



Ain Shams University
Faculty of Science
Physics Department

Synthesis and Characterization of Core-Shell Composites of Nano-sized Ferrites

A Thesis Submitted for the Degree of Ph.D of Science

In

Solid State Physics

To

Physics Department, Faculty of Science, Ain Shams University

By

Ibrahim Ali Abdullah Alsugia

M. Sc. In Physics, Damascus University, 2012

2017



Ain Shams University
Faculty of Science
Physics Department

Synthesis and Characterization of Core-Shell Composites of Nano-sized Ferrites

A Thesis Submitted for the Degree of Ph.D of Science

In

Solid State Physics

To

Physics Department, Faculty of Science, Ain Shams University

By

Ibrahim Ali Abdullah Alsugia

M. Sc. In Physics, Damascus University, 2012

2017



Ain Shams University
Faculty of Science
Physics Department

Synthesis and Characterization of Core-Shell Composites of Nano-sized Ferrites

A Thesis Submitted for the Degree of Ph.D of Science
in Solid State Physics

To

Physics Department, Faculty of Science, Ain Shams University

By

Ibrahim Ali Abdullah Alsuqia

M. Sc. In Physics, Damascus University, 2012

Supervised by

PROF. DR. Adel Abd El –Sattar

PROF. DR. Hesham El-Sayed

Professor of Solid State Physics

Professor of Solid State Physics

Faculty of Science -Ain Shams University

Faculty of Science -Ain Shams University

2017

To My Family

To my father and my mother

Words cannot express how grateful I am to my mother and father. My Parents, whose has been a source of sympathy, and for their continuous encouragement and inspiration to me. This work is the result supplication

To my brothers who supported me at the beginning of Education

To my wife who stood long by my side until my success saw the light

To my beloved daughters Raghad and Dima, the smiles and flowers that were born in my life.

To all who helped me and wished me good luck

ACKNOWLEDGEMENT

First, I thank Allah, the most Beneficent, the most Merciful, who gave me the ability to do this work and I am asking His support for further success in my scientific work.

This work is dedicated to my loving family, for I would not be who I am today without their love, friendship and everlasting support. I am forever thankful.

I would like to express my deep gratitude to Prof. Dr. Adel Abd El-Sattar Mohammed for his supervision, guidance and continuous support to me, during the study period and his handling upscale and humble, which had a major impact in the completion of this work.

I would like to express my deep gratitude to Prof. Dr. Hesham Mohammed El-Sayed for her supervision, guidance and continuous support to me, during the study period, great efforts, fruitful discussions, and for his help to overcome all the difficulties and obstacles that I faced during the work.

The valuable discussions and continuous assistance which were so willingly given by supervisors during the course of this work will never be forgotten.

Ibrahim Ali Abdullah Alsuqia

2017



CONTENTS

<i>ACKNOWLEDGEMENT</i>	i
<i>CONTENTS</i>	ii
<i>List of Tables</i>	vii
<i>List of Figures</i>	viii
<i>Abstract</i>	xvi
<i>SUMMARY</i>	xviii
Chapter 1	2
Theoretical Background	2
1.1 Introduction to Nanotechnology	2
1.2 Classification of Nanomaterials	6
1.3 The Importance of Nanomagnetism	7
1.4 Magnetic Nanoparticles	8
1.5 Fundamental Theory of Magnetism	10
1.5.1 Origins of Magnetism	10
1.5.2 Classification of Magnetic Materials	13
I. Diamagnetic Materials	13
II. Paramagnetic Materials	14
III. Ferromagnetic Materials	16
IV. Antiferromagnetic Materials	17
V. Ferrimagnetic Materials	18
1.6 Types of Ferrites	19
1.6.1 Soft Ferrites	19
1.6.2 Hard Ferrites	20
1.7 Crystal Structure of Ferrites	20
1.7.1 Spinel Ferrites Structure	21
1.7.2 Types of Spinel Ferrites	23
I. Normal Spinel Ferrites	23

II. Inverse Spinel Ferrites.....	24
III. Random Spinel Ferrites	24
1.8 Application of ferrite:.....	24
1.9 Magnetization Curves and Hysteresis Loops	25
1.10 Domain Theory.....	27
1.11 Magnetism of Nanomagnetic Materials.....	30
1.11.1 Superparamagnetism	30
1.11.2 Measuring of Blocking Temperature (ZFC-FC) Curves	32
1.12 Magnetic Exchange Interactions.....	34
1.12.1 Direct exchange	35
1.12.2 Indirect Exchange (Double exchange)	36
1.12.3 Superexchange.....	37
1.13 Magnetic Anisotropy in Magnetic Nanoparticles.....	38
I. Magnetocrystalline Anisotropy	39
II. Stress Anisotropy	39
III. Shape Anisotropy.....	40
IV. Surface Anisotropy	40
1.14 Core-Shell Model of Magnetic Nanocomposites.....	41
1.15 Exchange Bias.....	45
1.16 Application of Core-Shell Nanoparticles	52
Chapter 2.....	56
Literature Review	56
2.1 Cobalt Ferrite Nanoparticles	56
I. Change of Preparation Methods of Cobalt Ferrite	56
II. Doping of Cobalt Ferrite Nanoparticles	62
III. Change of Thermal Treatment of Cobalt Ferrite Nanoparticles	67
2.2 Core-Shell Nanoparticles	72
2.3 Core-Shell-Shell Structure Nanoparticles	81

<i>Aim of the Work</i>	84
Chapter 3	86
Experimental Techniques	86
3.1 Methodology and Experimental Techniques	86
3.2 Synthesis Method for Magnetic Nanoparticles	86
3.3 Experimental Details	90
3.3.1 Materials	90
3.3.2 Synthesis of Cobalt Ferrite, Nickel and Zinc Ferrite by the Hydrothermal Method:	90
3.3.3 Synthesis of the CoFe₂O₄-NiFe₂O₄ Core-Shell Magnetic Nanocomposite	91
3.3.4 Preparation of CoFe₂O₄-ZnFe₂O₄ Core-Shell Magnetic Nanocomposite	92
3.3.5 Synthesis of the CoFe₂O₄-ZnFe₂O₄-NiFe₂O₄ Core-Shell-Shell Magnetic Nanocomposite	93
3.4 Characterization Techniques	93
3.4.1 Structural Characterization	94
3.4.1.1 X-Ray Diffraction (XRD)	94
3.4.1.2 Fourier Transform Infrared Spectroscopy	96
3.4.1.3 Transmission Electron Microscopy	97
3.4.1.4 Energy Dispersive X-ray Spectroscopy (EDX)	99
3.4.2 Magnetic Characterization	101
3.4.2.1 Vibrating Sample Magnetometer (VSM)	101
3.4.2 The Initial Permeability and Curie Temperature (T_c) Measurements:	105
3.4.3 Measuring of Specific Power Absorption Rate (SAR)	107
Chapter 4	111
Results and Discussion	111
Physical and Magnetic Properties	111

4.1 CoFe₂O₄ Magnetic Nanoparticle.....	111
4.1.1 X-Ray Diffraction Analysis	111
4.1.2 FT–IR Spectra Analysis	112
4.1.3 TEM Spectrum Analysis	114
4.1.4 The Magnetic Properties	116
4.1.4.1 Room Temperature Hysteresis Loop Measurements	116
4.1.4.2 Zero Field Cooling (ZFC) Magnetization Measurements	117
4.1.4.3 Temperature Dependence of The Hysteresis Loops:	118
4.2.1 X-Ray Diffraction Analysis	121
4.2.2 FT–IR Spectra Analysis	125
4.2.3 TEM and EDX Spectrum Analysis	127
4.2.4 The Magnetic Properties	132
4.2.4.1 Room Temperature Hysteresis Loop Measurements	132
4.2.4.2 Zero Field Cooling (ZFC) Magnetization Measurements	134
4.1.4.3 Temperature Dependence of the Hysteresis Loops:	135
4.2.4.2 Change of Hysteresis Parameters with Temperature.....	138
4.3 CoFe₂O₄-ZnFe₂O₄ Core-Shell Magnetic Nanocomposite.....	142
4.3.1 X-Ray Diffraction Analysis	142
4.3.2 FT–IR Spectra Analysis	145
4.3.3 TEM and EDX Spectrum Analysis	147
4.3.4 The Magnetic Properties	152
4.3.4.1 Room Temperature Hysteresis Loop Measurements	152
4.3.4.2 Zero Field Cooling (ZFC) Magnetization Measurements	154
4.3.4.3 Temperature Dependence of The Hysteresis Loops:	155
4.3.4.4 Change of Hysteresis Parameters with Temperature.....	157
4.4 CoFe₂O₄-ZnFe₂O₄-NiFe₂O₄ Core-Shell-shell Magnetic	
Nanocomposite	162
4.4.1 X-Ray Diffraction Analysis	162

4.4.2 FT–IR Spectra Analysis	165
4.4.3 TEM Analysis	167
4.4.4 The Magnetic Properties	169
4.4.4.1 Room Temperature Hysteresis Loop Measurements	169
4.4.4.2 Zero Field Cooling (ZFC) Magnetization Measurements	172
4.4.4.3 Temperature Dependence of the Hysteresis loops:	174
4.4.4.4 Change of Hysteresis Parameters with Temperature	176
4.5 Specific Absorption Rate (SAR)	180
Chapter 5.....	185
Conclusion	185
Reference	188
Publication.....	221
.....	221
الملخص العربي	I

