

Faculty of Science Ain Shams University

Physico-Chemical Properties of Polymer Nanocomposites

By
Islam Ali Elsayed Ali

A Thesis submitted to the

Faculty of Science

Ain Shams University

In Partial Fulfillment of the Requirements for the

Master Degree of Science

In

Chemistry

Faculty of Science
Ain Shams University
Cairo, Egypt



Physico-Chemical Properties of Polymer Nanocomposites

By

Islam Ali Elsayed Ali
A Thesis submitted to the
Faculty of Science
Ain shams University
Fulfillment of the Requirements

In Partial Fulfillment of the Requirements for the Master Degree of Science

In

Chemistry

Under the Supervision of

Prof.Dr / Mahmoud Refae Mahmoud Akl

Prof. of organic chemistry,
Chemistry Department,
Faculty of Science,
Ain Shams University

Dr / Gamal Abdel Aziz Meligi

Assistant Prof. of organic chemistry,
Chemistry Department,
Faculty of Science,
Ain Shams University,

Prof.Dr / Zakaria Ismaiel Ali

Prof. of radiation chemistry,
National Center for Radiation
Research and Technology,
Atomic Energy Authority

Supervisors

A Thesis Title

Physico-Chemical Properties of Polymer Nanocomposites

Researcher Name:

Islam Ali Elsayed Ali

Supervisors

Prof.Dr / Mahmoud Refae Mahmoud Akl

Prof. of organic chemistry,
Chemistry Department,
Faculty of Science,
Ain Shams University

Dr / Gamal Abdel Aziz Meligi

Assistant Prof. of organic chemistry-Chemistry Department, Faculty of Science, Ain Shams University,

Prof.Dr / Zakaria Ismaiel Ali

Prof. of radiation chemistry,
National Center for Radiation
Research and Technology,
Atomic Energy Authority

Prof.Dr.Hamed Derballa

Head of Chemistry Department

Referees

A Thesis Title

Physico-Chemical Properties of Polymer Nanocomposites

Researcher Name:

Islam Ali Elsayed Ali

Supervisors

Prof.Dr / Mahmoud Refae Mahmoud Akl

Prof. of organic chemistry, Chemistry Department, Faculty of Science, Ain Shams University

Dr / Gamal Abdel Aziz Meligi

Assistant Prof. of organic chemistry, Chemistry Department, Faculty of Science, Ain Shams University,

Prof.Dr / Zakaria Ismaiel Ali

Prof. of radiation chemistry, National Center for Radiation Research and Technology, Atomic Energy Authority

Referees committee:

Prof.Dr / Zakaria Ismaiel Ali Prof.Dr / Mahmoud Refae Mahmoud Akl Prof. of radiation chemistry, Prof. of organic chemistry, Chemistry Department, National Center for Radiation Faculty of Science, Research and Technology, Ain Shams University Atomic Energy Authority Dr / Hamada Abdelwahab Ibrahim Prof.Dr / Maher Helmy Helal Prof. of organic chemistry, Assistant Prof. of organic chemistry, Chemistry Department, Chemistry Department, Faculty of Science, Faculty of Science, Helwan University Alazhar University,

Prof.Dr.Hamed Derballa
Head of Chemistry Department

Acknowledgment

ACKNOWLEDGMENTS

First and foremost, with a deep sense of gratitude, I want to thank Allah AlrahmanAlrahiem for allowing me to perform this thesis smoothly even though I face some obstacles throughout my work and peace is upon Prophet Mohammed. This thesis is the result of the work whereby I have been accompanied and supported by many people. It is a pleasant opportunity for me to express my gratitude for all of them.

Firstly, I would like to thank my advisor, **Prof.Dr.Mahmoud Refae Mahmoud Akl** .I appreciate that he let me work on this project which I like too much and I can do well on it. During those years in my study and research as a graduate student, I learned a lot from him. He taught me how to become a real scientist. I really appreciate his supports and advices from my heart. Without him, I could not achieve so much today. Secondly, **Dr.Gamal Abdel aziz Meligi** by his outstanding leader-ship paved the inroads on which I could develop the skills and knowledge requisite of a scientist. He welcomed me into his laboratory and gives me the chance to work freely. I appreciate him for his patience and confidence in me and so many valuable discussions on our experiments. I dedicate this work to the spirit of the late **Prof. Dr.Zakaria Isamiel Ali.** He gave me freedom to learn on my own, lending advice when it was appropriate. He allowed me to set my own goals and achieve them, rather than dictating what I should or should not do. And if some experiments

weren't going my way, or things didn't turn out how I thought they would, he went beyond simply giving scientific advice by telling an anecdote about his days as a graduate student, bringing smiles to all of the lab members.

This thesis could not have been completed without the endless love and blessings from my family. I wish to thank my parents, who taught me the value of hard work and rendered me enormous support during the whole tenure of my research work. I would like to thank my small family for the patience to go through this endeavor hand-in-hand with me. Without her dearly accompaniment, both physically and spiritually, there would be no such an achievement.

Islam Ali Elsayed Ali

2015

Aim Of The Work

Aim of the Work

Nanocomposites are of great interest in recent years because they are considered to be novel functional materials with a wide range of potential applications in bio and chemical sensors, electronics, catalysis and optics. A number of production techniques have been reported for preparation of metallic colloids using metal salts as starting materials, such as chemical, photochemical, electrochemical, radiolytic, and sonochemical reduction. Due to its unique advantages, the irradiation-based strategy, as a wrathful tool, has been extensively used to prepare nanoscale particles and materials.

In this thesis, we have developed a novel approach to synthesize silver / polystyrene/polyvinylpyrrolidone (Ag/PS/PVP) nanocomposite hybrid material which is based on the seeded growth of Ag nanoparticles within PS/PVP matrix. In this synthetic strategy, we use gamma-irradiation, to utilize the reorganized seed points, or nucleation sites, to initiate the growth of Ag nanoparticles directly on the polymer backbone. Gamma irradiation of Ag/PS/PVP nanocomposite can reduce metal ions to zero valent metal particles, avoiding the use of additional reducing agents and the consequent side reactions. Furthermore, the amount of zero valent nuclei can be controlled by varying the irradiation dose. Homogeneous formation of silver nanoparticles (AgNPs) is favorable as it results in uniformly dispersed nanoparticles. Through this process we are assured of successful producing of PS polymer filled with high monodispersed silver nanoparticles (AgNPs). Polystyrene (PS), which is a water insoluble polymer, has important advantages of good

mechanical and acoustic optical properties, where this parameter determines the photo induced response in the newly suggested composite. In our experiment, polyvinylpyrrolidone (PVP) was used as a polymer capping reagent, utilizing the interactions of silver ions with aminogroup in the poly vinyl pyrrolidone molecules.



Contents

Title	page
1.Introduction	I
1.1 Nanoscience and Nanotechnology	_
1.1.1 Classification of nanomaterials	1
1.2 Silver Nanoparticle	3
1.3 Polystyrene	8
1.3.1 Synthesis of polystyrene	9
1.4 Metal – Polymer Nanocomposites	10
1.4.1 Methods for preparation of Nanocomposites	35
1.5 Mechanism of PVP in the preparation of Nanocomposites	36
1.6 Ionizing radiation	38
1.6.1 Advantage of Ionizing radiation	39
II Literature Review	40
III Materials and Experimental Techniques	70
3.1 Materials	70
3.2 Synthesis of polystyrene	72
3.3 Synthesis of Ag/ PS/PVP nanocomposite microsphere	72
3.4 Irradiation process	73
3.5 Characterization Techniques	77
3.5.1 Spectroscopic analysis	77

	1
3.5.1.1 Ultraviolet / visible spectrophotometry, (UV/VIS)	77
3.5.1.2 Fourier Transform Infrared (FTIR) Spectroscopy	,,
	78
3.5.1.3 Sample preparation	81
3.5.2 Transmission Electron Microscope (TEM)	01
Measurements	82
3.5.3 X-ray diffraction (XRD) Measurements	83
3.5.4 Thermogravimetric analysis, (TGA)	03
	83
4.I Results and Discussion	0.5
4 1 111/4 . 1 4 77 1 1 (177/2/10) 41	85
4. I.1Ultraviolet-Visible (UV/VIS) Absorption	
Spectroscopic Studies of Ag/PS Nanocomposites.	87
4.I.1.1 Effect of irradiation dose on the optical properties	
of Ag/PS/PVP nanocomposite microsphere.	87
	07
4. I.1.2 Effect of Ag ⁺ Ion Concentration on the Optical	
Properties of (PS/PVP) at different Irradiation doses (20,	95
40, 80, and 120 kGy).)3
4. I.1.3 Effect of PVP Content on the Optical Properties of	
Ag/PS Nanocomposites at different dose (40, 120 kGy).	104
4. I.1.4 Estimation of the Optical Band Gap Energy of	
Ag/PS/PVP Nanocomposites microsphere	107
4.I.2 Transmission electron microscope (TEM) of Ag/PVA	
nanocomposites	114
4. I.2.1The effect of irradiation dose on the morphology of	
Ag/PS/PVP Nanocomposites	114
4. I.2.2Effect of AgNO ₃ on the morphology by TEM of the	119
Ag/PS/PVP Nanocomposite.	119
4.I.3 X-Ray diffraction Analysis	
	123

4.I.3.1 Effect of γ-irradiation on XRD of the as-prepared samples	123
4.I.3.2 Effect of AgNO ₃ concentration on the XRD pattern of the as-prepared samples	130
4. I.3.3Determination of particle size of Ag/PS/PVP	
nanocomposite microspheres.	135
4.I.4 Fourier Transform Infrared (FTIR) Spectroscopy	
Investigation	138
4. I.4.1 FTIR spectroscopic analysis of irradiated	
PS/PVP/Ag Nanocomposite microsphere	142
4.I.5 Thermogravimetric analysis (TGA)	151
4. I.5.1Kinetics analysis of Ag/PS/PVP nanocomposites microsphere	154
4. I.5.1.1Study of thermal gravimetric analysis (TGA) for	
different concentration of AgNo ₃ at the same irradiation	156
dose (80 kGy).	
4. I.5.1.2 Study of thermal gravimetric analysis for the	
same concentration of AgNO ₃ at different Irradiation doses	162
$(1*10^{-2} \text{ M})$	
Conclusion	169
References	172
Summary	
Arabic summary	

List of Abbreviations