UNIVERSITI TEKNOLOGI MARA

MICROWAVE ASSISTED SYNTHESIS OF SALPHEN AND SALICYLALDAZINE COMPLEXES: CHARACTERIZATION AND ANTIMICROBIAL STUDIES

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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ABSTRACT

This thesis reported on the synthesis, characterization as well as biological application of Ni(II), Pd(II), Zn(II), Cd(II) salphen and thiocarbohydrazone Schiff base metal complexes. Both type of ligands, denoted as L1 and L2 for salphen and thiocarbohydrazone were synthesized using microwave irradiation technique. The syntheses began by preparing L1 and L2 ligands in 2:1 ratio. Each type of ligand was further reacted using microwave irradiation with metal acetate salts to formed L1 and L2 metal complexes. A total of 8 ligands and 32 metal complexes were synthesized and characterized using elemental analysis, melting point, molar conductance, magnetic susceptibility, Fourier Transform Infrared (FTIR), ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR) and UV-visible spectroscopy. The FTIR, NMR and UV-visible data of L1 complexes showed peak shifting trend of -OH, C=N, and C-$O_{phenolic}$ indicated that the metal ions of the dinuclear complexes were coordinated to the ligand through azomethine nitrogen, and deprotonated phenolic oxygen in a tetradentate ONNO manner. On the other hand, spectroscopic analyses obtained for L2 series were in contrary with the proposed structure. In addition, Single crystal X-ray diffraction (SCD) was conducted on both ligand series to further elucidate the structures. Based on the crystallographic data, it was revealed that L1 series ligand formed as proposed, whereas L2 ligands formed an azine-type ligand instead of thiocarbohydrazone, in which the C=S and N-N functional groups were appeared missing. This absence of C=S and N-N functional groups were supported by the spectroscopic data obtained. As for the metal complexes of both series, the magnetic properties of Pd(II) and Ni(II) complexes of L1 and L2 is suggested to be square planar and tetrahedral geometry respectively, whereas for Zn(II) and Cd(II) of both series may exist as tetrahedral of square planar. Low conductance values of these complexes indicate non electrolyte in nature. All synthesized compounds were tested for antimicrobial properties against Gram-negative and Gram-positive bacteria. Screening of these compounds revealed that metal complexes showed higher inhibition activity compared to free ligands, in agreement with the Tweedy's chelation theory and Overtone concept of permeability. Most of the compound tested show moderate inhibition values of 7-13 mm. Among all the tested compounds, Cd(II) complexes with -H and -OMe derivatives on both ligand series were found to be potent in resisting the growth of most Gram positive bacteria tested. Nevertheless, the Cd(II) complexes showed moderate activity in comparison with standard antibiotic, gentamicin.

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