STUDY ON THE SUPRAMOLECULAR INTERACTION OF INCLUSION COMPLEX OF β-CYCLODEXTRIN WITH DITHIZONE AND DITHIZONE-ZINC AND ITS ANALYTICAL APPLICATION

SITI NURUR RAIHAN BINTI MOHD KAMAL

FACULTY OF SCIENCE
UNIVERSITY OF MALAYA
KUALA LUMPUR

2011

STUDY ON THE SUPRAMOLECULAR INTERACTION OF INCLUSION COMPLEX OF β-CYCLODEXTRIN WITH DITHIZONE AND DITHIZONE-ZINC AND ITS ANALYTICAL APPLICATION

SITI NURUR RAIHAN BINTI MOHD KAMAL

DISSERTATION SUBMITTED IN FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE

DEPARTMENT OF CHEMISTRY
FACULTY OF SCIENCE
UNIVERSITY OF MALAYA
KUALA LUMPUR

2011

UNIVERSITI MALAYA

ORIGINAL LITERARY WORK DECLARATION

Name of Candidate: SITI NURUR RAIHAN BINTI MOHD KAMAL
(I.C/Passport No: 860906-56-5244)

Registration/Matric No: SGR 090130

Name of Degree: MASTER OF SCIENCE (EXCEPT MATHEMATICS AND SCIENCE PHILOSOPHY)

Title of Project Paper/Research Report/Dissertation/Thesis ("this Work"):

STUDY ON THE SUPRAMOLECULAR INTERACTION OF INCLUSION COMPLEX OF β-CYCLODEXTRIN WITH DITHIZONE AND DITHIZONE-ZINC AND ITS ANALYTICAL APPLICATION

Field of Study: ANALYTICAL CHEMISTRY

I do solemnly and sincerely declare that:

(1) I am the sole author/writer of this Work;
(2) This Work is original;
(3) Any use of any work in which copyright exists was done by way of fair dealing and for permitted purposes and any excerpt or extract from, or reference to or reproduction of any copyright work has been disclosed expressly and sufficiently and the title of the Work and its authorship have been acknowledged in this Work;
(4) I do not have any actual knowledge nor do I ought reasonably to know that the making of this work constitutes an infringement of any copyright work;
(5) I hereby assign all and every rights in the copyright to this Work to the University of Malaya ("UM"), who henceforth shall be owner of the copyright in this Work and that any reproduction or use in any form or by any means whatsoever is prohibited without the written consent of UM having been first had and obtained;
(6) I am fully aware that if in the course of making this Work I have infringed any copyright whether intentionally or otherwise, I may be subject to legal action or any other action as may be determined by UM.

Candidate's Signature Date: 30 April 2012

Subscribed and solemnly declared before,

Witness's Signature Date: 30 April 2012

Name: Dr Sharifah Mohamad

Designation: Lecturer

ABSTRACT

The non-covalent interaction of beta-cyclodextrin (β-CD), dithizone (H_2Dz) and dithizone-zinc (H_2Dz-Zn) complex was studied in detail. Solid samples were studied using solid state ^{13}C Cross Polarization and Magic Angle Spinning spectroscopy (^{13}C CP/MAS NMR), Fourier Transform Infrared spectroscopy (FTIR), Thermal Gravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), Powder X-Ray Diffraction (XRD) and Energy Dispersive X-ray (EDX) to investigate the formation of the inclusion complex. Spectrophotometric technique was also used to assess the formation of inclusion complex in liquid form. The characterization results supported the formation of inclusion complex. The results showed that β-CD formed a complex with H_2Dz in a ratio of 2:1 and with H_2Dz-Zn in a ratio of 1:1. A spectrophotometry method was developed based on the enhancement of the absorbance of dithizone-zinc complex produced through complex formation in the presence of β-CD. A linear relationship between absorbance and concentration of zinc was obtained in the range of 0.1 – 9.0 mg/L with correlation coefficient of 0.996. The detection limit obtained was 0.005 mg/L and the relative standard deviation (R.S.D) was 2.05%. The developed method was applied in real samples and the recoveries obtained were from 85 – 95%.

ABSTRAK

Interaksi bukan kovalen antara kompleks beta-siklodekstrin (β-CD) dan ditizon (H_2Dz) serta ditizon-zink (H_2Dz-Zn) telah dikaji dalam dua bahagian. Sampel pepejal telah dikaji dengan menggunakan kaedah Spektroskopi Resonans Magnet Nukleus Keadaan Pepejal ^{13}C (solid state ^{13}C CP/MAS NMR), Spektroskopi Inframerah (FTIR), Analisis Termagravimetri(TGA), Kalorimetri Pengimbasan Pembezaan (DSC), Belauan sinar-X (XRD) dan Tenaga Sinar-X Serakan (EDX) untuk menyiasat pembentukan kompleks. Teknik spektrofotometri juga digunakan untuk menyiasat pembentukan kompleks dalam medium cecair. Keputusan pencirian menunjukkan bahawa telah berlaku pembentukan kompleks. Keputusan menunjukkan bahawa β-CD membentuk kompleks bersama H_2Dz di dalam nisbah 2:1 manakala bersama H_2Dz-Zn, ia membentuk kompleks di dalam nisbah 1:1. Disebabkan peningkatan serapan hasil daripada kompleks H_2Dz-Zn, maka kaedah spektrofotometri telah dibina untuk mengesan zink bersama kehadiran β-CD. Perhubungan linear di antara serapan dan kepekatan zink telah didapati di antara julat 0.1 – 9.0 mg/L dengan pekali korelasi 0.996. Had pengesanan yang ditemui adalah 0.005 mg/L dan sisihan piawai relatif ialah 2.05%. Kaedah yang dibina telah diaplikasikan kepada sampel sebenar dan kebolehdapatan yang didapati adalah di antara 85 – 95%.

ACKNOWLEDGEMENT

Alhamdulillah, with the blessing of Allah, I am able to complete my research and dissertation according to the time frame given. First of all, I would like to express my sincere gratitude to my supervisor, Dr. Sharifah Mohamad for her guidance, encouragement, supports, comments and suggestions throughout my research and upon preparing this dissertation.

I would also like to thank all my lab mates at Analytical Research Laboratory (K012), Department of Chemistry, Faculty of Science, University of Malaya for their supports and helps when I needed. Not to forget, my family members, especially both of my parents, Mohd Kamal Bin Abd. Rahman and Rasida Bt Osman who have been supporting me in my studies and also to all science officers in Department of Chemistry, Department of Physics and Combicat for their valuable help, guidance and kindness. Not to forget, to all my friends for their direct or indirect supports and encouragements throughout my studies.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF SYMBOLS AND ABBREVIATIONS

^{13}C CP/MAS NMR – 13C Cross Polarization and Magic Angle Spinning spectroscopy

Abs. - Absorbance

Conc. – Concentration

DMF – Dimethylformamide

DSC – Differential Scanning Colorimetric

EDX – Energy Dispersive X-ray

H_2Dz – Dithizone

MW – Molecular Weight

TGA – Thermal Gravimetric Analysis

UV-Vis – Ultraviolet - Visible

XRD – X-ray Diffraction

Zn – Zinc

β-CD – β-cyclodextrin

β-CD-H_2Dz – β-cyclodextrin-dithizone

β-CD-H_2Dz-Zn – β-cyclodextrin-dithizone-zinc

LIST OF APPENDICES