UNIVERSITI TEKNOLOGI MARA

SYNTHESIS, CHARACTERIZATION, AND CORROSION STUDIES OF THIOSEMICARBAZONE DERIVATIVES ON MILD STEEL IN 1 M HCI AND ANTIBACTERIAL STUDIES OF NICKEL(II) AND PALLADIUM(II) COMPLEXES

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MSc

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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 reports on the synthesis, characterization, corrosion investigation of thiosemicarbazone ligands as well as antibacterial screening of thiosemicarbazone ligands and their nickel(II) and palladium(II) complexes, respectively. A series of thiosemicarbazone ligands namely L1, L2, L3, and L4 were successfully prepared from the condensation reaction between benzaldehyde and its derivatives with 4-phenylthiosemicarbazide. The synthesized ligands were reacted with nickel(II) acetate tetrahydrate and palladium(II) acetate in a 2:1 ratio using a microwave-assisted synthesis method, yielding nickel(II) and palladium(II) thiosemicarbazone complexes. A total of four ligands and eight complexes were successfully isolated and characterized through elemental analysis, melting point, Fourier Transform Infrared, ^{1}H, and ^{13}C Nuclear Magnetic Resonance, and magnetic susceptibility studies. All spectral and physicochemical data supported the proposed chemical structures. The diamagnetism of all complexes suggested their square planar geometry. Coordination to metal centers occurs through two thionic-sulfur and two imine nitrogen donor atoms, assenting to the indication through FTIR and NMR spectroscopy. All ligands were employed as corrosion inhibitors in protecting mild steel against 1 M hydrochloric acid (HCl). It was revealed that all ligands were found to be effective as corrosion inhibitors with high inhibition efficiencies (>90%) as the concentration increases. The inhibition efficiency (IE%) was affected by the presence of substituents such as chloro, hydroxyl, and methyl groups. L3 compound that contains chloro substituent has the highest IE% which is 94.35%. The presence of inhibitors was proven to be successfully inhibited and adsorbed on the mild steel surface by forming a film formation that protects from a corrosive environment. The order of effectiveness of the compounds as corrosion inhibitors is L3 > L2 > L4 > L1. All tested compounds are mixed-type inhibitors, predominantly as a cathodic inhibitor and the inhibition of these compounds obeyed the Langmuir adsorption isotherm. From surface morphological studies, the mild steel surface was not severely affected or corroded in the presence of inhibitors after 24 hours of immersion in 1 M HCl and has significantly reduced the roughness, the occurrence of pits and cracks of mild steel due to protective surface film formation. Moreover, a surface analysis study also was done using X-Ray Photoelectron Spectroscopy (XPS) to provide valuable quantitative and chemical state information from the mild steel surface which being adsorbed by the inhibitors. The oxide species chemisorbed and physisorbed on the mild steel surface. The aromatic rings in the L3 structure facilitate the adsorption of chloro, multiple bonds of C=N, and C=S on the metal/solution interface. Furthermore, the ligands and complexes were also tested against different bacterial strains (two *Gram-positive* and three *Gram-negative*) where the complexes exhibit significantly higher antibacterial activity than the free ligands. The increase in bactericidal activity of the complexes is due to the effect of the metal ion on the normal cellular process. These complexes disturb the respiration process of the cell and thus block the synthesis of proteins which restricts further growth of the organism. Additionally, the significant importance among these compounds owing to their unique chelating properties due to the presence of essential N and S donor atoms. The palladium(II) complexes are more active against bacteria than the nickel(II) complexes. While there is a significant increase in complex bactericidal activity compared to free ligands, metal salts, and control (DMSO), the complexes show moderate activity compared to standard drug gentamicin.

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