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FORMULATION AND EVALUATION OF A RUST REMOVAL SOLUTION

A DISSERTATION SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS OF THE BACHELOR OF SCIENCE EDUCATION HONOURS DEGREE IN CHEMISTRY (HBScEdCh).

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APPROVAL FORM

The undersigned certify that they have supervised, read and recommend to the Bindura University of Science Education for acceptance a research project entitled: Formulation and evaluation of a rust removal solution

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In partial fulfilment of the requirements for the ***BACHELOR OF SCIENCE EDUCATION HONOURS DEGREE IN CHEMISTRY*** **(HBScEdCh).**

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DECLARATION

I .Ruvhura.Prettymore........ declare that this research project is my own work and has not been copied from any source without the acknowledgement of the source.

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ABSTRACT

Corrosion, and specifically rusting, is a significant problem that affects the performance, lifespan, and economic viability of metal structures and components across a wide range of industries. Traditional rust removal methods, which often rely on the use of hazardous mineral acid-based solutions, pose risks to both the metal being treated and the environment.

This study aims to develop and evaluate the performance of a rust removal solution that combines chemical and natural approaches, including the use of mineral acids, organic acids, and tannins extracted from banana peels. The proposed solution aims to address the limitations of existing rust removal methods by improving performance, safety, and environmental sustainability.

The specific objectives of this study include the preparation and characterization of rusted metal samples, the formulation of various rust removal solutions, the assessment of their effectiveness, and the optimization of the best-performing solution. The study will also compare the developed solution against traditional mineral acid-based formulations, evaluating factors such as removal efficiency, environmental impact, and cost-effectiveness.

The successful formulation and evaluation of a rust removal solution with improved performance, safety, and sustainability can contribute to the preservation of metal infrastructure, the promotion of worker safety, and the adoption of environmentally responsible practices in various industries. This study has the potential to provide a more accessible and cost-effective alternative to traditional rust removal methods, particularly in the context of developing countries.

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TABLE OF CONTENTS

Contents **Page**

Cover page (i)

Approval form (ii)

Abstract (iii)

Acknowledgements (iv)

Contents page (v)

CHAPTER ONE: THE PROBLEM

1.0 Introduction 1
1.1 Background information 1
1.2 Justification of the study 2
1.3 Aim 2
1.4 Objectives 3
1.5 Scope and delimitations 3
1.6 Significance of the study 4
1.7 Summary 5

CHAPTER TWO: LITERATURE REVIEW

2.0 Introduction 7
2.1 Rust formation and corrosion processes 7
2.2 Traditional rust removal methods 8
2.2.1 Mechanical rust removal methods 8
2.2.2 Chemical rust removal method 8
2.2.3 Electrochemical rust removal method 9
2.3 Limitations of traditional rust removal methods 9
2.4 Towards eco-friendly and sustainable rust removal solutions 10
2.4.1 Tannin – based rust removal formulation 10
2.4.2 Organic acid-based rust removal formulations 11
2.4.3 Combination of organic and mineral acids 11

2.4.4 waste-derived rust removal formulations 12
2.5 factors influencing the effectiveness of rust removal solutions 13
2.5.1 Chemical composition 13
2.5.2 Concentration of active ingredients 14
2.5.3 Contact time 15
2.5.4 Surface characteristics of the rust metal 15
2.6 characteristics of rust removal effectiveness 15
2.6.1 Visual inspection 15
2.6.2 Weight loss measurements 16
2.6.3 Surface analysis 16
2.6.4 Corrosion resistance evaluation 16
2.6.5 Environmental impact 16
2.7 Challenges and future directions 17

CHAPTER THREE: RESEARCH METHODOLOGY

3.0 Introduction 20
3.1 Materials and equipment 20
3.1.1 Material utilized in the research 20
3.1.2 The equipment employed in this study 20
3.2 Artificial rusting 20
3.3 Formulation based on phosphoric acid 21
3.4 Formulation based on acidified tannins 21
3.4.1 Extraction of tannins 21
3.5 Phytochemical detection 21
3.6 Formulation based on tannins 21
3.7 Based on hydrochloric acid 22
3.8 Based on oxalic acid 22
3.9 Treatment of rusted samples 22
3.10 Characteristics of rusted samples 22

CHAPTER FOUR: RESULTS AND DISCUSSION

4.0 Introduction 23
4.1 Results for artificial rusting 23
4.2 Results on the Formulation Based on Phosphoric Acid 24

CHAPTER FIVE: DISCUSSION AND CONCLUSION

5.0 Introduction 28

5.1 Discussion 28

5.2 Conclusion 29

List of figures

Fig 4.1 Results for artificial rusting 23

Fig 4.2 Results on the Formulation Based on Phosphoric Acid 24

Fig 4.3 illustrates the effect of contact time on rust removal efficiency. 24

Fig 4.4 Effect of contact time on rust removal (30% phosphoric acid present, pH 1.8) 25

Fig 4.5 effects of contact time on rust removal 26

Fig 4.6 effects of contact time on rust removal 27

List of tables

Table 3.1: Rust removal characterization 22

Reference list

CHAPTER 1: INTRODUCTION

1.1 Background Information

Corrosion is a major problem that affects the performance and lifespan of metal structures and components across various industries. Rusting, which is a specific type of corrosion affecting iron and steel, is particularly prevalent and can lead to significant economic losses due to the replacement of damaged parts, reduced efficiency, and safety hazards (Singh, 2015). The global cost of corrosion has been estimated to be around 3-4% of a country's gross domestic product (GDP) (NACE International, 2016). In developing countries, the impact of corrosion can be even more severe, with estimates suggesting losses as high as 5-6% of GDP (NACE International, 2016).

Rust is a hydrated iron oxide (predominantly Fe_2O_3) that forms on the surface of iron and steel when exposed to moisture and oxygen in the environment (Rajendran, 2012). The rusting process involves the oxidation of iron atoms, which leads to the formation of iron oxide compounds that are voluminous and have a reddish-brown appearance (UK Essays, 2018). Rust can significantly degrade the structural integrity and appearance of metal components, leading to premature failure and the need for costly repairs or replacements.

Various methods have been employed to remove rust and prevent further corrosion, including mechanical, chemical, and electrochemical techniques (Mika, 2014). Chemical methods, commonly referred to as "pickling," involve the use of acidic solutions to dissolve and remove the rust layer from the metal surface (Singh, 2015). Mineral acids such as hydrochloric acid (HCl), sulfuric acid (H_2SO_4), and phosphoric acid (H_3PO_4) are widely used in industrial settings for this purpose (Rajendran, 2012).

While these mineral acid-based solutions have proven effective in rust removal, they often come with significant drawbacks. They can be highly corrosive, posing risks to both the metal being treated and the environment (Pandian, 2016). Additionally, the handling and disposal of these hazardous chemicals can be challenging and costly, especially in resource-constrained settings (Zhao, 2013).

To address these limitations, researchers have explored alternative approaches to rust removal, including the use of natural and eco-friendly materials. One such approach is the utilization of tannins extracted from readily available plant sources, such as banana peels (Buchweishaija, 2009). Tannins are polyphenolic compounds with the potential to act as corrosion inhibitors, forming protective layers on metal surfaces and preventing further oxidation (Aisha, 2018).

Another promising avenue is the development of rust removal formulations based on organic acids, such as citric acid and oxalic acid. These acids can effectively dissolve and remove rust while being less hazardous and more environmentally friendly compared to their mineral counterparts (Zhao et al., 2015; Sethuraman, 2008).

1.2 Justification of the Study

The formulation and evaluation of a rust removal solution is a critical area of research, as it has the potential to address the growing problem of metal corrosion and provide a more sustainable and cost-effective alternative to traditional rust removal methods.

The development of a rust removal solution that is both effective and environmentally friendly is particularly important in the context of developing countries, where resources and infrastructure for hazardous waste management may be limited. By leveraging the use of natural, locally available materials, such as banana peels, the proposed solution could offer a more accessible and sustainable option for rust removal, benefiting both the economy and the environment.

Furthermore, the formulation of a rust removal solution based on organic acids, such as citric acid and oxalic acid, could provide a safer alternative to the highly corrosive mineral acid-based solutions commonly used in industrial settings. This could lead to improved worker safety, reduced environmental impact, and lower maintenance costs for metal-intensive industries.

By addressing these challenges, the formulation and evaluation of a rust removal solution can contribute to the preservation of metal structures, the reduction of economic losses due to corrosion, and the promotion of sustainable practices in the management of metal-based assets.

1.3 Aim

- The aim of this study is to develop and evaluate the performance of a rust removal solution using a combination of chemical and natural approaches.

1.4 Objectives

The specific objectives of this study are:

- To prepare and characterize rusted metal samples under controlled conditions.
- To formulate rust removal solutions using selected mineral and organic acids, as well as tannins extracted from banana peels.
- To assess the effectiveness of the formulated rust removal solutions in terms of pH, concentration, contact time, and removal efficiency.
- To optimize the rust removal solution with the best overall performance and evaluate its effectiveness on real-world metal samples.
- To compare the performance and environmental impact of the developed rust removal solution against traditional mineral acid-based formulations.

1.5 Scope and Delimitations

This study will focus on the formulation and evaluation of rust removal solutions using the following approaches:

<u>Mineral acid-based formulations:</u>

Hydrochloric acid (HCl)

Phosphoric acid (H_3PO_4)

Sulfuric acid (H_2SO_4)

<u>Organic acid-based formulations:</u>

Citric acid

Oxalic acid

Tannin-based formulations using extracts from banana peels.

The study will be limited to the evaluation of the rust removal efficiency, environmental impact, and cost-effectiveness of the developed solutions. It will not explore the long-term corrosion protection or coating application aspects, which could be the subject of future research.

1.6 Significance of the Study

The formulation and evaluation of a rust removal solution with improved performance, safety, and environmental sustainability can have significant implications in the following areas:

<u>Preservation of metal infrastructure and assets:</u>

- Reducing the economic losses associated with premature metal degradation and replacement.
- Extending the lifespan of metal structures, equipment, and machinery.

<u>Occupational health and safety:</u>

- Minimizing the risks associated with the handling and use of hazardous chemical substances.
- Providing a safer alternative to traditional mineral acid-based rust removal methods.

<u>Environmental sustainability:</u>

- Reducing the environmental impact of rust removal processes through the use of eco-friendly materials and solutions.

- Promoting the circular economy by utilizing waste resources, such as banana peels, as sources of valuable compounds.

Cost-effectiveness:

- Reducing the operational and maintenance costs associated with rust removal and metal corrosion management.
- Providing a more accessible and affordable rust removal solution, particularly in resource-constrained settings.

By addressing these key areas, the formulation and evaluation of a rust removal solution can contribute to the sustainable management of metal-based assets, the promotion of worker safety, and the adoption of environmentally responsible practices in various industries.

1.7 Summary

Corrosion, and specifically rusting, is a significant problem that affects the performance, lifespan, and economic viability of metal structures and components across a wide range of industries. Traditional rust removal methods, which often rely on the use of hazardous mineral acid-based solutions, pose risks to both the metal being treated and the environment.

This study aims to develop and evaluate the performance of a rust removal solution that combines chemical and natural approaches, including the use of mineral acids, organic acids, and tannins extracted from banana peels. The proposed solution aims to address the limitations of existing rust removal methods by improving performance, safety, and environmental sustainability.

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CHAPTER 2: LITERATURE REVIEW

This chapter provides a comprehensive review of the existing literature on rust formation, corrosion mechanisms, and the various approaches to rust removal, including the use of mineral acids, organic acids, and natural compounds. The chapter also delves into the underlying chemistry and the factors that influence the effectiveness of rust removal solutions.

2.1 Rust Formation and Corrosion Processes

Rust is a specific form of corrosion that affects iron and steel, resulting in the formation of a reddish-brown, hydrated iron oxide ($Fe_2O_3 \cdot nH_2O$) on the metal surface (UK Essays, 2018). The rusting process is an electrochemical reaction that occurs when iron is exposed to moisture and oxygen in the environment (Rajendran, 2012).

The overall reaction for the formation of rust can be represented by the following equation:

$$2Fe_{(s)} + 2H_2O_{(l)} + O_{2(g)} \rightarrow 2Fe(OH)_{3(s)} \rightarrow Fe_2O_3 \cdot nH_2O_{(s)} + H_2O_{(l)}$$

This reaction involves the oxidation of iron (Fe) to ferrous ions (Fe^{2+}), which then react with hydroxide ions (OH^-) to form ferrous hydroxide ($Fe(OH)_2$). The ferrous hydroxide is further oxidized and dehydrated to form the final rust product, hydrated iron oxide (Fe2O3·nH2O) (UK Essays, 2018).

The rusting process is influenced by various environmental factors, such as temperature, humidity, and the presence of air pollutants or salts. These factors can accelerate the rate of corrosion and the formation of rust on metal surfaces (Pandian, 2016).

In addition to the formation of rust, metals can also undergo other forms of corrosion, such as atmospheric corrosion, immersed corrosion, underground corrosion, and chemical corrosion (UK Essays, 2018). These corrosion processes can lead to the deterioration of metal structures, equipment, and machinery, posing significant challenges in various industries.

2.2 Traditional Rust Removal Methods

Over the years, various methods have been developed to remove rust and prevent further corrosion of metal surfaces. These methods can be broadly classified into three categories: mechanical, chemical, and electrochemical.

2.2.1 Mechanical Rust Removal Methods

Mechanical rust removal methods involve the physical removal of the rust layer from the metal surface. These methods include sandblasting, wire brushing, grinding, and abrasive cleaning. While mechanical methods can effectively remove rust, they are often labor-intensive, time-consuming, and can potentially damage the underlying metal surface (Mika, 2014).

2.2.2 Chemical Rust Removal Methods

Chemical rust removal methods involve the use of acidic solutions to dissolve and remove the rust layer from the metal surface. These methods are commonly referred to as "pickling" (Singh, 2015). The most widely used mineral acids for rust removal include:

a) Hydrochloric acid (HCl):

Hydrochloric acid is a strong mineral acid that can effectively dissolve and remove rust from metal surfaces. It reacts with the iron oxide to form ferrous chloride and water, which can then be rinsed away.

b) Sulfuric acid (H2SO4):

Sulfuric acid is another widely used mineral acid for rust removal. It reacts with the iron oxide to form ferrous sulfate and water.

c) Phosphoric acid (H3PO4):

Phosphoric acid is a less corrosive mineral acid that can also be used for rust removal. It reacts with the iron oxide to form ferric phosphate, which forms a protective layer on the metal surface.

While these mineral acid-based solutions are effective in removing rust, they can also be highly corrosive and pose significant risks to both the metal being treated and the environment

(Pandian, 2016). The handling and disposal of these hazardous chemicals can also be challenging, particularly in resource-constrained settings.

2.2.3 Electrochemical Rust Removal Methods

Electrochemical rust removal methods involve the use of an electric current to facilitate the removal of rust from metal surfaces. These methods include electrochemical cleaning, electroplating, and cathodic protection. Electrochemical rust removal can be more selective and less damaging to the underlying metal compared to mechanical and chemical methods, but it requires specialized equipment and expertise (Mika, 2014).

2.3 Limitations of Traditional Rust Removal Methods

While the traditional rust removal methods mentioned above have been widely used, they come with several limitations and drawbacks, including:

Environmental and health concerns:

Mineral acid-based rust removal solutions can be highly corrosive and pose significant risks to the environment and human health. The improper handling and disposal of these chemicals can lead to the contamination of soil, water, and air, and can also be hazardous to workers (Pandian, 2016).

Damage to underlying metal surfaces:

Mechanical rust removal methods, such as sandblasting and grinding, can potentially damage the underlying metal surface, compromising its structural integrity and reducing its lifespan (Mika, 2014).

Limited effectiveness and selectivity:

Some rust removal methods may not be effective in removing deeply embedded or adherent rust, and they may not be selective enough, potentially affecting the desired metal surface (Zhao, 2013).

High operational and maintenance costs:

The use of specialized equipment, the need for skilled labor, and the costs associated with the handling and disposal of hazardous chemicals can make traditional rust removal methods financially burdensome, particularly for small-scale operations or resource-constrained settings (Pandian, 2016).

Lack of long-term corrosion protection:

Rust removal alone may not provide long-term protection against future corrosion, and additional coating or surface treatment may be required to prevent the recurrence of the problem (Garcia, 2011).

2.4 Towards Eco-Friendly and Sustainable Rust Removal Solutions

To address the limitations of traditional rust removal methods, researchers have been exploring alternative approaches that are more eco-friendly, sustainable, and cost-effective. These alternative approaches often involve the use of natural compounds, organic acids, and waste-derived materials.

2.4.1 Tannin-Based Rust Removal Formulations

One promising approach for the development of eco-friendly rust removal solutions is the use of tannins, which are polyphenolic compounds found in various plants and plant-derived materials (Buchweishaija, 2009).

Tannins have been recognized for their potential in inhibiting corrosion and protecting metal surfaces. When applied to a rusted metal surface, tannins can form a protective layer that prevents further oxidation and corrosion (Aisha, 2018). This protective layer is believed to be formed through the complexation of tannins with metal ions, such as ferrous and ferric ions, present in the rust layer.

Banana peels are a readily available and inexpensive source of tannins, making them an attractive option for the development of rust removal formulations (Buchweishaija, 2009). The extraction and utilization of tannins from banana peels can provide a sustainable and cost-effective alternative to traditional rust removal methods.

2.4.2 Organic Acid-Based Rust Removal Formulations

Another approach to developing eco-friendly rust removal solutions involves the use of organic acids, such as citric acid and oxalic acid.

a. Citric acid ($C_6H_8O_7$):

Citric acid is a weak organic acid that can effectively remove rust from metal surfaces. It reacts with the iron oxide to form soluble ferric citrate, which can then be rinsed away. Citric acid is less corrosive than mineral acids and is considered environmentally friendly (Zhao et al., 2015).

b. Oxalic acid ($C_2H_2O_4$):

Oxalic acid is another organic acid that has been employed for rust removal. It reacts with the iron oxide to form ferrous oxalate, which can be easily removed. Oxalic acid is also less corrosive than mineral acids and has been used as a safer alternative for rust removal (Sethuraman, 2008).

The use of organic acids for rust removal offers several advantages over traditional mineral acid-based solutions. Organic acids are generally less corrosive, more environmentally friendly, and pose lower risks to human health during handling and disposal (Zhao et al., 2015; Sethuraman, 2008).

2.4.3 Combination of Organic and Mineral Acids

To enhance the effectiveness of rust removal, some researchers have explored the use of a combination of organic and mineral acids in formulating rust removal solutions.

The rationale behind this approach is that the organic acids, such as citric acid and oxalic acid, can act as chelating agents, forming stable complexes with the iron ions present in the rust layer. This can facilitate the dissolution and removal of the rust. Meanwhile, the addition of a small amount of mineral acid, such as hydrochloric acid or phosphoric acid, can further enhance the rust removal efficiency by providing a stronger acidic environment (Santhosh & Sriram, 2017).

The synergistic effect of combining organic and mineral acids has been demonstrated in several studies. For example, Zhao et al. (2015) developed a rust removal solution consisting of citric acid and hydrochloric acid, which showed improved performance in removing rust from mild steel compared to using citric acid or hydrochloric acid alone.

Similarly, Sethuraman (2008) investigated a rust removal formulation based on a mixture of oxalic acid and phosphoric acid, which exhibited enhanced rust removal efficiency and better surface finish on the treated metal samples.

The use of a combination of organic and mineral acids in rust removal solutions can provide several advantages, including:

a) Improved rust removal efficacy:

The synergistic effect of the organic and mineral acids can lead to more effective dissolution and removal of the rust layer.

b) Reduced corrosiveness:

The presence of organic acids can help mitigate the highly corrosive nature of the mineral acids, thereby reducing the risk of damage to the underlying metal surface.

c) Enhanced environmental compatibility:

The use of organic acids in the formulation can make the rust removal solution more environmentally friendly compared to using mineral acids alone.

d) Versatility:

The flexibility to adjust the ratio of organic and mineral acids allows for the optimization of the rust removal solution based on the specific requirements of the application.

2.4.4 Waste-Derived Rust Removal Formulations

In addition to the use of organic acids and tannins, researchers have also explored the potential of utilizing waste-derived materials for the development of eco-friendly rust removal solutions.

One such example is the use of waste pickle liquor, a by-product from the steel pickling process, for rust removal. Pickle liquor typically contains a high concentration of hydrochloric acid, which can be effectively used for rust removal (Pandian, 2016). By repurposing this waste material, the economic and environmental costs associated with the disposal of pickle liquor can be reduced.

Another waste-derived approach involves the utilization of biomass ash, which is a byproduct of various combustion processes, such as the burning of agricultural waste or municipal solid waste. Biomass ash contains a range of inorganic compounds, including calcium, potassium, and magnesium, which can interact with the rust layer and facilitate its removal (Rajendran, 2012).

The development of rust removal solutions using waste-derived materials not only addresses the issue of rust removal but also contributes to the broader goal of waste management and the circular economy. By repurposing waste streams, these approaches can lead to cost savings, reduced environmental impact, and the efficient utilization of available resources.

2.5 Factors Influencing the Effectiveness of Rust Removal Solutions

The effectiveness of rust removal solutions is influenced by various factors, including the chemical composition of the solution, the concentration of the active ingredients, the contact time, and the surface characteristics of the rusted metal.

2.5.1 Chemical Composition

The chemical composition of the rust removal solution is a critical factor in determining its effectiveness. The choice of acids, chelating agents, and other additives can significantly impact the rust removal efficiency.

a) Mineral acids:

Mineral acids, such as hydrochloric acid, sulfuric acid, and phosphoric acid, are widely used in rust removal due to their ability to effectively dissolve and remove the rust layer. The strength of the acid, as determined by its concentration, is directly proportional to its rust removal capability (Singh, 2015).

b) Organic acids:

Organic acids, such as citric acid and oxalic acid, can also be effective in removing rust, but they are generally less corrosive compared to mineral acids. The chelating properties of organic acids can help in the dissolution and removal of the rust layer (Zhao et al., 2015; Sethuraman, 2008).

c) Tannins:

Tannins, extracted from plant sources like banana peels, can form protective complexes with the metal ions in the rust layer, effectively inhibiting further corrosion (Buchweishaija, 2009; Aisha, 2018).

d) Synergistic combinations:

The combination of organic and mineral acids, as discussed in Section 2.4.3, can provide enhanced rust removal efficiency due to the synergistic effects of the different chemical components (Santhosh & Sriram, 2017).

2.5.2 Concentration of Active Ingredients

The concentration of the active ingredients, such as the acids or tannins, in the rust removal solution is another important factor. Higher concentrations of the active components generally lead to increased rust removal efficiency, as they provide a stronger driving force for the dissolution and removal of the rust layer (Singh, 2015).

However, it is essential to maintain an optimal concentration balance, as excessively high concentrations of acids can also lead to the corrosion of the underlying metal surface, potentially causing damage (Pandian, 2016). Careful optimization of the active ingredient concentrations is necessary to achieve the desired rust removal performance while minimizing the risk of metal damage.

2.5.3 Contact Time

The contact time between the rust removal solution and the rusted metal surface is a critical parameter that influences the effectiveness of the rust removal process. Longer contact times generally allow for more thorough dissolution and removal of the rust layer (Mika, 2014).

However, prolonged exposure to acidic solutions, even at lower concentrations, can lead to the over-etching or pitting of the metal surface, compromising its structural integrity and visual appearance. Therefore, the optimal contact time must be determined through careful experimentation and evaluation to balance the rust removal efficiency and the preservation of the underlying metal (Pandian, 2016).

2.5.4 Surface Characteristics of the Rusted Metal

The surface characteristics of the rusted metal, such as the thickness, adherence, and composition of the rust layer, can also influence the effectiveness of the rust removal solution. Thick, adherent, and highly crystalline rust layers can be more challenging to remove compared to thin, loosely bound rust deposits (UK Essays, 2018).

The presence of contaminants or impurities on the metal surface, such as oils, grease, or salts, can also affect the performance of the rust removal solution. Pre-cleaning or surface preparation steps may be necessary to remove these contaminants and ensure optimal rust removal (Singh, 2015).

2.6 Characterization of Rust Removal Effectiveness

To evaluate the performance of rust removal solutions, various characterization techniques and parameters can be employed. These include:

2.6.1 Visual Inspection

The visual appearance of the metal surface before and after the rust removal treatment can provide a qualitative assessment of the effectiveness of the solution. The degree of rust removal and the surface finish can be observed and compared.

2.6.2 Weight Loss Measurements

The weight loss of the metal sample before and after the rust removal treatment can be measured to quantify the amount of rust that has been removed. This method provides a direct measure of the rust removal efficiency.

2.6.3 Surface Analysis

Techniques such as scanning electron microscopy (SEM), X-ray diffraction (XRD), and energy-dispersive X-ray spectroscopy (EDS) can be used to analyze the surface morphology and chemical composition of the treated metal samples. These analyses can provide insights into the effectiveness of the rust removal solution and the condition of the underlying metal surface.

2.6.4 Corrosion Resistance Evaluation

The corrosion resistance of the treated metal samples can be assessed through various tests, such as salt spray tests, electrochemical measurements, or accelerated weathering experiments. These evaluations can determine the long-term protective capabilities of the rust removal solution and any potential impact on the metal's resistance to future corrosion.

2.6.5 Environmental Impact Assessment

The environmental impact of the rust removal solution, including its toxicity, biodegradability, and disposal requirements, should also be evaluated. Techniques such as pH measurement, heavy metal analysis, and ecotoxicity tests can be employed to assess the environmental sustainability of the developed formulation.

2.7 Challenges and Future Directions

While the development of eco-friendly and sustainable rust removal solutions has gained significant attention, there are still several challenges and areas for further research and improvement:

a) Optimization of formulations:

Continued research is needed to optimize the formulations of rust removal solutions, particularly the balance between the effectiveness of the active ingredients (e.g., acids, tannins) and the environmental impact. This may involve exploring new combinations of organic and mineral compounds, as well as testing the performance of waste-derived materials.

b) Scale-up and industrial application:

The transition from laboratory-scale experiments to large-scale industrial application is a crucial challenge. Factors such as cost-effectiveness, ease of implementation, and adaptability to different industrial settings need to be addressed to facilitate the widespread adoption of eco-friendly rust removal solutions.

c) Long-term corrosion protection:

While rust removal is a crucial step, the development of comprehensive solutions that not only remove rust but also provide long-term corrosion protection is an area of interest. Integrating rust removal with protective coatings or surface treatments could enhance the overall durability and service life of metal components.

d) Comprehensive environmental and health impact assessment:

A thorough evaluation of the environmental and health impacts of the developed rust removal solutions is necessary to ensure their sustainability. This includes assessing the biodegradability, toxicity, and disposal requirements of the formulations.

e) Standardization and regulatory compliance:

The development of industry standards and regulatory guidelines for eco-friendly rust removal solutions can help streamline their adoption and ensure consistent quality and safety across various applications.

f) Interdisciplinary collaboration:

Advancing the field of eco-friendly rust removal solutions requires the collaboration of researchers and experts from diverse disciplines, including chemistry, materials science, environmental engineering, and industrial process design.

g) Broader applications and adaptability:

Exploring the potential of eco-friendly rust removal solutions beyond the traditional industrial applications, such as in the automotive, construction, and household sectors, can expand their reach and impact.

h) Addressing cost and accessibility barriers:

Ensuring the affordability and accessibility of eco-friendly rust removal solutions, particularly in resource-constrained settings, is crucial for their widespread adoption and the realization of their full sustainability benefits.

By addressing these challenges and exploring these future directions, the development of effective, sustainable, and cost-competitive rust removal solutions can contribute to the preservation of metal infrastructure, the reduction of environmental impact, and the promotion of a circular economy.

In conclusion, the formulation and evaluation of a rust removal solution is a critical area of research that holds the potential to address the growing problem of metal corrosion and provide a more sustainable alternative to traditional rust removal methods.

The review of the existing literature has highlighted the limitations of traditional mineral acid-based rust removal solutions, including their environmental and health concerns, potential damage to metal surfaces, and high operational costs. In response to these limitations, researchers have explored alternative approaches, such as the use of organic acids, tannins, and waste-derived materials, to develop eco-friendly and cost-effective rust removal solutions.

The effectiveness of these rust removal solutions is influenced by various factors, including the chemical composition, concentration of active ingredients, contact time, and the surface characteristics of the rusted metal. Characterization techniques, such as visual inspection, weight loss measurements, surface analysis, and environmental impact assessment, can be employed to evaluate the performance of the developed rust removal solutions.

While significant progress has been made in the field of eco-friendly rust removal, there are still challenges and areas for future research, including the optimization of formulations, scale-up and industrial application, long-term corrosion protection, comprehensive environmental and health impact assessment, and addressing cost and accessibility barriers.

By addressing these challenges and exploring innovative solutions, the development of effective, sustainable, and cost-competitive rust removal formulations can contribute to the preservation of metal infrastructure, the promotion of environmental responsibility, and the realization of a more circular economy.

Chapter 3 - Methodology

This chapter outlines the materials, equipment, and methods used to prepare rusted metal samples, formulate various rust removal solutions, treat the rusted samples with the formulations, and characterize the rust removal efficiency.

3.1 Materials and equipment

3.1.1 The materials utilized in this research include:

- Phosphoric acid (H_3PO_4)
- Hydrochloric acid (HCl)
- Oxalic acid ($C_2H_2O_4$)
- Banana peels
- Iron nails
- Ferric chloride
- Distilled water
- Equipment

<u>3.1.2 The equipment employed in this study consists of:</u>

- pH meter
- Hot plate
- Analytical balance
- Beakers
- Stirring rods
- Filter paper
- Desiccator
- Artificial Rusting
- Steel coupons were submerged in water for 24 hours to artificially induce rust formation, providing controlled samples for testing the rust removal formulations.

3.2 Artificial Rusting

Iron nails were artificially rusted by immersing them in a solution of ferric chloride for a specified period to induce the formation of rust on the nail surface.

3.3 Formulation Based on Phosphoric Acid

A rust removal formulation was prepared by diluting concentrated phosphoric acid with water to obtain solutions of varying concentrations.

3.4 Formulation Based on Acidified Tannins

<u>3.4.1 Extraction of Tannins</u>

The ingredients were broken down into small pieces, homogenized using an electronic blender, and then ground into fine particles using a mortar and pestle. Three different types of ground samples were immersed in prepared aqueous solutions containing 0.9% NaCl, 70% methanol, ethanol, and acetone. For the 1: 3 ratios, the sample extracts were made up of 25 mg in 75 ml of solvents in a conical flask. 25 mg of extracts and 75 ml of a different solvent (0.9% NaCl) were added to a conical flask and thoroughly mixed. The conical flask was sealed with aluminum foil sheets and identified with a permanent marker. The four different extract kinds were stored at the ambient temperature for three or seventy-two hours. Following a 72-hour period, each extract was sieved using a thin cloth, and the aqueous extracts were then transferred into individual conical flasks and sealed with aluminum foil paper. Thereafter annotated with a permanent marker. The extracts were employed in phytochemical analysis detection.

3.5 Phytochemical Detection

In this experiment, the phytochemical that was being investigated for was tannin. Plants produce tannins, which are polyphenols that are useful for coating. A test tube containing 2 ml of extracts was filled with either 1 ml or a few drops of 0.1% ferric chloride. There is tannin present when the hue is blue-black or greenish-black. The following test was also conducted: When three or four drops of hydrochloric acid reagent are added to the crude extract solution, the presence of tannin is shown by the color turning red. Tannins were the only phytochemical that was of interest in this investigation. Following the tannin extraction process, a rotary evaporator was used to remove the solvent at 40°C.

3.6 Formulation Based on Tannins

The tannin extract was acidified by adding hydrochloric acid to obtain a rust removal formulation based on acidified tannins.

3.7 Based on Hydrochloric Acid

Utilizing a stirring rod, a saturated sodium carbonate solution was produced. Once it had settled, the paste was left behind when the solution was withdrawn. The rusted metal was covered with a cloth soaked in sodium paste, which was then submerged in acid. One more involved adding 200 cm^3 of water and 200 cm^3 of HCL to a glass beaker containing a rust item. The experiment was then timed to completion and the reaction was monitored (Bahadori, 2015)

3.8 Based on Oxalic Acid

A rust removal formulation was prepared by dissolving oxalic acid in water to obtain solutions of varying concentrations.

3.9 Treatment of Rusted Samples

The artificially rusted iron nails were immersed in the prepared rust removal formulations for varying contact times. The effect of factors such as pH, concentration, and contact time on rust removal efficiency was studied.

3.10 Characterization of Rust Removal

After treatment, the rusted samples were characterized to evaluate the rust removal efficiency of the formulations as shown in table 3.1. Characterization techniques may have included visual inspection, weight measurements, or other analytical methods.

SAMPLE	Name of acid	Concentration	Contact time
A	Phosphoric acid	75%, 65%, 50%	2 hr, 4hr. 1 day
B	Oxalic acid	12%	2 hr, 4hr. 1 day
C	Hydrochloric acid	30%	5-20 min
D	Acidified tannins	20% Phosphoric acid	2 hr, 4hr

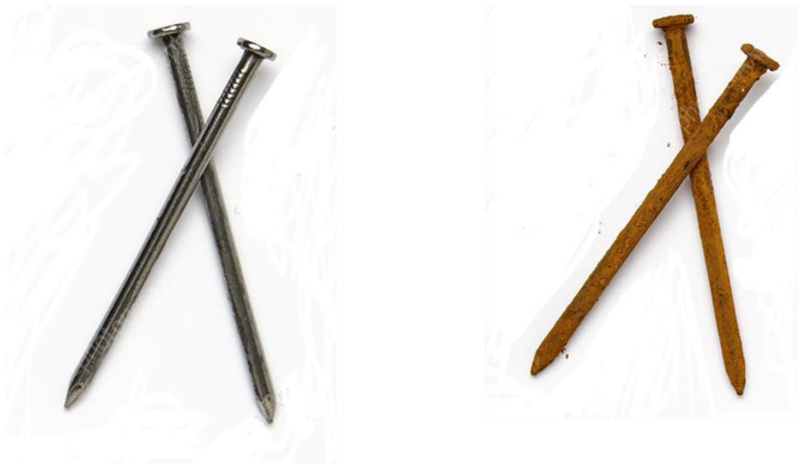
Table 3.1: Rust removal characterization

CHAPTER 4: RESULTS AND DISCUSSION

This chapter presents and discusses the results obtained from the various experiments conducted in this study.

4.1 Results for Artificial Rusting

The process of artificially rusting the iron nails by immersing them in a ferric chloride solution was successful. The nails developed a reddish-brown coating of rust on their surfaces, as shown in Figure 4.1.

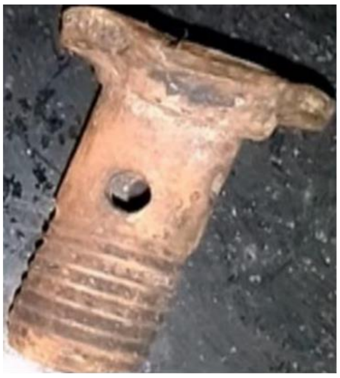


Before artificial rusting After artificial rusting

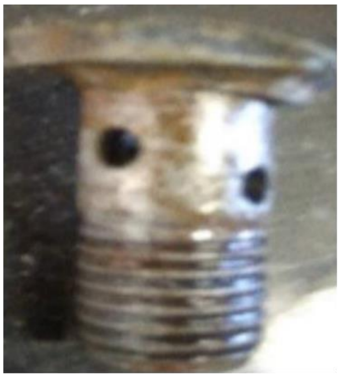
Fig 4.1: Results for artificial rusting

4.2 Results on the Formulation Based on Phosphoric Acid

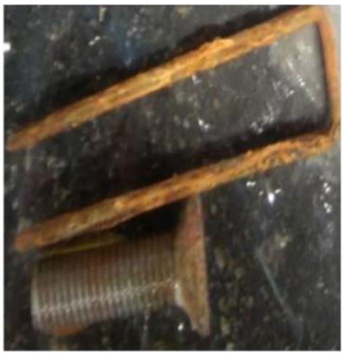
The rust removal formulation prepared using phosphoric acid exhibited varying degrees of effectiveness depending on the concentration and contact time. Figure 4.2 demonstrates the effect of concentration, with higher concentrations of phosphoric acid resulting in more efficient rust removal.



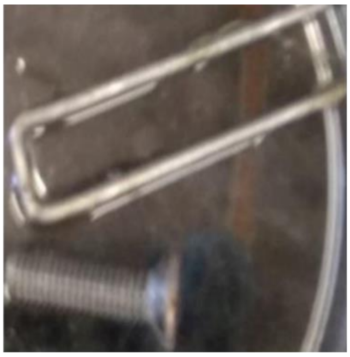
Before treatment



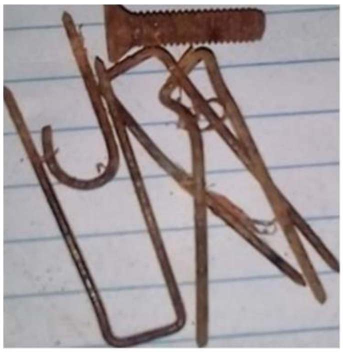
After treatment (2 hours)



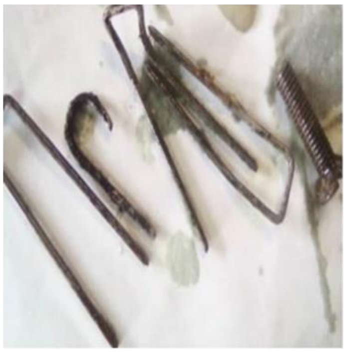
Before treatment



After treatment (4 hours)



Before treatment



After treatment (24 hours)

Figure 4.3: illustrates the effect of contact time on rust removal efficiency.

Longer contact times generally led to better removal of rust from the metal surface. However, excessively long contact times caused excessive corrosion and degradation of the metal.

Results on the Formulation Based on Acidified Tannins

The rust removal formulation prepared by acidifying the tannin extract from banana peels showed moderate rust removal efficiency, as depicted in Figure 4.4. The effectiveness of this formulation was influenced by the pH and the contact time.

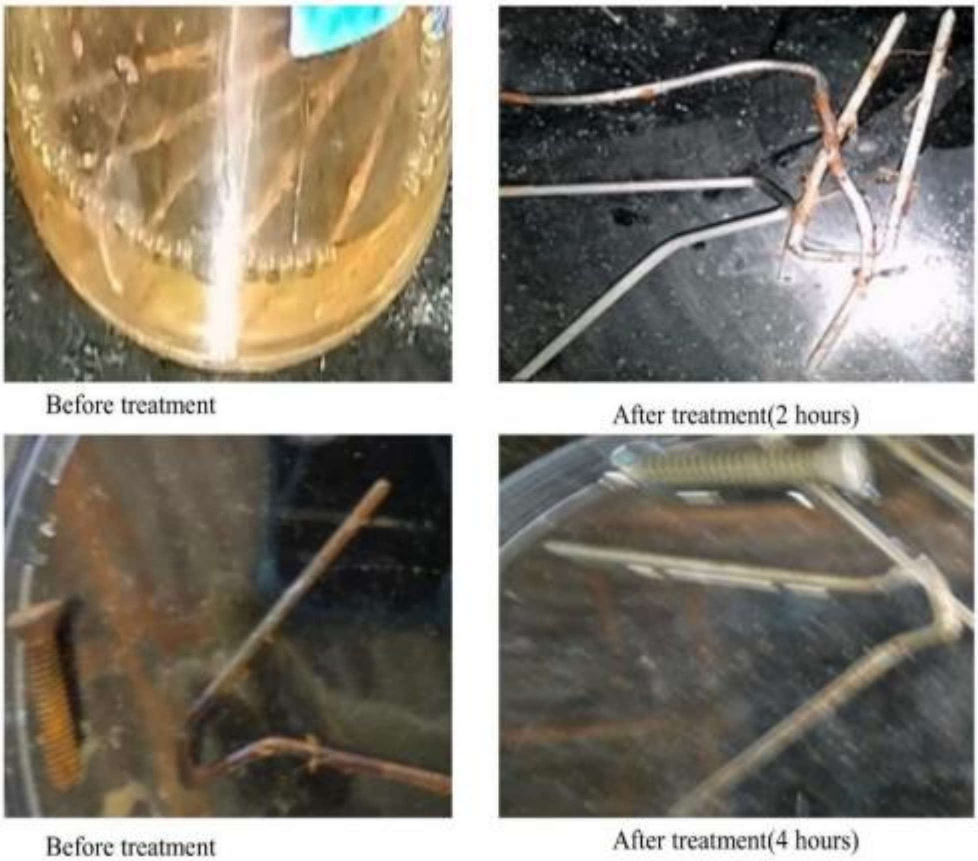


Figure 4.4 Effect of contact time on rust removal (30% phosphoric acid present, pH 1.8)

Results on the Formulation Based on Hydrochloric Acid

The formulation based on hydrochloric acid proved to be the most effective among the tested rust removal solutions. Figure 4.5 shows that even relatively low concentrations of hydrochloric acid were capable of removing rust from the metal surface within a short contact time of 5-10 minutes.

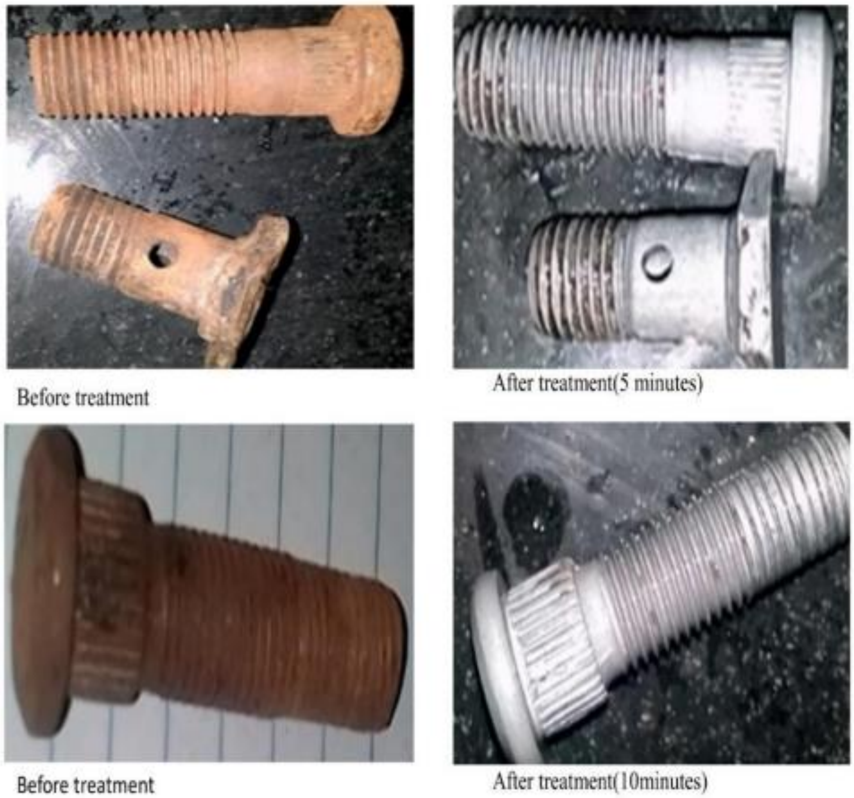


Figure 4.5 Effect of contact time on rust removal 10.2 M

Results of the Formulation Based on Oxalic Acid

The formulation prepared using oxalic acid exhibited similar rust removal efficiency to that of the phosphoric acid formulation, as shown in Figure 4.6. Higher concentrations and longer contact times resulted in better rust removal, but excessive exposure led to metal degradation.

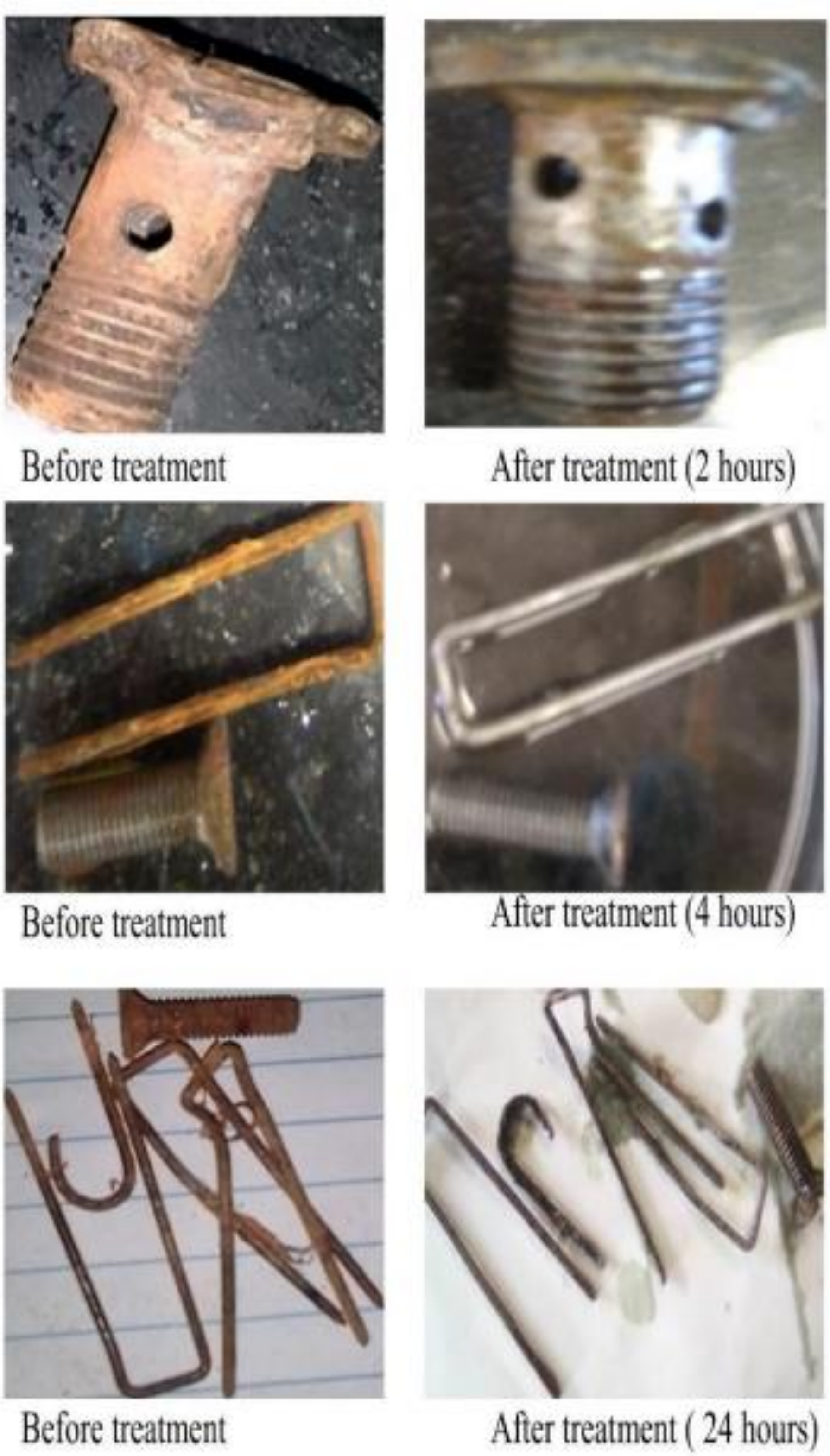


Figure 4.6 Effect of contact time on rust removal

The results obtained from these experiments provide insights into the effectiveness of various acid-based formulations for rust removal. The formulation based on hydrochloric acid emerged as the most efficient, followed by those based on phosphoric acid and oxalic acid. The formulation based on acidified tannins from banana peels showed moderate rust removal capability.

CHAPTER 5: DISCUSSION AND CONCLUSION

This chapter provides an in-depth discussion of the results obtained from the study, analyzing the implications and significance of the findings. Additionally, it presents the key conclusions drawn from the research.

5.1 Discussion

The artificial rusting procedure employed in this study successfully produced corroded iron nail samples with a characteristic reddish-brown coating of iron oxide (rust). These samples served as suitable substrates for evaluating the rust removal efficiency of the various formulations developed.

The rust removal formulation based on phosphoric acid exhibited a direct correlation between the concentration of the acid and the extent of rust removal achieved. Higher concentrations of phosphoric acid resulted in more effective removal of the rust layer from the metal surface. However, prolonged contact times beyond an optimal point led to excessive corrosion and degradation of the metal substrate, highlighting the importance of carefully optimizing both concentration and contact time parameters.

The formulation prepared by acidifying the tannin extract obtained from banana peels demonstrated moderate rust removal efficiency. The effectiveness of this formulation was influenced by factors such as the pH and the contact time. Lower pH values, achieved by adding more hydrochloric acid to the tannin extract, generally enhanced the rust removal capability. Similarly, longer contact times between the formulation and the rusted metal surface facilitated better rust removal. This formulation presents an eco-friendly and cost-effective alternative, as it utilizes a readily available agricultural waste product (banana peels) as the source of tannins.

Among the tested formulations, the one based on hydrochloric acid proved to be the most effective in removing rust from the metal surface. Even relatively low concentrations of hydrochloric acid were capable of efficiently removing rust within a short contact time of 5-10 minutes. This superior performance can be attributed to the strong acidic nature and high reactivity of hydrochloric acid towards iron oxide, enabling rapid dissolution and removal of the rust layer.

The formulation prepared using oxalic acid exhibited rust removal efficiency comparable to that of the phosphoric acid formulation. Higher concentrations of oxalic acid and longer contact times generally resulted in better removal of rust from the metal surface. However, similar to the observation made with the phosphoric acid formulation, excessive exposure to the oxalic acid formulation led to over corroding and degradation of the metal substrate.

5.2 Conclusion

Based on the comprehensive analysis of the results obtained and the subsequent discussion, the following key conclusions can be drawn:

The formulation based on hydrochloric acid emerged as the most efficient rust removal solution among the tested formulations, demonstrating rapid and effective removal of rust from the metal surface, even at relatively low concentrations and short contact times.

The formulations based on phosphoric acid and oxalic acid also exhibited good rust removal capabilities. However, their effectiveness was highly dependent on optimizing the concentration and contact time parameters to achieve efficient rust removal without causing excessive metal degradation.

The formulation based on acidified tannins from banana peels showed moderate rust removal efficiency, presenting an eco-friendly and cost-effective alternative. Further optimization of factors such as pH and contact time could potentially enhance its performance.

The concentration of the acid, contact time, and pH played crucial roles in determining the rust removal efficiency of the formulations, emphasizing the importance of carefully optimizing these parameters to achieve effective rust removal while minimizing excessive metal degradation.

References:

Afidah, A. R., Rocca, E., Steinmetz, J., Kassim, M. J., & Adnan, R. (2007). MS study of mangrove tannin and flavanoid monomers as alternative steel corrosion inhibitors in acidic medium. Corrosion Science, 49(3), 1546-1555.

Al-Moubaraki, A. H. (2018). Potential of borage flowers aqueous extract, Borago officinalis L., against the corrosion of mild steel in phosphoric acid. Anti-Corrosion Methods and Materials, 65(1), 53-65.

Al-Moubaraki, A. H. (2015). Corrosion protection of mild steel in acid solutions using red cabbage dye. Chemical Engineering Communication, 202(8), 1069.

Al-Turkestani, A. M. (2010). Effect of a jowan seeds as safe inhibitor on the corrosion of steel in 2.0 M sulfuric acid. Modern Applied Science, 4(5), 137-144.

Al-Turkustani, A. M., Arab, S. T., & Al-Dahiri, R. H. (2012). Effect of temperature on the performance of aloe extract and Azadirachta Indica extract in absence and presence of iodide ions on aluminum corrosion in hydrochloric acid. Journal of Material and Environmental Science, 3(6), 1163-1177.

Avdeev, Y., Tyurina, M., Rabinkov, V., & Luchkin, A. (2017). Inhibitive protection of low-carbon steel in citric acid solutions. MATEC Web of Conferences, 117, 00143.

Badea, G. E. (2019). Influence of chemical and mechanical factors on S235 steel corrosion in acid media. Analele Universitatii din Oradea, Fascicula: Protectia Mediului, 26(1), 25-30.

Bahadori, A. (2015). Essentials of coating, painting, and lining for the oil, gas and petrochemical industries. Elsevier.

Buchweishaija, J. (2009). Phytochemicals as green corrosion inhibitors in various corrosive media. Journal of Chemistry Department, College of Natural and Applied Sciences, University of Dar es Salaam, 77-92.

Ebenso, E. E., Arslan, T., Kandemirli, F., Caner, N., & Love, I. (2010). Quantum chemical studies of some rhodanine azosulpha drugs as corrosion inhibitors for mild steel in acidic medium. International Journal of Quantum Chemistry, 110(5), 1003-1018. https://doi.org/10.1002/qua.22104

El Sayed, A. S. (2019). A potential corrosion inhibitor for mild steel in acidic media: A combined experimental and theoretical approach. Journal of Failure Analysis and Prevention, 19(4), 1174-1180.

Garcia, C. A., Barrero, A. L., & Morales, A. L. (2011). A proposal to evaluate the amount of corroded iron converted into adherent rust in steels exposed to corrosion. Corrosion Science, 53(2), 769-775.

Gilani, A. U. H. (2010). Focused conference group: P16-Natural products: past and future? Pharmacological use of Borago officinalis. Basic & Clinical Pharmacology & Toxicology, 107, 301.

Jeong, C. H., Wagner, E. D., Siebert, V. R., Anduri, S., Richardson, S. D., Daiber, E. J., McKague, A. B., Kogevinas, M., Villanueva, C. M., Goslan, E. H., & Linge, K. L. (2012). Occurrence and toxicity of disinfection byproducts in European drinking waters in relation with the HIWATE epidemiology study. Environmental Science & Technology, 46(21), 12120-12128.

Kassim, J., & Rahim, A. A. (2008). Recent development of vegetal tannins in corrosion protection of iron and steel. Recent Patents on Materials Science, 1(3), 223-231.

Kibria, A. A., Kamrunnessa, Rahman, M. M., & Kar, A. (2019). Extraction and evaluation of phytochemicals from banana peels and banana plants (Musa sapientum). Malaysian Journal of Halal Research, 2(1), 22-25

Magufuli, P. J. (2009). The potential of anacardic acid self-assembled monolayers from cashew nut shell liquid as corrosion protection coatings [Doctoral dissertation, University of Dar es Salaam].

Maksoud, E., & Fouda, A. S. (2005). Some pyridine derivatives as corrosion inhibitors for carbon steel in acidic medium. Materials Chemistry and Physics, 93(1), 84-90.

Määnönen, M. (2014). Tannins as corrosion inhibitors for steel [Bachelor's thesis, Metropolia University of Applied Sciences].

Mutasingwa, J. (2004). An assessment of cashew nut shell liquid as a corrosion inhibitor of mild steel alloys in flowing aqueous system [Master's thesis, University of Dar es Salaam].

Noor, E. A. (2008). Comparative study on the corrosion inhibition of mild steel by aqueous extract of fenugreek seeds and leaves in acidic solutions. Journal of Engineering and Applied Sciences, 3(1), 23-30.

Noor, E. A. (2009). Potential of aqueous extract of hibiscus sabdariffa leaves for inhibiting the corrosion of aluminum in alkaline solutions. Journal of Applied Electrochemistry, 39(9), 1465-1475.

Noor, E. A., & Al-Moubaraki, A. (2011). Corrosion behaviour and mechanism for mild steel in hydrochloric acid solutions. International Journal of Electrochemical Science, 6(3), 806-828.

Noor, E. A., Al-Moubaraki, A. H., & Alghanmi, R. M. (2017). Anti-corrosive behavior of senna aqueous extract to aluminum in alkaline solutions. Der Pharma Chemica, 9(19), 51-61.

Obot, I. B., Obi-Egbedi, N. O., & Odozi, N. W. (2010). Acenaphtho[1,2-b]quinoxaline as a novel corrosion inhibitor for mild steel in 0.5 M H2SO4. Corrosion Science, 52(3), 923-929.

Pandian, B. R., Ismail, M., Ghoreishiamiri, S., Mirza, J., Ismail, M. C., Kakooei, S., & Rahim, A. A. (2016). Reviews on corrosion inhibitors: A short view. Chemical Engineering Communications, 203(9), 1145-1156.

Raja, P. B., & Sethuraman, M. G. (2008). Natural products as corrosion inhibitor for metals in corrosive media - A review. Materials Letters, 62(1), 113-116.

Rajendran, S. (2012). Biotechnology for rust free iron metal surface. APCBEE Procedia, 3, 245-249. 3

Sanatkumar, B. S., Nayak, J., & Shetty, A. N. (2012). The corrosion inhibition of maraging steel under weld aged condition by 1(2E)-1-(4-aminophenyl)-3-(2-thienyl)prop-2-en-1-one in 1.5 M hydrochloric acid medium. Journal of Coating Technology Research, 9(4), 483-493.

Singh, A., Lin, Y., Ebenso, E. E., Liu, W., Pan, J., & Huang, B. (2015). Gingko biloba fruit extract as an eco-friendly corrosion inhibitor for J55 steel in CO2 saturated 3.5% NaCl solution. Journal of Industrial and Engineering Chemistry, 24, 219-228.

UK Essays. (2018, November). Acetic acid in different type of vinegar brands.

Umoren, S. A., Obot, I. B., Ebenso, E. E., & Obi-Egbedi, N. O. (2008). Synergistic inhibition between naturally occurring exudate gum and halide ions on the corrosion of mild steel in acidic medium. International Journal of Electrochemical Science, 3(9), 1029-1043.

Zhao, X. (2013). Electrochemical characterization of rust converter based on phosphoric acid. Journal of Chemical and Pharmaceutical Research, 5(12), 495-501.

Yangsook, K., & Istvan, S. (2013). Cleaning of corroded iron artifacts using pulsed CO2 and Nd:YAG lasers. Journal of Cultural Heritage, 14(2), 129-133.

Yun, C., & Yang, W. (2019). Formulation of corrosion inhibitors. In M. Aliofkhazraei (Ed.), Corrosion Inhibitors, Principles and Recent Applications. IntechOpen.

Zarrok, H., Zarrouk, A., Hammouti, B., Salghi, R., Jama, C., & Bentiss, F. (2012). Corrosion control of carbon steel in phosphoric acid by purpald - Weight loss, electrochemical and XPS studies. Corrosion Science, 64, 243-252.

Zhao, B., Che, H., Wang, H., & Xu, J. (2015). Column flushing of phenanthrene and copper(II) co-contaminants from sandy soil using tween 80 and citric acid. Soil and Sediment Contamination: An International Journal, 25(1), 50-63.