SYNTHESIS, CHARACTRIZATION AND APPLICATION OF NOVEL METALLONANOCOMPOSITE SYSTEMS IN VARIOUS PROCESSES OF ENVIRONMENTAL AND INDUSTERIAL IMPACT

A Thesis Submitted By

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B.Sc. and M.Sc.

For the Award of

Ph.D. degree in Chemistry

Under supervision of

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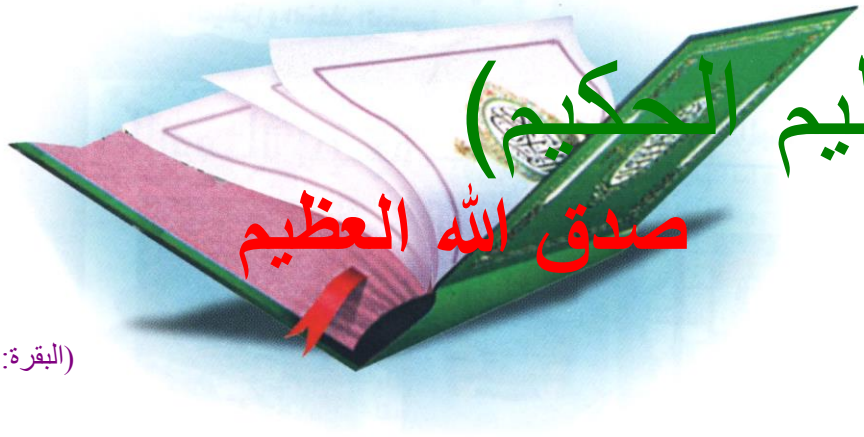
2016

بِسْمِ ٱللَّهِ ٱلرَّحْمَٰنِ ٱلرَّحِيمِ

(قالوا سبحانك لا علم لنا
الا ما علمتنا انك انت
العليم الحكيم)



صدق الله العظيم

(البقرة: الآية٣٢)

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Thesis submitted to: Faculty of Science, Ain-Shams University

For the award of: Ph.D. degree in Chemistry

By Safaa Rushdy Atia Fouda

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Prof. Dr.

To my Father,mother, the dearest children of mysister (Mohmmed, Basant and Malk), sister(Marwa) and my brother (Moustafa)

Acknowledgement

In the name of Allah, Most Gracious, Most Merciful

It is my duty, as a start to praise Almighty Allah, lord of the world, whose guidance, blessings and help enabled me to take my first step on the path of improving my knowledge through this humble effort.

I would like to express my deepest respect and most sincere gratitude to my supervisor, ***Prof. Salah A. Hassan***, Professor of Physical Chemistry, Faculty of Science, Ain-Shams University, who initiated and assigned this study. His valuable advice, help, guidance, encouragement at all stages of my work, stimulating discussion and also reviewing critically the manuscript. His constructive criticism and comments from the initial conception to the end of this work are highly appreciated. I am greatly indebted to his assistance and understanding in matters of non-academic concern, which have helped me endure some difficult times during my study period.

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Also all thanks to ***Dr. Nour El-Din Ahmed Abd-El Star*** Lecture of Organic Chemistry, Faculty of Science, Ain-Shams University, who helped me more in this work, her advices were very useful in

solving all the problems that I faced specially through nanocomposites synthesis and discussion.

A very special appreciation is due to **my parents, my sister and my brother** not only for their constant encouragement but also for their patience and understanding throughout. May God bless them in all their endeavors because without their unreserved support, completion of this study would not have been possible.

Also, I would like to thank my lab friends, for their cooperation and assistance during the achievement of this work.

Finally, I thank **each person** who had helped me in accomplishing this work especially my colleagues.

Candidate

Safaa Rushdy

2016

Abstract

Name: Safaa Rushdy Fouda

Thesis title: *Synthesis, Characterization, and Application of Novel metallonanocomposite systems in various processes of Envirnomental and Industrial impact.*

In the present work, magnetite nanoparticles (M NPs) were incorporated into poly (amido amine) (PAMAM, G=2) dendrimer matrix to form the mother composite M NPs ($1*10^{-3}$ M)@PAMAM. Dendrimer-modified composites were synthesized through conjugation with rice straw ash (RSA) or montomorillonite (MMT). Hybrid composites were obtained by applying ($1*10^{-3}$ M, $3*10^{-3}$ M and $6*10^{-3}$ M) M NPs, namely M NPs @PAMAM@RSA and M NPs @PAMAM@MMT. In addition polyaniline based-RSA composite was prepared, followed by application of M NPs to form M NPs @PANI@RSA hybrid nanocomposite with the same molar ratios.

Structural characteristics of the various samples were investigated through XRD, FTIR techniques. Morphological textural characteristics were estimated from TEM, SEM and low-temperature adsorption-desorption isotherms of N_2 and pore size distribution analysis. The stability of colloidal system and conductivity were investigated by DLS and zeta potential.

Different linkages and interactions during the synthesis processes were investigated.

All the synthesized hybrid composites were applied in removal of hazardous dyes (e.g., XO, MG and ROS.HCl), ions (e.g., Br^-, NO^-_3, and NH_4^+) and heavy metals (e.g., Hg).

Keywords:

Magnetite nanoparticles; PAMAM dendrimer; Polyaniline; Hybrid nanocomposites; Montomorillonite; Rice Straw Ash

Aim of the Work

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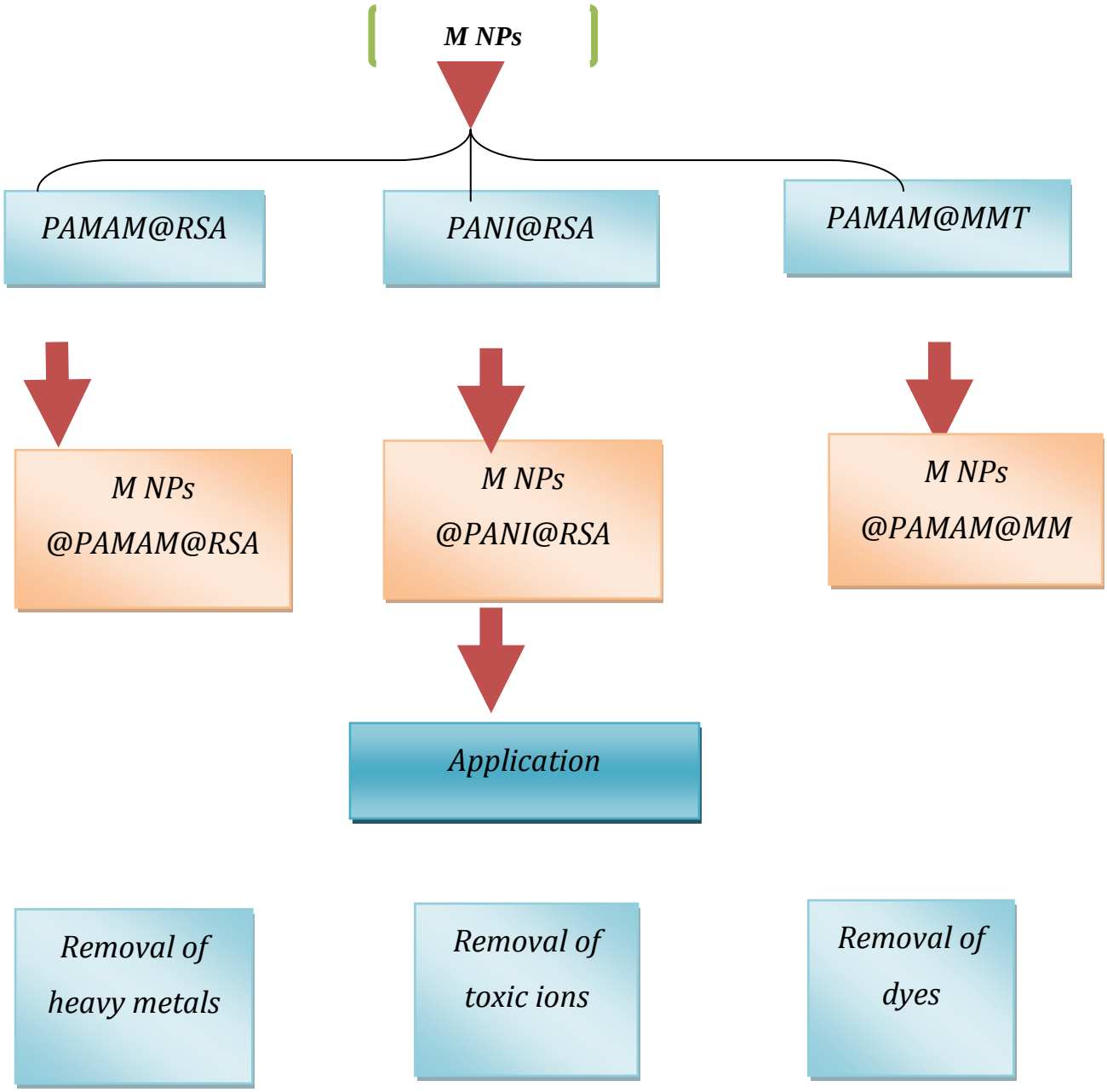
THE AIM OF WORK

This work is aimed at manipulation of novel nanocomposites based on M NPs@PAMAM dendrimer and using them as efficient nancatalysts and adsorbents for removal of dyes and toxic ions from wastewater.

This study included the following items:-

- Synthesis of magnetite nanoparticles in the absence and in the presence of PAMAM dendrimer as mother nanocomposite for further modification.
- Synthesis of modified MMT by intercalation of PAMAM dendrimer into Na-MMT matrix with applying the magnetite nanoparticles in different ratios ($1*10^{-3}$ M, $3*10^{-3}$ M and $6*10^{-3}$ M) M NPs @PAMAM@MMT.
- Synthesis of RSA-conjugated PAMAM dendrimer with applying magnetite nanoparticles of different concentrations M NPs @PAMAM@RSA.
- Synthesis of RSA-conjugated polyaniline with applying magnetite nanoparticles of different concentrations
  M NPs @PANI@RSA.
- Performing physicochemical characterization of different composites, by adopting XRD, FTIR, N_2-physisorption, TEM, SEM and DLS techniques.
- Study of the catalytic removal (adsorption) efficiencies of the as-synthesized composites towards the dyes ,e.g., XO, MG, ROS-HCl dyes) and ions, e.g., Br^-, NO_3^- and NH_4^+ as well as heavy metals, e.g., Hg.
- Examination of adsorption kinetic models in all cases.

PLAN OF WORK


M NPs
PAMAM@RSA
PANI@RSA
PAMAM@MMT
M NPs @PAMAM@RSA
M NPs @PANI@RSA
M NPs @PAMAM@MM
Application
Removal of heavy metals
Removal of toxic ions
Removal of dyes

Abbreviations

Poly(amidoamine) dendrimer	PAMAM
Sodium montmorillonite	Na^+-MMT
Cation exchange capacity	CEC
Magnetite nanoparticles	M NPs (Fe_3O_4)
Rice straw ash	RSA
X-ray diffraction	XRD
Fourier Transform infrared	FTIR
Transmission electron microscope	TEM
Scanning electron microscope	SEM
Specific Surface area	S_{BET}
BET-C energetic constant	C_{BET}
Monolayer coverage	V_m
Pore size distribution	P_aSD
Dynamic light scattering	DLS
Zeta potential	Z
Xylenol orange	XO
Malachite green	MG
Rosaniline-hydrochloride	ROS-HCl
The initial concentration of dye in liquid phase	C_o
Dye concentration in liquid phase at equilibrium	C_e
The volume of the dye used	V
The mass of adsorbent used	M
Maximum wavelength of absorbance	λ_{Max}

Contents

II: Experimental