, and effective electrode with improved characteristics to distinguish DA from AA, UA and EP, reduce the signal noise and overcome low selectivity and sensor fouling and degradation over time during the determination of DA trace amount in biofluid.

The limitations of EC sensors prompted continued development to increase the sensitivity, selectivity, and biocompatibility of these sensors. Also, considerable efforts have been devoted to develop optical based methods to detect and quantitate DA and cover its whole physiological concentrations. These optical methods include colorimetry and spectrophotometry (Chen et al., 2015; Palanisamy et al., 2016), fluorescence spectrometry (H. Wang et al., 2002; Khattar & Mathur, 2013; Kruss et al., 2014; Zhao et al., 2016; Kruss et al., 2017), ultraviolet-visible (UV-vis) spectrophotometry (Barreto et al., 2008), electrochemiluminescence (ECL) (Qi et al., 2009; Yu et al., 2011), surface-enhanced Raman spectroscopy (SERS) (Bu et al., 2013; Ranc et al., 2014; An et al., 2015; Pu et al., 2015; Lu et al., 2016), chemiluminescence (CL) (Deftereos et al., 1993), photoelectrochemical (PEC) (Yan et al., 2015), photoluminescence (PL) (Sun & Wang, 2012), solid phase spectrophotometry (SPS) (Taghdiri & Mohamadipour-taziyan, 2012), resonance Rayleigh scattering (RRS) (Dong et al., 2013), and surface plasmon resonance (SPR) spectroscopy (Matsui et al., 2005; Kumbhat et al., 2007; Sebok et al., 2013; Kamali et al., 2015; Raj et al., 2016; Jiang et al., 2017; Manaf et al., 2017; Cao & Mcdermott, 2018; Sharma & Gupta, 2018; Sun et al., 2019; Yuan et al., 2019). Spectroscopic methods are advantageous because they are cheap and their repeatability is good. Add to that, the sensitivity of most of them is better or comparable with EC methods. However, complicated procedures still required to develop colorimetric and fluorescent probes for DA sensing. Although SERS-based DA sensors exhibit higher sensitivity and selectivity towards DA when compared to other techniques, they need expensive equipment for analysis, which limits their application to assess DA. Therefore, significant and growing efforts have lately been directed toward overcoming these limitations and developing label-free optical biosensors.

1.3 SPR Based Sensor

SPR is a quantum electromagnetic phenomenon that happens whenever light interacts with free electrons at the metal-dielectric interface (Ishimaru et al., 2005; Pitarke et al., 2006). When light-matter interaction occurs, photons of the incoming p-polarized light cause free electrons on the noble metal usually gold (Au) surface of the SPR chip to collectively oscillate, resulting in surface plasmon polariton (SPP). Once the incoming light's wave vector matches that of the SPP mode, the resonance takes place and the incoming light's energy is coupled into the SPP mode, causing energy loss at the resonance angle and reducing the intensity of the reflected light, resulting in an SPR dip (Pitarke et al., 2006; Phillips & Cheng, 2008; Wang et al., 2012; Zainudin et al., 2018; Daniyal et al., 2019).

SPR sensor is an optical sensor that works by exciting the surface plasmons. This light-based SPR sensor is a type of refractometric sensing device that monitors changes in the refractive index of the sensing medium that occur in response to binding events (Homola, 2008). SPR sensors have showed remarkable progress in terms of both technological development and applications to detect different analytes. Today, SPR based optical biosensor is considered among the most advanced technologies for studying biomolecular interactions (Kan & Li, 2016). This label-free sensor offers accurate and fast detection of biological and chemical analytes with good sensitivity. It has proven its potential for the application in different important fields such as clinical and medical diagnostics (Chung et al., 2005; Uludag & Tothill, 2012; Yanase et al., 2014; Omar et al., 2019), food-safety analysis (Situ et al., 2010; Zainuddin et al., 2018), environmental protection (Fen et al., 2012; Fen et al., 2013; Sadrolhosseini et al., 2017; Roshidi et al., 2019; Daniyal et al., 2019) and others. Over the last two decades, this powerful analytical technique has received extensive attention and has been developed in different configurations to detect a diversity of analytes.

1.4 Sensing Layers and Sensitivity Enhancement

SPR biosensors have demonstrated their effectiveness compared to other techniques due to their substantial features including high specificity, good sensitivity, low cost, real-time sensing capabilities, no labelling required, and ease of preparation. However, employing these sensors to detect analytes with low molecular weights or at low concentrations necessitates surface modification to improve their sensitivity. Because of their exceptional optical, magnetic, and electrical characteristics, nanomaterials have shown significant promise in sensing field (Zeng et al., 2014), batteries (Khan et al., 2019; Kang et al., 2021), electronics, medicine, and other domains. Incorporating nanomaterials into SPR biosensors was a viable and potentially effective approach of enhancing sensitivity. As a result, a variety of materials are expected to function well as active layers on the SPR chip for DA detection, and the material selection controls the performance of the developed sensor.

Carbon-based nanomaterials have piqued the scientific community's interest due to their unusual electrical, mechanical, chemical, thermal, and optical capabilities among other types of nanomaterials. They have been employed as a plasmonic layer in the construction of SPR based sensors to improve the sensor sensitivity, and to offer a large surface area and good compatibility for the immobilization of diverse biomolecules such as enzymes, DNA, antibodies, and antigens (Zeng et al., 2014; Gupta et al., 2019). Carbon quantum dots (CQDs) are nanoparticles (NPs) with extremely small sizes, usually less than 10 nm, that have lately sparked the interest due to their unique physicochemical properties, ease of synthesis, low-cost, high-water solubility, environmental friendliness, low concentrations of toxicity, good biocompatibility, and chemical stability (Hu et al., 2017; Wu et al., 2022; Dong et al., 2022). Graphene is a single layer of graphite with extraordinary physicochemical characteristics (Du et al., 2014; Dai et al., 2021). Graphene quantum dots (GQDs) have recently emerged as a distinct class of carbon-based nanomaterials, exhibiting various appealing qualities such as low toxicity, biocompatibility, photostability, and a comparatively high quantum yield when compared to other carbon nanomaterials. Amongst various carbonaceous materials, graphene oxide (GO) has demonstrated significant potential in biosensing. It has piqued the curiosity of many people because to the extraordinary properties derived from its electronic arrangement, namely the sp^2/sp^3 coexisting structure (Chen et al., 2011; Teymourian et al., 2013; D. Yuan et al., 2014).

Chitosan (CS) is a nontoxic biodegradable biopolymer with a high molecular weight found abundantly in nature. CS is derived from chitin, a natural organic molecule that may be taken from the exoskeletons of crustaceans and insects. This polymer has a lot of amino ($–NH_2$) and hydroxyl (–OH) functional groups (Sonsin et al., 2021). CS biopolymer and its derivatives were selectively utilized to stabilize plasmonic NPs, semiconductor NPs, luminescent NPs, and photoluminescent complexes in the field of optically active materials (Marpu & Benton, 2018). Because of the distinct properties of the carbon-based nanomaterials and the biopolymer chitosan, they were effectively employed in the preparation of SPR sensor chips as active layers to improve SPR sensor sensitivity towards DA and enhance the sensor performance.

1.5 Problem Statement

Dopamine (DA) insufficiency in the human body causes major neurological disorders such as Parkinson’s disease (PD) (Hsu et al., 2012), and Alzheimer’s disease (Zhu et al., 2016). As a result, there is an urgent need for extremely sensitive and selective sensors capable of monitoring DA concentrations and making relevant measurements accurately in real time. To date, several techniques have been developed for DA detection, including electrochemical (EC) methods (Shin et al., 2017), and various types of optical methods such as colorimetry and spectrophotometry (Palanisamy et al., 2016), fluorescence (Kruss et al., 2017), surface-enhanced Raman spectroscopy (SERS) (Lu et al., 2016) and surface plasmon resonance (SPR). The detection of DA employing SPR sensors is still limited and in its early stages (Matsui et al., 2005; Kumbhat et al., 2007; Sebok et al., 2013; Kamali et al., 2015; Raj et al., 2016; Jiang et al., 2017; Manaf et al., 2017; Cao & Mcdermott, 2018; Sharma & Gupta, 2018; Sun et al., 2019; Yuan et al., 2019). SPR sensors are currently among the most advanced technologies that have satisfied the requirement for more relevant details on biomolecular interactions (Omar & Fen, 2018), and have shown efficient in the detection of various biological analytes and medical diagnostics (Yanase et al., 2014; Omar et al., 2019). Despite the significant benefits of high specificity, cheap cost, sensing capabilities in real-time, and ease of preparation of SPR sensor, its sensitivity necessities improvements to detect analytes in extremely low concentrations. Because of their unique optical, magnetic, and electrical characteristics, nanomaterials have shown promising potential in sensing and other domains (Zeng et al., 2014). The use of nanomaterials with SPR sensor provides exceptional prospects and a feasible approach to boost its sensitivity. Therefore, in this work, SPR sensor was developed by modifying the gold chips with different thin films prepared using carbon-based nanomaterials and the biopolymer chitosan for highly sensitive detection of DA.

Though the preliminary results of the previously developed DA SPR sensors are encouraging, however, their reports on the sensing performance are very limited. Just a few works indicated the sensor sensitivity (Manaf et al., 2017; Sharma & Gupta, 2018; Sun et al., 2019) and affinity constant (Cao & Mcdermott, 2018). Therefore, it is of interest to evaluate the sensing performance of all sensor films in terms of sensitivity, binding affinity, limit of detection, and accuracy.

Moreover, the reported studies did not investigate DA binding behaviour on the sensor surface using structural measurements. In addition, the characterization of the optical properties of DA and the sensor film, as well as the determination of the sensor film thickness were reported only by Manaf et al. (2017). Therefore, it is important to characterize the structural and optical properties of all sensor films and determine their thickness before and after interactions with DA.

For more reliability of the appropriateness of the developed SPR sensor to detect DA, the sensing layers with the best performance towards DA were selected for a thorough evaluation of their efficiency in terms of selectivity, repeatability, reproducibility and stability. In addition, the kinetic behaviour of DA solution in contact with the chosen sensor films was studied. This nanomaterials-based SPR sensor represents an advantageous possibility to detect extremely low concentrations of DA rapidly, easily, inexpensively and reliably.

1.6 Research Objectives

The main objectives of this study are stated as follows:

1. To develop an SPR optical sensor using Au/CQDs, Au/GQDs, Au/GO, Au/CS, Au/CS-CQDs, and Au/CS-GQDs thin films for DA detection.
2. To investigate the performance of DA sensing for all sensor films.
3. To characterize the structural and optical properties, as well as the thickness of sensor films before and after interactions with DA.
4. To investigate the selectivity, repeatability, reproducibility, stability and kinetic behaviour of DA on the best-performing sensor films.

1.7 Thesis Organization

This thesis consists of six chapters. Chapter 1 provides an introduction on DA and its important role in human body, and briefly mentions the methods and challenges of DA detection. SPR based sensor has also been included throughout this chapter, where the general principle of SPR was introduced, as well as sensing layers and the necessity for the sensitivity enhancement. The problem statement is also involved and research objectives are outlined. Chapter 2 presents DA sensors including EC sensors based on carbon nanomaterials and chitosan, optical sensors such as colorimetric and spectrophotometric sensors, SERS sensor, and fluorescence sensor. It also covers SPR phenomenon and sensing applications in medical diagnosis. In addition, DA detection using SPR sensors is discussed in details. The performance characteristics of SPR sensor, the function of SPR technique as a refractometer, as well as the structural properties of carbon-based materials and chitosan are also addressed in this chapter. Chapter 3 focuses on theory of surface plasmon in the aspects of the evanescent wave and surface plasmon dispersion, surface plasmon polaritons at a single interface, multilayer systems and Fresnel analysis, and SPR sensorgram. Chapter 4 describes the methodology of preparation the sample solutions and sensor films, the experimental procedure of incorporation the thin films to SPR system for target sensing, theoretical analysis method, and the structural characterization techniques of sensor films. Chapter 5 shows the results and discussion of SPR optical sensor based on Au, Au/CQDs, Au/GQDs, Au/GO, Au/CS, Au/CS-CQDs, and Au/CS-GQDs thin films. Finally, chapter 6 concludes the research findings made during this study and summarizes the performance of all developed sensing layers. The suggestions and recommendations for future work have been also stated in this chapter.

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LIST OF PUBLICATIONS

Journals

Published Papers

Faten Bashar Kamal Eddin, Yap Wing Fen. (2020) Recent advances in electrochemical and optical sensing of dopamine, *Sensors*, 20, 1039. (**Q1**)

Faten Bashar Kamal Eddin, Yap Wing Fen. (2020) The principle of nanomaterials based surface plasmon resonance biosensors and its potential for dopamine detection, *Molecules*, 25, 2769. (**Q2**)

Faten Bashar Kamal Eddin, Yap Wing Fen, Nur Alia Sheh Omar, Josephine Ying Chyi Liew, and Wan Mohd Ebtisyam Mustaqim Mohd Daniyal. (2021) Femtomolar detection of dopamine using surface plasmon resonance sensor based on chitosan/graphene quantum dots thin film, *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 263, 120202. (**Q1**)

Faten Bashar Kamal Eddin, Yap Wing Fen, Nurul Illya Muhamad Fauzi, Wan Mohd Ebtisyam Mustaqim Mohd Daniyal, Nur Alia Sheh Omar, Muhammad Fahmi Anuar, Hazwani Suhaila Hashim, Amir Reza Sadrolhosseini and Huda Abdullah. (2022) Direct and sensitive detection of dopamine using carbon quantum dots based refractive index surface plasmon resonance sensor, *Nanomaterials*, 12, 1799. (**Q1**)

Faten Bashar Kamal Eddin, Yap Wing Fen, Amir Reza Sadrolhosseini, Josephine Ying Chyi Liew, and Wan Mohd Ebtisyam Mustaqim Mohd Daniyal. (2022) Optical property analysis of chitosan-graphene quantum dots thin film and dopamine using surface plasmon resonance spectroscopy, *Plasmonics*, 17, 1985–1997. (**Q3**)

Faten Bashar Kamal Eddin, Yap Wing Fen, Josephine Ying Chyi Liew, Wan Mohd Ebtisyam Mustaqim Mohd Daniyal. (2022) Plasmonic refractive index sensor enhanced with chitosan/Au bilayer thin film for dopamine detection. *Biosensors*, 12, 1124. (**Q1**)

Faten Bashar Kamal Eddin, Yap Wing Fen, Josephine Ying Chyi Liew, Nurul Illya Muhamad Fauzi, Wan Mohd Ebtisyam Mustaqim Mohd Daniyal and Huda Abdullah. (2023) Development of plasmonic-based sensor for highly sensitive and selective detection of dopamine, *Optics and Laser Technology*, 161, 109221. (**Q1**)

Faten Bashar Kamal Eddin, Yap Wing Fen, Josephine Ying Chyi Liew, Hong Ngee Lim, Wan Mohd Ebtisyam Mustaqim Mohd Daniyal, and Nur Alia Sheh Omar. (2023) Simultaneous measurement of the refractive index and thickness of graphene oxide thin films for potential in dopamine sensing using surface plasmon resonance spectroscopy, *Optik*, 278, 170703. (**Q2**)

Submitted Papers

Faten Bashar Kamal Eddin, Yap Wing Fen, Josephine Ying Chyi Liew, Hong Ngee Lim, Nur Alia Sheh Omar, Nurul Illya Muhamad Fauzi and Wan Mohd Ebtisyam Mustaqim Mohd Daniyal. (2023) Improved dopamine sensing characteristics of a plasmonic platform with chitosan-carbon quantum dots active layer, *Applied Physics B*. (**Q3**)

Faten Bashar Kamal Eddin, Yap Wing Fen, Josephine Ying Chyi Liew, Hong Ngee Lim, Nur Alia Sheh Omar, Nurul Illya Muhamad Fauzi and Wan Mohd Ebtisyam Mustaqim Mohd Daniyal. (2023) Performance analysis of plasmonic sensor modified with chitosan-graphene quantum dots based bilayer thin film structure for real-time detection of dopamine, *Sensors and Actuators A*. (**Q1)**

Faten Bashar Kamal Eddin, Yap Wing Fen, Josephine Ying Chyi Liew, Hong Ngee Lim, Nur Alia Sheh Omar, Nurul Illya Muhamad Fauzi and Wan Mohd Ebtisyam Mustaqim Mohd Daniyal. (2023) Structural, optical and sensing characteristics of graphene quantum dots/gold thin film in contact with dopamine solution, *Applied Physics A*. (**Q2)**

Awards

1. Malaysian Solid-State Science & Technology (Mass) Award, Received in Physics Excellence Appreciation Ceremony (MAKeF 2022), Department of Physics, UPM, December 2022.
2. Graduate Journal Papers Publishing Award, Received in Physics Excellence Appreciation Ceremony (MAKeF 2022), Department of Physics, UPM, December 2022.
3. Graduate Journal Papers Publishing Award, Received in Physics Excellence Appreciation Ceremony (MAKeF 2021), Department of Physics, UPM, December 2021.
4. Bronze Medal Award at Virtual Materials Technology Challenges 4.0 (v-MTC4.0) on 2nd September 2020 at Universiti Putra Malaysia.

Seminar and Conferences

As oral presenter:

Faten Bashar Kamal Eddin, Yap Wing Fen, Amir Reza Sadrolhosseini and Nur Alia Sheh Omar (2020, September). The potential of graphene oxide based surface plasmon resonance biosensor for dopamine detection. Poster presented at Virtual Materials Technology Challenges 4.0 (v-MTC4.0), Universiti Putra Malaysia.

Faten Bashar Kamal Eddin, Yap Wing Fen, Amir Reza Sadrolhosseini, Josephine Ying Chyi Liew, and Hong Ngee Lim (2021, June). design and development of surface plasmon resonance sensor for dopamine detection. Oral presentation 2021 Physics

Postgraduates Online Seminar Series Organized by Department of Physics UPM and Supported by The MASS Chapter UPM.

Faten Bashar Kamal Eddin, Yap Wing Fen, Nur Alia Sheh Omar, Josephine Ying Chyi Liew and Wan Mohd Ebtisyam Mustaqim Mohd Daniyal (2021, August). Highly sensitive optical sensor for dopamine using nanomaterials based surface plasmon resonance spectroscopy. Oral presentation at the 12th International Fundamental Science Congress (iFSC) 2021.