

**STUDY ON THE SUPRAMOLECULAR  
INTERACTION OF INCLUSION COMPLEX OF  
 $\beta$ -CYCLODEXTRIN WITH DITHIZONE AND  
DITHIZONE-ZINC AND ITS ANALYTICAL  
APPLICATION**

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**FACULTY OF SCIENCE  
UNIVERSITY OF MALAYA  
KUALA LUMPUR**

**2011**

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## ABSTRACT

The non-covalent interaction of beta-cyclodextrin ( $\beta$ -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  $\beta$ -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  $\beta$ -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 ( $\beta$ -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  $\beta$ -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  $\beta$ -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%.

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## LIST OF SYMBOLS AND ABBREVIATIONS

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

Abs. - Absorbance

Conc. – Concentration

DMF – Dimethylformamide

DSC – Differential Scanning Colorimetric

EDX – Energy Dispersive X-ray

$\text{H}_2\text{Dz}$  – Dithizone

MW – Molecular Weight

TGA – Thermal Gravimetric Analysis

UV-Vis – Ultraviolet - Visible

XRD – X-ray Diffraction

Zn – Zinc

$\beta$ -CD –  $\beta$ -cyclodextrin

$\beta$ -CD- $\text{H}_2\text{Dz}$  –  $\beta$ -cyclodextrin-dithizone

$\beta$ -CD- $\text{H}_2\text{Dz}$ -Zn –  $\beta$ -cyclodextrin-dithizone-zinc

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