Ain Shams University
Faculty of Science
Chemistry Department

Preparation of new graphite based composite electrodes with some metal oxide nanoparticles and their applications in energy

A Thesis

Submitted to Chemistry Department – Faculty of Science
Ain Shams University in Partial Fulfillment for Requirements of the
Master Degree of Science (M.Sc.) In Chemistry

By

Ahmed Sayed Essa Abd El-Razik
B.Sc. in Chemistry and Physics, Faculty of Science
Ain Shams University
2010
Under Supervision of

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2018

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Approval Sheet

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2018

بِسْمِ اللّهِ الرَّحْمنِ الرَّحِيمِ

قَالُواْ سُبْحَانَكَ لاَ عِلْمَ لَنَا
إِلاَّ مَا عَلَّمْتَنَا إِنَّكَ أَنتَ
الْعَلِيمُ الْحَكِيمُ

صدق اللَّهُ العظيم

سورة البقرة – الاية (٣٢)

To the soul of my Father

To my Family

To my best teachers and friends

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At First I'd like to knee praying to ALLAH for helping me to do this work.

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Abstract

Cobalt/graphite electrodes are prepared by potentiostatic electrodeposition of cobalt from sulphate baths with and without citrate ions on graphite discs. The electrocatalytic activity of the prepared electrodes toward oxygen evolution reaction is investigated by cyclic voltammetry technique, electrochemical impedance spectroscopy and electrochemical quartz microbalance techniques. The electrocatalytic activity increases by increasing the concentration of Co^{+2} ions in the electrodeposition bath up to 0.5 M. The Co/Graphite electrode obtained from electrodeposition bath containing 0.5M Co^{2+} and 0.5M citrate ions, $Co_{(0.5\text{-Cit})}/G$ exhibits better catalytic activity toward oxygen evolution reaction than that obtained from citrate free baths. Furthermore, $Co_{(0.5\text{-Cit})}/G$ electrode gains extra OER activity when undergoes a successive cycling potentiodynamic polarization in 1M NaOH solution as a result of the formation of cobalt oxyhydroxide phase. Greater stability of $Co_{(0.5\text{-Cit})}/G$ electrode is verified by a chronopotentiometric test.

Key words

Cobalt, Citrate, Chronoamperometry, Chronopotentiometry, Cyclic Voltammetry, Electrochemical Impedance Spectroscopy, Electrochemical Quartz Crystal Microbalance, Oxygen Evolution Reaction

Abbreviations

symbol	Name
G	**bare graphite**
SD-Co$_{(x)}$/G	**electrodeposited cobalt on stationary graphite disk from bath containing x M $CoSO_4$.**
SD-Co$_{(x,Cit)}$/G	**electrodeposited cobalt on stationary graphite disk from bath containing xM $CoSO_4$ + xM sodium citrate ($Na_3C_6H_5O_7.2H_2O$).**
RD-Co$_{(x)}$/G	**electrodeposited cobalt on 1600 rpm rotated graphite disk from bath containing x M $CoSO_4$.**
RD-Co$_{(x-Cit)}$/G	**electrodeposited cobalt on 1600 rpm rotated graphite disk from bath containing xM $CoSO_4$ + xM sodium citrate ($Na_3C_6H_5O_7.2H_2O$).**
Co$_{(0.5)}$/Au/Q	**electrodeposited on Au coated quartz crystal from 0.5 M $CoSO_4$ bath cobalt**
Co$_{(0.5-Cit)}$/Au/Q	**electrodeposited cobalt on Au coated quartz crystal from 0.5 M $CoSO_4$ + 0.5M sodium citrate ($Na_3C_6H_5O_7.2H_2O$) bath.**
OER	**Oxygen Evolution Reaction**
HER	**Hydrogen Evolution Reaction**
EIS	**Electrochemical Impedance Spectroscopy**
CV	**Cyclic Voltammetry**
eQCM	**Electrochemical Quartz Crystal Microbalance**
XRD	**X-ray Powder Diffraction**
FESEM	**Field Emission Scanning Electron Microscopy**

Contents

Chapter 1
Introduction and Literature survey

Chapter 2
Experimental Work

Chapter 3
Results and Discussion

List of figures

List of tables

Chapter 1

Introduction

and

Literature survey

Introduction

1.1. Literature Survey

The global challenge for discovering sustainable and renewable energy sources as alternative to the irrecoverable fossil fuels has been grown up in the last few decades and might extend for the next two decades [1, 2]. Electrochemical science and technology can play a vital role for the indirect storing of extra renewable energy through converting the electricity into chemical energy [3]. For instance, water splitting into hydrogen and oxygen by electrolysis is considered as a hopeful substitute to electricity storage systems using batteries [4].

Recently, many intensive studies focused on constructing high efficient and low cost electrocatalysts mostly for water splitting reactions: oxygen evolution reaction (OER), and hydrogen evolution reaction (HER). Owing to, hydrogen creation from water splitting reactions is highly obstructed by the sluggish kinetics of the multistep 4-electron OER. Consequently, there is a great necessity for fabricating high efficient electrocatalysts of low overpotential to overcome this energy barrier. Basically, iridium dioxide, and ruthenium dioxide are inordinate examples for high performance electrocatalysts with low overpotential and Tafel