UNIVERSITI TEKNOLOGI MARA

SYNTHESIS, CHARACTERIZATION, CORROSION INHIBITION PERFORMANCE AND MECHANISTIC INSIGHT OF TRIDENTATE HYDRAZONE LIGANDS ON MILD STEEL IN CO_2-SATURATED 3.5% NaCl SOLUTION

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ABSTRACT

Corrosion of mild steel in CO_2-saturated environment poses a significant problem in the petroleum industry, where conventional inhibitors often raise environmental and sustainability concerns. In response to the growing demand for affordable and eco-friendly anti-corrosion agents, this study focuses on the development of sustainable tridentate hydrazone ligands as potential corrosion inhibitors. Four ligands (AL01-AL04) derived from benzohydrazide and 2-hydroxybenzohydrazide were successfully synthesized and characterized using melting point, FTIR, NMR, elemental analysis, UV-Vis, and mass spectroscopy. Spectroscopic data confirmed the presence of characteristic *ν*(C=N) and azomethine proton signals (N=CH) in all synthesized compounds, indicate the successful formation of hydrazone ligands. Single crystal X-ray diffraction further supported the proposed structure for AL03 and AL04. Subsequently, the inhibition performance of AL01-AL04 as corrosion inhibitors on mild steel in CO_2-saturated 3.5% NaCl solution was evaluated through electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP). The ligands showed moderate to high inhibition efficiency between 59-78 (η%) with AL01 being the most effective inhibitor at 500 ppm. The effectiveness of hydrazone ligands in reducing the corrosion rate of mild steel was in the order as AL01 > AL03 > AL02 > AL04. Based on the polarization curves, the ligands have been classified as anodic, cathodic, and mixed-typed inhibitors. Furthermore, the adsorption behaviour and inhibition mechanism of tridentate hydrazone ligands were investigated by employing surface analysis techniques namely, FESEM-EDX, AFM, and XPS. These techniques have confirmed the formation of a protective layer attributed to the adsorption of the ligands on mild steel surface by the reduction in surface roughness and the presence of carbon, nitrogen, and oxygen peaks, attributed to the chemical composition of the inhibitor. In addition, adsorption isotherm modelling was applied to determine the best-fitting model in describing adsorption behaviour of inhibitors. It has been deduced that AL01 follows Langmuir isotherm, demonstrated both chemisorption and physisorption mechanism, consistent with the formation of monolayer protection. Whereas, Freundlich isotherm exhibited by AL02-AL04 indicates multilayer adsorption and dominated by physisorption. In addition to the presence of heteroatoms, pyridine, and aromatic rings that facilitates the adsorption of hydrazone ligands onto the mild steel surface, DFT calculations further corroborate the experimental findings on adsorption and inhibition capabilities of the ligands by confirming that the inhibitors with additional hydroxyl group, AL01 and AL03 had better interaction with Fe atom, hence, the higher corrosion inhibition efficiency. This work contributes to the development of less toxic organic corrosion inhibitors which aligns with sustainable development goals, SDG 9 and SDG 14, and in line with MYSTIE 10-10 objectives in Energy and Advanced Materials by fostering innovative and sustainable industrial practices and reducing the consumption of harmful inhibitors.

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CHAPTER 1
INTRODUCTION

1.1 Research Background

1.1.1 Hydrazone Schiff Base as Inhibitors

According to Alwi et al. (2023), the product from condensation reaction between a primary amine and a carbonyl compound (aldehyde or ketone) is classified as organic compounds characterized by the azomethine group (C=N), namely Schiff base. It has been widely utilized in scientific and technological applications such as pharmaceuticals, catalysts, synthetic dyes, and corrosion inhibition. The study of Schiff bases as corrosion inhibitors has gained a significant amount of attention among researchers in past years due to their cost-effectiveness, eco-friendliness, efficient inhibitory capabilities, and ease of synthesis with high yields (Kumari et al., 2014; Chaouiki et al., 2020; Abdallah et al., 2022; Altalhi, 2023).

Corrosion inhibitors are chemical compounds used to mitigate corrosion by introducing a small amount of the compounds into a solution that contains corrosive agents. Specifically, organic corrosion inhibitors function through the adsorption onto the metal surface and forming a protective layer. This layer prevents the corrosive agents from getting to the metal surface and inhibits the electrochemical reactions at the interface between the metal and the solution. The effectiveness of the organic inhibitors largely depends on the presence of heteroatoms such as nitrogen (N), oxygen (O), or sulphur (S), certain functional groups, and conjugated π-electrons. These particular features provide adsorption sites that allow the inhibitor molecules to interact with the metal surface *via* chemisorption or physisorption (Altalhi, 2023; Vaidya et al., 2023).

A prominent example of organic compounds that meet the characteristics of efficient inhibitors is hydrazone Schiff bases. Hydrazone Schiff bases can be obtained by reacting the hydrazide with carbonyl compounds like aldehydes or ketones (Singh et al., 2018; Kargar et al., 2024). Figure 1.1 represents a general structure of the hydrazide-hydrazone (R_1-C(=O)-NH-N=CH-R_2), which consists of an azomethine group (-NH-N=C-), and nitrogen (-NH) connected to a carbonyl group (C=O) (Popiołek, 2017; Han et al., 2021; Raczuk et al., 2022). The azomethine group has both