شبكة المعلومات الجامعية

# بسم الله الرحمن الرحيم


HOSSAM MAGHRABY

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# التوثيق الالكتروني والميكروفيلم

HOSSAM MAGHRABY

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# جامعة عين شمس

## التوثيق الإلكتروني والميكروفيلم

## قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها

علي هذه الأقراص المدمجة قد أعدت دون أية تغيرات

## يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار

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# بعض الوثائق الأصلية تالفة

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# بالرسالة صفحات

# لم ترد بالأصل

HOSSAM MAGHRABY

# *Study the Reaction Conditions for the Chemically Polymerized Aniline Films.*



*A Thesis*
Submitted in partial fulfillment
for the degree of M. Sc.
(in Chemistry)

*Presented by*

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(B. Sc. in Chemistry)

*Supervised by*

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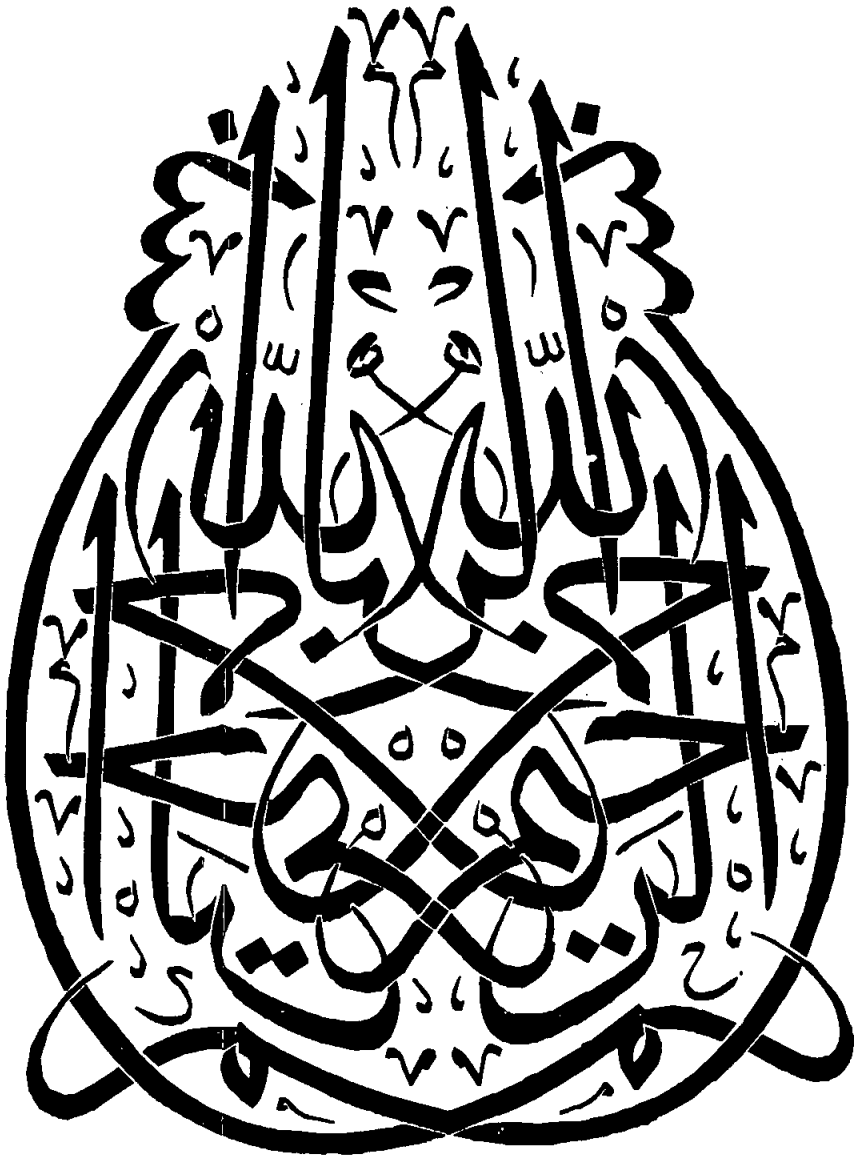

# *Dedication*

I would like to dedicate this work to my parents, my brothers and my sisters, especially for my grand brother *Dr.Eng. Khaled Khodary Esmaiel Shenashen* and his family and thank them for always giving me their prays, confidence, encouragement and continuous support.

*To Yara, Nada, Gehad, Khaled and Ahmed*

Candidate

*Mohamed Al-Khodary Esmaiel Shenashen*

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# *Curriculum Vitae*

*Name* :*Mohamed Al-Khodary Esmaiel Shenashen*

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# *Note*

Besides the research work materialized in this thesis, the candidate has attended the following courses for one year in the following topics:-

1- Chemical kinetics
2- Electrochemistry
3- Solid-state chemistry
4- Molecular spectroscopy
5- Organic reaction mechanism
6- Coordination and inorganic chemistry
7- Organometallic chemistry
8- Physical organic chemistry
9- German language

He had successfully passed the final examination of these courses held in September 2001.

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# *Acknowledgment*

***In the name of Allah the ALL-Merciful, the Ever-Merciful.***
All praise is for Allah, lord of the world, who guided and helped me specially during this work and in all life fields.

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"No task is ever accomplished single-hand" and I'm indebted to all stuff of the chemistry department in faculty of science, Tanta university, and my laboratory company *Mohamed Esmail Whdan, Enas H. ElGhezawy, Mohamed Azam and Ahmed M. Abou Elmagd*, for their assistance during the preparation this work.

Candidate

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# *Summary*

Conducting polymers have emerged as an important class of electronic materials because of their potential applications in solid state batteries, electrochromic displays and microelectronic devices, etc. a large number of conducting polymers such as polyacetylene, polypyrrole and polythiophene etc, have been synthesized and characterized in recent past. Among these polyaniline( PANI) family of conjugated polymers has been considered as extremely interesting and unusual member of polymers having the delocalized p electrons. Conducting polyanilines are basically poly (paraphenylene amineimine) in which its oxidation state can be varied from the fully oxidized (pernigraniline) to fully reduced (leucoemeraline) states. The most stable emeraldine state refers to 50% oxidized form of PANI.

A numbers of studies on PANI relating to its structure, morphology, electrochromic and solid state properties have been reported in the literature. In spite of these interesting developments, the basic chemistry and physics of PANI has not yet been completely elucidated. The present thesis is a systematic attempted towards the solution of some problems encountered in relation to the synthesis and characterization

The first chapter divided to two parts. The first part deals with an up-to-date review in relation to PANI polymer. The aim of this part is to enlighten research works done on PANI after a state of the art on the synthesis, mechanism of polymerization, PANI doping, deposition methods of thin PANI films and application of PANI. The second part of the introduction deals with the basic theory of the technique used which is the quartz crystal microbalance (QCM). This part includes an introduction about piezoelectricity, development of piezoelectric quartz crystal mass sensing, QCM solution phase sensing and application of QCM sensors

Chapter two deals with an investigation to the optimum reaction conditions for the PANI films. The QCM technique was used to determine the reaction conditions for PANI film deposition during the oxidative polymerization of aniline with ammonium persulfate (APS) as an oxidizing agent in aqueous acidic medium. The effect of the initial molar ratios of the APS/aniline on the yield and the thickness as well as on the growth rate of the PANI films was investigated. The most efficient initial molar ratio of APS/aniline was found to be ~1.15 ± 0.1. This was in close agreement with the optimum reaction conditions for the PANI formed in the bulk

Third chapter involves a study to the kinetics of PANI film deposition during the oxidative polymerization of aniline with APS in aqueous acidic medium. The formation PANI film is half-order with respect to APS and first-order with respect to aniline. The activation energy of the polymerization is 39.79 kJ/mol. This is in close agreement with the activation energy obtained for the PANI formed in the bulk. The UV-visible spectra for PANI films grown onto glass supports immersed into the bulk solution during the polymerization process were measured. This revealed that the trends of the growth of PANI film thickness with time and the growth of the PANI film absorbance with time are similar except the induction periods. The detection of the induction period using the UV-visible spectroscopy delayed to longer times, in comparison to the induction period detection using the QCM experiment under the same conditions. The sensitivity of the QCM technique to the oligomer sites formation is superior compared with the UV-visible absorption detection.

The fourth chapter investigates the influence of HCl on the PANI film formation. It was concluded that the yield of PANI film deposition increased with increasing the acid concentration from 0.025 up to 0.1M., and decreased when the concentration of HCl increased from 0.1M to 0.4 M. In addition, for solutions of HCl concentration higher than 0.1 M, a degradation for the deposited PANI film started to appear at the early stages of polymerization. The effect of temperature on the yield formation was studied. The yield of PANI film deposition increased when the temperature decreased.

# Aim of the work

Study the oxidative polymerization of aniline to deposit polyaniline films on some substrates such as gold and glass using the chemical method. The oxidative polymerization of aniline was carried out using ammonium persulfate. The weight and thickness of the formed films were measured using the quartz crystal microbalance technique. The study included a determination of the optimum concentration conditions of the polyaniline film deposition in the aqueous acid medium. The kinetics and the spectral properties of the film formation were evaluated. Also, the effect of HCl concentrations and temperature on the film formation were studied.