

**ENHANCING THE ANTICORROSIVE AND ANTIMICROBIAL
ACTIVITIES OF POLYURETHANE COATING USING 4-NITRO-O-
PHENYLENEDIAMINE COUPLED WITH APTMS-SiO₂**

BY

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AUGUST, 2024

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**A DISSERTATION SUBMITTED TO THE SCHOOL OF POSTGRADUATE
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COLLEGE OF SCIENCE AND TECHNOLOGY, COVENANT
UNIVERSITY, OTA, OGUN STATE, NIGERIA**

AUGUST, 2024

ACCEPTANCE

This is to attest that this dissertation is accepted in partial fulfilment of the requirement for the award of the degree of Master of Science (M.Sc.) in Industrial Chemistry in the department of Chemistry, College of Science and Technology, Covenant University, Ota, Nigeria.

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DECLARATION

I, **ADEBAMIRO, OLUWAFAYOKUNMI MODUPE (22PCC02438)**, declare that this dissertation is a representation of my work, and is written and implemented by me under the supervision of Dr. Tolutope Oluwasegun Siyanbola of the Department of Chemistry, College of Science and Technology, Covenant University, Ota, Ogun State. I attest that this dissertation has not been submitted elsewhere for the award of a masters' degree. All Information cited from published and unpublished literature has been duly acknowledged.

ADEBAMIRO, OLUWAFAYOKUNMI MODUPE

Signature and Date

CERTIFICATION

We certify that the dissertation titled “**ENHANCING THE ANTICORROSIVE AND ANTIMICROBIAL ACTIVITIES OF POLYURETHANE COATING USING 4-NITRO-O-PHENYLENEDIAMINE COUPLED WITH APTMS-SIO₂**” is the original work carried out by **ADEBAMIRO, OLUWAFAYOKUNMI MODUPE (22PCC02438)** in the Department of Chemistry, Covenant University, Ota, Ogun State, Nigeria under the supervision of Dr. Tolutope.O. Siyanbola of the Department of Chemistry. We have examined and found this proposal acceptable as part of the requirements for the award of the degree of Master of Science in Industrial Chemistry.

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DEDICATION

I dedicate my work to Almighty God, the acquisition of knowledge, and the progress of scientific inquiry, expecting it to enhance our comprehension of the subject matter.

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TABLE OF CONTENTS

CONTENTS	PAGES
COVER PAGE	
TITLE PAGE	
ACCEPTANCE	iii
DECLARATION	iv
CERTIFICATION	v
DEDICATION	vi
ACKNOWLEDGEMENT	vii
TABLE OF CONTENTS	viii
LIST OF FIGURES	xiii
LIST OF SCHEMES	xv
LISTS OF TABLES	xvi
LIST OF ABBREVIATIONS AND ACRONYMS	xvii
ABSTRACT	xix
CHAPTER ONE: INTRODUCTION	1
1.1 Background to the Study	1
1.2 Statement of the Research Problem	5
1.3 Research Questions	5
1.4 Aim and Objectives	5
1.4.1 Aim of the study	5
1.4.2 Objectives of the study	5
1.5 Justification for the Study	6
1.6 Scope of the Study	6
CHAPTER TWO: LITERATURE REVIEW	7
2.1 Seed Oils	7
2.2 Castor oil	8
2.3 Poly urethanes and polyurea coatings	11
2.3.1 Formation of Polyols	12

2.3.2 Formation of Polyamines	12
2.3.3 Formation of Polyurea Polyols.	13
2.3.4 Polyurethanes from Polyols without Natural Hydroxyl Groups	13
2.3.4.1 Synthesis of polyols by epoxidation accompanied by oxirane ring using seed oils	13
2.3.4.2 Synthesis of polyols by ozonolysis using seed oils	16
2.3.4.3 Synthesis of polyols through transesterification using seed oils	17
2.3.4.4 Synthesis of polyols by thiol-ene using seed oil	19
2.3.5 Diisocyanates	20
2.3.5.1 Vegetable Oil-derived isocyanate	20
2.3.6 Chain extenders	21
2.3.7 Vegetable oil-based Polyurethane coatings	21
2.3.7.1 High solids Polyurethane coatings	21
2.3.7.2 Hyperbranched Polyurethane coatings	22
2.3.7.3 Waterborne Polyurethane coatings	22
2.3.7.4 UV curable Polyurethane coatings	23
2.3.7.5 Nanocomposite Polyurethane coatings	24
2.3.7.6 Non-isocyanate Polyurethane (NIPU) coatings	24
2.4 Nanoparticles	25
2.4.1 Synthesis of Nanoparticles	26
2.4.1.1 Bottom-up approach	26
2.4.1.2 Top-down approach	27
2.5 Applications of Bio-based Polyurethanes (PU)	28
2.6 4- Nitro-o- phenylenediamine	28
2.7 Antimicrobial activity	29
2.8 Different research on Polyurethane	30
2.9 Gap in Literature	30

CHAPTER THREE: MATERIALS AND METHODS	31
3.1 Materials	31
3.1.1 Apparatus	31
3.2 Methods	31
3.2.1 Castor Bean Seeds Processing	31
3.2.2 Extraction of castor oil from castor seed	32
3.2.3 Physico-chemical analysis of castor oil (CSO)	33
3.2.3.1 Acid Value (AV)	33
3.2.3.2 Iodine Value (IV)	33
3.2.3.3 Viscosity (V)	33
3.2.3.4 Refractive Index (RI)	34
3.2.3.5 Moisture content (MC)	34
3.2.3.6 Saponification value (SV)	34
3.2.4 Solubility Analysis	35
3.2.5 Synthesis of poly(epichlorohydrin-triol) (PECH-triol)	35
3.2.6 Synthesis of polyurethane	35
3.2.7 Synthesis of hybrid polyurethane urea (PUU) with 4NPD (0.1%)	37
3.2.8 Synthesis of hybrid polyurethane urea with 4NPD (0.3%)	38
3.2.9 Synthesis of hybrid polyurethane urea with 4NPD (0.5%)	38
3.2.10 Synthesis of APTMS-SiO ₂ (ASiO ₂)	38
3.2.11 Synthesis of hybrid polyurethane urea with modified Silica (PUU-ASiO ₂) (1%)	39
3.2.12 Synthesis of hybrid polyurethane urea with modified Silica (PUU-ASiO ₂) (1.5%)	40
3.2.13 Synthesis of hybrid polyurethane urea with modified Silica (PUU-ASiO ₂) (3%)	41
3.3 Spectroscopic Analysis	41
3.3.1 Fourier Transform Infrared Spectroscopy (FT-IR)	41
3.3.2 Nuclear Magnetic Resonance (NMR)	41

3.3.3 X-ray Diffraction (XRD)	42
3.3.4 Thermogravimetric Analysis (TGA)	42
3.3.5 Antimicrobial Analysis	42
3.3.6 Salt Spray Test	42
3.3.7 Water Contact Angle	42
3.3.8 Scanning Electron Microscopy (SEM)	42
CHAPTER FOUR: RESULTS	43
4.1 Physico-chemical characterisation of Polyols	43
4.1.1 Solubility test	45
4.2 Spectroscopic characterisation of Castor oil	45
4.3 Spectroscopic characterisation of PECH-triol	47
4.4 Spectroscopic Characterisation of Silica and Modified Silica	47
4.5 Spectroscopic characterisation of PU and polymeric hybrid composite	48
4.6 Thermogravimetric Analysis and Differential Thermogravimetric Analysis	52
4.7 X-ray Diffraction Analysis	53
4.8 Scanning Electron Micrograph	54
4.9 Water contact angle	55
4.10 Salt Spray test	55
4.11 Antimicrobial evaluation	57
CHAPTER FIVE: DISCUSSION	58
5.1 Solubility analysis	58
5.2 Physico-chemical analysis	58
5.3 FT-IR Spectra analysis	60
5.3.1 Castor seed (<i>Ricinus communis</i>) Oil	60

5.3.2 Silicon Oxide and APTMS-Silicon Oxide	60
5.3.3 Poly(epichlorohydrin-triol) (PECH-triol)	61
5.3.4 Polyurethane urea hybrid coatings (PUU)	61
5.3.5 Polyurethane urea hybrid coating containing modified silica (PUU-ASiO ₂)	61
5.4 NMR Analysis	62
5.4.1 Castor seed (<i>Ricinus communis</i>) Oil	62
5.4.2 Polyurethane coatings	62
5.4.3 Polyurethane urea hybrid coatings	63
5.4.4 Polyurethane urea hybrid coatings embedded with ASiO ₂	63
5.5 Thermogravimetric analysis and differential thermogravimetric analysis	64
5.6 X-ray diffraction analysis	65
5.7 Scanning Electron Micrograph	67
5.8 Salt-spray test	68
5.9 Contact angle	69
5.10 Antimicrobial analysis	69
CHAPTER SIX: CONCLUSION AND RECOMMENDATION	70
6.1 Summary	70
6.2 Conclusion	71
6.3 Contributions to Knowledge	71
6.4 Recommendations	71
REFERENCES	73

LIST OF FIGURES

Figure 1.1: Formation of polyurethane	4
Figure 2.1: Structure of castor oil	10
Figure 2.2: Jatropha oil hydroxyl group induction through epoxidation accompanied by oxirane ring opening mechanism	15
Figure 2.3: Synthesis of polyols from vegetable oils through a hydroformylation/hydrogenation Process	16
Figure 2.4: Ozonolysis followed by the oxidation of vegetable oil triglyceride	17
Figure 2.5: Production of Polyols through the transesterification mechanism	18
Figure 2.6: Synthesis of polyols based on soybean oil using the thiol-ene coupling method	19
Figure 2.7a: Structure of HMDI	20
Figure 2.7b: Structure of TDI	20
Figure 2.8: Preparation of fatty acid-based aliphatic diisocyanate	21
Figure 2.9: Synthesis process showing both the top-down approach and bottom-up approach	26
Fig 2.10: Structure of 4- nitro-o-phenylenediamine	29
Figure 3.1: Conversion of Castor bean seed to Castor Oil	32
Figure 4.1: Films of PUU	44
Figure 4.2: Films of PUU- ASiO ₂	44
Figure 4.3: ATR-FTIR spectrum of castor oil	45
Figure 4.4: ¹ H-NMR spectrum of Castor oil	46
Figure 4.5: ATR-FTIR of PECH-triol	46
Figure 4.6: ATR-FTIR of Silica and APTMS-Modified Silica	47
Figure 4.7: FT-IR spectrum of PUU	48
Figure 4.8: FT-IR spectrum of PUU-ASiO ₂	48
Figure 4.9: ¹ H NMR spectrum of PU	49
Figure 4.10: ¹ H NMR spectrum of PUU	50
Figure 4.11: ¹ H NMR spectrum of PUU-ASiO ₂	51
Figure 4.12: TGA spectrum of PUU-ASiO ₂	52

Figure 4.13: DTG of PUU-ASiO ₂	52
Figure 4.14: XRD of SiO ₂ and ASiO ₂	53
Figure 4.15: XRD of PUU	53
Figure 4.16: XRD of PUU-ASiO ₂	54
Figure 4.17: SEM of Silica and Modified silica	54
Figure 4.18: Contact angle of PUU-ASiO ₂	55
Figure 4.19a: PUU before Salt spray	56
Figure 4.19b: PUU after Salt Spray	56
Figure 4.20a: PUU- ASiO ₂ before Salt spray	56
Figure 4.20b: PUU- ASiO ₂ after Salt Spray	56

LIST OF SCHEMES

Scheme 3.1: Synthesis of poly(epichlorohydrin-triol)(PECH-triol)	35
Scheme 3.2: Synthesis of polyurethane	36
Scheme 3.3: Synthesis of polyurethane-urea coating	37
Scheme 3.4: Synthesis of APTMS-modified silica	39
Scheme 3.5: Synthesis of polyurethane-urea coating with APTMS-SiO ₂	40

LISTS OF TABLES

TABLES	LIST OF TABLES	PAGES
Table 2.1:	Different fatty acids found in vegetable seed oil	8
Table 2.2:	Classifications of nanoparticles generated by diverse processes	27
Table 3.1:	Parameters for reaction during the synthesis of Polyurethane	36
Table 3.2:	Parameters for response during the synthesis of Polyurethane-urea(0.1%)	37
Table 3.3:	Parameters for reaction during the synthesis of Polyurethane-urea (0.3%)	38
Table 3.4:	Parameters for reaction during the synthesis of Polyurethane-urea (0.5%)	38
Table 3.5:	Parameters for reaction during the synthesis of PUU-ASiO ₂ (1%)	39
Table 3.6:	Parameters for reaction during the synthesis of PUU-ASiO ₂ (1.5%)	41
Table 3.7:	Parameters for reaction during the synthesis of PUU-ASiO ₂ (3.%)	41
Table 4.1:	Physico-chemical characterisation of Castor (<i>Ricinus communis</i>) Oil	43
Table 4.2:	Solubility of CSO, PECH-triol and 4NPD in different organic solvents	45
Table 4.3:	Antimicrobial activities of PUU and PUU-ASiO ₂	57
Table 5.1:	Thermogravimetric comparison between PUU and PUU-ASiO ₂	65
Table 5.2:	Xray diffraction values of PUU and PUU-ASiO ₂	67

LIST OF ABBREVIATIONS AND ACRONYMS

4NPD:	4-nitro-o-phenylenediamine
ASiO ₂ :	(3-Amino Propyl)trimethoxysilane- silicon(iv)oxide
ATR:	Attenuated Total Reflectance
CSO:	Castor seed oil
DMF:	Dimethylformamide
DMSO:	Dimethylsulfoxide
DTG:	Differential Thermogravimetric Analysis
FT-IR:	Fourier Transform- Infrared Spectroscopy
HDDA:	1,6-hexanediol diacrylate
HMDI:	Hexamethylene diisocyanate
MIBK:	Methyl isobutyl ketone
NaCl:	Sodium chloride
NIPU:	Non-isocyanate polyurethane
NM:	Nanomaterial
NMR:	Nuclear Magnetic Resonance
PECH-triol:	Poly(epichlorohydrin-triol)
PO:	Palm oil
PU:	Polyurethane
PUA:	Polyurethane acrylate
PUU:	Polyurethane Urea
PUR:	Poly Urea

ROP:	Ring-opening Polymerization
SEM:	Scanning Electron Microscopy
SFFO:	Safflower oil
SFO:	Sunflower oil
SMO:	Silica Modified
TDI:	Toluene diisocyanate
Tg:	Glass transition temperature
TGA:	Thermogravimetric Analysis
TMPTA:	Trimethylolpropane triacrylate
TPGDA:	Tripropylene glycol diacrylate
TPSO:	Thevetia peruviana seed oil
UN SDG:	United Nations Sustainable Development goals
UV:	Ultraviolet
VOC:	Volatile organic compound
VSO:	Vegetable seed
WPUD:	Water-bone polyurethane dispersion
XRD:	X-ray diffraction

ABSTRACT

The research explores the synthesis and properties of polyurethane-urea coatings derived from castor seed oil (CSO), Toluene diisocyanate (TDI) and 4-nitro-o-phenylenediamine (4NPD) as a sustainable alternative to petrochemical-based polymers. Given the increasing environmental concerns and the need for biodegradable materials, this study focuses on utilising renewable plant biomass materials, particularly non-edible vegetable oils, to produce environmentally friendly polymeric coatings. The experimental approach involved the synthesis of poly(epichlorohydrin-triol) (PECH-triol) and its subsequent reaction with CSO and toluene diisocyanate (TDI) to form polyurethane. The inclusion of 4NPD and APTMS-SiO₂ aimed to enhance the attendant properties of the resultant coatings. Comprehensive analyses were conducted to characterise the synthesised materials. Fourier Transform Infrared Spectroscopy (FT-IR) and Nuclear Magnetic Resonance (NMR) confirmed the successful incorporation of 4-nitro-o-phenylenediamine 4NPD and APTMS-SiO₂ into the polyurethane matrix. X-ray Diffraction (XRD) analysis revealed the crystallinity of the coatings. The salt spray test shows that the more the nanomaterial, the higher it resists corrosion. The water contact angle demonstrates that the 3% APTMS-SiO₂ is more hydrophobic than the others, while Thermogravimetric Analysis (TGA) demonstrated their thermal stability. Furthermore, antimicrobial tests showed significant activity against various bacterial and fungal strains. It is hereby concluded that 4NPD enhances the coating strength and adhesives. It was also observed that PUU-ASiO₂ with 3% ASiO₂ show a more substantial effect on the attendant property of Polyurethane-urea coatings.

Keywords: 4-nitro-o-phenylenediamine, Antimicrobial, Polyurethane-urea, Nanomaterial, Castor oil