OPTIMIZATION OF EXTRACTS AND CONSTITUENTS FROM *ALPINIA GALANGA* AS CORROSION INHIBITOR FOR MILD STEEL IN ACIDIC MEDIUM

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Dedicated to the Glory of Almighty God and to the memories of my late loving parents, Madam Alice Aduke Ajeigbe who I lost during the course of this program and Pa Joseph Ajeigbe Osinkolu that departed when I was barely ten years old.

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ABSTRACT

In terms of environmental impacts and cost considerations, the use of green additives particularly from plant origin have been found as a viable alternative approach to synthetic organic inhibitors in combatting the menace of corrosion. However, owing to the composition matrix complexity of plant extracts, efforts are seldom made to engage their isolated constituents for corrosion inhibition; hence their optimal utilization is hindered. In this research, corrosion inhibition properties of the rhizomes of *Alpinia galanga* and its constituents were investigated experimentally and theoretically on mild steel in hydrochloric acid solution using weight loss and electrochemical methods, and surface characterization techniques namely attenuated total reflection-Fourier transform infrared spectroscopy (ATR-FTIR), scanning electron microscopy (SEM), field emission scanning electron microscopy (FESEM) and energy dispersive X-ray spectroscopy (EDX). Explorations using response surface methodology (RSM) as the optimization tool and quantitative structure-activity relationship (QSAR) modelling of the plant's major phenylpropanoids were carried out. At room temperature, efficiencies were highest at the uppermost concentrations of all the inhibitors in the following order: hexane extract (90.2%), essential oils (87.9%), and methanol extract (74.2%) while for the phenylpropanoid constituents; 1'-acetoxychavicol acetate (84.6%), methyl eugenol (83.6%), eugenol acetate (82.1%), eugenol (76.3%) and p-hydroxycinnamic acid (30.4%). Optimal efficiencies of 90.3% and 91.17% were attained for hexane extract and essential oil components, respectively, at optimized concentration, temperature, and time. Investigations revealed that mixed mode interactions for all the inhibitors and their effectiveness were supported by the surface characterization techniques. Inhibition efficiencies decreased with increasing temperature for all inhibitors except for the essential oil fraction which increased steadily. The Langmuir isotherm model showed the best fit, giving negative values of adsorption energies with thermodynamics and kinetics parameters supporting the principles of electrostatic interaction. The structural requirements of the phenylpropanoids for effective inhibition were clarified while electrostatic interaction-related descriptors were selected by penalization methods in the constructed QSAR models.

ABSTRAK

Dari segi kesan alam sekitar dan pertimbangan kos, penggunaan bahan tambahan hijau terutamanya yang berasal daripada tumbuhan telah didapati sebagai satu pendekatan alternatif berdaya maju di sebalik perencat organik sintetik dalam manangani ancaman kakisan. Walau bagaimanapun, oleh sebab kerumitan matriks komposisi ekstrak tumbuhan, usaha yang melibatkan juzuk terpencil jarang dibuat untuk perencatan kakisan, justeru penggunaan optimumnya terhalang. Dalam kajian ini, sifat perencatan kakisan bagi rizom *Alpinia galanga* dan juzuknya dikaji secara eksperimen dan teori terhadap keluli lembut di dalam larutan asid hidroklorik menggunakan kaedah kehilangan berat dan kaedah elektrokimia, dan teknik pencirian permukaan iaitu spektroskopi inframerah transformasi Fourier-pantulan total dilemahkan (ATR-FTIR), mikroskopi elektron pengimbas (SEM), mikroskopi elektron pengimbas pemancaran medan (FESEM) dan spektroskopi serakan tenaga sinar-X (EDX). Eksplorasi menggunakan kaedah permukaan tindak balas (RSM) sebagai alat pengoptimuman dan pemodelan hubungan struktur-aktiviti kuantitatif (QSAR) fenilpropanoid utama tumbuhan tersebut telah dijalankan. Pada suhu bilik, kecekapan adalah tertinggi pada kepekatan tertinggi bagi semua perencat mengikut susunan berikut: ekstrak heksana (90.2%), minyak pati (87.9%), dan ekstrak metanol (74.2%), sementara bagi juzuk fenilpropanoid; 1'-asetoksikavikol asetat (84.6%), metil eugenol (83.6%), eugenol asetat (82.1%), eugenol (76.3%), dan asid p-hidroksisinnamik (30.4%). Kecekapan optimum masing-masing 90.3% dan 91.17% dicapai bagi ekstrak heksana dan komponen minyak pati pada kepekatan, suhu, dan masa optimum. Kajian mendedahkan bahawa mod campuran interaksi semua perencat dan keberkesanannya adalah disokong oleh teknik pencirian permukaan. Kecekapan perencatan berkurangan dengan peningkatan suhu bagi semua perencat kecuali pecahan minyak pati yang semakin meningkat. Model isoterma Langmuir adalah padanan yang paling sesuai memberikan nilai tenaga penjerapan negatif dengan parameter termodinamik dan kinetik yang menyokong prinsip interaksi elektrostatik. Keperluan struktur fenilpropanoid untuk perencatan berkesan telah dijelaskan manakala petunjuk berkaitan interaksi elektrostatik telah dipilih dengan kaedah pembetulan dalam model-model QSAR yang dibina.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

14BDCD	-	1,4-benzenedicarboxaldehyde
34DHBD	-	3,4-dihydroxybenzaldehyde
34DMBD	-	3,4-dimethoxybenzaldehyde
4ABD	-	4-acetoxybenzaldehyde
4CAEE	-	4-Coumaryl alcohol ethyl ether
4H3MBD	-	4-hydroxy-3-methoxybenzaldehyde
4H3MCA	-	4-hydroxy-3-methoxycinnamic acid
4HBD	-	4-hydroxybenzaldehyde
4HCA	-	4-hydroxycinnamic acid
ACA	-	1'-acetoxychavicol acetate
AFM	-	Atomic Force Microscopy
ANOVA	-	Analysis of Variance
ASTM	-	American Society for Testing and Materials
ATR-FTIR	-	Attenuated Total Reflectance – Fourier Transform Infrared Spectroscopy
B3LYP	-	Becke, three-parameter, Lee-Yang-Parr
CAD	-	Cinnamaldehyde
CC	-	Column Chromatography
CCD	-	Central Composite Design

CE	-	Chloroform Extract
CMA	-	Cinnamic acid
DFT	-	Density Functional Theory
DOE	-	Design of experiments
EA	-	Eugenol acetate
EDX	-	Energy Dispersive X-Ray
EIMS	-	Electron Ionization Mass Spectral
EIS	-	Electrochemical Impedance Spectroscopy
EN	-	Elastic Net
EO	-	Essential Oil
EUG	-	Eugenol
FESEM	-	Field Emission Scanning Electron Microscopy
GC-MS	-	Gas Chromatography–Mass Spectrometry
GDP	-	Gross Domestic Product
GDS	-	Glow Discharge Spectroscopy
GNP	-	Gross National Product
HE	-	Hexane Extract
HNMR	-	Proton Nuclear Magnetic Resonance
HPLC	-	High Performance Liquid Chromatography
ISO	-	International Standard Organization
LASSO	-	Least Absolute Shrinkage and Selection Operator
ME	-	Methanol Extract
MEUG	-	Methyl eugenol
MLR	-	Multiple Linear Regression

MM2	-	Molecular Mechanics
MOPAC	-	Molecular Orbital Package
MSE_{test}	-	Mean squared errors of test data set
MSE_{train}	-	Mean squared error of training data set
OFAT	-	One Factor At a Time
OLS	-	Ordinary Least Squares
PMLR	-	Penalized Multiple Linear Regression
Q^2_{ext}	-	Coefficient of external validation
Q^2_{int}	-	Coefficient of internal validation
QSAR	-	Quantitative Structure–Activity Relationship
R^2	-	Coefficient of determination
RBS	-	Rutherford Backscattering Spectrometry
RR	-	Ridge Regression
RSM	-	Residual Surface Methodology
RSS	-	Residual Sum of Squares
SEM	-	Scanning Electron Microscopy
TLC	-	Thin Layer Chromatography
UV-vis	-	Ultraviolet–visible spectroscopy
VLC	-	Vacuum Liquid Chromatography
XPS	-	X-ray Photoelectron Spectroscopy
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

T	-	Temperature (K)
t	-	Time
R	-	Universal Gas constant $(8.3145\ J\ mol^{-1}\ K^{-1})$
h	-	Planck's constant $(6.62606896 \times 10^{-34}$ Js)
N	-	Avogadro $(6.02214078 \times 10^{23}\ mol^{-1})$
C_{inh}	-	Inhibitor concentration
ϑ	-	Surface coverage
K_{ads}	-	Equilibrium constant for the adsorption process
ΔG_{ads}	-	Standard free energy of adsorption $(kJ\ mol^{-1})$
E_a	-	Activation energy $(kJ\ mol^{-1})$
ΔH^*	-	Enthalpy $(kJ\ mol^{-1})$
ΔS^*	-	Entropy $(kJ\ mol^{-1})$

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Chapter synopsis

The chapter identified the fundamentals and the basis for this research. The background information on the subject matter was stated giving justification for the study. Cost of corrosion is enormous and efforts geared towards controlling it using economical corrosion inhibition additives of low toxicity and ecological acceptability is worthwhile. The past and present approaches in the field of corrosion inhibition, the areas not yet addressed and the gap that this research seeks to fill were clearly mentioned. In this chapter, the objectives, scope and means to accomplish the stated objectives were outlined. The significance of the of the research work were equally stated.

1.2 Research Background

Metals and various alloys of metals have excellent combinations of properties which make their applications indispensable in engineering and various environments (acidic, neutral and alkaline). Even in their normal application domain, metals and metal alloys become unstable and corrode. Corrosion is insidious in its behavior and may not be immediately apparent until its effects become conspicuous eliciting into production losses, equipment failures, compromised safety and problematic effluents.

According to (Landolt, 2007), corrosion is interpreted from the Latin word "*corrodere" as* "to chew away", "to attack". As a result of man's increasing activities and technological developments, problems due to corrosion can assume a colossal level if not promptly attended to. Corrosion is a persistent environmental and technological issue which continues to be of great relevance globally. Corrosion is therefore a major concern environmentally and industrially and efforts must be geared up at mitigating or minimizing this global menace. Corrosion is a risk to both the environments and production processes and as such the deleterious consequences of the corrosion process have become a problem of worldwide significance. Corrosion is detrimental, persistent and insidious in its action. Its effect is threatening to big as well as small industries. Its total prevention and elimination is practically impossible, hence the only effective antidote lies in controlling it.

Acids are extensively used industrially mostly in pickling, descaling, cleaning, oil well acidizing in oil recovery and petrochemical processes (Schweitzer, 2009). In the acidic medium, various types of corrosion inhibitors have been used for mild steel. Most of the reported acid corrosion inhibitors are synthetic organic compounds containing aromatic rings or heterocyclic atoms such as nitrogen, oxygen, sulphur and phosphorus, or compounds having multiple bonds in their molecule through which they are adsorbed on the metal surface (Deng and Li, 2012a; Hooshmand Zaferani *et al.*, 2013; Ji *et al.*, 2011; Li and Deng, 2012; Rani and Basu, 2012; Singh *et al.*, 2012c).

Adsorption of inhibitor molecules on metal surface has been shown to depend on certain physicochemical properties of the inhibitor group, such as functional groups, electron density at the donor atom, π-orbital character, and the electronic structure of the molecule (Singh, *et al.*, 2012c). Most organic inhibitors act by adsorption at the metal/solution interface (Rani and Basu, 2012). This phenomenon could take place either as electrostatic attraction between the charged metal and the charged inhibitor molecules; dipole-type interaction between uncharged electron pairs in the inhibitor with the metal; the π-electrons bonds interaction with the metal and combination of all of the above. The adsorption process has also been shown to depend on the electronic characteristics of the inhibitor, the nature of the surface, the temperature and pressure of reaction, steric effect, multilayer adsorption and a varying degree of surface site activity (Muthumegala *et al.*, 2011).

Several works have been carried out on the use of synthetic organic inhibitors to inhibit corrosion in different environments. Amino acids (Ashassi-Sorkhabia *et al.*, 2004; Khadom *et al.*, 2010), aliphatic and aromatic amines, aromatic acids, thiosemicarbazide derivatives, phenol, Schiff bases, surfactants, thiophenes , pyridine derivatives, tetrazole derivatives, benzimidazole derivatives (Obayes *et al.*, 2014; Tang *et al.*) and many others have been used. The mechanism of corrosion inhibition by most organic compounds is via adsorption to metal surfaces in which the metal active sites are blocked. The efficiency of inhibition of such organic compounds depends on the mechanical, structural and chemical properties of the adsorption layers formed under experimental conditions.

Plant products are organic in nature, and some of the constituents including tannins (Rahim *et al.*, 2007), organic and amino acids , alkaloids (Raja *et al.*, 2013a), and pigments are known to exhibit corrosion inhibiting action. In addition, plant extracts have become important not only because they are cheap renewable sources of materials but they are also ecologically acceptable. Moreover, they are also found to be easily extracted by simple procedures at low cost (Singh, *et al.*, 2012c). Extracts from various parts of plants have been used for corrosion inhibition on mild steel in different acid solutions.

Alpinia galanga, as well as turmeric and ginger belong to the Zingiberaceae family. The Zingiberaceae are perennial plants that produce aromatic rhizomes and are shown to possess good antioxidant properties. It has been reported that the antioxidant activities in plants are mainly dependent on their redox properties

(Mahae and Chaiseri, 2009). These redox properties have been shown to be a requirement for corrosion inhibition (Deng and Li, 2012a; Hooshmand Zaferani, *et al.*, 2013; Li and Deng, 2012; Li *et al.*, 2012b; Rani and Basu, 2012; Singh, *et al.*, 2012c).

1.3 Problem Statements

Despite the facts that the synthetic compounds showed good anticorrosive activity, most of them are highly toxic to both human beings and environment which has limited their use. These inhibitors may result into temporary or permanent damage to organ systems like kidneys or liver. It can also result into disturbance in the biochemical and enzymatic activities at some sites in the body (Patel *et al.*, 2013). These identified hazardous effects and high cost of organic corrosion inhibitors compounds have motivated an alternative in the natural organic compounds. Recently, widespread efforts have been devoted to the use of natural products, particularly plant extracts as corrosion inhibitors. This stems from the fact that the rich phytochemical constituents of plants have extensive potentials as economical, benign, readily available and renewable sources of organic compounds of potential industrial significance (Singh, *et al.*, 2012c). Mostly, all the plants' phytoconstituents namely; phenolics, flavonoids, terpenoids, alkaloids, tannins, saponins, amino acids, carbohydrates among others have molecular and electronic structures bearing close resemblances with those of classical corrosion inhibitors and many have been established to possess corrosion inhibition properties on metals (Mejeha *et al.*, 2012; Obi-Egbedi *et al.*, 2012).

Unfortunately, this abundant nature's phytochemicals have remained largely underutilized and their scope of application still remains narrow predominantly limited to medicine and nutrition. Equally, due to the complexity in composition matrix of plant extracts, efforts are seldom made to engage their isolated pure constituents for corrosion inhibition. This has limited the identification of the constituent(s) responsible for corrosion inhibition and therefore the mechanism of inhibition is somehow very indistinct. In addition to this, structural variations resulting into synergistic or antagonistic interactions of the constituents towards corrosion inhibition of the extracts are somewhat difficult to determine and hence maximum utilization of the plant constituents as potential inhibitors has only been given far too little attention.

The conventional experimental investigations are mostly costly, time-consuming, environmentally threatening and empirical research describing the optimization of corrosion inhibition process has not been significantly investigated. Corrosion inhibition measurement procedures have been limited to One Factor At a Time (OFAT) interactions for the process variables. Additionally, Quantitative Structural-Activity Relationship (QSAR) has been applied widely in the study of organic compounds as corrosion inhibitors and also in the study of antioxidant properties of plants, however, limited attention has emerged so far to the potential application of QSAR studies using plants as green corrosion inhibitors. Traditional QSAR studies in corrosion are principally based on quantum chemical descriptors, until now there exists only limited approaches adopting molecular descriptors derived from Dragon. This approach is able to leverage plant-based knowledge in corrosion studies. Its use will help to identify the roles of plant constituents towards corrosion inhibition by understanding the structural requirements for enhanced inhibition efficiency. This will further help to generate more effective inhibitors.

A. galanga belonging to the Zingiberaceae family has been chosen based on relating phylogenic and phytochemical considerations whose approach is premised on the existence of similar biochemical properties in closely related plant species. It is pertinent to note that turmeric and ginger which also belong to the Zingiberaceae family as *A. galanga* have previously been investigated to be good corrosion inhibitors on mild steel in acidic medium (Al-Fakih *et al.*, 2015a; Fouda *et al.*, 2013). *A. galanga* has been recognized as an antioxidant and a therapeutic agent for several diseases (Jaju *et al.*, 2009; Yasuhara *et al.*, 2009). Its major constituents are phenolics which have resemblance with structures of common organic corrosion inhibitors, however, its extracts of various solvent systems and its phenylpropanoid constituents are yet to be considered as corrosion inhibitors.

1.4 Research Objectives

In this research, rhizomes of *A. galanga* and its phenylpropanoid constituents are being employed as inhibitors for mild steel corrosion in hydrochloric acid. The following are the various objectives of the work:

i. To carry out phytochemical screening, extraction with different solvent systems, isolation, characterization and establishment of the corrosion inhibition properties of the major constituent compounds of *A. galanga*.

ii. To evaluate the interactive effects of the process variables and carry out process optimization of the corrosion inhibition for the extracts of *A. galanga*.

iii. To determine the adsorption and thermodynamic properties and establish models of adsorption for the extracts and the major phenylpropanoid of *A. galanga* with a view to proposing the mechanism for the corrosion inhibition process.

iv. To develop QSAR models of green corrosion inhibition of the phenylpropanoids of *A. galanga* using new molecular descriptors.

1.5 Scope of study

The focus of the research is to experimentally and theoretically investigate the rhizomes of *A. galanga* and its phenylpropanoid constituents as corrosion inhibitors on mild steel in hydrochloric acid solution.

The work is limited to extraction, screening, quantification and characterization of phytochemicals of *A. galanga* using hydrodistillation, Soxhlet extraction, VLC, TLC, CC, HPLC, GC-MS and ^{1}H NMR spectroscopy. The corrosion inhibition proficiencies of the various inhibitors were established on mild steel from 100 mg/L to 1000 mg/L of all the inhibitors in 1 M HCl solution at temperatures ranging from 300 K to 333 K. The concentration ranges (100 mg/L to 1000 mg/L for all inhibitors and 100 mg/L to 1000 mg/L for the essential oils) were chosen based on results obtained from preliminary experiments carried out in the study. The range of temperature between 300 K and 333 K was adopted to simulate the latent working temperature of inhibitor applicability in the field. Inhibition time range between 1 hour to 24 hours was chosen to get the most effective time required for maximum efficiency. Experimental determinations of corrosion inhibition efficiencies were carried out by using weight loss, Polarization and Electrochemical Impedance Spectroscopy (EIS) techniques. Investigation and characterization of the surface adsorption of the extracts and constituents as corrosion inhibitors on mild steel using adsorption isotherms were accomplished by Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR), Scanning Electron Microscopy (SEM), Field Emission Scanning Electron Microscopy (FESEM) and Energy Dispersive Spectroscopy (EDX). The adsorption characteristics, as well as kinetics and thermodynamic properties were established for hexane extract and the isolated 1'-acetoxychavicol acetate on mild steel.

Optimization of the corrosion inhibition process for the crude extract of *A. galanga* on mild steel in 1 M HCl was achieved using Response Surface Methodology (RSM) by adopting Central Composite Design (CCD). Development of Quantitative Structure- Activity Relationship (QSAR) models from fifteen phenylpropanoids of *A. galanga* and related compounds using molecular descriptors generated by Dragon software. Penalized regression method was used by adopting the methods of Ridge Regression (RR), Least Absolute Shrinkage and Selection Operator (LASSO) and Elastic Net (EN) for the selection of descriptors and estimation. The mechanism for the inhibition process was proposed based on the adsorption isotherms and theoretical findings.

1.6 Significance of study

The cost of corrosion is enormous ranging from direct to indirect costs and efforts geared towards controlling it using economical corrosion inhibition additives of low toxicity and ecological acceptability is worthwhile. Corrosion research often requires several experimental runs resulting into high cost of investigation as well as energy and time expenditure. This work leads to the optimization of inhibition properties of the plant and its constituents and the optimal conditions for the process variables. The work is able to rationalise the mechanism of corrosion inhibition of extracts with contributions from constituents. The results will help to provide structural requirements and understanding of existence of interactions for enhanced inhibition activities on mild steel. This approach furnishes information on the propensities to make extrapolation guide leading to the generation of novel corrosion inhibitor analogues that are structurally allied to the ones under study. It is envisaged that the modelling approach adopted can be extended to other family of compounds to provide valuable considerations for the design and generation of novel, green and efficient corrosion inhibitors.

1.7 Thesis Layout

The thesis is composed of six chapters in all. In chapter one is chronologically presented the preliminary components of the research work consisting of the background, the problem statement, research objectives, scope and the significance of the study.

Chapter two highlights the literature details of previously undertaken related works on corrosion and corrosion control with emphasis on the use of organic corrosion inhibitors. This chapter presents the importance, the electrochemical concepts, the principles of corrosion and its control, as well as presenting some theoretical basis for corrosion investigation. Exhaustive analysis of literature reveals that the rhizomes and phenylpropanoids of *A. galanga* have not so far been reported as corrosion inhibitors for any metal or mild steel in acid media.

In chapter three is presented the methodology involving the phytochemical identification, extraction, isolation and characterisation protocols for the rhizomes of *A. galanga*. This is followed by experimental determination of the inhibition efficiencies of the extracts and the various pure compounds using weight loss and electrochemical techniques. Various surface analytical techniques involving the use of GDS, ATR-FTIR, SEM, FESEM and EDX were equally presented to support the efficiency of the inhibitors. The chapter ends with development of the QSAR modelling methods using descriptors generated by Dragon software.

Chapter four presents the various results from the polarization measurements, electrochemical impedance and weight loss experiments as well as their interpretations based on established theories and offers the theoretical statements on findings. The interpretations of experimental results were premised on the composition of the mild steel specimen, constituents of the various extracts and the molecular nature of the inhibitor compounds. The behaviour of the extracts and the pure compounds as corrosion inhibitors were examined kinetically and thermodynamically, coupled with surface characterization techniques to ascertain the mechanism of interaction between the inhibitors and metal surface. The process of adsorption of the inhibitors was established using various adsorption isotherms.

Chapter five presents the theoretical insight into the study by establishing the procedure for the statistical modelling of the inhibition process using Design of Experiment. The chapter discusses the optimization of the process variables as accomplished through Response Surface Methodology (De Wael, *et al.*) by adopting Central Composite Design (CCD). The chapter further gives insight to the QSAR modelling of the phenylpropanoids of *A. galanga* as corrosion inhibitors using descriptors generated by Dragon software. As a result of the high dimensional nature of the data, the use of penalized methods of variable selection involving RR, LASSO and EN was adopted.

Chapter six concludes the thesis. The conclusion drawn on the use of *Alpinia galanga* as an eco-friendly corrosion inhibitor on mild steel in acidic medium is presented. Practical recommendations on importance of findings to the industry are emphasized. The chapter lastly shows further windows for future research

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