UNIVERSITI PUTRA MALAYSIA

SYNTHESIS OF SCHIFF BASE HYDRAZONE FROM PALMITIC HYDRAZIDE AND CORROSION INHIBITORY PROPERTIES VIA ELECTROCHEMICAL METHODS

NOOR KHAIRIN MOHD

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

April 2017

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Chair: Assoc. Professor Nor Azowa Ibrahim, PhD
Faculty: Science

In view of the emerging importance of non-toxic and green corrosion inhibitors to replace chromate (CrO_4^{2-}), molybdenate (MoO_3^-), nitrate (NO_2^-) and silicate (SiO_4^{4-}) in protecting metallic surfaces in acid pickling for metal processing industry, acidified oil wells and boiler cleaning process; researchers have recently focused on organic corrosion inhibitors. Schiff bases are amongst the molecules of choice that are able to exhibit corrosion inhibitory activities and have been predominantly investigated as corrosion inhibitors for numerous metals in various acidic media. The lone pair electrons and π-electrons at imine bond may interact with *d*-orbital of metal through electron donation and forms a layer. This layer protects the metal surface from being attacked by corrosive species. The objectives of this study are firstly to synthesize Schiff base hydrazones (SBHs) from palmitic hydrazide and various aldehydes; and secondly to investigate inhibitive activities of those SBHs on mild steel in acid solution *via* electrochemical method.

Eight SBHs were successfully synthesized from PH and various aldehydes containing linear alkyls, branched alkyl and aryls through one-pot synthesis. Commonly, acid catalyst is required in the synthesis of Schiff base as to increase electrophilicity of carbonyl for aldehyde and to allow dehydration to form Schiff base; and an efficient water removal system is applied to improve conversion. In this study, SBHs were prepared by heating the reactants in dimethylformamide at 90^{o}C for 2 hours without using any acid catalyst. Opportunely, SBHs with high yields (92-100%) and high purity (92-99%) were successfully obtained. The effect of solvent used, reactant mol ratio and reaction time on synthesis of SBHs were also investigated. Chemical characterization was conducted using gas chromatography, Fourier transformed-infrared (FT-IR), CHN elemental analysis, ^{1}H and ^{13}C nuclear magnetic resonance and gas chromatography-mass spectroscopy.

The inhibitory properties were examined on mild steel (MS) coupons in 1 M HCl solution with concentration varied from 25, 50, 75, 100 and 200 mg/L at temperature ranged from 25 to 55°C using electrochemical techniques: linear polarization and electrochemical impedance spectroscopy as well as surface analysis using scanning electron microscopy-energy dispersive x-ray (SEM-EDX). The presence of studied inhibitors had shown appreciable inhibition efficiency (IE%) in which the increment of IE% values exceed 50% when the concentration of inhibitor and temperature were increased from 25 to 200 mg/L and 25 to 55°C, respectively.

The adsorption of SBHs onto the MS surface obeyed the Langmuir adsorption isotherm with the standard free energy of adsorption (ΔG_{ads}) of between -31 and -43 kJ/mol. According to these values, these SBHs adsorbed spontaneously onto MS surface through both physisorption and chemisorption processes. Other thermodynamic parameters such as activation energy (E_a), entropy of adsorption (ΔS_{ads}), enthalpy of adsorption (ΔH_{ads}), enthalpy of activation (ΔH^o) and entropy of activation (ΔS^o) were also determined.

SEM-EDX analysis was conducted on MS coupons after immersing in 1 M HCl solution and inhibited 1 M HCl solution containing 200 mg/L for 3 hours and 30 days. In general, significant changes of MS surface were clearly seen after 30 days of immersion. Smooth surfaces with invisible of scratch lines were observed for MS coupons obtained from experiments containing SBHs except for SBHs with a branched alkyl (HB1) and phenyl with methoxy group (HA2). In EDX analysis, significant differences in ferum, chlorine and oxygen contents were observed for MS coupons that were immersed in blank and inhibited solutions containing PH and HB1 that probably attributed to the formation of corrosion products like $FeCl_2$ and $Fe(OH)_2$.

All SBHs except for solutions containing HB1 and HA2 showed inhibitory properties that better than PH. This could be due to the lone pair of electrons on the nitrogen at imine and heteroatoms of SBHs structures. These electrons allow them to efficiently adsorb onto metal surface thus inhibiting the surface. Branching effect and incapability of phenyl group to lay flat orientation on the MS surface for HB1 and HA2 molecules, respectively could be the main reasons for these two inhibitors did not perform well as corrosion inhibitors.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SINTESIS MENGHASILKAN SCHIFF BES HIDRAZON DARIPADA HIDRAZIDA PALMITIK DAN KAJIAN SIFAT PERENCAT PENGARATAN MENGGUNAKAN KAEDAH ELEKTROKIMIA

Oleh

NOOR KHAIRIN MOHD

April 2017

Pengerusi: Profesor Madya Nor Azowa Ibrahim, PhD
Fakulti: Sains

Kepentingan terhadap perencat kakisan tidak toksik dan hijau untuk menggantikan perencat bukan organik seperti kromat (CrO_4^{2-}), molybdenate (MoO_3^-), nitrat (NO_2^-) dan silikat (SiO_4^{4-}) untuk melindungi permukaan logam dalam proses *acid pickling* untuk industri pemprosesan logam, telaga minyak berasid serta proses pembersihan dandang telah menyebabkan para penyelidik telah beralih tumpuan kepada perencat kakisan bersumberkan organik. Schiff bes adalah antara molekul pilihan yang berupaya merencat kakisan dan telah banyak dikaji sebagai perencat kakisan untuk pelbagai logam dalam medium asid. Elektron pasangan tunggal dan elektron π pada ikatan imina boleh berinteraksi dengan orbital-*d* logam secara pemindahan elektron seterusnya membentuk lapisan yang dapat melindungi permukaan logam daripada serangan spesies menghakis. Sehubungan ini, objektif pertama kajian ini adalah untuk mensintesis hidrazon Schiff bes (SBHs) dari palmitat hidrazida (PH) dan pelbagai aldehid; dan seterusnya mengkaji aktiviti perencatan kakisan oleh SBHspada keluli lembut dalam larutan asid melalui kaedah elektrokimia.

Lapan SBHs telah berjaya disintesis daripada PH dan pelbagai aldehid mengandungi alkil rantai linear, alkil rantaian bercabang dan fenil melalui sintesis satu periuk. Kebiasaannya, pemangkin asid diperlukan dalam sintesis Schiff bes bagi meningkatkan sifat keelektrofilan karbonil bagi aldehid dan untuk membenarkan dehidrasi bagi membentuk Schiff bes; dan sistem pembuangan air yang cekap digunakan untuk meningkatkan hasilan tindak balas. Walaubagaimanapun, dalam kajian ini SBHs telah disediakan secara memanaskan bahan tindak balas dalam pelarut dimethylformamide pada 90^{o}C selama 2 jam tanpa menggunakan sebarang pemangkin asid dan SBHs dengan hasil yang tinggi (92-100%) dan ketulinan yang tinggi (92-99%) telah berjaya diperolehi. Kesan parameter-paramer tindak balas seperti jenis pelarut yang digunakan, nisbah mol bahan tindak balas dan masa tindak balas terhadap penghasilan SBHs juga telah disiasat. Pencirian kimia dijalankan dengan menggunakan kromatografi gas, fourier berubah-inframerah (FT-IR), CHN analisis unsur, ^{1}H dan ^{13}C resonans magnetik nuklear dan gas kromatografi jisim spektroskopi.

Sifat-sifat perencatan SBHs telah dikaji pada kupon keluli lembut (MS) dalam larutan 1 M HCl dengan kepekatan bahan perencat sebanyak 25, 50, 75, 100 dan 200 mg/L pada suhu antara 25 hingga 55°C menggunakan teknik elektrokimia: polarisasi linear dan spektroskopi impidan elektrokimia serta analisis permukaan menggunakan imbasan mikroskop elektron-penyerakan tenaga sinar-x (SEM-EDX). Kehadiran perencat telah meningkatkan kecekapan perencatan (IE%) di mana kenaikan nilai IE% melebihi 50% apabila kepekatan perencat dan suhu ditingkatkan masing-masing dari 25 ke 200 mg/L dan 25 ke 55°C.

Penjerapan SBHs ke permukaan MS mematuhi penjerapan isoterma Langmuir dengan tenaga bebas penjerapan (ΔG_{ads}) antara -31 dan -43 kJ/mol. Berdasarkan nilai-nilai ΔG_{ads} ini, SBHs ini terjerap secara spontan ke permukaan kupon MS secara serapan fizikal dan kimia. Parameter-parameter termodinamik lain seperti tenaga pengaktifan (E_a), entropi penjerapan (ΔS_{ads}), entalpi penjerapan (ΔH_{ads}), entalpi pengaktifan (ΔH_o) dan entropi pengaktifan (ΔS_o) juga diukur.

Analisis SEM-EDX telah dijalankan ke atas kupon MS yang telah direndam dalam larutan 1 M HCl dan larutan 1 M HCl berperencat yang berkepekatan sebanyak 200 mg/L selama 3 jam dan 30 hari. Secara umumnya, perubahan ketara permukaan MS telah jelas dilihat selepas 30 hari rendaman. Permukaan yang licin tanpa garisan calar diperhatikan untuk kupon MS yang direndam dalam larutan asid yang mengandungi SBHs kecuali SBHs yang mempunyai rantaian alkil bercabang (HB1) dan fenil dengan kumpulan metoksi (HA2). Dalam analisis EDX, perbezaan kandungan ferum, klorin dan oksigen yang ketara diperhatikan untuk kupon MS yang telah direndam di dalam larutan kawalan dan larutan yang mengandungi PH dan HB1 dan ini boleh dikaitkan dengan pembentukan produk kakisan seperti $FeCl_2$ dan $Fe(OH)_2$.

Kesemua SBHs kecuali HB1 dan HA2 menunjukkan sifat perencatan yang lebih baik daripada PH. Ini boleh dikaitkan dengan kewujudan pasangan elektron tunggal pada atom nitrogen imina yang membolehkan molekul SBHs ini menjerap dengan cekap ke permukaan logam itu serta melindungi permukaan. Kesan struktur alkil bercabang dan ketidakupayaan kumpulan fenil untuk berorientasi sesatah dengan permukaan kupon MS untuk HB1 dan molekul HA2, masing-masing boleh menjadi sebab utama bagi kedua-dua perencat ini tidak menunjukkan prestasi yang baik sebagai perencat kakisan.

ACKNOWLEDGEMENTS

All praises to **ALLAH**. Thanks to almighty **ALLAH** for giving me strength, good health and ability to complete this thesis. Alhamdulillah…

I wish to express my immeasurable appreciation and deepest gratitude to these persons who have contributed in making this study possible. Firstly, I owe an enormous debt of gratitude to my supervisors: **Assoc. Prof. Dr. Nor Azowa Ibrahim, Dr. Siti Mariam Mohd Nor, Prof. Dato' Dr. Wan Md. Zin Wan Yunus, Assoc. Prof. Dr. Mariyam Jameelah** and **Dr. Yeong Shoot Kian**, who have encouraged, guided, gave supports and advised throughout my study. I truly appreciate their trust, guidance and understanding. Without their guidance, this thesis would not have been possible. Words can be never enough to thank their kindness.

I would like to express my special gratitude and thanks towards my family for the encouragement in completion of this study. My beloved and supporting husband, **Shmsualkamar Abdullah** who is always patience, love and care whenever I am in a terrible need of help and support; and his help really reduces my physical and mental burdens; and also to my kids: **Alya Qistina, Faiq Nuqman** and **Amnah Qaleesya**.

My thanks and appreciations also go to my beloved mum, **Siti Rohaya Mohd Dali** and all my siblings: **Mohammad Khairi, Khaizul Nizam, Mohammad Mokhriq, Nurul Sahidah, Siti Maisara** and **Nur Fatiah**. To my late father **Allahyarham Mohd Bin Johari**, many thanks for all your support and encouragement. Al-Fatihah!

I wish to thank to Malaysian Palm Oil Board (MPOB) for providing financial support for this study. Last but not least, I would like to thank all staff of Advanced Oleochemical Technology Division (AOTD) and Faculty of Science, UPM who has assisted me in conducting experiments and analyses. Thank you so much.

I certify that a Thesis Examination Committee has met on 20 April 2017 to conduct the final examination of Noor Khairin Mohd on her thesis entitled “Synthesis of Schiff Base Hydrazones from Palmitic Hydrazide and Their Corrosion Inhibitive Properties Via Electrochemical Methods” in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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Mohamed Ibrahim Mohamed Tahir, PhD
Lecturer
Faculty of Science
Universiti Putra Malaysia
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Faculty of Science
Universiti Putra Malaysia
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Chemistry
Institut Teknologi Sepuluh Nopember
Indonesia
(External Examiner)

NOR AINI AB. SHUKOR, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 8 August 2017

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

Nor Azowa Ibrahim, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Siti Mariam Mohd Nor, PhD
Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

Prof. Dato' Wan Md Zin Wan Yunus, PhD
Professor
Faculty of Science and Technology
National Defence University of Malaysia
(Member)

Mariyam Jameelah Ghazali, PhD
Associate Professor
Faculty of Engineering and Built Environment
Universiti Kebangsaan Malaysia
(Member)

Yeong Shoot Kian, PhD
Senior Researcher,
Advanced Oleochemicals Technology Division
Malaysian Palm Oil Board
(Member)

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This is to confirm that:

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Signature: ______________________________
Name of Member of Supervisory Committee: Mariyam Jameelah Ghazali

Signature: ______________________________
Name of Member of Supervisory Committee: Yeong Shoot Kian

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BSTFA	*N,O*-Bis-(trimethylsilyl)-trifloroacetamide
C_{dl}	Double layer capacitant
CE	Counter electrode
CI	Corrosion inhibitor
CPE	Constant phase element
CR	Corrosion rate
E_{HUMO}	The highest unoccupied molecular orbital
E_{LUMO}	The lowest unoccupied molecular orbital
EDL	Electrical double layer
EDX	Energy dispersive x-ray
EI-MS	Electron impact mass spectroscopy
EIS	Electrochemical impedance spectroscopy
FAMEs	Fatty acid methyl esters
FID	Flame ionization detector
FTIR	Fourier Transformed Infrared Spectroscopy
GC	Gas chromatography
IPA	Isopropyl alcohol
LP	Linear polarization
MP	Melting point
MS	Mild steel
NMR	Nuclear magnetic resonance
OCP	Open circuit potential
PH	Palmitic hydrazide
R_{ct}	Charge transfer resistance
R_{s}	Solution resistance

RE	Reference electrode
SEM	Scanning electron microscopy
SBHs	Schiff base hydrazones
SAMs	Self-assembled monolayers
TLC	Thin layer chromatography
WE	Working electrode
XRD	X-ray diffractometry
d	Thickness
ε	Local dielectric constant
ε_o	Permittivity of free space
η	Overpotential
β_a	Anodic current
β_c	Cathodic current
ΔG	Free energy
$\Delta G_a^{\ddagger}$	Free energy of activation
ΔG^o_{ads}	Standard free energy of adsorption
ΔH^o_{ads}	Enthalpy of adsorption
ΔH_a	Enthalpy of activation
ΔS^o_{ads}	Entropy of adsorption
ΔS_a	Entropy of activation
θ	Surface coverage
ω	Angular frequency
c	Concentration
E_{corr}	Corrosion potential
F	Faraday constant (96485 $Cmol^{-1}$)
f	frequency
h	Planck’s constant (6.626×10^{-34} Js)

I	Current
I_{corr}	Corrosion current
i_a	Anodic reaction
i_c	Cathodic reaction
J	Reflux of substance
K_{ads}	Equilibrium constant of adsorption
Q	Total electric charge
M	Molar mass
N	Avogadro number (6.022 x 10^{23} mol^{-1})
R	Universal gas constant (8.314 J $mol^{-1}K^{-1}$)
T	Absolute temperature
S	Surface area
Z	Impedance
z	Number of electron

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Steel pickling is part of finishing processes in the manufacture of steel products. Pickling is a metal surface treatment used to remove impurities such as stains, inorganic contaminants, rust or scale from ferrous metals, copper and aluminum alloys. These impurities are removed by dissolution in acids like hydrochloric acid (HCl), sulfuric acid (H_2SO_4), hydrofluoric acid (HF) and nitric acid (HNO_3). In this process, iron oxides dissolve in acid (in case of HCl) form ferrous salt and water as shown in Eq. 1.1, Eq. 1.2 and Eq. 1.3.

$$Fe_2O_3\,(s) + Fe\,(s) + 6HCl\,(aq) \longrightarrow 3FeCl_2\,(aq) + H_2O\,(aq) \qquad \textbf{Equation 1.1}$$

$$Fe_3O_4\,(s) + Fe\,(s) + 8HCl\,(aq) \longrightarrow 4FeCl_2\,(aq) + H_2O\,(aq) \qquad \textbf{Equation 1.2}$$

$$FeO\,(s) + 2HCl\,(aq) \longrightarrow FeCl_2\,(aq) + H_2O\,(aq) \qquad \textbf{Equation 1.3}$$

However, HCl acid also may react with base steel (Fe) by this mechanism (Eq. 1.4)

$$Fe\,(s) + 2HCl\,(aq) \longrightarrow FeCl_2\,(aq) + H_2\,(g) \qquad \textbf{Equation 1.4}$$

In this process, hydrogen gas is released from the metal surface due to the metal-acid interaction. The exposure of hydrogen to the metal could bring a problematic issue known as hydrogen embrittlement. The diffusing of hydrogen through the metal makes it brittle. In the case of inhibitor usage, the inhibitor forms a barrier on the metal surface and the hydrogen gas that obtained due to acid -metal interaction is strongly inhibited (Uhlig, 1971). Therefore, hydrogen embrittlement risk on metal parts may be reduced with addition inhibitor into pickling baths. Inhibitor also inhibit or reduce acid attack on the steel itself while allowing preferential attack on the iron oxides (Al-Sabagh *et.al*, 2011). Similar to a process for scale removal treatment that conducted for steam boilers in which HCl acid solutions is applied. These processes require the use of efficient corrosion inhibitors to protect the metal surface from corrosive species. Chromate (CrO_4^{2-}), molybdenate (MoO_3^{-}), nitrate (NO_2^{-}), silicate (SiO_4^{4-}) and phosphate (PO_4^{3-}) are amongst efficient inhibitors that have been extensively used for several decades (Rudnick, 2009). These compounds inhibit corrosion on metal surfaces by providing passivation protection to metallic surfaces (Koeppen *et al*., 1972). Unfortunately, they are carcinogenic to human and harmful to the environment as such their use was prohibited in some countries. The health, safety and environmental issues related to these inorganic inhibitors have encouraged researchers to develop organic corrosion inhibitors from renewable resources that are able to exhibit excellent inhibition with low toxicity.

In recent years, several types of organic compounds were investigated on several metals in acidic solutions. Organic inhibitors are categorized under film-forming types due to their ability to bond with a metallic surface. Many types of organic inhibitors were investigated including alkaloids, amino acids, organic dyes, tannins, aldehydes and honey. Most of these organic inhibitors were chemically synthesized and very few of them were extracted from plants. These organic inhibitors have shown excellent inhibition properties on various metals in acidic media. Compounds containing lone pair electrons (heteroatoms) and π-electrons were found to be very efficient in inhibiting metals from corrosion in corrosive environments. These high electron density moieties function as active centers for inhibiting activity. They facilitate electron transfer between the molecules (inhibitors) and the metal surface. The adsorption of organic inhibitors onto metal with solution interface might also occur through several interactions (Danaee *et al.,* 2013) as follows:

a) Electrostatic attraction between the charged molecules and the charged metal
b) Interaction of unshared electron pair in the molecule with the metal
c) Interaction of π-electrons with the metal
d) Combination of all above.

Theoretically, the adsorption of organic inhibitors onto metal surfaces starts with displacement of water molecules on the corroding surface (Eq. 1.5) followed by a formation of a barrier (El-Maksoud, 2008). The barrier subsequently slows down the anodic reaction, cathodic reaction or both reactions that leads to a decrease in corrosion rate.

$$[\text{Inhibitor}]_{sol} + [nH_2O]_{ads} \longrightarrow [\text{Inhibitor}]_{ads} + [nH_2O]_{sol}$$ **Equation 1.5**

where *n* is the number of water molecule to be displaced by an inhibitor molecule. The value of *n* is not influenced by coverage and charge of an electrode but, it depends on the cross-section area of organic molecules (El-Maksoud, 2008). Meanwhile, the size and orientation of an inhibitor molecule might affect the number of water molecules to be displaced (Papavisanam, 2000). Many techniques were utilized to measure the performance of organic inhibitors as corrosion inhibitors on different metals includes weight loss analysis, electrochemical analyses, quantum chemical analysis and Mylius thermometric technique and surface analysis.

In recent times, some efforts to develop nitrogenous compounds containing long alkyl chains were described in the literatures. Beside the adsorption of the active center of an inhibitor molecule, it was also postulated that the alkyl chains may arrange themselves to form a hydrophobic layer thus providing additional inhibition. The hydrophobic layer protects the metal surface from being attacked by corrosive species (Chen *et al*., 2000; Miksic *et al*., 2009; Negm *et al*., 2011). An illustrative diagram of an organic inhibitor containing long alkyl chain is shown in Figure 1.1.

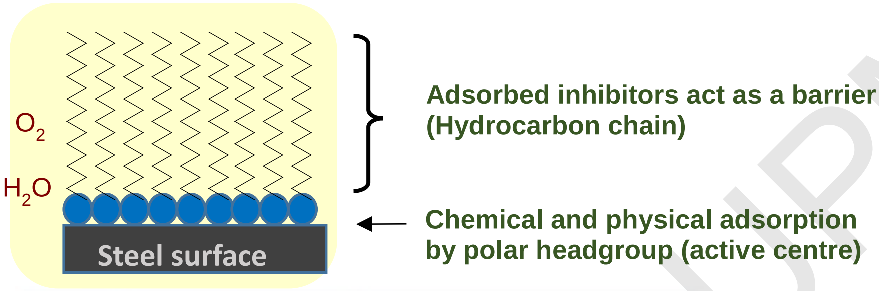
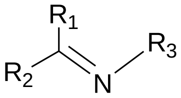


Figure 1.1: Illustration on adsorption of organic inhibitor bearing long alkyl chain on a metal surface

Amongst several types of organic inhibitors, Schiff bases are interesting molecules for corrosion inhibitors. They have been recently reported as effective corrosion inhibitors toward several metallic surfaces. The presence of heteroatoms and π-electrons combined in one molecule enables them to have several interactions with metals in acidic media. According to research works done by Liu *et al*. (2014), the excellent inhibition of Schiff bases was due to formation of self-assembled monolayers (SAMs) that resulted from their adsorption on the metallic surfaces.

A Schiff base or also synonymous with an imine or azomethine (Figure 1.2) was discovered by Hugo Schiff, a German chemist in 1864 (Brodowska and Lodyga-Chruscinka, 2014). Hugo Schiff introduced a condensation reaction between an amine with a carbonyl compound that conducted under an azeotropic distillation with simultaneous removal of water. The removal of water was facilitated using molecular sieves or a Dean-Stark apparatus (Taguchi and Westheimer, 1971; Moffett and Rabjohn, 1963; Kuehne, 1959).

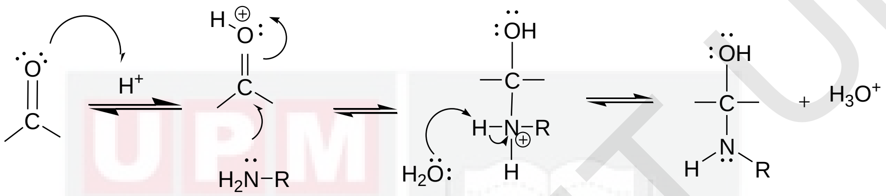


R_1 and R_2 = alkyl, aryl or H
R_3 = alkyl or aryl

Figure 1.2: General structure of Schiff base

Since then, many efforts have been devoted by researchers in improving the yields. Some researchers introduced an *in-situ* dehydration method using dehydrating solvents such as tetramethyl orthosilicate or trimethyl orthoformate (Love and Ren, 1993; Look *et al*., 1995). In different studies, Chakraborti and his colleagues (2004) recommended that the efficiency of the reaction greatly dependent on the use of highly electrophilic carbonyl compounds and strongly nucleophilic amines. Bronsted-Lowry or Lewis acids that used to activate the carbonyl group of aldehydes, catalyze the nucleophilic attack of amine and dehydrate the system or eliminate water was also introduced. Basically, this condensation reaction starts with a formation of an unstable addition product so called hemiaminal followed by losing water to give an imine or a Schiff base. Acid catalyst is required to increase the electrophilicity of the carbonyl and to protonate the OH group to allow dehydration process as shown in Figure 1.3.

a) Acid-catalyzed addition of the amine to the carbonyl group



b) Acid-catalyzed dehydration

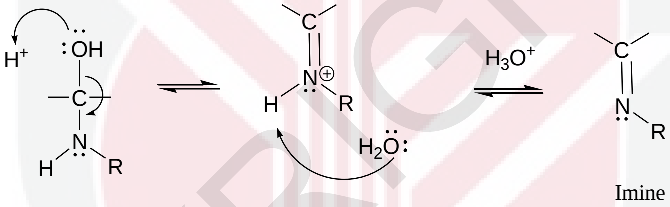
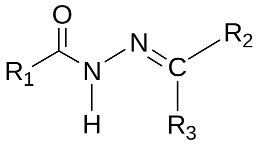


Figure 1.3 Acid-catalyzed condensation

However, this reaction is reversible and fairly unstable, unless the imine group consists an extended system of conjugation (Xavier and Srividhya, 2014; Liu *et al*., 2014). In addition, dehydration process is the rate determining step and the process could also occur in both acidic and basic conditions (Mishra *et al*., 2013). Meanwhile, Schiff base hydrazones (SBHs) are characterized by an imine and an amide groups as shown in Figure 1.4. SBHs are produced from a condensation reaction of a hydrazide with an aldehyde or a ketone.



R_1, R_2 and/or R_3 is H, alkyl and/or aryl

Figure 1.4: General structure of SBHs

In the present investigation, SBHs were prepared from palmitic hydrazide and various aldehydes bearing linear alkyl chains, branched alkyl, aryls, saturation and unsaturation bond. Since the carbonyl compound is aldehyde as such the R2 is H atom. The effect of linear alkyl chain and different types of aryl groups of R3 on yield of product was investigated. A large number of works on the preparation of SBHs from various hydrazides and carbonyl compounds were reported in the literatures. In most cases, reactions were carried out *via* reflux using various solvents: ethanol, acetone, methanol and water in the presence of a catalyst or an oxidizing agent such as ceric ammonium nitrate (Dabiri *et al.*, 2009; Al-Qalaf *et al*., 2008), glacial acetic acid (Liu *et al*., 2014) and Lewis acid (Jha *et al*., 2013). Conversely, Chamarthi *et al.* (2012) introduced a successful reaction system that conducted without the use of catalyst.

The presence of lone pair electrons at heteroatoms and π-electrons in the structures is expected to provide high electron density active center and to make strong adsorption with metal surface. Additionally, the development and utilization of these palmitic acid derivatives could add value to palm oil as palmitic acid is one of major components of palm oil (Sumathi *et al*., 2008).

1.2 Problem Statement

Inorganic inhibitors are very efficient in inhibiting metallic surface from corrosion. These inorganic inhibitors are widely utilized in acid pickling and scale removal treatment for steam boilers. However, some of them are carcinogenic and their use gave negative impact on the environment. Their usage was also banned in some countries. This has encouraged scientist to develop inhibitors from organic sources that are able to exhibit excellent inhibitory properties with low toxicity.

1.3 Research Aim and Scope

The aim of this study is to produce palm-based derivatives namely SBHs to be utilized as corrosion inhibitors. PH was prepared from methyl palmitate and hydrazine monohydrate through hydrazinolysis process. Palmitic acid is major component of palm oil and developing of this fatty acid for application of corrosion inhibitors may give value addition to oleochemicals especially palm oil. PH was further reacted with several aldehydes containing linear alkyls, branched alkyl, aryl, saturation and unsaturation bonds. In designing corrosion inhibitors, SBHs were expected to exhibit some inhibitory activities on metal surface due to the presence of imine bond. Unshared electrons and π-electrons at imine may adsorb on the metal surface, form protective layer thus inhibiting the surface. Aldehyde with linear alkyls, branched alkyl, aryl, saturation and unsaturation bonds were selected as these functionalities might have different effects on inhibitory properties. SBHs with phenyl and unsaturation were expected to have excellent inhibitory activities due to increase number active centers. However, the effect of linear alkyl and branched alkyls are also interested to be investigated. Their presence could interrupt the adsorption of active center on metal surface.

In the synthesis of SBHs, the aldehydes used might also have different reactivity due to steric hindrance and resonance effect. The resonance effect could change the electrophilicity of carbonyl. All synthesized compounds were characterized using several chromatographic and spectroscopic methods. The corrosion inhibitory properties were tested on mild steel (MS) coupon in 1 M HCl solution using electrochemical analysis employing linear polarization (LP) and electrochemical impedance spectroscopy (EIS) techniques. Inhibitory activities of SBHs are expected to occur at imine that acts as active center for adsorption. Here, the effect of linear alkyl, branched alkyl, phenyl, saturation and unsaturation groups on the adsorption of these SBHs on metal surface were investigated.

Subsequently, this study also aims to understand the cumulative effects of inhibition properties, thermodynamic parameters and adsorption isotherm of the synthesized PH and SBHs. The inhibition properties of the synthesized PH and SBHs was also determined by capturing the morphology of MS coupons that were exposed to inhibited corrosive solution and blank corrosive solutions using scanning electron microscopy (SEM) and analyzing the surface composition through energy dispersive x-ray (EDX). Adsorption mechanism of these inhibitors on MS coupons was determined by fitting the surface coverage (θ) with several isotherm models. Free energy of adsorption (ΔG_{ads}), equilibrium constant of adsorption (K_{ads}), entalphy of adsorption (ΔH_{ads}) and entropy of adsorption (ΔS_a) will be also determined. Thermodynamic activation parameters such as activation energy (E_a), entalphy of activation (ΔH_a), entrophy of activation (ΔS_a) and adsorption isotherm of the PH and SBHs was obtained by conducting experiments at different temperatures.

This research embarks on the following objectives

a) To synthesize Schiff base hydrazones (SBHs) from palmitic hydrazide and various aldehydes.
b) To determine the effect of reaction time, solvent and reactant mol ratio for synthesis of SBHs.
c) To identify the corrosion inhibitory properties of PH and SBHs on mild steel in acidic solution through electrochemical analysis including linear polarization and electrochemical impedance spectroscopy.
d) To determine thermodynamic activation parameters, adsorption isotherm, and adsorption mechanism of PH and SBHs on mild steel in the acidic solution.
e) To characterize the surface morphology of mild steel after immersed in the test solution containing SBHs.

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