UNIVERSITI TEKNOLOGI MARA

SYNTHESIS OF GOLD NANOPARTICLES EMBEDDED GRAPHENE OXIDE FOR FIBRE OPTIC GAS SENSOR APPLICATIONS

MOHD RAFAL BIN SAHUDIN

Thesis submitted in fulfillment of the requirements for the degree of **Master of Science** (Mechanical Engineering)

College of Engineering

October 2022

ABSTRACT

In this work, graphene oxide (GO) was successfully synthesized with the optimization formulation obtained using 1g of graphite, 3g of potassium permanganate (KMnO₄), 25 mL of sulfuric acid (H₂SO₄), 5 mL of hydrogen peroxide (H₂O₂) with 20°C of reaction temperatures and 3 hours of reaction time. The AuNPs were successfully synthesized with the optimum condition using 0.254 mM of gold chloride (HAuCl₄) and 40 mM of trisodium citrate (NaCt). The composite structure of AuNPs embedded to GO (GO-AuNPs) was performed by direct mixing of GO solution to AuNPs solution at various volume ratios (1:05, 1:1, 1:2.5, 1:5, 1:7.5, 1:10 and 1:15). Then, fabrication of GO, AuNPs, and composite GO-AuNPs (at various volume ratios) encapsulated optical fibre probes was performed via dipping process for 24 hours with the aid of vortex mixing. The detection using indoor air exposed the highest light intensity signal on the fibre optic sensor generated from the GO-AuNPs-based sensor at a volume ratio of 1:5 and 1:10. For spectral analysis, the highest CO₂ detection was detected by fibre optic sensors with GO-AuNPs (1:5) as a coating material with 62.7% signal improvement from uncoated sensor probe, 26.4% higher than the signal from GO-coated and 33.9% better than AuNPs-coated sensor. The highest N2 detection was obtained by the sensor fabricated of GO-AuNPs (1:10) with the signal enhancement of 59.8% from uncoated probe sensors. In the sensitivity analysis which represents the ratio of a change in output intensity to a change in gas concentration, the results of GO-AuNPs (1:5)-based sensor demonstrated excellent sensitivity enhancement to CO₂ detection (28.02% at 25 L/min) and GO-AuNPs (1:10)-based sensor demonstrated high sensitivity to N_2 (12.4% at 25 L/min). The signal also proved that the composite (GO-AuNPs) coating probe provides 6 times higher signal than single-element coating (GO or AuNPs). Based on the stability analysis, GO-AuNPs (1:5) sensors, and GO-AuNPs (1:10) sensors were found to be very stable against CO₂ and N₂. From the selectivity analysis, the results show that GO-AuNPs (1:5), GO-AuNPs (1:10) and AuNPs-based sensors have a high selectivity for CO_2 . Meanwhile, GO-based sensors are the most sensitive to N_2 gas, which is supported by the results of selectivity analysis. In summary, the CO_2 and N_2 detection signal from the uncoated probe was really low while all coated probes generated a higher signal, hence proving that the composite (GO-AuNPs) coating materials are very useful in enhancing the signal of the gas detection. This newly integrated configuration of the sensing platform should help in developing versatile optical fibre-based sensors, thus creating enormous sensor application possibilities, especially for gas sensor applications.

ACKNOWLEDGEMENT

Firstly, I wish to thank Allah SWT for providing me with the opportunity to embark on my Master's degree and for completing this long and challenging journey successfully. My gratitude and thanks to my supervisor Dr. Siti Rabizah Makhsin, my co-supervisors; Prof. Ir. Dr. Hj. Muhammad Azmi Ayub, Prof. Ts. Dr. Nor Hayati Saad and Prof. Dr. Mohammed Zourob.

I am greatly appreciative and thankful for all technical staff and postgraduate students, Micro-Nano Electromechanical System Laboratory (MiNEMs), School of Mechanical Engineering, Nano-Electronic (NET) Research Group, School of Electrical, Centre for Functional Materials & Nanotechnology (CFMN), Institute of Science (IOS), Photonics And Materials Research Group, Faculty of Applied Sciences, Universiti Teknologi MARA, Shah Alam, Selangor, Analytical Unit, Faculty of Pharmacy, Universiti Teknologi MARA, Puncak Alam, Centre For Research And Instrumentation Management (CRIM), Universiti Kebangsaan Malaysia and Institute for Research in Molecular Medicine (INFORMM), Universiti Sains Malaysia, Pulau Penang. This work is financially supported by the fundamental research grant scheme; FRGS grant 600-IRMI/FRGS 5/3(341/2019), Ministry of Higher Education Malaysia. This project wouldn't have been possible without the help of my supervisor, friends and colleagues.

Finally, this thesis is dedicated to the loving memory of my wife and mother for the vision and determination to educate me. This piece of victory is dedicated to both of you. Alhamdulillah.

TABLE OF CONTENTS

Page

CONFIRMATION BY PANEL OF EXAMINERS AUTHOR'S DECLARATION			ii
AUT	iii		
ABS	iv		
ACF	KNOWL	v	
TAE	BLE OF	vi	
LIST OF TABLES			
LIST	Г OF FI	GURES	xi
LIST	Г OF SY	MBOLS	xvi
LIS	Г OF AI	BBREVIATIONS	xvii
CHA	APTER	ONE INTRODUCTION	1
1.1	Resea	urch Background	1
1.2	Proble	em Statement	2
1.3	Objec	ctives	4
1.4	Scope and limitation of study		5
1.5	Signif	ficance of Study	6
CHA	APTER '	TWO LITERATURE REVIEW	7
2.1	Introd	luction	7
2.2	AuNF	7	
	2.2.1	Properties of AuNPs	7
	2.2.2	The synthesis method of AuNPs	8
	2.2.3	Characterization of AuNPs	12
	2.2.4	AuNPs in sensing applications	15
2.3	Graphene oxide (GO)		19
	2.3.1	Properties of GO	19
	2.3.2	The synthesis method of GO	20
	2.3.3	Characterization of GO	26

	2.3.4	GO in sensing applications	31
2.4	Fibre optic sensors		
	2.4.1	The basic configuration of fibre optic	32
	2.4.2	Fibre optic in sensing applications	35
	2.4.3	Fabrication of fibre optic sensors	36
	2.4.4	AuNPs and GO-based fibre optic sensors	38
2.5	Fibre optic in gas sensors applications		
	2.5.1	Configuration and mechanism of gas sensors	40
	2.5.2	Factors influencing the performance of the gas sensors	43
	2.5.3	Sensing materials for fibre optic gas sensors	44
2.6	Summ	nary	49
СНА	PTER	THREE RESEARCH METHODOLOGY	50
3.1	Introd	uction	50
3.2	Mater	faterials and chemicals	
3.3	Phase 1: Synthetization of GO, AuNPs and AuNPs-GO (Objective 1)		
	3.3.1	Synthesis of GO	53
	3.3.2	Synthesis of AuNPs	55
	3.3.3	AuNPs embedded GO (GO-AuNPs)	57
3.4	Phase 2: Characterization of GO, AuNPs and GO-AuNPs (Objective 1)		
	3.4.1	UV-Vis spectroscopy	58
	3.4.2	SEM / FESEM	58
	3.4.3	EDX spectroscopy	59
	3.4.4	XRD spectroscopy	59
	3.4.5	Fourier Transform infrared spectroscopy (FTIR)	59
	3.4.6	Raman spectroscopy	60
	3.4.7	TEM	60
	3.4.8	Zetasizer particle size analyzer	60
3.5	Phase 3: Fabrication and characterization of GO-AuNPs encapsulated o		
	fibre probe (Objective 2).		
	3.5.1	Fabrication of sensing region	60
	3.5.2	Characterization of coating materials onto fibre optic sensor	62
3.6	Phase	4: Fibre optic gas sensor; setup and sensing performance (Object	ive 3)
			62