Green Synthesis and Characterization of Zinc Oxide and Aluminum Oxide Nanoparticles; Corrosion-Inhibition and Antimicrobial Study

Taiwo Felicia OWOEYE
LCU/PG/002976

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Certification

This is to certify that Taiwo Felicia OWOEYE with matriculation number LCU/PG/002976 carried out this research work titled ‘‘Green Synthesis and Characterization of Zinc Oxide and Aluminum Oxide Nanoparticles: Corrosion-Inhibition and Antimicrobial Study in the Department of Chemical Sciences, Faculty of Natural and Applied Sciences, Lead City University Ibadan, Oyo state, for the award of Doctor of Philosophy Degree in Industrial Chemistry and that this has not been previously submitted.

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Dr Abayomi O. Bamisaye Date
(Supervisor)

…….. ……

Prof. McDonald O. Ighodaro Date
(Head of the Department)

Dedication

This research work is dedicated to God almighty, my husband, my twin sister, and my children.

Acknowledgement

I am thankful to God for assisting me in completing this thesis and guiding me through my program at Lead City University. Without God, I am nothing. I also extend my gratitude to Lead City's management, particularly the entire faculty and staff of the postgraduate College, and the department's postgraduate coordinators, Dr. Abayomi O. Bamisaye and Dr. Olumuyiwa O. Ogunlaja, for their availability and support during my program. I sincerely appreciate Covenant University for granting me permission to conduct my bench work in their chemistry laboratory.

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Abstract

The green technique for the synthesis of metal oxide Nanoparticles (NPs) is quick, simple, cost-effective, and environmentally friendly. This study aims to biogenically synthesize zinc oxide (ZnONP) and aluminum oxide (Al_2O_3NP) nanoparticles using *Adonida merrilli* (NM), *Washingtonia robusta* (M), *Cassia javanica* (C), and *Casuarina equisetifolia* (E) leaf extracts. The nanoparticles were characterized using UV-Vis spectroscopy, X-ray diffractometer (XRD), Fourier transform infrared spectroscopy (FTIR), Energy dispersive X-ray analyzer (EDX) and Scanning electron microscopy (SEM). The Corrosion inhibition studies were carried out in 0.5 M H_2SO_4 using gravimetric and potentiodynamic polarization methods. The samples were examined in an acidic media at varying concentration ranges of 500, 1000, 1500 and 2000 ppm. The antimicrobial study of the NPs was carried out on *Salmonella typhi (Sa), Escherichia coli (Ec), Staphylococcus aureus (St),* and *Pseudomonas aeruginosa (Pa)*. The FT-IR analysis shows metal oxide absorption band values of 857 cm^{-1} (E-ZnONP), 700 cm^{-1} (C-ZnONP), 663 cm^{-1} (M-ZnONP), 715 cm^{-1} (NM-ZnONP). UV-Vis shows a plasmon resonance band value of 292 nm (C), 300 nm (C-ZnONP), 335 nm and 401nm (C-Al_2O_3Np), 290 nm (NM), 302 nm (NM-ZnONP), 336 nm and 401 nm (NM-Al_2O_3NP), 296 nm (M), 300 nm (M-ZnONP), 333 nm and 401 nm (M-Al_2O_3NP),

288 nm (E), 308 nm (E-ZnONP), 317 nm and 401 nm (E-Al_2O_3NP). The SEM micrographs and the XRD spectra show spherical crystalline nanoparticles with an average particle size of 73.82 nm (NM-ZnONP), 10.13 nm (C-Al_2O_3NP), 41.85 nm (C-ZnONP), 71.50 nm (M-ZnONP), 22.06 nm (E-ZnONP), 8.63 nm (E- Al_2O_3NP), 5.86 nm (NM Al_2O_3-NP), 8.68 nm (M-Al_2O_3NP). The recorded Zone of inhibition (ZI) and Minimum Inhibitory Concentration (MIC) values of E-ZnONP are 30.00 mm (Sa), 18.00 mm (Ec), 30.00 mm (St), and 30.00 mm (Pa), with MIC of 31.25 mg/mL (Sa), 0.00 mg/mL (Ec), 31.25 mg/mL (St), 31.25 mg/mL (Pa). C-ZnONP, ZI of 32.00 mm (Sa), 19.00 mm (Ec) , 34.00 mm (St), 40.00 mm (Pa), and MIC of 31.25 mg/mL (Sa), 0.00 mg/mL (Ec), 31.25 mg/mL (St), 31.25 mg/mL (Pa), M-ZnONP, ZI of 36.00 mm (Sa), 30.00 mm (Ec), 36.00 mm (St), 38.00 mm (Pa), with MIC of 31.25 mg/mL (Sa), 250.00 mg/Ml (Ec), 125.00 mg/mL (St), 62.50 mg/ mL (Pa). NM-ZnONP, ZI of 30.00 mm (Sa), 32.00 mm (Ec), 40.00 mm (St), 36.00 mm (Pa) and MIC of 7.81 mg/mL (Sa),7.81 mg/mL (Ec),1.95 mg/mL (St), 3.90 mg/mL (Pa). C-Al_2O_3NP, ZI of 0.00 mm (Sa), 22.00 mm (Ec), 21.00 mm (St), 20.00 mm (Pa)], with zero MIC, while M-Al_2O_3NP recorded ZI of 0.00 mm (Sa), 19.00 mm (Ec), 25.00 mm (St), 22.00 mm (Pa) with Zero MIC values. The optimum recorded corrosion inhibitory efficiency values are 92.93% (500 ppm), 81.49% (1000 ppm), 82.99% (1500 ppm), 88.84% (2000 ppm) for E-ZnONP, 61.41% for M- Al_2O_3NP, 86.63% (500 ppm) for C extracts in 0.5 M H_2SO_4. E-ZnONP, C-ZnONP, M-ZnONP, and NM-ZnONP. E-ZnONP showed optimal corrosion resistance, while NM-ZnONP exhibited superior antimicrobial activity with low MIC values.The findings of this study showed that the biogenically synthesized nanoparticles possessed excellent therapeutic properties and were also viable for corrosion mitigation.

Keywords: Green Synthesis, Corrosion Inhibition, Plant extract, Metal-Oxide Nanoparticles, Surface Chemistry.

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List of Acronyms

Abbreviation	Meaning

PM	Particulate matter
EPA	Environmental Protection Agency
GNP	Gross National Product
NACE	National Association of Corrosion Engineers
GDP	Gross domestic product
EDX	Energy dispersive X-ray analyzer
UV-VIS	Ultraviolet-Visible spectrophotometer
SEM	Scanning electron microscopy
XRD	X-ray diffractometer
FT-IR	Fourier transform infrared spectroscopy
SOCIs	Synthetic organic corrosion inhibitors
AFM	Atomic Force Microscope
TICIs	Traditional inorganic corrosion inhibitors
Cis	Commercial Inhibitors
GCIs	Green Corrosions Inhibitors
SPR	Surface Plasmon Resonance
nm	Nanometer
AgNPs	Silver Nanoparticles
AuNPs	Gold Nanoparticles
ZnO-NPs	Zinc Oxide Nanoparticle
FE-SEM	Field Emission Scanning Electron Microscope
TEM	Transmission electron microscopy
DLS	Dynamic light scattering
PCS	Photon correlation spectroscopy
SP	Scanning probe microscopy
AFM	Atomic force microscopy
BET	Brunauer-Emmet-Teller
VSM	Vibrating sample magnetometers
ICP-MS	Inductively coupled mass spectrometer
NP	Nanoparticles
ATR	Attenuated total reflectance

pH	Potential of hydrogen
Au-NCs	Gold nanoclusters.
GLE	Golpar leaf extract
MS	Mild Steel
G XRD	Glancing incidence X-ray Diffraction
CA	Contact Angle
PDS	Potentiodynamic polarization spectroscopy
EIS	Electrochemical impedance spectroscopy
DNA	Deoxyribonucleic acid
SeNPs	Selenium nanoparticles
AIDS	Acquired immunodeficiency syndrome
HR-TEM	High resolution-transmission electron microscopy
OFET	Organic field-effect transistor
HRSEM-EDS	High resolution Scanning electron microscopy - energy dispersive
MOGAC	Moringaoleifera gum activated carbon
MEMSs	Micro electromechanical systems
DNA	Deoxyribonucleic acid
IDE	Interdigitated electrode
PEMN	Plant extract mediated nanoparticles
CI	Corrosion inhibitor
GT	Green tea extract
rebars	Reinforcing bars
ROCP	Rebar open circuit potential
OCP	Open circuit potential
VOCs	Volatile organic compounds
GC-MS	Mass Spectrometer for Gas Chromatography
MIC	Minimum Inhibitory Concentration