



Ain Shams University
Faculty of Science
Chemistry Department

Preparation and characterization of carbon-based nanostructured materials for advanced electrochemical Capacitors

A Thesis Submitted to

By

Dalia Mohamed Ahmed Elgendy

B.Sc. (Botany- Chemistry) 2002

M.Sc. (Chemistry) 2010

Supervised by

Prof. Dr. Essam Elsayed Foad Elsherbini

Professor of Physical Chemistry,
Faculty of Science, Ain Shams University

Prof. Dr. Nabil Abdel Ghany El-Manakhly

Professor of Electrochemistry, National Research Centre

Dr. Nageh Khalf Allam

Associate Professor of Physics
The American University in Cairo

2018

**Preparation and characterization of carbon-
based nanostructured materials for
advanced electrochemical
Capacitors**

A Thesis

**Submitted for Degree of Ph.D
(Physical Chemistry)**

By

Dalia Mohamed Ahmed Elgendy

B.Sc. (Botany- Chemistry) 2002

M.Sc. (Chemistry) 2010

**Department of Chemistry
Faculty of Science
Ain Shams University
2017**

APPROVAL SHEET FOR SUBMISSION

Title of the Ph.D. Thesis: Prepration and characterization of carbon-based nanostructured materials for advanced electrochemical capacitors

Name of the candidate: Dalia Mohamed Ahmed Elgendy

This thesis has been approved for submission by the supervisors:-

1. Prof. Dr. Essam Elsayed Foad Elsherbini
(Ain-Shams University)

Signature:

2. Prof. Dr. Nabil Abdel Ghany. El-Mankhly
(National Research center)

Signature:

3. Associate. Prof. Dr. Nageh K. Allam
(American University in Cairo)

Signature:

Approval

Prof. Dr. Ibrahim H. A. Badr
Chairman of Chemistry Department

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

قالوا

سببنا انك لا تعلم لنا
إلا ما علمتنا إنك أنت
العليم العظيم

صدق الله العظيم

سورة البقرة الآية: ٣٢

Dedication

To

My mother and the soul of my father



I am very grateful to **Allah the Most Merciful** Who blessed my efforts, showed me the way and provided me with power to present this work in an acceptable form.

*I wish to express my sincere appreciation to **Prof. Dr. Essam Fouad ELsherbiny** Professor of Physical Chemistry, Faculty of Science, Ain Shams University. **Prof. Dr. Nabil Abdel Ghany El-Manakhly**, Professor of Electrochemistry National Research centre and, Associate. **Prof. Dr. Nageh Khalf Allam Associate**, Professor of Physics, the American university in Cairo suggesting the problem, their keen supervision, continuous and valuable help, fruitful criticism and encouragement during the execution of this work.*

*Firstly, I would like to express my great thanks to **Prof. Essam ELsherbiny**, who made it possible to complete this work in its final form and may Allah reward and bless him.*

*I would like first to express my deep gratitude to my advisor, **Prof. Nabil Mankhly** and for giving me the opportunity and for his invaluable guidance and inspiration throughout this work, and encouraged me in every possible way. He has helped me develop my professional skills and I will emulate his drive and work ethic throughout my career.*

*It is my pleasure to express my deep gratitude to **Dr. Nageh Allam**, Thank you very much for being so nice to me and keeping your door open every time I come to bother you and may Allah reward you and bless your valuable effort with me.*

*I would like to express my sincere appreciation to **Eng. Ali Hanafy**, and thanks to great efforts with me.*

*I would like to express my sincere appreciation to all colleagues in the National Research center and all colleagues in the Physics Department, Faculty of Science in the American University in Cairo for their valuable help& advice. May Allah reward you and bless your valuable efforts with me. Specially thank to **Hayam, Safaa ELsayed, Basma, Zeinab, Maryam, Basant Elsebai and Ahmed**.*

I would be remiss if I did not take a moment to thank my family: my mother, my brothers and my sisters and I am so very grateful for them.

Finally, I would like to express my sincere appreciation to all those who helped me make it and make it possible to complete this work in its final form.

 **Dalia Mohamed Ahmed Elgendy**

CONTENTS

Serial No.	Item	Page no.
	Acknowledgement	
	Abstract	i
	LIST OF ABBREVIATIONS	ii
	List of Figures	iii
	1. CHAPTER (1) Introduction.	1
	1.1.Necessity for Energy storage.....	1
	1.2.History of supercapacitors	3
	1.2.1.Energy and Power	5
	1.3. What are supercapacitors?.....	7
	1.4.Types of supercapacitors	8
	1.4.1 Electric Double Layer Capacitors (EDLCs)	9
	1.4.1 .1 Materials for Electric Double Layer Capacitors	10
	1.4.2 Pseudocapacitors	12
	1.4.3 Hybridessupercapacitors.	15
	1.4.3.1 Composite electrodes	15
	1.4.3.2 Asymmetric hybrid capacitors	15
	1.1.5. Electrolytes for supercapacitors	16
	1.1.5.1. Aqueous electrolytes	17
	1.1.5.2. Organic electrolytes	17
	1.1.5.3. Ionic liquid electrolytes	17
	1.2. LITERATURE REVIEW	18
	1.2.1. Preparation of Graphene by Different Methods.	20
	 1.2.2 Electrocatalysis for Supercapacitors Application.	22
	1.3.Aim of the work:	32
	2. CHAPTER (2) Experimental 1.4.Types of supercapacitors	33
	2.1. Materials and Reagents	33
	2.2. Synthesis	33
	2.2.1. Synthesis of Spongy Graphene Oxide (SGO)	33
	2.2.2. Preparation of adenine-functionalized graphene oxide (FGO)	35

2.2.3. Preparation of Reduced Grapheneoxide (RGO) and Functionalized Graphene (FG)	35
2.2.4. Preparation of functionalized graphene Silver (FG-Ag Nano particles)	36
2.2.5. Preparation of TiO ₂ and HydrogenatedTiO ₂ (H-TiO ₂)Nanotubes Powder	38
2.2.6. Preparation of functionalized graphene (FG-TiO ₂ H) Nano particles)	39
2.2.7. Preparation of Covalently functionalized graphene with guansine monophosphate disodium salt at room temperature	41
2.3. Experimental Techniques	42
2.3.1. Physicochemical characterization	42
2.3.1.1. X-ray diffraction (XRD)	42
2.3.1.2. Fourier Transform Infrared Spectrometer (FT-IR)	43
2.3.1.3. Ramen spectroscopy	44
2.3.1. 4. Thermal Gravimetric Analysis (TGA) under Nitrogen	45
2.3.1.5. X-ray photoelectron Spectroscopy (XPS)	45
2.3.1.6. Field Emission scanning Electron Microscope (FESEM).	46
2.3.1.7. Energy dispersive X-ray spectroscopy (EDX)	46
2.3.1.8. Transmission electron microscope (TEM).	47
2.3.2. Electrochemical characterization	48
2.3.2. 1. Preparation of electrodes and electrochemical measurements	48
2.3.2.2. Cyclic Voltammetry	50
2.3.2.3. Glvanostatic Charge and Discharge	50
3. CHAPTER (3) Results and Discussion	51
Part 1: Adenine-functionalized Spongy Graphene for Green and High-Performance Supercapacitors	51

3.1.Characterization of grphene oxide (GO)and functionalize graphene (FG).....	52
3. 1.1. X-ray Diffraction (XRD)	52
3. 1.2. UV- vis. Spectroscopy	53
3. 1.3. Fourier Transform Infrared Spectrometer (FT-IR)	55
3. 1.4. Thermal Gravimetric Analysis (TGA)	56
3. 1.5. Ramen Spectroscopy	58
3. 1. 6. Field Emission Scanning Electron Microscopy (FESEM).	60
3. 1.7. Transmission Electron Microscope (TEM)	61
3. 2. Electrochemical Measurements	62
3. 2.1. Cyclic Voltammetry	62
3. 2. 2.Glvano static Charge and Discharge	65
Part 2: Synergistic effect of silver and adenine on boosting the super capacitance performance of spongy graphene	70
3 .3. Characterization of Functionalized graphene Silver (FG-Ag).	71
3. 3.1. X-ray Diffraction (XRD)	71
3. 3.2. UV- vis.Spectroscopy	72
3. 3.3. Fourier Transform Infrared Spectrometer (FT-IR)	74
3. 3.4. Thermal Gravimetric Analysis (TGA)	75
3. 3.5. Ramen Spectroscopy	78
3. 3.6. Field Emission Scanning Electron Microscopy (FESEM)	79
3. 3.7. Energy Dispersive X-ray Spectroscopy (EDX)	81
3. 3.8. Transmission Electron Microscope (TEM).	81
3.4. Electrochemical Measurements	83
3.4.1. Cyclic Voltammetry	83
3.4.2.Glvano static Charge and Discharge	86
Part 3: Black Tatiana/Spongy Graphene Nano composites for High-Performance Supercapacitors	91
3.5. Characterization of Functionalize graphene Titanium (FGTio2H)	92
