PREPARATION OF SILICA AEROGEL EMBEDDED GLASS FIBRE MATS FOR THERMAL INSULATION APPLICATIONS

Dissertation submitted to the Christ College (Autonomous) in partial fulfilment of the requirement for the Degree of

MASTER OF SCIENCE

IN

CHEMISTRY

Submitted by

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2022-2024

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I, Aarsha Augustine, hereby declare that this project work entitled, "**PREPARATION OF SILICA AEROGEL EMBEDDED GLASS FIBRE MATS FOR THERMAL INSULATION APPLICATIONS**," is an authentic record of the project work carried out by me under the guidance and supervision of Dr. Riju Davis, Principal Scientist, and Dr. S. Ananthakumar, Chief Scientist and Head of Division, Materials Science and Technology Division (MSTD), CSIR-NIIST, Thiruvananthapuram. The report is submitted to the University of Calicut to partially fulfill the requirements for the Master of Science degree in Chemistry from Christ College (Autonomous), Irinjalakuda. The present work or any part of this project work has not been submitted earlier for any degree or diploma of any other university.

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I am immensely thankful to **Dr. Rani Varghese**, Head of the Department of Chemistry and Assistant Professor, Christ College (Autonomous), Irinjalakuda, **Dr. V. T. Joy**, former Head of the Department of Chemistry and Associate Professor, Christ College (Autonomous), Irinjalakuda and **Dr. Tom Cherian**, Assistant Professor, Christ College (Autonomous), Irinjalakuda for their great support and encouragement.

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Last but not the least, I thank my friends and family for their immense help, cooperation and encouragement during the course of this journey.

Aarsha Augustine

ABSTRACT

High-performance engineered materials having very low thermal conductivities have found important applications in industrial processes that require temperature shielding and thermal insulation. These materials play an important role to maintain temperature or minimize the overall heat loss of substances transported between containers during manufacturing processes. Silica aerogel, owing its superior properties, such as ultra-low dielectric constant, low thermal conductivity, extremely low density and high bulk porosity, has become a highly sought component in the architecture of thermal insulation materials. This project work was designed to prepare thermal insulation mats using silica aerogel embedded in glass substrates. To accomplish this, we prepared and characterized silica aerogel and incorporated it into thin layered glass matrices. Glass fiber and glass wool were used as substrates to prepare these insulation mats. The high-temperature shielding properties of these insulation mats were investigated. These results were compared to a benchmark product that is currently used in industrial applications. The temperature shielding properties of the materials are found to be in close range to that of the available product. However, further investigation and optimization are required to improve the overall performance and qualification of the materials for the intended application.

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ELECTROCHEMICAL SYNTHESIS OF REDUCED GRAPHENE OXIDE AND NITROGEN DOPED REDUCED GRAPHENE OXIDE FOR SUPERCAPACITOR APPLICATIONS

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ADWAITH A.P

CONTENTS

ABBREVIATIONS USED

1. N-rGO - Nitrogen doped Reduced Graphene Oxide
2. EDLC - Electrical Double Layer Capacitance
3. EIS - Electrochemical Impedance Spectroscopy
4. GCD - Galvanostatic Charge Discharge
5. rGO - Reduced Graphene Oxide
6. SCs - Supercapacitors
7. CV - Cyclic Voltammetry

A COMPARATIVE APPROACH ON VARIANT RATIOMATERIC SYNTHESIS OF AMINO ACID BASED GRAPHENE QUANTUM DOTS FOR ELECTROCHEMICAL SENSING APPLICATIONS

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I am privileged to acknowledge my deep gratitude to **Dr. Achu Chandran**, Senior Scientist at MSTD, CSIR-NIIST, Thiruvananthapuram. His unwavering guidance and insightful advice were instrumental in shaping the success of this study.

I also extend my sincere appreciation to **Dr. C. Anandharamakrishnan**, Director, CSIR-NIIST, and **Dr. Ananthakumar S**, HOD OF MSTD, CSIR-NIIST, for providing the necessary infrastructural support.

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ABSTRACT

Graphene quantum dots (GQDs) are carbon nanomaterials with sizes less than 10 nm, known for their unique properties, including low toxicity, high solubility, tunable photoluminescence, and biocompatibility. These features have made GQDs highly attractive in the realm of carbon nanomaterials, as evidenced by the burgeoning literature on this subject. Various methods for synthesizing GQDs from organic, inorganic, and biomaterials have been reported. This study focuses on the synthesis of GQDs using amino acids, methionine and tryptophan as precursors. Amino acid-based GQDs are expected to be less toxic and more biocompatible compared to those synthesized from other organic or inorganic materials. We investigate the effects of varying amino acid proportions on the structural, heteroatom doping, and functional characteristics of the resulting GQDs. Additionally, the electrochemical sensing properties of these GQDs are explored for sensing various analytes with biological and environmental significance.

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(a) CV for 1 mM HMFA
(b) DPV for 1 mM HMFA

Fig 3.8 Profile of CV and DPV obtained for the sensing of HMFA on the N,S-GQDs/GCE concentration range from 1μM- 1Mm

Fig 3.9 DPV of LOD obtained for HMFA on the N,S-GQDs

Fig 4.0

(a) CV profiles of the N,S-GQD/GCE in 0.1 M NaOH containing 1 mM HMFA at various scan rates (10-100 mVs^{-1})

(b) Calibration plot of the current vs the scan rate

(c) Calibration plot of the current vs the square root of the scan rate

Fig 4.1

(a) CV diagram of interference studies on N,S-GQD/GCE for the detection of HMFA in the presence of other analytes
(b) DPV diagram of interference studies on N,S-GQD/GCE for the detection of HMFA in the presence of other analytes

ABBREVIATIONS

1. GQDs – Graphene Quantum Dots
2. QDs – Quantum Dots
3. CDs – Carbon Dots
4. NPs – Nano Particles
5. GO – Graphene Oxide
6. GSs – Graphene Sheets
7. DMF – Dimethyl formamide
8. DMSO – Dimethyl sulfoxide
9. PL – Photoluminescence
10. MW – Microwave
11. PS-PDMS – Polystyrene-b-polydimethylsiloxane
12. BCPs - Block copolymers
13. NDs – Nano Dots
14. MWCNTs – Multi-walled carbon nanotubes
15. UV – Ultraviolet
16. NMR – Nuclear magnetic resonance
17. CCs – Carbon clusters
18. CA – Citric acid
19. Glu – Glucose
20. AAs – Amino acids
21. EC – Electrochemical
22. RE – Reference electrode
23. WE – Working electrode
24. CE – Counter electrode
25. CV – Cyclic voltammetry
26. Try – Tryptophan
27. NIR – Near Infrared
28. Meth – Methionine
29. TEM – Transmission Electron Microscopy
30. XRD – X-ray diffraction
31. XPS – X-ray Photoelectron Spectroscopy
32. FTIR – Fourier Transform Infrared Spectroscopy

33. GCE – Glassy carbon electrode
34. DPV – Differential pulse voltammetry
35. 2- EF - 2-Ethyl furan
36. 2-MF - 2-Methyl furan
37. Acn - Acrolein
38. Acr - Acryl amide
39. DMF - 2,5-Dimethyl furan
40. HMFA - 5-Hydroxy methyl -2- furaldehyde
41. Asc – Ascorbic acid

REDUCTION KINETICS OF NICKEL OXIDE DISPERSED ON MCM-41

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I, ALEENA VARGHESE (Reg.No.CCAWMCH004) do hereby declare that, this dissertation work entitled "**REDUCTION KINETICS OF NICKEL OXIDE DISPERSED ON MCM-41"** submitted to the University of Calicut in partial fulfilment of the requirement for the award of the degree of Master of Science was carried under the guidance of Dr. Robinson P. Ponminiessary, Dean of Academics, Assistant Professor, Department of Chemistry, Christ College, Irinjalakuda and it is a record of original project work carried out by me and it has not previously formed the basis for the award of, any degree, diploma fellowship or other similar titles of recognition by any other university or institutions.

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ACKNOWLEDGEMENT

I extend my sincere and heartfelt gratitude to my project guide Dr. Robinson P. Ponmaniessary, dean of academics, assistant professor, Department of Chemistry, Christ College, Irinjalakuda for his valuable and inspiring guidance, critical assessment and constant encouragement at all stages of this project. I am greatly indebted to him for the completion of this work in the specified period.

I express my sincere thanks to Dr. Rani Varghese, Head, PG Department of Chemistry, for granting permission to carrying my project. I would like to express my gratitude to all the faculty members in the Department of Chemistry for their inspiration and guidance to complete this work.

I wish to acknowledge my gratitude to Rev. Dr. Jolly Andrews, C.M.I, Principal of Christ College, Irinjalakuda and Library staff of the Christ College for providing the timely help and necessary facilities.

I cheerfully express my profound thanks to all my classmates for their support and co-operation.

Above all I humbly thank God Almighty, whose sustaining grace has been sufficient for me to complete this endeavour.

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TABLE OF CONTENTS

ABSTRACT

Nickel oxide was supported on MCM-41 using the precipitation method. Reduction kinetics of supported and unsupported nickel oxide was studied using non isothermal methods to investigate the change in activation energy and the reduction reaction mechanism. Activation energy was determined using the Kissinger's method and Straink's method. Model discrimination was done using the Hancock-Sharp analysis and Malek's method. The calculated and the experimental TPR data was compared to check the reliability of the estimated kinetic parameters. The supported nickel oxide showed an increase in activation energy due to the influence of the support. The reaction mechanism shifted from Avrami-Erofeev model in the unsupported to three-dimensional diffusion-controlled model in the supported nickel oxide. The higher activation energy in the case of supported nickel oxide indicates the diffusion of vacancies is the rate limiting step. The reduction mechanism of nickel oxide on supported on MCM-41 was proposed in agreement with literature and current study.

Key words: Nickel Oxide, MCM 41, Reduction kinetics, Temperature Programmed Reduction, Diffusion control.

TETRADENTATE BIS AMIDATE COPPER(II) COMPLEX AS FUNCTIONAL MODELS OF OXIDASE ENZYMES

Dissertation submitted to the Christ College (Autonomous) in partial fulfilment of the requirement for the Degree of

MASTER OF SCIENCE

IN

CHEMISTRY

Submitted by

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I offer my special thanks to our former HoD, chemistry department, Christ College (Autonomous), Dr. V T Joy, Class teacher Dr. Tom Cherian for all his support. I express my sincere thanks to Dr. Rani Varghese, Head, PG department of Chemistry, for granting permission to carry out my project. And also I thank Dr. Titto Varghese, Assistant Professor, department of chemistry for providing the support during my course

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Above all I humbly thank God Almighty, whose sustaining grace has been sufficient for me to complete this endeavour.

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CONTENT

ABSTRACT

This dissertation reports the synthesis, characterization, and also the phenoxazinone synthase and catechol oxidase activity of a monomeric copper(II) complex Cu(L) with bis-amide tetradentate ligand H_2L: *N*,*N*′-(1,2-phenylene)dipicolinamide. The ligand and complex were characterized using a variety of spectroscopic techniques, including ^{1}H and ^{13}CNMR spectroscopy, IR spectroscopy, UV-visible spectroscopy, and HRMS spectrometry. The UV-vis spectra revealed two peaks, corresponding to the LMCT and d-d transitions. The generated complex demonstrated both phenoxazinone synthase and catechol oxidase mimicking activity. The copper(II) complex showed PHS activity with significant turnover number of $1.19 \times 10^{4}\ h^{-1}$ and catechol-mimicking activity with a TON of $3.17 \times 10^{5}\ h^{-1}$.

POLYMORPHISM IN ISATIN BASED COMPOUNDS: SYNTHESIS, CRYSTALLIZATION, THERMAL, MICROSCOPIC AND MECHANICAL CHARACTERIZATION

Dissertation submitted to the Christ College (Autonomous) in partial fulfillment of the requirement for the Degree of

MASTER OF SCIENCE

IN

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CONTENTS

CHAPTER 3: EXPERIMENTAL&CHARACTERIZATION

CHAPTER 4: RESULT AND DISCUSSION

ABBREVIATIONS

- $ClCH_3$- (Z)-5-chloro-3-(p-tolylimino)indolin-2-one.
- 4-$ClCH_3$- (Z)-4-chloro-3-(p-tolylimino)indolin-2-one.
- $BICH_3$ (Z)-5-bromo-3-(p-tolylimino)indolin-2-one.
- BIH (Z)-5-bromo-3-(phenylimino)indolin-2-one.
- BII (Z)-5-bromo-3-((4-iodophenyl)imino)indolin-2-one.
- CIH (Z)-5-chloro-3-(phenylimino)indolin-2-one.
- FIC (Z)-3-((4-chlorophenyl)imino)-5-fluoroindolin-2-one.
- $FICH_3$ (Z)-5-fluoro-3-(p-tolylimino)indolin-2-one.
- $HICH_3$ (Z)-3-(p-tolylimino)indolin-2-one.
- HIF (Z)-3-((4-fluorophenyl)imino)indolin-2-one.
- HIH (Z)-3-(phenylimino)indolin-2-one.
- IIH (Z)-5-iodo-3-(phenylimino)indolin-2-one.
- IIB (Z)-3-((4-bromophenyl)imino)-5-iodoindolin-2-one.
- IIC (Z)-3-((4-chlorophenyl)imino)-5-iodoindolin-2-one.
- IIF (Z)-3-((4-fluorophenyl)imino)-5-iodoindolin-2-one.
- $IICH_3$ (Z)-5-iodo-3-(p-tolylimino)indolin-2-one.
- III (Z)-5-iodo-3-((4-iodophenyl)imino)indolin-2-one.
- F- Flake
- B- Block
- N- Needle
- DCM- Dichloromethane
- EtOAc- Ethyl Acetate

ABSTRACT

The design of flexible molecular crystals by utilizing polymorphism is an emerging area of research. Herein we studied polymorphism in a set of halogen-substituted isatin compounds by controlling crystallization conditions. A detailed investigation by optical hot-stage microscopy, DSC, and PXRD measurements allowed us to confirm the polymorphism in a set of halogen-substituted isatin compounds. The obtained polymorphic forms in this set of halogen-substituted isatin compounds showed distinct macroscopic mechanical responses. It was found that the different polymorphs of IIH, IIF, IIC, IIB, IICH_3, III, CIH, HIF and 4-CICH_3 showed different mechanical compliance: the needle crystals are elastically deformable, while the block-type crystals are brittle in nature. Distinct mechanical responses with varying functional groups also observed: that is by replacing H with halogens or CH_3. This study is of great scientific significance to engineer mechanical compliance of organic crystals by packing structure modification instead of chemical structure optimization.

Design and Synthesis of Fluorescent and Colorimetric Pyrene Schiff Base-Based Sensors for Zn^{2+} and Cu^{2+} Ions

Dissertation submitted to the Christ College (Autonomous) in partial fulfilment of the requirement for the Degree of

MASTER OF SCIENCE

IN

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This is to certify that the project work entitled **"Design and Synthesis of Fluorescent and Colorimetric Pyrene Schiff Base-Based Sensors for Zn^{2+} and Cu^{2+} Ions"** is an authentic record of the work carried out by SREERAG SHANIL (Reg.No. CCAWMCH007) under the co-guidance of Dr. Tom Cherian, Asst. Professor, Department of Chemistry, Christ College (Autonomous), Irinjalakuda, for the partial fulfilment of the award of the degree of Master of Science in Chemistry during the academic year 2022-2024.

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ACKNOWLEDGEMENT

Words cannot express my gratitude to my guide, Dr. Chinna Ayya Swamy. P, for giving me the opportunity to conduct my project with all the resources available. I am also grateful to him for guiding me through the right track and for his constant support, that proved monumental towards the success of the work. I am sincerely thankful for his dedication, and arduous efforts and for his marvellous help in preparing this dissertation.

I express my heartfelt gratitude to my co-guide Ms. Afrin A. for her instructions and persistent help. My sincere gratitude also goes to other research scholars for their feedback, suggestions, attention and valuable comments.

I offer my special thanks to our Head of the Department of Chemistry, Christ College (Autonomous), Irinjalakuda, Dr. Rani Varghese for moral support and invaluable advice. I also express my gratitude towards Dr. Tom Cherian, Assistant Professor of Department of Chemistry, Christ College (Autonomous) Irinjalakuda, for his support and guidance as an internal project guide. I am indebted to all my teachers for their inspiration and motivation and also to our lab assistants for their patience and collaboration.

I pay special regard to my family members and friends for their encouragement, love, assistance and wise counsel.

Above all, I owe a debt of gratitude to God Almighty for steering me in the right direction for successfully carrying out my project.

ANGEL ROSE

CONTENTS

Abbreviations

The following abbreviations appear in this thesis:

BODIPY	Borondipyrromethane
°C	Degree Celsius
caled	calculated
cm	centimetres
cm^{-1}	wavenumber(s)
δ	Chemical shift in parts per million
d	doublet
DCM	Dichloromethane (CH_2Cl_2)
DMF	Dimethylformamide
dd	doublet of doublets
ESIPT	Excited state intramolecular proton transfer
equiv	equivalents
g	gram(s)
hr	hour(s)
Hz	Hertz
HRMS	High Resolution Mass Spectrometry
μ	micro
μM	micromolar
M	Molar
m	meter, multiplet
m/z	Mass to charge ratio
mg	milligram
min	minute
ml	millilitre
mmol	millimole
mol	mole
MHz	Megahertz
m.p.	melting point

NMR	Nuclear magnetic resonance
nm	nanometres
p	para
PET	Photoinduced electron transfer
s	second
SBs	Schiff bases
TFA	Trifluoroacetic acid
TMS	Tetramethyl silane
UV	Ultraviolet
UV-vis	Ultraviolet-visible
λ	wavelength
Φf	Fluorescent quantum yield

Abstract

We present the design and synthesis of two Schiff base derivatives, **Pyr-SB-1** and **Pyr-SB-2**, tailored for the sensitive detection of Zn^{2+} and Cu^{2+} ions through fluorescence *turn-on* and colorimetric mechanisms. These receptors were characterized by significant changes in absorption and fluorescence spectra upon metal ion binding, with **Pyr-SB-2** demonstrating superior fluorescence enhancement compared to **Pyr-SB-1**. The selective response towards Zn^{2+} ions was particularly notable, amidst the presence of competing metal ions, underscoring their potential for biomedical and environmental applications. The colorimetric shift from yellow to green upon metal coordination highlights their utility as dual-mode sensors. Future efforts will focus on elucidating binding stoichiometry, determining detection limits, and expanding sensor capabilities through the incorporation of alternative fluorophores.

SYNERGISTIC CORROSION INHIBITION OF ALUMINIUM ANODES IN ALKALINE MEDIA USING ZnO AND NEEM EXTRACT

Dissertation submitted to the Christ College (Autonomous) in partial fulfilment of the requirement for the Degree of

MASTER OF SCIENCE

IN

CHEMISTRY

Submitted by

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DECLARATION

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This is to certify that the thesis entitled " ***Synergistic Corrosion Inhibition of Aluminium Anodes in Alkaline Media Using ZnO and Neem Extract*** *"submitted by* ***Ms. ANN RANJITH (Reg No:CCAWMCH008)*** *to the* ***University of Calicut*** *towards the partial fulfilment of requirement for the award of the degree of* ***Master of Science in Chemistry*** *is a bonafide record of the work carried out by her at the Material Science and Technology Division of CSIR-National Institute for Interdisciplinary Science and Technology (NIIST), Thiruvananthapuram under my guidance during April to June 2024.*

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I begin by expressing my heartfelt gratitude to the Almighty God for granting me the strength, wisdom, and perseverance to complete this project. His divine guidance has been a constant source of inspiration and motivation throughout this work.

I am extremely thankful to CSIR National Institute for Interdisciplinary Science and Technology (CSIR-NIIST), Thiruvananthapuram for giving me this valuable project opportunity. I am grateful to Dr. Anandharamakrishnan, Director of CSIR – NIIST, for granting us access to the laboratory facilities necessary to complete our project successfully.

I would like to extend my heartfelt thanks to my supervisor, Dr. T P D Rajan, Senior Principal Scientist at the Materials Science and Technology Division, National Institute for Interdisciplinary Science and Technology (CSIR-NIIST) in Thiruvananthapuram. His guidance, encouragement, and valuable advice throughout the project have been greatly appreciated and respected.

I wish to express my sincere gratitude to Mr. Muhammad M, Senior Research Fellow at CSIR-NIIST, for his invaluable guidance and support during the project.

I am deeply grateful to Dr. Rani Varghese, Head, of the Department of Chemistry and Assistant Professor, Christ College (Autonomous), Irinjalakuda, Dr. V. T. Joy, former Head of the Department of Chemistry and Associate Professor, Christ College (Autonomous), Irinjalakuda, and Dr. Tom Cherian, Assistant Professor Christ College (Autonomous), Irinjalakuda, and Dr. Jibin.A.K Assistant Professor Christ College (Autonomous), Irinjalakuda, for their invaluable support and encouragement.

Finally, I would want to thank my family and friends for all of their help, cooperation and inspiration along this journey.

Ann Ranjith

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Fig 4.9	SEM micrographs of protective layer formed on commercial pure Al surface anode after immersing in 4M NaOH solution with 0.2M ZnO (a-b), and in 4M NaOH with 0.2M ZnO + 3 g/L neem extract (c-d).

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ABSTRACT

Aluminium metal is highly regarded as a superior choice for anode material in Metal-Air Battery due to its advantageous capacity and energy density. Nevertheless, the challenge of anodic parasitic corrosion significantly impedes the advancement of Aluminium-Air Battery performance. In the present work, attempts were made to investigate the corrosion inhibition effects of both ZnO and neem extract in alkaline solution on commercially pure aluminium. The synergistic effect of ZnO and neem extract on the aluminium anode in a 4 M NaOH was studied using hydrogen evolution experiment, electrochemical measurements, and surface analysis method. The H_2 evolution shows that it effectively reduces self-corrosion of the aluminium anode and increases inhibition efficiency up to 98%, at the optimum concentration 0.2M ZnO and 3g/L neem extract .Scanning electron microscopy (SEM) is used to examine the surface morphology of aluminium and reveal the chemical composition of the extract as well as the formation mechanism of the protective film on the aluminium surface. The interaction between ZnO particles and neem extract is responsible for the formation of this film. The surface characterization was carried out using infrared spectroscopy techniques.

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MULTICOMPONENT SYNTHESIS OF CHROMENE DERIVATIVES

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DECLARATION

I ARUN KJ **(Reg No. CCAWMCH009)** do hereby declare that the dissertation entitled, "MULTICOMPONENT SYNTHESIS OF CHROMENE DERIVATIVES" submitted to the University of Calicut in partial fulfilment of the requirement for the award of degree of Master of Science is a genuine record of project work done by me under the guidance of Dr.Arun.S, Assistant Professor of the department of chemistry, Christ College (Autonomous) Irinjalakuda and has not been submitted to any university or institution for the award of any degree or diploma.

I further declare that the results presented in this work and consideration made therein contribute to the advancement of knowledge in Chemistry.

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ACKNOWLEDGEMENT

First and foremost, I would like to extend my sincere and heartfelt gratitude to my guide Dr.Arun.S, Department of Chemistry ,Christ College (Autonomous) Irinjalakuda for his valuable and inspiring guidance, critical assessment and constant encouragement throughout all stages of this work. I'm greatly indebted to him for the successful completion of this work within the specified period.

I express my sincere gratitude to Dr.Rani Varghese, Head of the Department of Chemistry and to all faculty members in the Department of Chemistry for their support.

I wish to acknowledge my gratitude to Rev.Dr.Jolly Andrews, CMI, Principal of Christ College (Autonomous) Irinjalakuda for providing timely help and necessary facilities.

I extend my heartfelt thanks to Seena Chakko and Ramya K, research scholars, Christ College (Autonomous) Irinjalakuda for their valuable support and ceaseless assistance throughout the work.

I must express my love and sincere gratitude to my parents for their cooperation and the support that they provided me during the project work. I cheerfully express my profound thanks to my dear friends for their support and cooperation.

Above all I humbly thank God Almighty who showered his abundant grace on me to make this project a success.

ARUN K J

ABBREVIATIONS USED

(1) MCRs - MULTICOMPONENT REACTIONS

(2) MDR - MULTICOMPONENT DOMINO REACTIONS

(3) SAR - STRUCTURE ACTIVITY RELATIONSHIP

(4) DOS - DIVERSITY ORIENTED SYNTHESIS

(5) NCE - NOVEL CHEMICAL ENTITIES

CONTENTS

Raman Spectroscopy Combined with Partial Least Squares Regression for Detection of Adulteration in Coconut Oil

Dissertation submitted to the Christ College (Autonomous) in partial fulfilment of the requirement for the Degree of

MASTER OF SCIENCE

IN

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I, **Athira P B (Reg No: CCAWMCH010)** do hereby declare that, this dissertation work entitled **"Raman Spectroscopy Combined with Partial Least Squares Regression for Detection of Adulteration in Coconut Oil"** submitted to the University of Calicut in partial fulfilment of the requirement for the award of the degree of Master of Science was carried under the guidance of **Dr. K. N. Narayanan Unni,** Senior Principal Scientist, CSIR - National Institute for Interdisciplinary Science and Technology (NIIST), Thiruvananthapuram, and it is a record of original project work carried out by me and it has not previously formed the basis for the award of, any degree, Diploma fellowship or other similar titles of recognition by any other university or institutions.

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I would like to express my deepest gratitude to all those who have supported me throughout the completion of this MSc project.

First and foremost, I am profoundly grateful to my external supervisor, **Dr. K. N. Narayanan Unni**, Senior Principal Scientist, National Institute for Interdisciplinary Science and Technology (NIIST), Thiruvananthapuram, for allowing me to do my M.Sc. project under his supervision. I thank him for his guidance, encouragement, and involvement throughout my research work.

I extend my sincere thanks to the Director of NIIST, **Dr. C. Anandharamakrishnan**, for providing the necessary infrastructure and lab facilities that were essential for conducting this research.

I express my gratitude to our Principal **Rev. Dr. Jolly Andrews CMI** and our Head of the Department, **Dr. Rani Varghese,** Department of Chemistry, Christ College (Autonomous), Irinjalakuda, for providing an opportunity to do the project work.

My heartfelt appreciation goes to my internal supervisor, **Dr. Tom Cherian**, Asst. Professor, Department of Chemistry, Christ College (Autonomous), for his constant guidance and sincere support.

I am extremely thankful to my co-guide, **Nandu A**, Ph D Scholar, National Institute for Interdisciplinary Science and Technology (NIIST), Thiruvananthapuram, for his persistent support, valuable suggestions, and for always being available to provide assistance and guidance throughout the project.

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Thank you all for your invaluable contributions and support.

Athira P B

ABSTRACT

This work investigates the application of Partial Least Squares Regression (PLSR) models for detecting and quantifying adulteration in edible oils using Raman spectroscopy. The study focuses on coconut oil adulterated with various oils such as corn, cottonseed, flaxseed, groundnut, nigerseed, palm kernel, palmolein, rice bran, soybean, and sunflower oils. Key quality parameters like iodine value, saponification value, and acid value were assessed to ensure oil quality. Raman spectroscopy provided molecular fingerprints of the oils, facilitating the identification of adulterants. The PLSR models were calibrated using MATLAB, demonstrating high accuracy with R^2 values nearing 0.999 and low RMSEC values across different systems. However, the prediction accuracy varied, highlighting the need for further optimization. For instance, the coconut-soybean oil system showed an R^2 of 0.999 in calibration but had a higher RMSEP in prediction. Similarly, the coconut-sunflower oil system exhibited excellent calibration results but required improvement in prediction accuracy. The research underscores the potential of combining Raman spectroscopy with PLSR models for rapid, non-destructive adulteration detection in edible oils, though future work should focus on enhancing predictive capabilities for practical applications in the food industry.

Keywords: Raman spectroscopy, Partial Least Squares Regression (PLSR), edible oil adulteration, Coconut oil.

CONTENTS

ELECTROCHEMICAL AND SPECTROSCOPIC STUDIES OF SPRAY COATED WO_3 FILMS

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DECLARATION

I, ATHIRA SUDEESH (Register No: CCAWMCH011), hereby declare that this project work entitled "**ELECTROCHEMICAL AND SPECTROSCOPIC STUDIES OF SPRAY COATED WO_3 FILMS**" is an authentic record of the project work carried out by me during my M.Sc. degree course under the guidance of Dr. BISWAPRIYA DEB, Senior Principal Scientist, Centre for Sustainable Energy Technologies, CSIR- National Institute for Interdisciplinary Science and Technology (CSIR- NIIST), Thiruvananthapuram. The report is submitted to University of Calicut to partially fulfill the requirements for the Master of Science Degree in CHEMISTRY from Christ College (Autonomous), Irinjalakuda. No part of this is reported for any other degree or diploma earlier.

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Date : 30th June, 2024

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This is to certify that the work embodied in the project report entitled **""Electrochemical and Spectroscopic studies of spray coated WO_3 films""** has been carried out by **Ms. Athira Sudeesh (CCAWMCH011)** under my supervision and the co-supervision of Mr. Nayan Dev Madhavan K at the Centre for Sustainable Energy Technologies (CSET), CSIR-National Institute for Interdisciplinary Science and Technology (CSIR-NIIST), Thiruvananthapuram during the period April to June 2024 in partial fulfilment of the Degree of Master of Science in Chemistry, Calicut university, Postgraduate and Research Department of Chemistry, Christ College (Autonomous). The present work or any part thereof has not been submitted elsewhere for the award of any other degree.

Nayan Dev Madhavan K
Research Scholar

Dr. Biswapriya Deb
Senior Principal scientist

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ACKNOWLEDGEMENT

With utmost respect, I wish to convey my deepest gratitude to my project supervisor, Dr. Biswapriya Deb, Senior Principal Scientist, AcSIR Associate Professor at the Centre for Sustainable Energy Technologies, CSIR- National Institute for Interdisciplinary Science and Technology (CSIR-NIIST), Thiruvananthapuram. His guidance, insightful suggestions, and unwavering encouragement have been pivotal in the successful completion of this project.

I am profoundly grateful to Dr. C. Anandharamakrishnan, Director, CSIR-NIIST, Thiruvananthapuram for providing the essential infrastructure for this project. I also extend my heartfelt thanks to Dr. K.N. Narayanan Unni, Head of the Centre for Sustainable Energy Technologies (CSET), and Dr. U.S. Hareesh, HRAD, CSIR-NIIST, Thiruvananthapuram, for offering me the opportunity to undertake this project work.

Words cannot adequately express my deep gratitude to Mr. Nayan Dev Madhavan K. for his hard work, support, and patience, which contributed to the successful and timely completion of my project. I feel fortunate to have had Dr. Shaiju S. S., Ms. Ranjana Venugopal, Ms. Chinnu V. Devan, Ms. Amritha P., Ms. Meritta James, Ms. Sivakrishna S. P., Ms. Anjitha Dinakaran, Ms. Arathy C. B., Ms. Meenu C. Nair, Ms. Anju C. L., Mr. Sandeesh, Mr. Salil N., and Mr. Nithin R. S. as seniors. Their shared knowledge, valuable suggestions, advice, and timely assistance have been invaluable to me.

I am also deeply thankful to Dr. Rani Varghese, Head of the Department of Chemistry and Assistant Professor, Christ College (Autonomous), Irinjalakuda, Dr. V. T. Joy, former Head of the Department of Chemistry and Associate Professor, Christ College (Autonomous), Irinjalakuda, and Dr. Tom Cherian, Assistant Professor, Christ College (Autonomous), Irinjalakuda for their immense support and encouragement.

I am grateful to my project mates, Farhana Fathima K., Fathima Nitha C. K., Nasheeda P. for their support and assistance throughout this work. I owe a deep debt of gratitude to my parents and family members for their unwavering love and support throughout my life. Above all, I am deeply thankful to God Almighty for blessing me and giving me the courage to persevere.

ATHIRA SUDEESH

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LIST OF FIGURES

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PREFACE

Electrochromic (EC) systems, as exemplified by active chromogenic glasses, offer the capability to modulate optical transparency through the adjustment of electric voltage or current. An electrochromic device (ECD) typically comprises a multi-layered sandwich structure, including an active layer, an electrolyte layer, and a charge-balancing counter layer, all positioned between two conductive glass plates. The device functions by electrically driving ion insertion or extraction from the electrolyte, facilitating a reversible redox reaction in the active or counter electrode. These materials are gaining significant traction in the global market, with applications in energy-saving smart windows, facades, aesthetic and privacy control glasses, non-emissive displays, smart labels, sensors, and signboards. Investigating electrochemical methods to enhance both the EC response and storage capacity is essential for maximizing the energy-saving potential of these glasses. While various materials are employed as light diffusers in windows to reduce glare and maximize daylight utilization, these materials generally lack smart window properties. In this study, we investigate the feasibility of integrating a light diffuser into smart windows using hazy WO_3 thin films for electrochromic devices. We aim to compare and evaluate the performance of hazy WO_3 films against transparent WO_3 films through a series of characterization techniques, demonstrating the comparison of hazy and transparent films and study elaborates increase in number of layers of hazy film and changes in important properties. This report provides a comprehensive analysis of the structural, morphological, optical, and electrochemical properties of these films, offering valuable insights into their potential applications in innovative and energy-efficient smart window technologies. The findings contribute to the advancement of smart window solutions, fostering energy efficiency and sustainability in modern architectural practices.

ABSTRACT

This work compares the properties and performance of transparent (T200) and hazy (H200) WO_3 films, fabricated via spray-coating, for smart window applications. XRD confirmed the amorphous structure of both films. Profilometer measurements showed T200 films, coated at lower temperatures, are thicker than H200 films.

OPM and SEM revealed morphological differences: hazy films had spherical-shaped pits, while transparent films had a tile-like structure. AFM confirmed higher roughness in hazy films, causing more light scattering. UV-VIS NIR spectroscopy showed lower transmittance for H200 due to increased scattering.

Electrochemical studies indicated Li^+ ion insertion with voltage changes, and GCD experiments showed higher capacitance values for hazy films with more layers. Cyclic stability tests showed high optical contrast for both films, with decreasing transmittance over time. Coloration efficiency was higher for T200, and response time analysis revealed faster responses for transparent films.

Transparent WO_3 films are suitable for applications requiring natural daylight and heat control, while hazy films are ideal for privacy and glare reduction. Both films offer dynamic control over window properties, contributing to sustainable building designs and innovative smart window solutions.

SPECTROPHOTOMETRIC AND COLORIMETRIC SENSOR FOR BILIRUBIN USING ZINC PHTHALOCYANINE

Dissertation submitted to the Christ College (Autonomous) in partial fulfilment of the requirement for the Degree of

MASTER OF SCIENCE

IN

CHEMISTRY

Submitted by

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DECLARATION

I hereby declare that the dissertation entitled "***Spectrophotometric and Colorimetric Sensor for Bilirubin using Zinc Phthalocyanine***" is the original work done by me, under the supervision and guidance of **Dr. Leena R**, Assistant Professor, Department of Applied Chemistry, Cochin University of Science and Technology (CUSAT), Kochi, in partial fulfilment of the requirements for the award of the degree of Master of Science in Chemistry. No part of this dissertation has been presented earlier to award any degree, diploma, or other titles of recognition.

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ACKNOWLEDGEMENT

At the very outset of this report, I would like to extend my sincere obligation to all the persons who have helped me in this endeavour. Without their active guidance, help, cooperation, and encouragement, I would not have made headway in this project.

I bow my head and offer my thanks to God Almighty for the never-failing grace and blessings to complete the project work.

I express my gratitude to Principal ***Rev. Dr. Jolly Andrews C.M.I*** *,our Head of the Department* ***Dr. Rani Varghese*** *& Co-guide Professor* ***Dr. Digna Varghese*** *for providing an opportunity to do the project work.*

I express my heartfelt gratitude to my external supervisor ***Dr. Leena R****, Assistant Professor, Department of Applied Chemistry, Cochin University of Science and Technology (CUSAT), Kochi, for allowing me to do my M.Sc. project under her supervision. I thank her for her guidance, continued assistance, encouragement, and involvement throughout my research work.*

I sincerely thank ***Ms. Shamna S****, Research Scholar, Department of Applied Chemistry, Cochin University of Science and Technology (CUSAT), Kochi, for her great support, guidance, and timely assistance during my project work.*

I like to thank my lab mates in CUSAT, Akhila, Jennifer, Sandhra & Digil, for their positive companionship and selfless support.

I also acknowledge with a deep sense of reverence, my gratitude towards my parents and members of my family, who has always supported me morally as well as economically.

Last but not least gratitude goes to all of my friends, who directly or indirectly helped me to complete this project report.

ABSTRACT

This project focuses on the development and characterization of a spectrophotometric and colorimetric sensor for bilirubin using Zinc(II) phthalocyanine pyridine (ZnPcPy). ZnPcPy was synthesized through a multistep process involving 4-nitrophthalonitrile, 3-hydroxypyridine, pentanol, zinc acetate, cobalt chloride, and potassium carbonate. Spectrophotometric analysis confirmed that ZnPcPy forms a stable complex with bilirubin, exhibiting a distinctive colour change whose intensity correlates with bilirubin concentration. Concentration studies, performed both spectrophotometrically and colorimetrically, demonstrated that the sensor can detect bilirubin with a detection limit of 2.3×10^{-7} M. Selectivity and interference studies against co-existing biomolecules further validated the specificity of ZnPcPy towards bilirubin. Practical utility and reproducibility assessments underscored the sensor's potential for clinical applications. This study establishes ZnPcPy as a promising candidate for bilirubin sensing due to its sensitivity, selectivity, and robust performance characteristics.

SEPARATION AND ESTIMATION OF BIOACTIVES FROM GREEN LEAVES EXTRACTS

Dissertation submitted to the Christ College (Autonomous) in partial fulfilment of the requirement for the Degree of

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IN

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Submitted by

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2. Dr. Mini Thomas
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DECLARATION

I, GREESHMA.K.V (Reg.No.CCAWMCH013) do hereby declare that, this dissertation work entitled **"SEPARATION AND ESTIMATION OF BIOACTIVES FROM GREEN LEAVE EXTRACTS"** submitted to the University of Calicut in Partial fulfilment of the requirement for the award of the degree of Master of Science was carried under the guidance of Dr.Robinson P. Ponminiessary, Dean of Academics,Assistant professor, Department of Chemistry, Christ College, Irinjalakuda and it is a record of original project work carried out by me and it has not previously formed the basis for the award of, any degree, Diploma fellowship or other similar titles of recognition by any other university or institutions.

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ACKNOWLEDGEMENT

I extend my sincere and heartfelt gratitude to my project guide Dr. Robinson P. Ponmaniessary, dean of academics, assistant professor, Department of Chemistry, Christ College, Irinjalakuda for his valuable and inspiring guidance, critical assessment and constant encouragement at all stages of this project. I am greatly indebted to him for the completion of this work in the specified period.

I express my sincere thanks to Dr. Rani Varghese, Head, PG Department of Chemistry, for granting permission to carrying my project. I would like to express my gratitude to all the faculty members in the Department of Chemistry for their inspiration and guidance to complete this work.

I wish to acknowledge my gratitude to Rev. Dr. Jolly Andrews, C.M.I, Principal of Christ College, Irinjalakuda and Library staff of the Christ College for providing the timely help and necessary facilities.

I cheerfully express my profound thanks to all my classmates for their support and co-operation.

Above all I humbly thank God Almighty, whose sustaining grace has been sufficient for me to complete this endeavour.

GREESHMA.K.V

TABLE OF CONTENTS

ABSTRACT

This work focuses on the quantitative estimation of bioactive compounds such as protein and total phenolic as well as a qualitative estimation of cyanide by using the leaf extracts. The aim behind this work was to let know people the existance of plant protein called Rubisco which claimed to be of zero allergens and high nutritive value. It also possesses high phenolic content which is a key factor for enhancing the antioxidant properties. Qualitative estimation for cyanide shows a positive result and indicate the need to remove them and make useful food supplements from plant leaves.

Key words: protein, total phenolic content, cyanide and FCR

ENHANCING THE CORROSION RESISTANCE OF THE ALUMINIUM ANODE IN ALKALINE ELECTROLYTE USING A GLYCEROL-BASED WATER-IN-SALT ELECTROLYTE WITH DL α-LIPOIC ACID ADDITIVES

Dissertation submitted to the Christ College (Autonomous) in partial fulfilment of the requirement for the Degree of

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IN

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2022-2024

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DECLARATION

I Nihala C.M, hereby declare that the project entitled "**Enhancing the corrosion resistance of the aluminium anode in alkaline electrolyte using a Glycerol-Based Water-in-Salt Electrolyte with DL α-Lipoic Acid additives**" is a piece of original work I completed at the Materials Science and Technology Division, National Institute for Interdisciplinary Science and Technology (CSIR-NIIST), Thiruvananthapuram, Kerala under the guidance of Dr T P D Rajan in partial fulfilment of the requirements for the completion of Degree of Master of Science in Chemistry from Christ Collage Irinjalakuda. I affirm that the work submitted for this project hasn't been submitted for credit toward any other degree or qualification and all cited sources of information, references, and data are appropriately acknowledged.

June 2024

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T.P.D. Rajan

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CERTIFICATE

This is to certify that the project work entitled **"Enhancing the corrosion resistance of the aluminium anode in alkaline electrolyte using a Glycerol-Based Water-in Salt Electrolyte with DL α-Lipoic Acid additives"** is an authentic record of the work carried out by NIHALA C.M (Reg.No.CCAWMCH014) under the co-guidance of Dr. Titto Varughese, Assistant Professor, Department of Chemistry, Christ College (Autonomous), Irinjalakuda, for the partial fulfilment of the award of the degree of Master of Science in Chemistry during the academic year 2022-2024.

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ABSTRACT

Aluminium metal is highly regarded as an excellent choice for the anode material in metal-air fuel cells. The self-corrosion of aluminium anode in aqueous alkaline electrolyte hinders the wider application of Al-air battery. The addition of corrosion inhibitors to the electrolyte significantly reduces the corrosion behaviour of Al anode. In this work, we introduce a glycerol system as a water-in-salt electrolyte to modify the conventional aqueous solution and investigate the effect of DL α-lipoic acid as a corrosion inhibitor to further reduce the corrosion of Al anode in 4M NaOH solution. The combination of 20% glycerol and 3mM lipoic acid exhibits an inhibition efficiency of 72% in Hydrogen evolution test. Electrochemical measurements such as Electron impedance spectroscopy, Potentiodynamic polarisation were carried out. The results indicate a negative shift in open circuit potential, suggesting that additives play a role in suppressing the cathodic corrosion process and addition of lipoic acid further decreases the corrosion current (I_{corr}), achieving a 67% efficiency. Surface morphology reveals the interaction of lipoic acid molecules on the surface of aluminium. Surface characterisation was carried out using ATR-FTIR Spectroscopy.