

**SYNTHESIS, MODIFICATION, AND CHARACTERISATION OF  
FUNCTIONAL POLYURETHANE COATING SYSTEMS FROM  
CASTOR OIL**

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**SEPTEMBER, 2024**

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**A THESIS SUBMITTED TO THE SCHOOL OF POSTGRADUATE  
STUDIES IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR  
THE AWARD OF DOCTOR OF PHILOSOPHY (PhD) DEGREE IN  
CHEMISTRY IN THE DEPARTMENT OF CHEMISTRY, COLLEGE OF  
SCIENCE AND TECHNOLOGY, COVENANT UNIVERSITY, OTA,  
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**SEPTEMBER, 2024**

## **ACCEPTANCE**

This is to attest that this thesis is accepted in partial fulfilment of the requirements for the award of the degree of Doctor of Philosophy in Industrial Chemistry in the Department of Chemistry, College of Science and Technology, Covenant University, Ota, Nigeria.

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## **DECLARATION**

**I, ADEBOYE, SAMUEL ADEPITAN (20PCC02309)**, declare that this research was carried out by me under the supervision of Prof. Kolawole O. Ajanaku and Dr. Tolutope O. Siyanbola of the Department of Chemistry, Landmark University and Department of Chemistry, College of Science and Technology, Covenant University, Ota respectively. I attest that this Thesis has not been presented either wholly or partially for the award of any degree elsewhere. All the sources of materials and scholarly publications used in this thesis have been duly acknowledged.

**ADEBOYE, SAMUEL ADEPITAN**

**Signature and Date**

## **CERTIFICATION**

We certify that the Thesis titled **SYNTHESIS, MODIFICATION, AND CHARACTERISATION OF FUNCTIONAL POLYURETHANE COATING SYSTEMS FROM CASTOR OIL** is the original work carried out by **ADEBOYE SAMUEL ADEPITAN (20PCC02309)** in the Department of Chemistry, Covenant University, Ota, Ogun State, Nigeria under the supervision of Prof. Kolawole O. Ajanaku and Dr. Tolutope O. Siyanbola of the Department of Chemistry, Landmark University and Department of Chemistry, College of Science and Technology, Covenant University, Ota respectively. We have examined and found this work acceptable as part of the requirements for the award of the degree of Doctor of Philosophy in Industrial Chemistry.

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**Signature and Date**

## **DEDICATION**

I dedicate this work to my darling wife, Olufunke Asake Adeboye, a faithful friend and an unflinching support in this journey.

## ACKNOWLEDGMENTS

My wholehearted appreciation goes to the Almighty God for the gift of life. He is my sustainer and my source of strength. This research could not have become a reality without His grace, wisdom, strength, and divine provisions. To God alone is all the glory eternally. I sincerely appreciate the visioner and Chancellor of this great institution, Covenant University, Ota, Bishop David O. Oyedepo, who is more than just the Chancellor but a father to me. He gave me a platform that made pursuing this Ph.D a comfortable and memorable journey. I acknowledge the Covenant University Management team for allowing me to accomplish this programme. The Vice-Chancellor, Prof. Abiodun H. Adebayo, the Deputy Vice-Chancellor, Prof. Olujide A. Adekeye, and the Registrar, Mrs. Regina A. Tobi-David. I acknowledge the Dean, School of Postgraduate Studies, Prof. Akan B. Williams; the Sub-Dean, School of Postgraduate Studies, Dr. Hezekiah O. Falola. I also appreciate the Dean of the College of Science and Technology, Prof. Timothy A. Anake. I am grateful to my supervisor, Prof. Kolawole O. Ajanaku, and co-supervisor, Dr. Tolutope O. Siyanbola, for their patience, encouragement, advice, and wisdom transferred throughout this programme. Despite your busy schedules, I am forever grateful for the sacrifice and the time you made for me. My earnest appreciation goes to the Head of Chemistry Department, Dr. Cyril O. Ehi-Eromosele, for his positive impact, support, and motivation, which started from the interview day and continued throughout the Ph.D programme. I must not fail to appreciate Dr. Anuoluwa A. Akinsiku, the Departmental PG Coordinator, for her support and motherly role during this programme. My profound gratitude goes to Prof. Olayinka O. Ajani, who was always ready to explain Organic Chemistry principles even without prior notice. Prof. J. A. Olugbuyiro, Dr Joseph Adekoya, the late Prof. Grace Olasehinde and her entire family, Dr. Olabisi Ademosun, and Dr. Gbolahan Oduselu, thank you all for your kind support and encouragement during this programme. I cannot forget to thank the technologists who helped at various times during this work: Mr. Bamidele Durodola, Mrs. Hassana Jonathan, Mrs. Taiwo Owoye, Miss Grace Ibiwoye, Mr Tunde Ogunleye, and Mr Gbenga Taiwo (Microbiology lab).

I profoundly thank Professor Omobola Okoh of the Department of Chemistry, University of Fort Hare, South Africa, for her assistance in procuring some scarce chemicals from South Africa and Mr. Jeff Lawrence, Senior Technician (Materials) at Teesside University, Middlesbrough, UK, who went beyond the call of duty to help with the analyses. I want to thank my colleagues and co-travellers along this Ph.D journey, Mrs Tolu-Bolaji, Mrs Omokehinde Taiwo, Mrs. Anike Jenbola, Miss Oluwadunni Elebiju, Miss Temitope

Ogunnupebi, and Dr. Augustina Dada. I want to thank Adedamola Adebawale and Oluwafayokunmi Adebamiro, who are part of the oleochemistry team, for their invaluable roles.

I must not forget to thank Prof. F.A. Dawodu, my M.Sc. supervisor at the University of Ibadan in the 1989/90 session, who laid a solid foundation for me in the world of Chemistry and practically demonstrated the chemistry of love to me through generosity that is beyond description. My gratitude goes to my children and my grandchild, Tioluwanimi, for their endurance, love, and support throughout the programme. I appreciate my brothers and sisters for their support. I want to thank my loving wife, sister, and friend, Prof. Olufunke A. Adeboye, for bearing the brunt of my absence from home while pursuing this programme. Thank you so much for your patience and understanding. May the good Lord reward your sacrifice in multiple ways.



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## LIST OF ACRONYMS AND ABBREVIATIONS

APTMS	Aminopropyltrimethoxysilane
AFM	Atomic Force Microscopy
ATR	Attenuated Total Reflectance
CSO	Castor Seed Oil
DSC	Differential Scanning Calorimetry
DTG	Differential Thermogravimetric Analysis
DMTA	Dynamic Mechanical Thermal Analysis
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
FROCs	Functional Renewable Organic Coatings
GPC	Gel Permeation Chromatography
GO	Graphene Oxide
HDIT	Hexamethylene diisocyanate trimer
IPDI	Isophorone diisocyanate
JCO	<i>Jatropha caucis</i> Oil
MIBK	Methyl isobutyl ketone
NIPU	Non-isocyanate polyurethane
NMR	Nuclear Magnetic Resonance
PMAA	Polymethacrylic acid
PU <sub>s</sub>	Polyurethanes
RA	Ricinoleic Acid
RCSO	<i>Ricinus communis</i> Seed Oil
SEM	Scanning Electron Microscopy
TEOS	Tetraethoxysilane
TGA	Thermogravimetric Analysis

TPSO	<i>Thevetia peruviana</i> Seed Oil
TDI	Toluene diisocyanate
TMP	Trimethylolpropane
UV	Ultraviolet
VOCs	Volatile Organic Compounds
WPU <sub>s</sub>	Waterborne Polyurethanes
XRD	X-ray Diffraction

## ABSTRACT

Environmental challenges have driven production science towards using biodegradable and sustainable feedstocks for product development. Developing sustainable and high-performance coating materials to address the environmental concerns and technical demands of modern industries has become so critical that plant seed oils are considered viable renewable feedstocks capable of substituting petrochemical-based materials in polymeric material preparation. This study reports the synthesis and characterisation of functional organic polyurethane coatings from castor bean seed oil (CSO) (*Ricinus communis* seed oil). Graphene nanoparticles were modified into graphene oxide and incorporated within the polyurethane polymer matrix in a one-pot synthesis. Also, aminopropyltrimethoxysilane (APTMS) was used to alter silica nanoparticles and was incorporated into the polyurethane system. Bisphenol A and trimethylolpropane (TMP) were used as extenders, and their influences on the coating properties were also examined in the urethane systems. Physicochemical analysis of the feedstock and prepared coating formulations was conducted. Structural evaluation of synthesised materials was performed using proton nuclear magnetic resonance ( $^1\text{H}$  NMR) and attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy. Synthesised urethane coatings were cured on silicon resin mould and mild steel. Thermal stability and crystallinity of pristine and composite films were studied using thermogravimetric analysis (TGA) and X-ray diffraction (XRD). Scanning electron microscopy (SEM) was used to analyse surface morphology. Water contact angle analysis revealed the hydrophobicity of the synthesised urethane films. Solubility, anticorrosive, and antimicrobial properties of prepared materials were evaluated. Spectroscopic analysis confirmed the structure of modified nanomaterials, pristine, and composite films. Surface morphology and photographic images showed successful incorporation of nanomaterials (graphene oxide and hybrid APTMS-modified silica) within the polymer matrix. Thermal stability, anti-corrosive, and antimicrobial properties of the coating films were enhanced with increasing percentages of nanomaterials in the polyurethane systems. Coating films exhibited improved hydrophobicity with rising percentages of modified nanoparticles. Film photographic retention tests showed no particle agglomeration and high transparency at 0.5% graphene oxide composition (0.5% PU-GO). It also shows that the polymer with 0.5% PU-GO is the most thermally stable. Similarly, composite films of modified silica in CSO showed enhanced thermal stability, hydrophobicity, antimicrobial activity, and corrosion resistance. The polymer with 5% PU-SNP was the most thermally stable at high temperatures. It also has the highest water contact angle and lowest corrosion rate, hence the most hydrophobic and corrosion resistant. In conclusion, the 0.5% loading of modified graphene oxide (0.5% PU-GO) nanoparticles is the optimum loading in applications requiring low and high temperatures. At the same time, 5% loading is the optimum loading of APTMS-modified silica (5% PU-SNP) in applications that require high temperature. These results present a viable, sustainable alternative for various industrial applications.

***Keywords: renewable polymers, castor oil, polyurethane, nanomaterial, coatings.***