

The Influence of Exfoliated Graphite on Sensing Properties of Chitosan/PVA Based Ammonia Sensors by original cor

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A thesis submitted in fulfillment of the requirements for the degree of Master of Science in Materials Engineering

> **School of Materials Engineering UNIVERSITI MALAYSIA PERLIS**

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iii

TABLE OF CONTENTS

	PAGE
DECLARATION OF THESIS	i
APPROVAL AND DECLARATION SHEET	ii
ACKNOWLEDGEMENT	iii
TABLE OF CONTENT	iv
LIST OF TABLES	xi
LIST OF FIGURES	xiii
LIST OF ABBREVIATIONS	xviii
LIST OF SYMBOLS	XX
ABSTRAK	xxi
ABSTRACT	xxii
CHAPTER 1 INTRODUCTION	
1.1 Research Background	1
1.2 Problem Statement	3
1.3 Research Objective	4
1.4 Scope of Study	4
CHAPTER 2 LITERATURE REVIEW	
2.1 Sensor	6
2.2 Gas Sensor	6
2.3 Chitosan	8
2.3.1 Properties of Chitosan	9

	2.3.2 Chitosan Based Sensor	10
	2.3.3 Chitosan for Sensor Application	11
2.4	Polyvinyl alcohol (PVA)	12
	2.4.1 Properties of PVA	13
	2.4.2 Application of PVA in Sensor Application	14
2.5	Graphite	15
2.6	Exfoliated Graphite (EG)	16
2.7	Applications of Exfoliated Graphite	19
2.8	Applications of Exfoliated Graphite Based Sensor	20
2.9	Electrochemical Deposition Technique	20
2.10	Volatile Compounds and Pollutants Gases	21
2.11	Ammonia	22
	2.11.1 Sources of Ammonia Contamination	23
	2.11.2 Ammonia Exposure Limit	24
	2.11.3 Current Ammonia Sensor	25
2.12	Optical Band Gap	25
2.13	Characteristics of Sensing Response	28
	·S	

CHAPTER 3 METHODOLOGY

3.1	Introduction of Methodology	31
3.2	Materials	34
3.3	The Preparation of Chitosan (w/v %) Gel Solution	34
3.4	The Preparation of PVA (w/v %) Solution	34
3.5	The Preparation of Exfoliated Graphite Powder	35

3.6	The Pre solutior	eparation of Chitosan/PVA/EG (w/v %) composite	35
3.7	Printed	Circuit Board (PCB) Patterning	35
3.8	Electro	chemical Deposition Technique	36
3.9	Hard Ba	ake	37
3.10	Electric	al Testing Upon Exposure of Analyte	37
3.11	Parame Concen	ter Study of Chitosan Solution with Various tration	38
3.12	Parame Solution	ter Study of Chitosan/PVA (Various PVA Concentration) n	39
3.13	Parame	ter Study of Chitosan/PVA (Various Ratio) Solution	40
3.14	Parame	ter Study of Exfoliated Graphite (EG) Powder	41
3.15	Gas Tes	sting	42
3.16	Charact	rerization	43
	3.16.1	Fourier Transform Infrared Spectroscopy (FTIR)	43
	3.16.2	Fourier Electron Scanning Electron Microscopy	44
		(FESEM)	
	3.16.3	Ultra Violet Visible (UV-VIS)	45
	3.16.4	X-Ray Diffraction (XRD)	46
	3.16.5	Four Point Probe Analyzer	47
Ć			

CHAPTER 4 RESULTS AND DISCUSSION

4.1	Introduction	49
4.2	Fabrication of Chitosan Films, CS/PVA Films and CS/PVA/EG Films	49
4.3	FTIR Studies of Films	50
	4.3.1 Functional Groups Studies of chitosan films	51

	4.3.2	Functional Group Studies of CS/PVA (Various PVA Concentration)	52
	4.3.3	Functional Group Studies of CS/PVA (Various Ratio)	54
	4.3.4	Comparison of Functional Group Studies of Chitosan Films and CS/PVA Films	55
	4.3.5	Functional Group Studies of Graphite powder, Graphite Intercalated Compound (GIC) powder and Exfoliated Graphite (EG) powder	56
	4.3.6	Functional Group Studies of CS/PVA/EG Film	57
	4.3.7	Comparison of Functional Group Between Chitosan Films and CS/PVA/EG Films	59
	Morpho	logy Studies of Films	60
	4.4.1	Surface Morphology of Various Concentration of Chitosan Film	60
	4.4.2	Surface Morphology of CS/PVA (Various PVA Concentration)	62
	4.4.3	Surface Morphology of CS/PVA (Various Ratio)	63
	4.4.4	Comparison of Surface Morphology of Chitosan and CS/PVA Film	65
	4.4.5	Surface Morphology of Graphite, Graphite Intercalated Compound (GIC) and Exfoliated Graphite (EG)	65
	4.4.6	Surface Morphology of CS/PVA/EG Film	68
ć	XRD St	udies of Films and Powder	70
Q	4.5.1	Diffraction of Various Concentration of Chitosan Films	70
	4.5.2	Diffraction of CS/PVA (Various PVA concentration) Film	71
	4.5.3	Diffraction of CS/PVA (Various Ratio) Film	73
	4.5.4	Comparison Diffraction of Chitosan and CS/PVA Film	74
	4.5.5	Diffraction of Graphite, Graphite Intercalated Compound (GIC) and Exfoliated Graphite (EG)	75

4.4

4.5

	4.5.6	Diffraction of CS/PVA/EG Film with Various Concentration of EG	76
	4.5.7	Comparison Diffraction of Chitosan and CS/PVA/EG Film	78
4.6	Optical	Band Gap	80
	4.6.1	Optical Band Gap of Chitosan Films	80
	4.6.2	Optical Band Gap of CS/PVA (Various PVA Concentration) Film	83
	4.6.3	Optical Band Gap of CS/PVA (Various Ratio) Film	86
	4.6.4	Comparison of Optical Band Gap of Chitosan and CS/PVA Films	88
	4.6.5	Optical Band Gap of CS/PVA/EG Film	89
	4.6.6	Comparison of Optical Band Gap of Chitosan Film and CS/PVA/EG Film	92
4.7	Conduc	tivity Studies of Films	93
	4.7.1	Conductivity Studies of Chitosan Film	93
	4.7.2	Conductivity Studies of CS/PVA (Various PVA Concentration) Film	95
	4.7.3	Conductivity Studies of CS/PVA (Various Ratio) Film	96
	4.7.4	Conductivity Studies of CS/PVA/EG Film	98
Č	4.7.5	Compare the Conductivity Studies of Chitosan and CS/PVA/EG Film	99
4.8	Element	t Analysis in Graphite, GIC and EG	101
	4.8.1	Element in Graphite Powder	101
	4.8.2	Element in Graphite Intercalated Compound (GIC) Powder	103
	4.8.3	Element in Exfoliated Graphite (EG) Powder	104
4.9	Measure	ement Technique for Electrical Testing	106
4.10	Electric	al Testing of Film Sensor	107

	4.10.1	Electrical Testing to Determine Best Concentration of Chitosan	107
	4.10.2	Electrical Testing to Determine Best Deposition Time of Chitosan	109
	4.10.3	Electrical Testing to Determine Best Deposition Voltage of Chitosan	110
4.11	Electrica Film Ser	al Testing of CS/PVA (Various PVA Concentration)	111
	4.11.1	Electrical Testing to Determine Best Concentration of CS/PVA (Various PVA Concentration)	111
	4.11.2	Electrical Testing to Determine Best Deposition Time of CS/PVA (Various PVA Concentration)	114
	4.11.3	Electrical Testing to Determine Best Deposition Voltage of CS/PVA (Various PVA Concentration)	115
4.12	Electrica	al Testing of CS/PVA (Various Ratio) Film Sensor	116
	4.12.1	Electrical Testing to Determine Best Ratio of CS/PVA (Various Ratio)	116
	4.12.2	Electrical Testing to Determine Best Deposition Time of CS/PVA (Various Ratio)	118
	4.12.3	Electrical Testing to Determine Best Deposition Voltage of CS/PVA (Various Ratio)	119
4.13	Electrica	a Testing of CS/PVA/EG Film Sensor	120
ć	4.13.J	Electrical Testing to Determine Best Concentration of CS/PVA (Various Ratio)	120
	4.13.2	Electrical Testing to Determine Best Deposition Time of CS/PVA/EG	122
	4.13.3	Electrical Testing to Determine Best Deposition Voltage of CS/PVA/EG	123
	4.13.5	Comparison Response of Chitosan and CS/PVA/EG	124
4.14	Sensing towards	Mechanisms of Chitosan and CS/PVA/EG Films Sensor Wet Air	125
4.15	Gas Sen Films Se	sing Study of Chitosan Films Sensor and CS/PVA/EG	127

	4.15.1	Response of CS/PVA/EG on Ammonia Exposure	127
	4.15.2	Electrical Testing of CS/PVA/EG Films Sensor Upon Ammonia Exposure	129
	4.15.3	Comparison Chitosan Film Sensor and CS/PVA/EG Film Sensor	131
	4.15.4	Mechanisms for Ammonia Sensing	132
4.16	Sensing	Properties of CS/PVA/EG toward Gas Exposure	133
	4.16.1	Sensitivity	133
	4.16.2	Response and Recovery	135
	4.16.3	Repeatability	136
	4.16.4	Stability	138
	4.16.5	Selectivity	138
		to,	
CHAP	FER 5	SUMMARY AND RECOMMENDATION	139
		XO	
5.1	Summa	ry	139
5.2	Recom	nendation	140
		KO'	
REFER	ENCES		142
LIST	F PUBL	JCATION	152

LIST OF TABLES

NO		PAGE
2.1	Summary of Chitosan for Sensor Application	11
2.2	Summary of PVA for Sensor Application	14
2.3	Summary of Exfoliated Graphite Based Sensor	20
2.4	Summary of Current Ammonia Sensor	25
2.5	Table of sensor characteristics	28
3.1	Preparation of Solutions	34
3.2	Variation concentration of chitosan solution	38
3.3	Concentration of chitosan/PVA (Various PVA Concentration) solution	39
3.4	Various ratio of chitosan/PVA solution	40
3.5	Variation weight percent of EG powder	41
4.1	Functional Group of chitosan films with various concentration	51
4.2	Functional Group of chitosan with various concentration of PVA films	53
4.3	Functional Group of CS/PVA with various ratio	54
4.4	Functional Group of graphite powder, GIC powder and EG powder	57
4.5	Table of functional group of CS/PVA/EG films with various concentration of EG	58
4.6	Position of Two-theta and d-spacing of various concentration of chitosan films	71
4.7	Position of Two-theta and d-spacing of CS/PVA with various concentration of PVA	73
4.8	Position of Two-theta and d-spacing of CS/PVA with various ratio	74

4.9	Position of Two-theta and d-spacing of Graphite, GIC and EG Powder	77
4.10	Position of Two-theta and d-spacing of CS/PVA/EG films with various concentration of EG	78
4.11	Strain Value of Chitosan and CS/PVA films	79
4.12	Strain Value of CS/PVA/EG films with various concentration of EG	79
4.13	Thickness and Energy Band Gap of Chitosan Film with various concentration of chitosan	82
4.14	Thickness and Band Gap of CS/PVA Film with various concentration of PVA	85
4.15	Thickness and Energy Gap of CS/PVA Films with (Various ratio)	87
4.16	Comparison of band gap if Chitosan and CS/PVA film	89
4.17	Thickness and Energy Gap of CS/PVA/EG Films with various concentration of EG	91
4.18	Conductivity of Chitosan films with various concentration	94
4.19	Conductivity of CS/PVA films with various concentration of PVA	96
4.20	Conductivity of CS/PVA films with various ratio	97
4.21	Conductivity of CS/PVA/EG films with various	99
	concentration of EG	
4.22	Percentage of Element in Graphite Powder	102
4.23	Percentage of Element in Graphite Intercalated Compound (GIC) Powder	104
4.24	Percentage Element in exfoliated Graphite (EG) Powder	105
4.25	Response and recovery of chitosan and CS/PVA/EG films towards gas exposure	135

NO		PAGE
2.1	Molecular structure of chitosan	8
2.2	Schematic interpretation of the polycationic state of chitosan in acid aqueous solutions	9
2.3	Molecular structure of PVA	12
2.4	Crystal structure of Graphite	16
2.5	Graphite Intercalation Compound (GIC)	18
2.6	HOMO-LUMO Concept	26
2.7	Excitation of Electron in Band Region	26
2.8	Band Gap of Conductor, Insulator and Semiconductor	28
3.1	Flow chart for phase one of preparation of films	32
3.2	Flow chart for phase two of preparation of films sensor	33
3.3	Gold pattern substrate on PCB	36
3.4	Electrochemical Deposition process of CS film sensor and CS/PVA/EG Film	36
3.5	Illustration of Electrical Set Up for Sensor Testing	38
4.1	FTIR spectra of chitosan films with various concentration	52
4.2	FTIR spectra of chitosan with various concentration of PVA	53
4.3	FTIR spectra of CS/PVA with various ratio	55
4.4	FTIR spectra of chitosan (100:0) and CS/PVA (95:5)	55
4.5	FTIR spectra of graphite, GIC and EG	57
4.6	FTIR spectra of CS/PVA/EG with various EG concentration	58
4.7	FTIR spectra of chitosan and CS/PVA/EG films	59
4.8	FESEM image of various concentration of chitosan films	61

LIST OF FIGURES

4.9	FESEM image of chitosan with various concentration of PVA	63
4.10	FESEM image of various ratio of CS/PVA films	64
4.11	FESEM image of chitosan and CS/PVA films	65
4.12	Morphology of Graphite, GIC and EG Powder	66
4.13	Morphology of CS/PVA/EG with various concentration of EG	69
4.14	Diffraction of various concentration of chitosan films	70
4.15	Diffraction of chitosan with various concentration of PVA films	72
4.16	Diffraction of CS/PVA with various ratio	74
4.17	Comparison diffraction of chitosan and CS/PVA with various ratio	75
4.18	Diffraction of Graphite, GIC and exfoliated graphite powder	76
4.19	Diffraction of CS/PVA/EG Film	77
4.20	Comparison Diffraction of Chitosan and CS/PVA/EG Film	78
4.21	Absorbance of chitosan films with various concentration	81
4.22	Plot to determine direct band gap energies of chitosan films with various chitosan concentration	81
4.23	Band Gap Energy and Splitting of energy level	83
4.24	Absorbance of CS/PVA with various concentration of PVA	84
4.25	Plot to determine direct band gap energies of CS/PVA films with various PVA concentration	84
4.26	Absorbance of CS/PVA films with various ratio	85
4.27	Plot to determine direct band gap energies of CS/PVA films with various ratio of CS/PVA concentration	87
4.28	Plot of comparison direct band gap energies of chitosan film and CS/PVA film	88

4.29	Absorbance of CS/PVA/EG films with various concentration of EG	90
4.30	Plot to determine direct band gap energies of CS/PVA/EG films with various EG concentration	90
4.31	Comparison absorbance of chitosan, CS/PVA and CS/PVA/EG films	92
4.32	Plot comparison direct band gap of chitosan, CS/PVA and CS/PVA/EG Film with various concentration of EG	92
4.33	Conductivity of chitosan films	94
4.34	Conductivity of CS/PVA (Various PVA Concentration) Film	95
4.35	Conductivity of CS/PVA (Various Ratio) Film	97
4.36	Conductivity of CS/PVA/EG (Various EG Concentration) Film	99
4.37	Comparison conductivity of Chitosan and CS/PVA/EG (Various EG Concentration) Film	101
4.38	Element Graphite Powder	102
4.39	Element in Graphite Intercalated Compound (GIC) Powder	103
4.40	Element in Exfoliated Graphite (EG) Powder	105
4.41	Response of chitosan films sensor toward wet air with various concentration of chitosan	108
4.42	Humidity (%) during electrical testing of chitosan with various concentrations	109
4.43	Temperature (°C) during electrical testing of chitosan with various concentrations.	109
4.44	Response (%) vs Time (s) of chitosan film sensor with different deposition time	110
4.45	Response (%) vs Time (s) of chitosan film sensor with different deposition voltage	111
4.46	Response of Chitosan/PVA film sensor towards wet air with different concentration of PVA	112

4.47	Humidity (%) during electrical testing of chitosan with various concentration of PVA	113
4.48	Temperature (°C) during electrical testing of chitosan with various concentration of PVA	113
4.49	Response of deposition time of CS/PVA film sensor towards with concentration of 5.00%	114
4.50	Response of deposition voltage of CS/PVA with concentration of 5.00%	115
4.51	Response of CS/PVA film sensor towards wet air with various ratio of CS/PVA	117
4.52	Humidity (%) during electrical testing of ratio CS/PVA	117
4.53	Temperature (°C) during electrical testing of ratio CS/PVA	118
4.54	Response of deposition time of CS/PVA with various ratio	119
4.55	Response of deposition voltage of CS/PVA with various ratio	120
4.56	Response of CS/PVA/EG film sensor towards wet air with various concentration of EG	121
4.57	Humidity (%) of CS/PVA with various concentration of EG	121
4.58	Temperature (°C) of CS/PVA with various concentration of EG	122
4.59	Response of deposition time of CS/PVA with various concentration of EG	123
4.60	Response of deposition voltage of CS/PVA with various concentration of EG	124
4.61	Comparison response of chitosan and CS/PVA/EG film sensor	124
4.62	Response of chitosan film sensor during exposure of ammonia with various concentration	128
4.63	Humidity (%) of chitosan film sensor during exposure of ammonia with various concentration	128

4.64	Temperature (°C) of chitosan film sensor during exposure of ammonia with various concentration	129
4.65	Response of CS/PVA/EG film sensor during exposure of ammonia with various concentration	130
4.66	Humidity (%) of CS/PVA/EG film sensor during exposure of ammonia with various concentration	130
4.67	Temperature (°C) of CS/PVA/EG film sensor during exposure of ammonia with various concentration	131
4.68	Comparison of maximum response chitosan film sensor and CS/PVA/EG film sensor towards ammonia gas	131
4.69	Graphic sensing mechanisms of ammonia gas	133
4.70	Calibration curve of ammonia with various concentrations for chitosan film sensor	134
4.71	Calibration curve of ammonia with various concentrations for CS/PVA/EG film sensor	135
4.72	Repeatability of Ammonia of maximum response for each cycles for CS film sensor	136
4.73	Repeatability of Ammonia of maximum response for each cycles for CS/PVA/EG film sensor	137
4.74	Standard deviation of ammonia with different concentration for chitosan film sensor	137
4.75	Standard deviation of ammonia with different concentration for CS/PVA/EG film sensor	137
4.76	Selectivity of CS/PVA/EG film sensor towards ammonia and hexanal	138

LIST OF ABBREVIATIONS

VOCs	Volatile Organic Compound
PVA	Polyvinyl alcohol
КОН	Potassium hydroxide
EG	Exfoliated Graphite
SnO ₂	Tin oxide
Cr_2O_3	Chromium (III) oxide
TiO ₂	Titanium dioxide
CNTs	Carbon nanotubes
NH ₃	Ammonia
CO_2	Carbon dioxide
NO _x	Nitrogen oxide
RFID	Radio-frequency identification
O ₂	Oxygen
NO	Nitric oxide
FTIR	Fourier Transform Infrared
TGA	Thermogravimetric analysis
FESEM	Field Emission Scanning Electron Microscope
AgNPs	Silver nanoparticles
SPR	Surface plasmon resonance
HRTEM	High-resolution transmission electron microscopy
AA	Acrylic acid

AN	Aniline
PANI	Polyaniline
РРу	Polypyrrole
MFC	Mass flow controller
GIC	Graphite intercalated compound
EMI	Electromagnetic shielding
N_2	Nitrogen
xGNP	Exfoliated graphite nanoplatelet
HNO ₃	Nitric acid
H_2SO_4	Sulphuric acid
PCB	Printed circuit board
CS	Chitosan
ATR	Attenuated total reflection
KBR	Potassium bromide
eV	Electron volt
A	Absorbance
m . C	meter
is i	Siemen
© S	Sensor response
V	Volts
R^2	Correlation coefficient
ppm	Part per million

LIST OF SYMBOLS



Pengaruh Grafit Terkelupas pada Sifat-Sifat Pengesan Ammonia yang Didasarkan pada Kitosan/PVA

ABSTRAK

Kitosan telah digunakan sebagai bahan pengesan untuk aplikasi pengesanan gas. Untuk mengukuhkan sifat-sifat kitosan sebagai bahan pengesan, kitosan (CS) telah diadun dengan polivinil alcohol (PVA) dan ditambahkan grafit terkelupas (EG). Kitosan filem, CS/PVA filem dan CS/PVA/EG filem telah dikaji untuk tujuan pencirian kesan EG ke atas microstruktur dan sifat elektrik daripada gabungan filem tersebut. Filem komposit disediakan dengan kaedah penuangan untuk tujuan pencirian. Ujikaji pencirian telah dibuat menggunakan alatan Spektrofotometer Ultra Lembayung-Nampak (UV-VIS), Probe Empat Titik (Ujian Kekonduksian), Pembelauan X-Ray (XRD), Mikroskop Elektron Pengimbas (FESEM) and Spektroskopi Infra-Merah (FTIR). Kitosan dengan kepekatan 1.75 (w/v) % telah diadun dengan polivinil alkohol (PVA) dengan kepekatan 5.00 (v/v) % dan dicampur dengan nisbah 95:5 ml dan ditambah dengan grafit terkelupas (EG). Keputusan menunjukkan penambahan EG dengan kepekatan 0.8 (w/v) % memberikan kekonduksian yang tertinggi dalam CS/PVA/EG filem.Filem CS/PVA/EG filem pengesan telah menjalani pembentukan di atas elektrod bersalut emas dengan menggunakan teknik pemendapan elektokimia untuk membentuk bahan pengesan untuk mengesan kehadiran gas. Kesimpulan telah dibuat bahawa masa pemendapan elektrokimia 6 minit dan voltan pemendapan elektrokimia 2.5 voltan adalah parameter terbaik untuk membentuk filem pengesan. Tahap terbaik ciri-ciri pengesan telah dapat dikenalpasti bahawa kitosan dengan kepekatan 1.75 (w/v) %, CS/PVA bernisbah 95:5 ml dengan kepekatan PVA (5.00 (v/v) %) dan EG dengan kepekatan 0.8 (w/v)% dengan mendedahkan filem pengesan ke atas udara basah. Parameter ini telah digunakan untuk membentuk filem pengesan untuk diujikaji dengan mendedahkan filem pengesan ke atas gas ammonia. Kitosan filem pengesan dan CS/PVA/EG filem pengesan telah didedahkan kepada gas ammonia dengan kepekatan yang berlainan seperti 10 ppm, 20 ppm, 30 ppm, 40 ppm dan 50 ppm untuk mengenalpasti tahap tindak balas daripada Kitosan filem pengesan dan CS/PVA/EG filem pengesan. Keputusan ujikaji elektrik telah menunjukkan bahawa Kitosan filem pengesan dan CS/PVA/EG filem pengesan teleh memberi tindak balas yang meningkat selaras dengan meningkatnya kepekatan gas ammonia. Kedua-dua filem pengesan telah menunjukkan ciri-ciri yang baik, stabil, kebolehupayaan untuk menghasilkan tindak balas yang sekata dan kebolehupayaan untuk kembali kepada tindak balas yang terendah, sensitive. CS/PVA/EG filem pengesan telah memberikan nilai tindak balas yang tinggi berbanding dengan kitosan filem pengesan sepanjang terdedahnya filem pengesan ke atas gas ammonia. Selain itu, CS/PVA/EG filem pengesan telah menunjukkan kebolehupayaan untuk membezakan nilai tindak balas sepanjang didedahkan dengan gas lain seperti gas hexanal.

The Influence of Exfoliated Graphite on Sensing Properties of Chitosan/PVA Based Ammonia Sensor

ABSTRACT

Chitosan was used as a sensing material for gas sensing applications. In order to nurture the chitosan properties as a sensing material, chitosan (CS) was blended with polyvinyl alcohol (PVA) and filled with exfoliated graphite (EG). The chitosan films, CS/PVA films and CS/PVA/EG films were characterized to study the effect of EG on microstructure and electrical properties of the composites films. The films casting method has been used to characterize the composite films. The characterization analysis was done using Ultra Violet-Visible (UV-VIS), Four Point Probe (Conductivity testing), X-Ray Diffraction (XRD), Fourier Electron Scanning Electron Microscopy (FESEM) and Fourier Transform Infrared Spectroscopy (FTIR). Chitosan with concentration 1.75 (w/v) % was blended with polyvinyl alcohol (PVA) with concentration 5.00 (v/v) % of ratio 95:5 ml and filled with exfoliated graphite (EG). The results showed that the addition of EG with concentration of 0.8 (w/v) % gives the highest conductivity value of CS/PVA/EG films. The CS/PVA/EG films sensor was deposited on the gold pattern electrode by using electrochemical deposition technique to fabricate the sensor for the ammonia detection. The best electrochemical deposition time and deposition voltage in this study were 6 minutes and 2.5 voltage to fabricate the films sensor. The optimum sensing properties were obtained with CS concentration of 1.75 (w/v) %, CS/PVA ratio of 95:5 ml with PVA concentration of 5.00 (v/v) % and EG concentration of 0.8 (w/v) % upon exposure to wet air. This parameter of CS films sensor and CS/PVA/EG films sensor was used to fabricate the films sensor to test upon exposure of ammonia gas. The CS film sensor and CS/PVA/EG film sensors were exposed to ammonia with different concentration i.e. 10 ppm, 20 ppm, 30 ppm, 40 ppm and 50 ppm to examine the sensing properties of the CS film sensor and CS/PVA/EG film sensors. The electrical results showed that the CS film sensor and CS/PVA/EG film sensors showed the increasing response value as the concentration of ammonia increases. Both sensors exhibit good response, stability, repeatability, recovery, sensitivity and selectivity during exposure to ammonia. CS/PVA/EG films sensor yield highest response compared to CS films sensors towards exposure of ammonia gas. In addition, CS/PVA/EG films sensor was selective towards exposure of different analyte such as hexanal.

CHAPTER 1

INTRODUCTION

1.1 Research Background

The detection of volatile compounds and pollutants gases is important to ensure the safety of mankind. The emission of volatile compounds and pollutant gases need to be controlled in order to concern by the harmful effects to health of humans. This harmful gas was mainly produced by industrial processes, engines combustion, transportations, manufacturing of fertilizers and etc (Wales et al., 2016). Volatile compounds and pollutants gases are categorized as toxic gases and have potential to cause cancer to human and give harm to environments (Celebioglu et al., 2016).

Ammonia (NH₃) in one of the toxic gases that exists in our surrounding environment include in air, soil and water. Exposure to high concentration of ammonia (>5000 ppm) lead to severely affects human health and contribute to death. Low exposure of low concentration (25-150 ppm) of ammonia cause the irritation of human skin, eyes and respiratory tract (Basova et al. 2016). Therefore, it is necessary to monitor the presence of ammonia in potential sites and fields of having the probability presence of ammonia such as agriculture, chemical industries, pharmaceutical, hydrogen fuels, defense and food processing industries. Therefore, the development of fast and real time detection of ammonia in atmosphere at room temperature (Mani et al. 2013).