UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF OIL PALM EMPTY FRUIT BUNCH TABLET AS CORROSION INHIBITOR FOR MILD STEEL ACID CORROSION

NUR IZZAH NABILAH BINTI HARIS

ITMA 2021 5

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By

NUR IZZAH NABILAH BINTI HARIS

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

July 2021

DEDICATION

To my beloved parents, Haris and Marjani.

To my sisters, Safura, Maisarah, and Batrisyia.

To my family and friends.

May this thesis be beneficial to anyone reading it.

“And We also sent down iron in which there lies great force and which has many uses for mankind…” (Al-Hadid:25)

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

DEVELOPMENT OF OIL PALM EMPTY FRUIT BUNCH TABLET AS CORROSION INHIBITOR FOR MILD STEEL ACID CORROSION

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Chairman : Shafreeza binti Sobri, PhD
Institute : Advanced Technology

The use of inhibitors is a prevailing corrosion control practice. However, the toxicological and environmental issues of conventional inhibitors have eloquently proliferated interest in developing safe and environmentally benign inhibitors. As considerable experimental evidence of nature-based inhibitors have been reported, their effectiveness are indisputable. Nonetheless, a breakthrough in this field is needed to go beyond the exploratory stage, seeking ways to implement the developed inhibitors in the industry. Considering the subject matter, this study aimed to develop an oil palm empty fruit bunch (OPEFB) tablet as a corrosion inhibitor for mild steel in 1 M hydrochloric acid (HCl). Two OPEFB treatments and one without treatment, that yield untreated, sodium hydroxide (NaOH) treated, and HCl treated OPEFB were used for inhibitor preparation. All inhibitors have been characterised and the results revealed that each inhibitor constitutes varying proportions of hemicellulose, cellulose, lignin, and extractives. The inhibition efficiencies of the inhibitors have been investigated using weight loss method at varying dosages from 0.1 to 0.5 g. The finding has shown that HCl treated OPEFB exhibited the highest inhibition efficiency (85.66%), followed by untreated (82.11%) and NaOH treated OPEFB (66.24%). All OPEFB inhibitors' adsorptions on mild steed obeyed Langmuir isotherm and are categorised as mixed-type adsorption. HCl treated OPEFB was selected to be further characterised and investigated. Its chemical composition was explored and the results unveiled that the inhibitor comprises sugar, fatty acid, amino acid, carotenoid, and phytosterol compounds. The dosage and immersion time were then optimised using response surface methodology (RSM) to obtain maximum inhibition efficiency. An optimum point at 0.33 g and 120 h that yields 87.11% of efficiency was predicted and validated. Next, the inhibition kinetics was studied using electrochemical methods. The results uncovered that HCl treated OPEFB was a mixed type inhibitor that inhibits both anodic and cathodic reactions simultaneously. Afterwards, 15 HCl treated OPEFB compounds were analysed computationally. The results revealed that the sugar compounds exhibited good characteristic, while the carotenoid compound showed a superior characteristic for corrosion inhibition. All compounds were adsorbed in a flat position on the metal surface. Finally, an OPEFB tablet was formulated to extend the effective inhibition time using D-optimal mixture method. Three formulations with excellent inhibition performances were selected for tensile strength, disintegration time, and dissolution profile evaluations. The findings unveiled that T3, with OPEFB to GA to HPMC ratio of 66:0:34, portrayed the best tablet properties. The inhibition performance of T3 at extended immersion times from 120 h to 720 h was then investigated. The finding unveiled that T3 showed persistent inhibition effect up to 360 h. Overall, this study's findings have shown that the OPEFB inhibitor tablet can inhibit mild steel acid corrosion effectively, and the tableting approach provides a promising way to implement nature-based inhibitors in the industry.

Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PEMBANGUNAN TABLET TANDAN KOSONG BUAH KELAPA SAWIT SEBAGAI PERENCAT KARAT UNTUK KAKISAN ASID KELULI LEMBUT

Oleh

NUR IZZAH NABILAH BINTI HARIS

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Pengerusi : Shafreeza binti Sobri, PhD
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Penggunaan bahan perencat adalah kaedah yang lazim digunakan untuk mengawal kakisan. Namun, masalah toksikologi dan alam sekitar perencat konvensional telah menyebabkan perhatian tertumpu kepada perencat alternatif yang lebih selamat dan mesra alam. Berdasarkan bukti saintifik, potensi dan keberkesanan perencat berasaskan bahan semula jadi tidak dapat dipertikaikan lagi. Walaubagaimanapun, kemajuan baru dalam bidang ini masih diperlukan bagi mencari cara untuk mengiplementasikan perencat ini di industri. Oleh itu, kajian ini dijalankan bertujuan untuk menghasilkan tablet berasaskan tandan kosong buah kelapa sawit (OPEFB) sebagai perencat kakisan bagi keluli lembut di dalam larutan 1 M asid hidroklorik (HCl). Dua kaedah rawatan OPEFB dan satu tanpa rawatan yang menghasilkan OPEFB yang tidak dirawat, OPEFB yang dirawat dengan natrium hidroksida (NaOH) dan HCl digunakan untuk penyediaan perencat. Semua perencat yang dihasilkan telah diperincikan dan keputusan menunjukkan bahawa setiap perencat OPEFB terdiri daripada sebatian hemiselulosa, selulosa, lignin dan ekstraktif dalam kadar berbeza. Kecekapan perencat OPEFB telah diuji menggunakan kaedah pengurangan berat dan kajian penjerapan isoterm pada dos yang berbeza dari 0.1 hingga 0.5 g. Hasil kajian menunjukkan bahawa OPEFB yang dirawat dengan HCl menghasilkan kecekapan perencatan tertinggi (85.66%), diikuti oleh OPEFB yang tidak dirawat (82.11%) dan OPEFB yang dirawat dengan NaOH (66.24%). Semua proses penjerapan perencat OPEFB pada keluli lembut mematuhi isoterm Langmuir dan dikategorikan sebagai penjerapan jenis campuran. OPEFB yang dirawat dengan HCl telah dipilih untuk diperincikan dan disiasat dengan lebih lanjut. Komposisi kimianya telah diperincikan dan keputusan menunjukkan bahawa perencat tersebut terdiri daripada sebatian gula, asid lemak, asid amino, karotenoid dan fitosterol. Kadar dos dan masa rendaman kemudian dioptimumkan menggunakan metodologi permukaan tindak balas (RSM) bagi mendapatkan kecekapan yang maksimum. Titik optimum pada 0.33 g dan 120 jam yang menghasilkan kecekapan sebanyak 87.11% telah diramalkan dan dibuktikan. Seterusnya, kinetik perencatan dikaji menggunakan kaedah elektrokimia. Hasil kajian menunjukkan bahawa perencat OPEFB merencat kakisan dengan menghalang kedua-dua tindak balas anodik dan katodik. Setelah itu, 15 sebatian perencat OPEFB dianalisis melalui analisis komputer. Hasil kajian menunjukkan bahawa sebatian gula menunjukkan sifat yang baik, manakala sebatian karotenoid menunjukkan ciri yang terbaik untuk merencat pengaratan. Semua sebatian diserap dalam kedudukan mendatar di atas permukaan logam. Seterusnya, perencat tablet OPEFB telah dihasilkan untuk menambah jangka masa perencatan menggunakan model campuran D-optimum. Tiga formulasi yang mempunyai kecekapan terbaik telah dipilih untuk analisis terperinci dari segi kekuatan, masa pemisahan, dan profil penguraian tablet. Hasil kajian menunjukkan bahawa T3, dengan nisbah OPEFB kepada GA kepada HPMC sebanyak 66:0:34, mempunyai prestasi terbaik. Kecekapan T3 pada durasi rendaman antara 120 hingga 720 jam juga disiasat. Hasil kajian menunjukkan bahawa T3 menunjukkan kesan perencatan konsisten hingga 360 jam. Secara keseluruhannya, penemuan kajian ini menunjukkan bahawa perencat tablet OPEFB dapat menghalang pengaratan bagi keluli lembut di dalam larutan berasid dengan berkesan, dan penggunaan perencat tablet OPEFB berpotensi untuk diaplikasikan di industri.

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Highest gratitude to Allah for bestowing me with knowledge, strength, and perseverance to complete this thesis. All accomplishment would have been impossible without His will and guidance.

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My profound indebtedness also goes to the examiners of my Master conversion to PhD defence, Prof. Dr. Robiah Yunus, Prof. Dr. Luqman Chuah Abdullah, and Assoc. Prof. Dr. Abdul Halim Abdullah. Their insightful comments and recommendations have brought this study to the next level. Not to mention, my deepest appreciation to the Thesis Examination Committees, Assoc. Prof. Salmiaton Ali, Assoc. Prof. Yusran Sulaiman, Dr. Faizah Mohd Yasin, and Prof. Song Guangling. Their meticulous evaluations and supportive criticisms have undoubtedly improved and increased the impact of my thesis.

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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:

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Date: 9 September 2021

Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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Signature: ______________________
Name of Member of Supervisory Committee: Nur Kartinee Binti Kassim

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LIST OF ABBREVIATIONS

AIL	Acid insoluble lignin
ANOVA	Analysis of variance
ASL	Acid soluble lignin
B3LYP	Becke’s three-parameter with Lee Yang Parr
CCD	Central composite design
CH_3COOH	Acetic acid
CO_2	Carbon dioxide
DFT	Density functional theory
DPPH	2,2-diphenyl-1-picrylhydrazyl
EDX	Energy dispersive X-Ray
EIS	Electrochemical impedance spectroscopy
EN	Electrochemical noise
FTIR	Fourier transform infrared
GA	Gum Arabic
GCMS	Gas chromatography-mass spectrometry
H_2S	Hydrogen sulphide
H_2SO_4	Sulphuric acid
H_3PO_4	Phosphoric acid,
HCl	Hydrochloric acid
HPLC	High-performance liquid chromatography
HPMC	Hydroxypropyl methylcellulose
IR	Infrared
LCMS	Liquid chromatography-mass spectroscopy
LPR	Linear polarisation resistance

MC	Monte Carlo
MD	Molecular dynamics
NaCl	Sodium chloride
NaOH	Sodium hydroxide
NMR	Nuclear magnetic resonance
NO_2	Nitrogen dioxide
NREL	National renewable energy laboratory
OFAT	One factor at a time
OPEFB	Oil palm empty fruit bunch
OPF	Oil palm fronds
PDP	Potentiodynamic polarisation
PKC	Palm kernel cake
RDF	Radial distribution function
RSM	Response surface methodology
SEM	Scanning electron microscopy
TAPPI	Technical association of pulp and paper industry
TG	Thermogravimetric
WL	Weight loss

CHAPTER 1

INTRODUCTION

1.1 Background of study

Metals and alloys are used for a myriad of purposes in a number of industrial sectors. Ferrous metal, also known as steel, accounts for 80% of all metallic materials worldwide (Mridha, 2016). Despite experiencing a minor demand slowdown due to COVID-19, steel production is still growing as it has been for the last 70 years, as evident in Figure 1.1 (Basson, 2020). In fact, it is estimated that the steel production will increase by 16.37% from 1869 million tonnes in 2019 to 2175 million tonnes in 2024 (Basson, 2020; *Global Steel Industry (China, India, EU, NAFTA & Japan): Insights & Forecast with Potential Impact of COVID-19 (2020-2024)*, 2020).

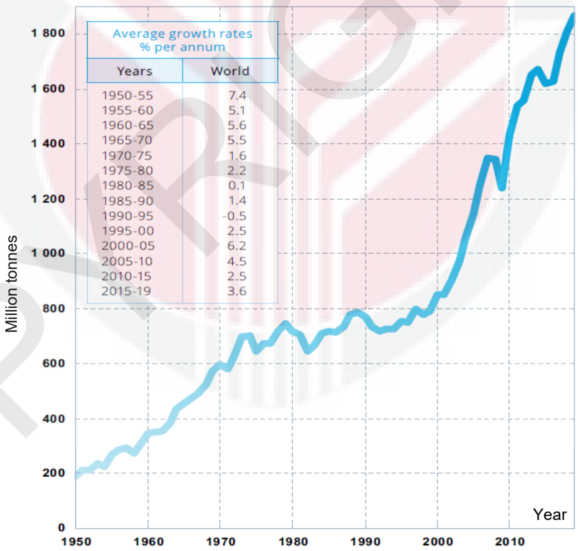

Figure 1.1: Crude steel production from 1950 to 2019 (Basson, 2020)

Steel is extensively used in the industries for building, mechanical equipment, and automotive due to its recyclability, strength, and reasonable price. The weightage of the global use of steel is shown in Figure 1.2. In spite of the extensive use, steel has a very concerning downside, which is its poor corrosion resistance. It is an ineluctable property as steel originally exists as iron ores in the form of oxides. Upon direct contact of metals to corrosive environments such as strong acidic, alkaline or saline solution, the iron would revert to its original state. The corrosion effect is extremely severe in the acidic medium, particularly in sulphuric acid (H_2SO_4), hydrochloric acid (HCl), and nitric acid (HNO_3) solution compared to other media (Yun Chen & Yang, 2020).

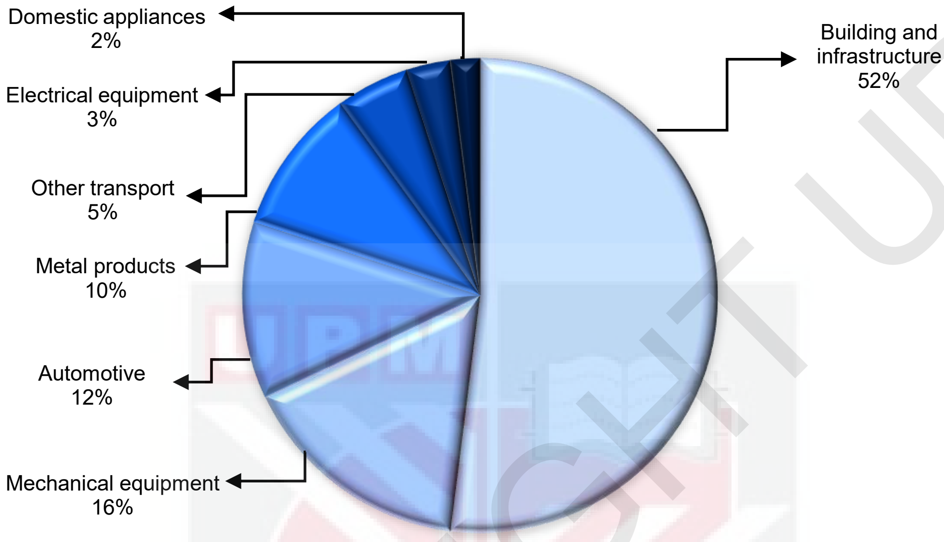

Figure 1.2: The global use of steel (Basson, 2020)

Corrosion is a critical worldwide concern since it substantially impacts the economy, safety, and the environment. Unsurprisingly, some of the popular catastrophic disasters, such as the Bhopal and Flixborough tragedy, blames metallic corrosion as the cause of the incident. The National Association of Corrosion Engineers estimated that corrosion expenses constitute approximately 4.2% of the global gross domestic product annually, equivalent to USD 2.5 trillion (C. Verma et al., 2021). Up to one-third of the expenses due to corrosion can be eliminated by incorporating adequate corrosion protection and prevention practices such as coating, plating, sacrificial anode, cathodic protection, and the use of corrosion inhibitors.

Corrosion inhibitors are substances that can minimise or eliminate metal corrosion once added to a corrosive solution. It is probably the most straightforward, inexpensive, and efficacious strategy employed on a reasonably regular basis in the industrial sector (Chauhan, Quraishi, et al., 2020). Conventional inorganic inhibitors such as chromates, nitrates, and phosphonates, are primarily chemical-based and harmful to human health and the environment (Popoola, 2019). Organic corrosion inhibitors, particularly those that are environmentally benign, are therefore of interest nowadays (Quraishi et al., 2020). The inhibition is mainly attributed to the inhibitor molecules' capability to adsorb on the metal surface, which is possible due to functional groups and heteroatoms in the chemical structure.

The “12 Principles of Green Chemistry” (Figure 1.3) initiated by Anastas and Warner (1998), and the Sustainable Development Goals (SDGs) (Figure 1.4) adopted by all members of the United Nations since 2015 had channelled the focus of research across the globe towards more sustainable and environmentally friendly products. Accordingly, researchers are making considerable efforts to substitute synthetic organic inhibitors that are toxic and costly to natural compounds that are minimally invasive. Throughout the years, plant extracts, mono and polymeric carbohydrates, ionic liquids, and drug molecules are becoming the leading clusters of organic corrosion inhibitors (Bharatiya et al., 2019; Umoren et al., 2019; C. Verma et al., 2017). Recently, the utilisation of biomass waste such as tomato peel, pistachio shell, oil palm frond, coconut leaf, and coffee husk as sustainable raw materials for corrosion inhibitors is thriving (S. Chen et al., 2020; Cordeiro et al., 2018; Halambek et al., 2020; Hussin et al., 2018; Shahmoradi et al., 2020). This rapid progress is explicable to the materials’ abundance, inexpensive cost, and worsening waste management problem.

Figure 1.3: Green chemistry principles (Anastas, P. T.; Warner, 1998)

Figure 1.4: Sustainable development goals (United Nations, 2019)

Oil palm empty fruit bunch (OPEFB) is one of the discarded solid waste from oil palm processing. As one of the major global producer of palm oil, it is estimated that Malaysia alone produces 22.6 million tonnes of OPEFB annually (Malaysian Palm Oil Board, 2019). The abundance of OPEFB as a lignocellulosic material provides an unlimited and inexpensive source for various applications. A large and growing body of literature has explored the potential of OPEFB to be converted to value-added products such as composite boards, conductive carbon paper, cellulose nanofiber, catalyst, and fuel for energy generation (Afriyanti et al., 2016; Destyorini & Indayaningsih, 2017; Hamzah et al., 2019; Okoye et al., 2019; Supian et al., 2019; Wahab et al., 2015). The building blocks of the OPEFB, which are cellulose, hemicellulose, and lignin, comprise a variety of heteroatoms such as nitrogen (N), sulphur (S) and oxygen (O). The presence of heteroatoms in OPEFB is a key criterion that qualifies it as a promising raw material for corrosion inhibition application. Heteroatoms typically have a single pair of electrons available to donate to iron atoms' unoccupied d-orbitals, resulting in strong chemical adsorption. Furthermore, heteroatoms can undergo protonation, which produces positively charged ions. These ions can be adsorbed on iron atoms through electrostatic or physical interactions, resulting in physical adsorption.

While comprehensive studies have been carried out on the use of OPEFB waste, there has been limited investigation on OPEFB utilisation as a corrosion inhibitor. Therefore, an attempt is made in this study to utilise OPEFB as a raw material for a corrosion inhibitor. Due to the exacerbating steel corrosion issue, an OPEFB inhibitor is developed for mild steel corrosion inhibition in 1 M HCl.

1.2 Problem statement

Corrosion inhibitors are crucial in the industry for corrosion prevention. Chromates, nitrites, and phosphates were once the most popular inorganic inhibitors. However, the atrocious toxicity of these compounds towards human and the environment has led to a ban on their use in certain parts of the world (K. M. O. Goni & A. J. Mazumder, 2019). Consequently, the industry opts for synthetic organic inhibitors such as pyridine, aniline, polyamine, and furfural that are less toxic but still quite costly to synthesis (Yun Chen & Yang, 2020). Most of these inhibitors are not biodegradable and possess issues upon disposal. Growing consciousness of environmental sustainability, economic and toxicological issues has called for better alternatives that are inexpensive, safe, and environmentally benign (Popoola, 2019). This demand offers a great opportunity for the exploration of nature-based or plant-based inhibitors.

To date, studies on natural inhibitors have been snowballing, and there is a proliferation of research papers addressing this matter in the literature. It is now well established that most plant-based inhibitors are effective in inhibiting corrosion, as technically, all natural materials comprise various functional groups and heteroatoms that can adsorb on metals (Miralrio & Vázquez, 2020). Nonetheless, far too little attention has been paid to go beyond the exploratory stage of proving the inhibition efficiency, that is, going a step further towards implementing the developed inhibitors in the industry (Popoola, 2019). The scant interest in commercialising plant-based inhibitors is due to foreseeable problems that arise when dealing with natural materials, such as processing and degradation issues.

Plants-based inhibitors require extraction processes that use different polarity of solvents ranging from water, methanol, ethyl acetate, dichloromethane to hexane (Miralrio & Vázquez, 2020). Although the extraction process is well accepted in the industry, concrete improvements of the process and better alternatives that use fewer resources and energy are still needed (Chaubey et al., 2021). Confronting the processing issues arises the idea of using natural products as inhibitors without any treatment. To the best of our knowledge, the use of untreated natural materials has not been closely investigated, except for plants' gums that are readily dissolvable (Guo, Zhang, et al., 2020). The scarcity in the use of unprocessed materials is due to the difficulties to properly understand the underlying mechanism of action since the unprocessed crude sample comprises a cocktail of hundreds of compounds (Chaubey et al., 2021). Besides, the compounds that do not participate in corrosion inhibition exist as contaminants in the system, which may cause problems later on (Chaubey et al., 2021). Despite the downsides, it opens up an opportunity to unravel better alternatives to utilise natural products with minimal treatment. A more viable solution is to make use of waste produced by the industrial processing of natural materials. For instance, alkaline and acid treatments on agricultural biomass are industrially performed to recover its pulp for paper production. The unwanted portion of the biomass disposed of during the process is seen as a potential source for corrosion inhibitors as it is readily available without needing excessive efforts to be processed.

Upon having a proper inhibitor source, there is still uncertainty whether the inhibitor is suitable for industrial application. This is due to the biodegradability of natural inhibitors, which causes their performances to deteriorate at longer immersion time, especially after 120 hours of usage (Asfia et al., 2020; Chahul et al., 2019; C. Wang et al., 2019). Although degradation is a positive feature from an environmental point of view, it is a huge drawback for implementing natural inhibitors in the industry. Several studies have tested incorporating plant-based inhibitors in polymer coatings, which can extend the shelf life of the inhibitor (Ong et al., 2021). However, this solution only applies to metal parts that can be coated. The industrial applicability of plant-based inhibitors for non-coated metal hitherto remains unresolved. A proposed solution to address this matter is to develop a controlled-release inhibitor tablet. This idea was triggered based on the concept of pharmaceutical controlled-release tablets and a patented invention of controlled release tablet for wastewater treatment (Haag & Relenyi, 2015). Technically, if an inhibitor is formed into a controlled-release tablet, the inhibitor particles are secured in a polymer matrix and, in consequence, prevent rapid degradation upon contact with a corrosive environment.

From another perspective regarding the raw material, OPEFB waste is a very concerning issue for palm oil-producer countries such as Malaysia, Indonesia and Thailand. This is due to the massive production of biomass from palm oil milling, approximately 4 kg of dry biomass produced per 1 kg of palm oil extracted (Mohammad Padzil et al., 2020). As the OPEFB waste is generated within the mill's compound, unsuitable as boiler fuel, and contains residual oil, it has attracted much interest from scientific and industrial parties. The cheap and abundant OPEFB waste is used as a raw material in this study to alleviate the waste management issue.

1.3 Research hypothesis

Keeping the above problems in mind, several hypothesis has been made to address the problems.

i. OPEFB is composed of various compounds with heteroatoms and heterocyclic structures capable of adsorbing on metals. It is also abundant and underutilised, making it a perfect source of material for a corrosion inhibitor.
ii. The utilisation of OPEFB for its cellulosic fibre is well acknowledged, and this requires alkaline, acidic, or other treatments that remove unnecessary compounds like hemicellulose and lignin. The discarded fraction of OPEFB is a possible prospect to be repurposed as a corrosion inhibitor.

iii. HCl treated OPEFB has the best corrosion inhibition efficiency compared to other OPEFB inhibitors as the acid treatment primarily isolates hemicellulose from the raw material. Hemicellulose is a heteropolymer with various heteroatoms in its structure and has excellent solubility in the corrosive medium. The presence of hemicellulose in HCl treated OPEFB contributes to its capability in inhibiting mid steel corrosion.
iv. As a head-start for implementing OPEFB inhibitor for commercial use, a prevailing technique in the pharmaceutical field is employed. A controlled-released OPEFB inhibitor tablet capable of releasing active compounds in a sustained manner, can prevent the active compounds from degrading when in contact with corrosive environment, extending the shelf life upon usage.

1.4 Research objectives

To address the problems and prove the hypothesis stated above, several objectives are set. This project's main goal is to develop an OPEFB tablet as a corrosion inhibitor for mild steel in 1 M HCl. The specific objectives targeted to achieve this goal is as follows:

i. To characterise the untreated, NaOH treated, and HCl treated OPEFB inhibitors and elucidate their inhibition efficiencies and adsorption isotherms on mild steel in 1 M HCl using weight loss measurement at different inhibitor dosages.
ii. To optimise the inhibition efficiency of the best OPEFB inhibitor using response surface methodology-central composite design (RSM/CCD) at different dosages and immersion times.
iii. To investigate the mechanism and kinetics of the best OPEFB inhibitor anticorrosion action using potentiodynamic polarisation (PDP) and electrochemical impedance spectroscopy (EIS).
iv. To analyse the OPEFB active compounds in terms of their electronic properties and interactions towards mild steel substrate using quantum chemical method and molecular dynamics (MD) simulation, respectively.
v. To formulate an OPEFB inhibitor tablet using D-optimal mixture design, determine the tablet properties, and compare its anticorrosion action with OPEFB powder inhibitor at extended immersion times.

1.5 Significance and contribution of study

This study's paramount importance and originality is that it explores and verifies the corrosion inhibition potential of OPEFB for the first time. More precisely, the contributions from each part of this project are as follows;

i. The characterisation of untreated and chemically treated OPEFB reveals valuable information regarding the essential properties in the corrosion inhibition field. This result is beneficial as a benchmark or reference for the utilisation of OPEFB for corrosion inhibition as well as other potential applications in future works. Besides, the in-depth chemical characterisation of the best inhibitor (HCl treated OPEFB) unveils various chemical compounds in the sample. Since the typical use of acid-treated lignocellulosic biomass is for xylose recovery, most sample characterisation in the literature only focused on the sugar compounds. The discovery of other compounds opens up an opportunity for this substance to be used for other purposes.

ii. The weight loss corrosion test divulges the best treatment of OPEFB for excellent corrosion inhibition. This result is pivotal for developing new inhibitors, as this rule is technically applicable for other lignocellulose materials with similar composition as OPEFB.

iii. The electrochemical test reveals the mode of inhibition and the corrosion kinetics of mild steel with the use of OPEFB. Understanding the impact of the OPEFB's incorporation towards the corrosion reaction is useful to improve the inhibition performance further.

iv. The computational studies of OPEFB compounds disclose each molecule's capability to inhibit corrosion from a theoretical perspective. With this information, it is possible to selectively purify the OPEFB for better corrosion inhibition in future studies. The challenge for the theoretical investigation of plant-based inhibitor is the presence of many compounds in the sample. Unlike the chemically synthesised inhibitors, which contain pure compounds, crude samples from plants comprise of hundreds of different compounds. Therefore, it is currently a norm to choose several compounds for theoretical studies based on assumptions. In this study, the sample is characterised for its chemical compounds, and molecules with relatively high abundance are selected for further investigation. This approach is more reliable as it eliminates the possibility of cherry-picking compounds. Hence, this method can be used as a basis for future investigation of nature-based inhibitors.

v. The innovation of the OPEFB corrosion inhibitor tablet unravels the issues that emerge when dealing with natural inhibitors. This pioneering discovery provides a colossal foundation to utilise nature-based inhibitors for commercial applications.

1.6 Scope and limitation of study

This study focuses on exploring the corrosion inhibition of untreated, NaOH treated, and HCl treated OPEFBs for mild steel in 1 M HCl. Herein, the scope of the study is described in a sequential manner starting from the preparation of OPEFB inhibitor to the performance test of OPEFB tablet for better understanding.

First of all, the choice of treatment for the OPEFB is on the basis of alkaline and acidic treatment in biomass processing. All treated OPEFBs are prepared according to typical laboratory procedures reported in the literature. The elemental composition, functional groups, thermal degradation property, antioxidant activity, and morphology of all OPEFB inhibitors are characterised prior to further analysis. Next, each OPEFB inhibitor's performance is evaluated using weight loss analysis at different dosages from 0.1 to 0.5 g at constant immersion time and temperature. The corrosion inhibition was tested for mild steel in mild acidic environment. The data is then fitted to Langmuir, Freundlich, and Temkin isotherm to explore the adsorption's nature.

HCl treated OPEFB with the utmost performance is selected for further investigation. Prior to any test, HCl treated OPEFB is characterised in terms of its structural (cellulose, hemicellulose, lignin, and extractives) and chemical compositions. Later, the inhibitor dosages (0.1 to 0.5 g) and immersion times (120 to 360 h) are optimised using RSM/CCD to obtain maximum inhibition efficiency. The morphology of mild steel immersed for 24 h in inhibited and uninhibited 1 M HCl solution is then analysed. Successively, electrochemical tests, namely PDP and EIS, are performed. Following this, the inhibitor compounds were examined computationally in terms of their electronic properties and interaction towards mild steel substrate.

Consecutively, an OPEFB tablet that includes binding polymers, namely gum Arabic (GA) and hydroxypropyl methylcellulose (HPMC), was formulated using D-optimal mixture design. Each formulation is analysed in terms of the corrosion inhibition performance at constant OPEFB ratio, immersion time, and temperature. Subsequently, the properties of three tablets; a control tablet with 100% OPEFB, and two best formulations from the mixture design, are evaluated. One tablet with the best tablet properties is further examined in the final part of this study. Ultimately, the anticorrosion action of the selected OPEFB tablet is compared with HCl treated OPEFB in powder form at extended immersion times, ranging from 120 h to 720 h. The elucidation of the tablet capability in inhibiting corrosion at prolonged time marks the end of the study.

Based on this study's scope mentioned above, a number of limitations can be pointed out. Firstly, it is comprehended that the study's overall scope may be too broad as it covers a conventional corrosion study and additional work for the application of the inhibitor in tablet form. Considering that very little is currently known about the use of OPEFB as a corrosion inhibitor, standard experimental and theoretical studies dealing with fundamental corrosion inhibition properties are the utmost priorities. From another perspective, the tableting of OPEFB inhibitor is also indispensable as it has a vast potential to solve a major problem in the corrosion field. Hence, it is necessary to scrutinise both aspects for a comprehensive study that is independent of existing research, original, and contributes significantly to the field of corrosion science. Regrettably, several aspects that are less critical and of low importance are being leveraged to achieve this project's goal in a reasonable time and expense.

Regarding the treatment of OPEFB, only alkaline and acidic treatments are assessed, and the parameters are not optimised for maximum yield. The studied parameter for weight loss measurement and electrochemical tests is limited to inhibitor dosage, and for optimisation is limited to inhibitor dosage and immersion time, respectively. The effect of different temperatures, corrosive solutions, metal type, and other parameters are not taken into account. Besides, starting from the optimisation study onwards, the OPEFB inhibitors are narrowed down to only one with the best performance for further investigation. In terms of the identification of chemical compounds, proper quantification is only focused on sugar compounds, while other compounds are detected qualitatively. This study is also unable to provide additional characterisation for lignin compounds. With respect to the computational analysis, examining all detected compounds is impractical without high-performance computers. Hence, this study only encompasses 15 compounds of significant abundance in the sample. Validating each compound's predicted performance through an experimental approach is also beyond the study's scope. Concerning the tableting of OPEFB, only the tablet formulation is prioritised, while other parameters such as the tablet diameter and compression force remained constant. Lastly, the formulations are also narrowed down for further test that examines the tablet properties and corrosion inhibition performance.

1.7 Structure of thesis

Chapter 1 highlights the background, problem, hypothesis, and objectives of this research. The significance and scope of the study are also elaborated in the chapter.

Chapter 2 presents a comprehensive literature review on related topics of the study. The main topics are corrosion, corrosion protection methods, corrosion inhibitors, experimental corrosion test, and theoretical corrosion investigations. Additionally, the information on OPEFB is also reviewed in this chapter.

Chapter 3 provides information on the materials and methodologies used in this study. Briefly, the methods of inhibitor preparation and characterisation, experimental corrosion inhibition tests, computational method, and tablet preparation are explained in detail.

Chapter 4 discusses the results obtained from the study. Generally, the results can be divided into four parts; inhibitors characterisation, experimental corrosion inhibition study, computational study, and tablet corrosion inhibition study.

Chapter 5 concludes the results and also states the recommendations for future work.

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