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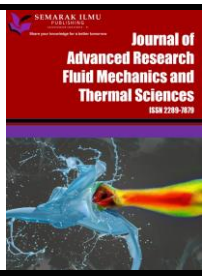

Effect of Temperatures on Green Synthesis of Amide-based Corrosion Inhibitors from Sustainable Source

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ABSTRACT

This research addresses the critical need for effective anticorrosion solutions, focusing on the development of environmentally friendly coatings using Palm Fatty Acid Distillate (PFAD), a byproduct of crude palm oil production, as a key component in amide inhibitors. Traditional inorganic corrosion inhibitors pose environmental and economic concern due to their expense and toxicity while PFAD based amides promote a sustainable and cost-effective alternative. The synthesis of PFAD based amides using ethylenediamine in ethanol at reflux at various temperatures (25, 40, 60 and 90˚C) revealed that higher synthesis temperatures enhance inhibition properties. Amides containing double bonds and nitrogen-functional groups demonstrated excellent efficiency in attaching to metal surfaces. This study evaluates the synthesized amide's inhibitory potential through physicochemical analysis using Fourier Transform Infrared Spectroscopy (FTIR) and corrosion inhibition efficiency using the Linear Polarization Resistance Method (LPRM). Physicochemical analysis using FTIR indicated the prominence of the amide group of N-H in the product with clear bonds at 1600 cm^{-1}, and visible amide groups of O-H at 3350 cm^{-1}. They showed the disappearance of the carboxylic acid (C=O) peak from the raw material of PFAD, indicating that the amide synthesis reaction occurred successfully. LPRM analysis revealed that the amides at 90°C achieved a 75% corrosion inhibition efficiency in a 3.5% NaCl solution, outperforming other tested temperatures. This efficiency is attributed to the adsorption of nitrogen-containing fatty amide molecules on the metal surface. These findings highlight the potential of PFAD based amides as green corrosion inhibitors, offering a promising solution for sustainable corrosion mitigation strategies.

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1. Introduction

Corrosion has emerged as a significant concern for industries, presenting potential catastrophic consequences through chemical and electrochemical interactions with equipment [1]. Despite ongoing advancements in corrosion-resistant materials, the use of corrosion inhibitors remains one of the most practical and cost-effective methods for controlling corrosion [2]. Corrosion inhibitors are chemical compounds introduced in trace quantities to fluids, aiming to mitigate corrosion caused by diverse processing conditions, fluctuating salt concentrations, and variable water levels encountered throughout various stages of oil and gas production processes [3]. Numerous inorganic and organic compounds have been identified as potential inhibitors to reduce corrosion [4].

However, many of these inhibitors are associated with toxicity, complex synthesis procedures, high costs, and lack of environmental friendliness [5]. The disposal of these toxic inhibitors, especially in the marine sector, poses risks to marine life and ecosystems. Consequently, the use of inorganic inhibitors, particularly those containing phosphate, chromate, and other heavy metals, has been increasingly restricted or banned to comply with environmental regulations. Non-green fatty acids used in corrosion prevention also face challenges due to improper disposal, which can lead to accumulation in landfills and subsequent absorption into soil and water systems [6]. This pollution can negatively affect ecosystems and biodiversity.

Consequently, there is significant interest in developing novel, environmentally friendly corrosion inhibitors through green synthesis process. Green chemistry involves integrating environmentally friendly principles into laboratory protocols to mitigate the adverse effects of harsh chemicals and chemical waste on both human health and the environment [7]. There is a significant demand for a coating method that is easy to implement, cost-effective, scalable, and environmentally friendly. This need arises from the limited availability of high-quality natural templates and straightforward manufacturing processes [8]. In a study conducted by Ali *et al.,* [4], the utilization of products derived from the reaction between palm oil-based fatty acids and monoethanolamine were proposed as environmentally friendly corrosion inhibitors in acidic environments. The incorporation of additional heteroatom groups from monoethanolamine is expected to enhance interactions with metal surfaces compared to conventional fatty acids or esters, thereby augmenting their corrosion prevention capabilities.

In addition, previous research by Al-Edan *et al.,* [9] reported that amines and their derivatives have been acknowledged for their efficacy as corrosion inhibitors for alloys and iron, owing to their relatively high solubility. The study suggests that the corrosion of mild steel when exposed to 1 M HCl can be mitigated using a synthesized compound named N-(4-aminobutyl) palmitamide (BAPA). The protective mechanism of BAPA is attributed to the presence of oxygen and nitrogen, which serve as highly effective electronic adsorption centers, thereby blocking the active sites of iron. Furthermore, Reyes-Dorantes *et al.,* [2] investigated the inhibitory effectiveness of fatty amides synthesized from crude rice bran oil combined with aminoethylethanolamine (AEEA) in a 1:3 mole ratio against the corrosion of APIX-70 steel in a CO_2-saturated solution containing 3.5% sodium chloride (NaCl). The fatty amides exhibited corrosion inhibitory properties at all tested temperatures (30, 50, and 70°C), with their effectiveness increasing in proportion to the concentration of the added inhibitor. The inhibitory mechanism was attributed to the adsorption of inhibitor molecules onto the metal surface, hindering the diffusion of aggressive ions from the electrolyte to the metal surface.

This study focuses on utilizing palm fatty acid distillate (PFAD) as the primary raw material for eco-friendly amide synthesis. PFAD's composition predominantly consists of free fatty acids (FFA), primarily palmitic acid and oleic acid, comprising around 80% of PFAD, with the remaining 20% comprising other saturated and unsaturated fatty acid [10]. Although PFAD has been explored for

biodiesel production, its potential for green amide synthesis as corrosion inhibitors remains underexplored. This research addresses this gap by investigating the impact of different synthesis temperatures on amides using PFAD through direct condensation reflux. The study involves comprehensive characterization of the synthesized amides using Fourier Transform Infrared Spectroscopy (FTIR) and evaluation of their corrosion inhibition efficiency via the Linear Polarization Resistance Method (LPRM). By repurposing PFAD, this work contributes to sustainable practices and aligns with global environmental initiatives, offering an eco-friendly and cost-effective alternative for corrosion mitigation.

2. Methodology

2.1 Chemicals and Materials

The optimization of the amidation reaction was conducted using PFAD as the model substrate. The PFAD utilized in this experiment was procured from Palm-Oleo Sdn. Bhd. (Rawang, Selangor), while Ethylenediamine, Ethanol and Hydrochloric acid (HCl) were obtained from Sigma-Aldrich (M) Sdn. Bhd.

2.2 Synthesis of Amide

Palm Fatty Acid Distillate (PFAD) and ethylenediamine were mixed and agitated at varied temperatures for 5 hours under reflux conditions in a 250-ml, three-necked round-bottomed flask as depicted in Figure 1.

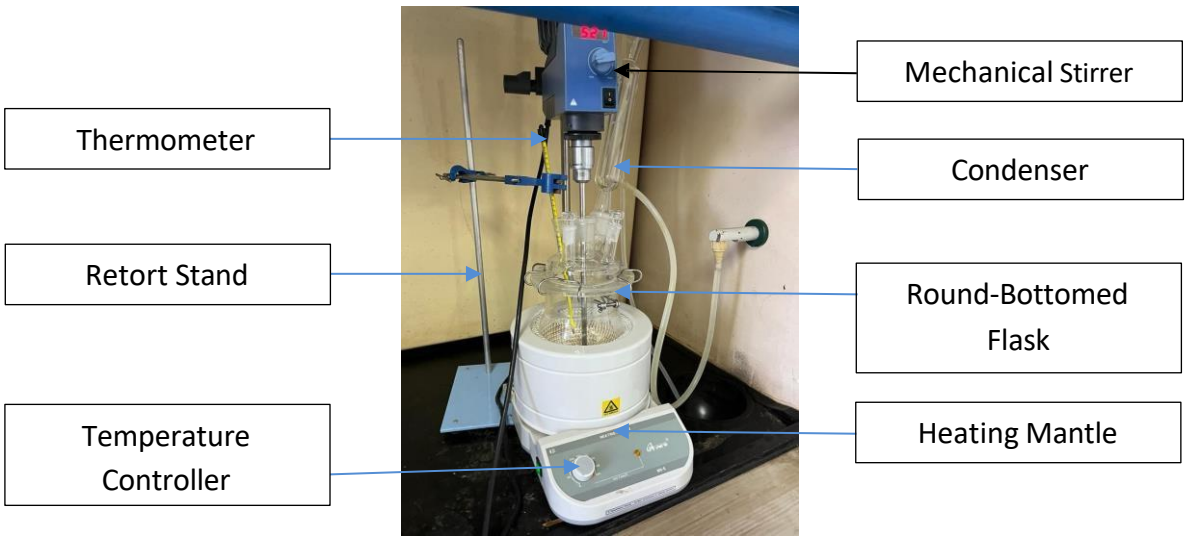

Fig. 1. experimental reflux setup

PFAD (20 mL), HCl (10 mL, 1M), and ethanol (120 mL, 1M) were combined and stirred for 10 minutes. Subsequently, the solution was transferred to the reaction vessel. Ethylenediamine (40 mL, 0.05M) was then added, and heating commenced. Following the reaction, the resulting products were transferred to a container and allowed to cool to room temperature for subsequent analysis. The impact of the reaction temperature on the yield of the amide product derived from PFAD and ethylenediamine was investigated, with temperatures set at 25°C, 40°C, 60°C, and 90°C. The set for the experiment were prepared and labeled as depicted in Table 1.

Table 1
Set of experiment

Set	Temperature (°C)
A	25
B	40
C	60
D	90

2.2.1 Characterization of amide

The liquid sample underwent FTIR analysis using a Shimadzu IR-Affinity FTIR spectrometer. This analytical technique allowed for the examination of the structure, connectivity, and chemical environment of atoms within the sample molecules based on their infrared absorption spectra.

2.3 Corrosion Testing Using Linear Polarization Resistance Method

The electrochemical measurements were conducted using a three-electrode setup consisting of a working electrode, an auxiliary electrode, and a reference electrode, employing the linear polarization method The metal sample (mild steel: 2cm x 2.3cm) surface was polished with abrasive paper with grades ranging from 500 to 1000. Subsequently, it was cleaned with acetone followed by distilled water and left to dry at room temperature. The mild steel specimen served as the working electrode and was immersed in 1000 ml of 3.5 wt.% NaCl solution, both with and without the presence of corrosion inhibitors. During the measurements, corrosion parameters including polarization resistance (R_p) in ohms (Ω), and efficiency inhibition (% IE) were measured using Eq. (1) and Eq. (2) [11].

$$R_p = \frac{\beta a \beta c}{2.303 \times i_{CORR} \times \beta a \beta c} \quad (1)$$

where,

R_P = Polarization resistance, Ω
$\beta_a.\beta_c$ = Beta Coefficients (V/decade)
I_{CORR} = Corrosion Current Density (A/cm^2)

$$IE\ (\%) = [\frac{R_{pi} - R_{po}}{R_{pi}}] \times 100 \quad (2)$$

where,

R_{pO} = Polarization Resistance (Blank), Ω
R_{pi} = Polarization Resistance (Inhibitor), Ω

3. Results and Discussion

3.1 Characterization of Amide

The analysis identified multiple peaks, indicating the presence of a structurally intricate substance. As depicted in Figure 2 a peak at 3350 cm^{-1} corresponds to O-H bonds suggesting the

presence of amide groups, consistent with observations made by Yu *et al.,* [12] who noted broadening in this spectral region due to the amide second overtone region. Additionally, C=O bonds related to amide groups were observed at wavenumbers 1640-1690 cm^{-1} and 1630-1740 cm^{-1}further confirming the formation of amides. Meanwhile, the raw material, PFAD showed C-N bonds in the 1200-1350 cm^{-1} range, which are characteristic of amine groups. The transformation from amine to amide is evidenced by the disappearance of these C-N bonds in the final product and the appearance of new peaks associated with the amide structure.

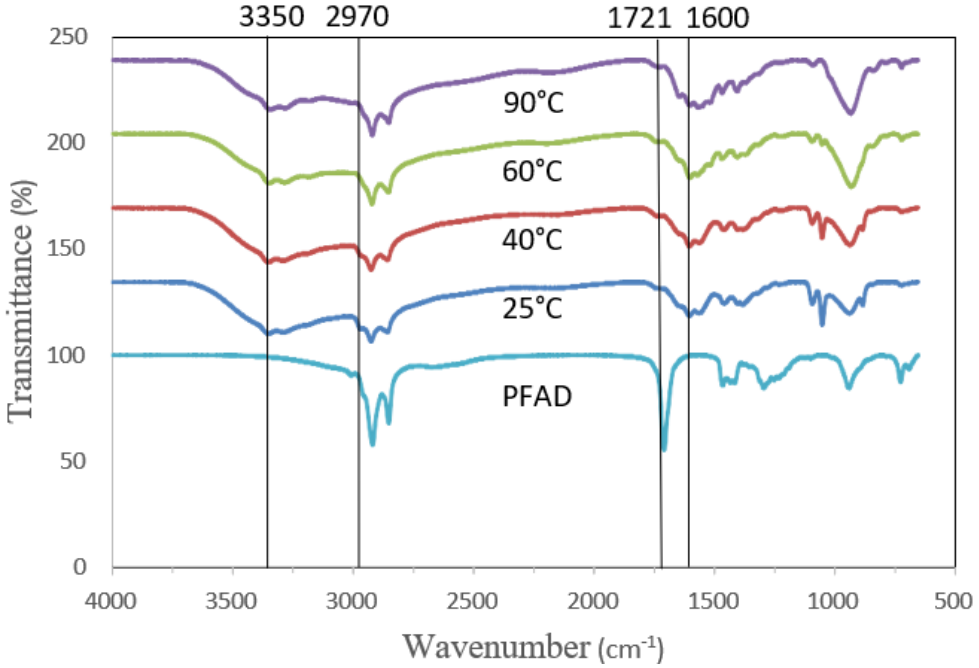

Fig. 2. FTIR spectra of PFAD-based amide

Further details on the expected functional group and compound presented in the PFAD based amide were tabulated in Table 2. The FTIR results obtained in this study were consistent with study recorded by Al-Edan *et al.,* [9] and Elsharif *et al.,* [13].

Table 2
Functional Group on PFAD-based Amide [8,12]

Wavenumber (cm^{-1})	Functional group	Expected Compund
3350	O-H	Amide
2970	O-H	Carboxylic Acid
1721	C=O	Carboxylic Acid
1600	N-H	Amide
1200-1350	C-N	Amine

Comparison of the final product with the PFAD spectrum revealed the disappearance of two carboxylic acid peaks at 2970 cm^{-1} and 1721 cm^{-1}, while an amide peak at 3350 cm^{-1} emerged in the final product compared to the PFAD spectrum. The absence of the carboxylic acid peaks at 2970 cm^{-1} and 1721 cm^{-1} can serve as an indicator of the reaction completion extent. These results are consistent with a study by Hoidy *et al.,* [14] on the characterization of n-hydroxy-n-methyl fatty amide (HMFA) from palm oil, which demonstrated distinct peaks in the FTIR spectrum of HMFA, including O-H stretching at 3420 cm^{-1}, C=O stretching of the amide group at 1650 cm^{-1}, and C-N stretching at 1041 cm^{-1}. These findings support the presence of fatty amide in PFAD-based amide.

On top of that, study by Ginting *et al.,* [15] demonstrated the characterization of fatty acid alkanolamide (FAA) from amidation of fatty acid and monoethanolamine supported the findings obtained in this study. In the FT-IR spectrum of PFAD and FAA, a shift in wavenumber was observed from 3007.20 cm^{-1}, corresponding to a broad peak from the OH group, to 3301.89 cm^{-1}, indicating the stretching vibration of the N-H group with a sharp peak. The presence of FAA compounds was corroborated by the emergence of stretching vibrations C-N-H and C-N at wavenumbers of 1557.03 cm^{-1} and 1466.08 cm^{-1}. These results are consistent with the findings obtained from the synthesis of fatty amide in this study.

In particular, spectral analysis revealed a distinct amide group at 3350 cm^{-1}, with the most prominent bond observed at 40°C. At 90°C, a sharp bond indicative of an amide bond appeared around 1600 cm^{-1}, suggesting the highest concentration of amide functional groups at this temperature. Thus, it can be inferred that the sample exhibits the highest concentration of amide functional groups at 90°C compared to other temperatures tested.

3.2 Corrosion Inhibition using Linear Polarization Testing Method

The Linear Polarization Resistance (LPR) technique was utilized to assess how well the synthesized amide could reduce corrosion rates. Electrochemical parameters such as polarization resistance (R_p) and inhibition efficiency (%) were plotted against various temperatures during the reflux reaction of amide derived from PFAD as depicted in Figure 3 while Table 3 summarizes corrosion parameters such as polarization resistance (R_p, measured in ohms, Ω), and inhibition efficiency (% IE) in a 3.5% NaCl solution, both with and without PFAD-based amide, at different synthesis temperatures.

Table 3
The resistance polarization (R_p) and inhibition efficiency obtained from LPR

Set (Based on Synthesis Temperature)	Resistance Polarization, R_p (Ω)	Inhibition Efficiency (%)
Blank	2.298×10^3	-
25	4.498×10^3	49
40	5.117×10^3	55
60	7.273×10^3	68
90	9.289×10^3	75

Based on Figure 3 and value tabulated in Table 3, the absence of the amide-based inhibitor resulted in a lower resistance polarization value (2.298×10^3 Ω), while its addition led to an increase in resistance polarization [16]. In the presence of PFAD-based amide acting as an inhibitor, a noticeable increase in the resistance polarization value (4.498×10^3 Ω). This occurrence is attributed to the adsorption process taking place on the metal surfaces. Higher R_p values indicate better corrosion resistance because it means the material is more resistant to the electrochemical processes that lead to corrosion. The polarization resistance value significantly impacts the corrosion rate [16]. According to Wong *et al.,* [17], polarization resistance (R_p) values are inversely proportional to corrosion rates (mpy). LPR tests demonstrated that the inclusion of the inhibitor led to the formation of a protective barrier between the metal surface and the aggressive medium [18]. This resulted in an increase in the R_p values.

The increase in resistance polarization corresponds to a decrease in the corrosion rate, indicating an improvement in corrosion inhibition efficiencies by adding the amides inhibitors. This might be due to the adsorption of the fatty amide inhibitors. A greater quantity of fatty amides was adsorbed,

either through physical or chemical means, thereby forming protective layers. Fatty amide molecules containing nitrogen (N) possess a pair of electrons (a lone pair) resulting from their sp^2 electron configuration, facilitating surface adsorption. The lone pair can effectively adsorb onto the surface of carbon steel, forming a protective layer [19].

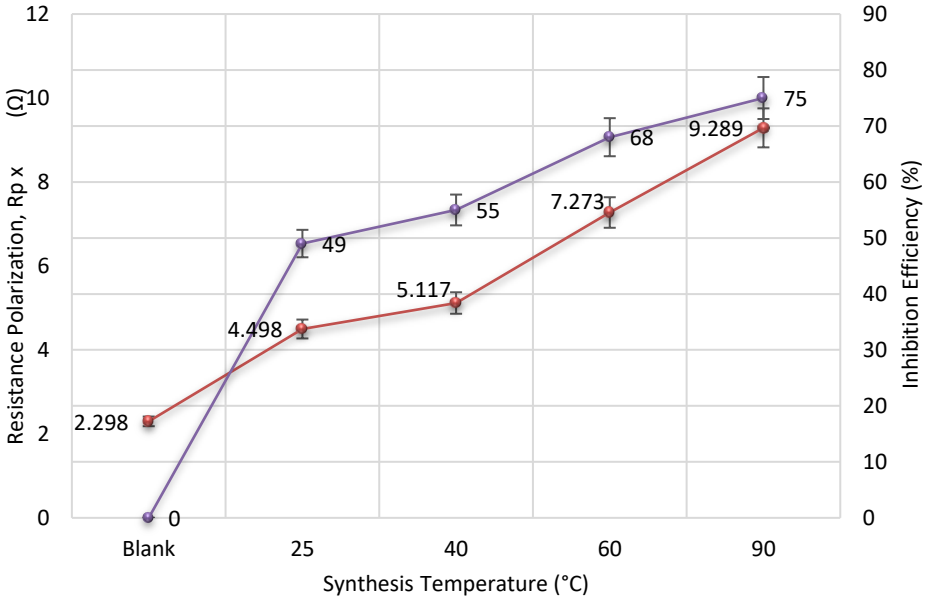

Fig. 3. Polarization resistance and inhibition efficiency of PFAD-based amide at different synthesis temperatures

At 90°C, PFAD-based amide exhibited optimal efficiencies of 75%, with a resistance polarization at 9.289 x 10^3 Ω establishing it as a highly effective inhibitor of mild steel corrosion in a 3.5% NaCl solution. Previous research conducted at various temperatures further supports these findings [8]. These results were also supported by the study of fatty amides from the reaction between crude palm oil and monoethanolamine by Ali *et al.,* [4]. Ali *et al.,* [4] suggested that inhibitor efficiency in the range 75%-80% exhibited effective corrosion inhibition properties of the fatty amide.

As the synthesis temperature increases, the resistance polarization (R_p) and inhibition efficiency both increases. This indicates that the higher synthesis temperature enhances the corrosion inhibition performance, likely due to improved formation or adherence of the protective layer on the metal surface, which reduces the corrosion rate.

In conclusion, the results highlight the promising corrosion inhibition capabilities of the synthesized PFAD-based amide, primarily attributed to the adsorption of nitrogen-containing fatty amide molecules onto the metal surface, thereby creating a protective barrier against corrosion. Moreover, due to PFAD's high content of free fatty acids (81.7%), with carbon atoms spanning from 2 to 18 per molecule, the resulting fatty amide inhibitor encompasses a variety of chain lengths of aliphatic amides [20]. This diversity augments the efficacy of a singular organic amine inhibitor molecule.

4. Conclusions

In conclusion, the eco-friendly synthesis of amides from PFAD demonstrates significant potential, aligning with green chemistry principles. Concerns about creating eco-friendly processes have led to the development of biological methods that use plant extracts for green synthesis. These methods are better than chemical processes because they are better for the environment, cheaper, use less

energy, and are easier to do. This study highlights the critical role of temperature in optimizing the formation and effectiveness of PFAD-based amides for corrosion inhibition. At a synthesis temperature of 90°C, the conditions were found to be optimal, as indicated by a significant peak in the FTIR spectra, confirming the presence of amide functional groups in the product. This corresponds with a corrosion inhibition efficiency of 75%, underscoring the promise of PFAD-based amides as green corrosion inhibitors. Further research and formulation enhancements could fully realize their potential, making them viable for practical applications where effective corrosion prevention is essential.

Conflict of interest

The authors have no conflict of interests related to this publication.

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