SYNTHESIS OF GRAPHENE BY PULLULAN-ASSISTED EXFOLIATION OF GRAPHITE USING ULTRASONIC BATH

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We hereby declare that we have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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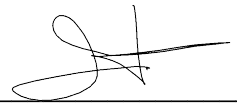

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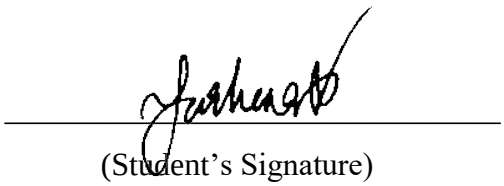

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I can still feel the warm touch of their loving hands.

As tears roll down my face, I know they are in a better place.

Al-Fatihah.

ABSTRAK

Grafin adalah asas karbon untuk semua dimensi grafit termasuk grafit, nanotiub karbon, fulerena, dan lain-lain. Menariknya, grafin dapat terkelupas dari grafit menggunakan pelarut yang sesuai sebagai medium pengelupasan. Oleh kerana sebahagian besar pelarut organik yang digunakan dalam sintesis grafin seperti N-metil-2-pirolidon (NMP) dan Dimetiformamida adalah bersifat toksik dan karsinogen, ia telah digantikan dengan pelarut endah alam sekitar untuk mengatasi masalah tersebut. Sebelum ini, polisakarida seperti gam Arab, kitosan, dan natrium alginat pernah digunakan sebagai pelarut untuk menghasilkan grafin. Namun, sebahagian besar polisakarida ini mempunyai kelikatan tinggi yang menyukarkan proses pengelupasan dan pencucian grafit. Oleh itu, dalam tesis ini, polisakarida dengan kelikatan rendah iaitu pullulan telah dipilih sebagai penyebar hijau dalam membantu pengelupasan grafit untuk menghasilkan grafin menggunakan mesin pembersih ultrasonik. Sifat kimia grafin berasaskan pullulan (graphene) disahkan dengan menggunakan Spektroskopi Ultraungu (UV-vis), Spektroskopi Foton X-Ray (XPS), dan Spektroskopi Inframerah Transformasi Fourier (FTIR). Sementara itu, Spektroskopi Raman, Mikroskopi Elektron Transmisi (TEM), dan Mikroskopi Daya Atom (AFM) dikendalikan untuk mengkaji analisis struktur graphene. Dari UV-vis yang diuji, hasilnya membuktikan kehadiran graphene pada panjang gelombang 269nm. XPS juga mendedahkan kandungan karbon yang tinggi dengan nilai nisbah 4.7 karbon kepada oksigen (C: O). Seperti yang dijangkakan dari pemerhatian FTIR, wujud keberadaan puncak C=C dan –OH dalam graphene setelah proses pengelupasan berlaku. Dengan jelas, terdapat intensiti kecacatan graphene yang kecil diperoleh dari Raman Spectroscopy. Panjang dan ketebalan rata-rata graphene yang terkelupas diukur oleh TEM dan AFM masing-masing pada julat ~ 800 nm dan ~ 4 nm (8 lapisan). Bagi mengkaji keadaan optimum untuk menghasilkan jumlah graphene yang tinggi, kesan parameter pemprosesan seperti masa sonikasi, jisim pullulan, dan jisim grafit telah diambil kira. graphene kemudian digunakan sebagai elemen pengesan untuk kertas foto graphene bagi menunjukkan kegunaan graphene. Mikroskopi Elektron Pengimbasan (SEM) digunakan untuk menyiasat morfologi kertas foto graphene. Dalam perangkaan sifat mekanik dan elektrik kertas foto graphene, ujian tegangan dan multimeter telah digunakan dengan sewajarnya. Pengukuhan graphene dalam kertas foto meningkatkan kekuatan tegangan hingga 21%. Mengagumkan, prestasi elektrik kertas foto graphene dengan faktor tolok (GF) yang tinggi (140) menunjukkan bahawa grafin berasaskan pullulan boleh digunakan sebagai dakwat konduktif elektrik. Secara keseluruhan, kaedah ini akan mewakili strategi termudah, paling efisien, dan paling endah alam sekitar untuk penyediaan grafin bebas toksik dan pengembangan sensor regangan berasaskan grafin yang fleksibel.

ABSTRACT

Graphene is the carbon basis for all graphitic dimensionalities like graphite, carbon nanotube (CNT), fullerene, and others. Interestingly, graphene can be exfoliated from graphite using a suitable solvent that acts as an exfoliating medium. Since most of the organic solvents used in the graphene synthesis like N-methyl-2-pyrrolidone (NMP) and Dimethylformamide are toxic and carcinogenic, an environment-friendly solvent was used to counter the problem. Previously, polysaccharides such as Arabic gum, chitosan, and sodium alginate were employed as solvents to produce graphene. However, the majority of these polysaccharides have high viscosity, which makes graphite exfoliation and washing process difficult. Therefore in this thesis, a lower viscosity polysaccharide which is pullulan was selected as a green dispersant in assisting the exfoliation of graphite in order to produce graphene under ultrasonic bath conditions. The chemical properties of pullulan-based graphene (graphene) was confirmed by utilizing Ultraviolet-Visible Spectroscopy (UV-vis), X-Ray Photon Spectroscopy (XPS), and Fourier Transformation Infrared Spectroscopy (FTIR). Meanwhile, Raman Spectroscopy, Transmission Electron Microscopy (TEM), and Atomic Force Microscopy (AFM) were used to study the structural analysis of graphene. From the UV-vis tested, the result proved the presence of graphene at a wavelength of 269nm. The XPS also reveals high carbon content with a value of 4.7 carbon to oxygen ratio (C:O). As expected, from the FTIR analysis, the existence of C=C and –OH peaks in the graphene was observed after the exfoliating process. Notably, a small defect intensity (ID/IG) of graphene was obtained from Raman Spectroscopy. The lateral size and thickness of the exfoliated graphene were measured by TEM and AFM at the range of 201 - 300 nm and ~2 nm (5 layers) respectively. To study the optimum condition to produce a high yield of graphene, the effect of processing parameters such as duration of sonication, pullulan, and initial mass of graphite were taken into account. The graphene was then enforced as a sensing element for graphene photo paper to demonstrate the use of graphene. Scanning Electron Microscopy (SEM) was used to investigate the morphology of the graphene photo paper. In order to get the mechanical and electrical properties of the graphene photo paper, tensile tests and multimeter have been utilized accordingly. The reinforcement of graphene in photo paper enhanced the tensile strength up to 21%. Impressively, the electrical performance of the graphene photo paper with 140gauge factor (GF) implies that the pullulan-based graphene could be applied as an electrically conductive ink. Overall, this work would represent the simplest, most environmentally friendly and most efficient strategy for the toxic-free graphene preparation and the development of photo paper for graphene-based strain sensor.

TABLE OF CONTENT

DECLARATION

TITLE PAGE

ACKNOWLEDGEMENTS **ii**

ABSTRAK **iii**

ABSTRACT **iv**

TABLE OF CONTENT **v**

LIST OF TABLES **ix**

LIST OF FIGURES **x**

LIST OF SYMBOLS **xii**

LIST OF ABBREVIATIONS **xiii**

CHAPTER 1 INTRODUCTION **14**

1.1 Research Background 14

1.2 Problem Statement 16

1.3 Research Objective 17

1.4 Research Scope 17

1.5 Thesis Outline 18

CHAPTER 2 LITERATURE REVIEW **20**

2.1 Introduction 20

2.2 Graphene History 21

2.3 Graphene Synthesis 23

2.3.1 Mechanical Exfoliation of Graphene 25

2.4 Liquid-Phase Exfoliation 29

2.4.1 Sonication Assisted Liquid-Phase Exfoliation 30

2.5 Biomolecule-assisted Exfoliation of Graphene 33

2.5.1 Pullulan as Stabilizing Agent 37

2.6 Development of Thin Film-Based Strain Sensor 39

2.6.1 Graphene-Based Strain Sensor 42

2.7 Characterization of Graphene and Graphene Paper 44

2.7.1 Ultra-Violet Spectroscopy 44

2.7.2 Transmission Electron Microscopy and Scanning Electron Microscopy 46

2.7.3 Atomic Force Microscopy 48

2.7.4 Raman Spectroscopy 50

2.7.5 X-Ray Photon Spectroscopy 52

2.7.6 Fourier Transformation Infrared Spectroscopy 53

CHAPTER 3 METHODOLOGY 54

3.1 Introduction 54

3.2 Materials 55

3.3 Preparation of Pullulan 55

3.4 Preparation of Graphene 56

3.5 Fabrication of Graphene/Paper 57

3.6 Characterization Works of Graphene and Graphene/Paper 57

3.6.1 Ultraviolet-Visible Spectroscopy 57

3.6.2 Fourier Transform Infrared Spectroscopy 58

3.6.3 Atomic Force Microscopy 59

3.6.4 Raman Spectroscopy 60

3.6.5 Transmission Electron Microscopy 62

3.6.6 X-ray Photon Spectroscopy 63

3.6.7 Scanning Electron Microscopy 64

3.6.8 Tensile Testing 65

3.6.9 Digital Multimeter 66

CHAPTER 4 RESULTS AND DISCUSSION 67

4.1 Introduction 67

4.2 Liquid-Phase Exfoliation of Graphene in Pullulan 68

4.2.1 Initial Evidence of Pullulan Modified Graphene 68

4.3 Properties of Pullulan Modified Graphene 69

4.3.1 Lateral Size of Sheet 69

4.3.2 Thickness of Graphene 71

4.3.3 Defect in Structure after Exfoliation 73

4.3.4 Validation of Graphene Functionalization 74

4.3.5 Chemical Composition of Graphene 77

4.4 Parameter Studies of graphene 79

4.4.1 Pullulan Mass 79

4.4.2 Initial Graphite Mass 80

4.4.3 Sonication Time 81

4.5 Application of Graphene as Smart Ink for Paper-Based Sensor 83

4.5.1 Morphology and Physical Changes 83

4.5.2 Mechanical Properties 85

4.5.3 Strain Sensor Performance 86

CHAPTER 5 CONCLUSION 89

5.1 Conclusions 89

5.2 Recommendations in Future Works 90

REFERENCES 91

APPENDICES **104**

APPENDIX A **105**

APPENDIX B **106**

APPENDIX C **107**

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