Facile Preparation of Chitosan/Clay Biocomposites and their Characterizations

A thesis
submitted to Bodoland University
for the award of the degree

of

Doctor of Philosophy

by

Madhabi Bhattacharjee
(FINAL.CHE0001 of 2017-2018)

Under the supervision of
Dr. Dhrubajyoti Haloi
Former Associate Professor

Department of Chemistry
Bodoland University, Kokrajhar
Assam-783370
24th March-2025

DECLARATION

I, Madhabi Bhattacharjee, hereby declare that the thesis entitled *"Facile Preparation of Chitosan/Clay Biocomposites and their Characterizations"* submitted in the fulfilment of the requirements for the degree of Doctor of Philosophy (PhD) at Bodoland University is my original work. I affirm that the thesis is based on my own research and has not been previously submitted for any other degree. All sources and references used in this work have been properly cited and acknowledged. I have not engaged in any form of academic misconduct, including plagiarism. Any assistance received in the preparation of this thesis has been acknowledged in the appropriate section. I understand that any violation of academic integrity or false statements in this declaration may have serious consequences.

Madhabi Bhattacharjee

Madhabi Bhattacharjee
(FINAL.CHE0001 of 2017-2018)

Date: 24/03/2025

ACKNOWLEDGEMENT

First of all, I would like to thank my supervisor Dr. Dhrubajyoti Haloi, Former Associate Professor, Department of Chemistry, Bodoland University, Assam, for accepting me as a research student and introducing me to this field. His passion, valuable guidance, constructive feedback, and cooperative spirit throughout this investigation assisted me in overcoming all the challenges encountered during the research process.

I would like to thank Bodoland University, Kokrajhar for giving me an opportunity to pursue my Ph.D. degree.

I want to convey my appreciation to all the faculty members in the Departments of Chemistry, Physics, Botany, and Zoology at Bodoland University for their valuable assistance and encouragement throughout the progression of my research work. Throughout the duration of my time here, the non-teaching staff in the Chemistry Department have been exceptionally kind to me.

I would like to acknowledge the help and co-operation of my lab mates from Department of Chemistry, Mr. Achinta Medhi and Dr. Maromi Roy. I am greatly indebted to Dr. Biswajit Nath for helping me at numerous occasions. I extend my gratitude to all my friends, seniors, and juniors for their timely assistance.

I express my gratitude to Dr. Nikhil K. Singha, Rubber Technology Centre, IIT Kharagpur; Dr. Nabendu B. Pramanik, ICT Mumbai - IOC Bhubaneswar Campus Bhubaneswar, Odisha; Dr. Mousmi Saikia, Department of Herbal Science & Technology, ADP College, Nagaon; Mr. Pranab Borah, Department of Botany, Cotton University, Guwahati; Dr. Jayanta Barman, Department of Physics, ADP College, Nagaon; Dr. Nishant R. M. Hulle, Department of Food Engineering & Technology, Tezpur University, Assam; Dr. Bikash Baishya, CBMR Lucknow; Dr. Manabendra Sarma, Department of Chemistry, IIT Guwahati; Mr. Haobam K. Singh, Department of Chemistry, IIT Guwahati and Mr. Rabu R. Changmai, Department of Chemistry, IIT Guwahati; Mr. Apurba Taye, Department of Applied Sciences, Tezpur University, Mr. Shyam Goswami, Drugs and Narcotics Division, Directorate of Forensic Science, Guwahati, for their help during different experiments related to my research work and cooperation in analysing different samples.

Finally, I want to express my lasting gratitude to my family for their unwavering love, blessings, patience, support, and encouragement.

Madhabi Bhattacharjee

Date: 24/03/2025

Madhabi Bhattacharjee

CERTIFICATE

This is to certify that the thesis entitled "***Facile Preparation of Chitosan/Clay Biocomposites and their Characterizations***" submitted by ***Miss Madhabi Bhattacharjee*** to Bodoland University was carried out under my supervision and is worthy of consideration for the award of the degree of Doctor of Philosophy of the University.

Dhrubajyoti Haloi

(Dhrubajyoti Haloi)

Dhrubajyoti Haloi, *MSc (GU), MTech & PhD (IIT Kgp)*
Associate Professor (Chemistry)
Dept. of Applied Sciences, Tezpur University
Mobile No: +91-78967-77085 / +91-88223-82803
E-mail: djhaloi@tezu.ernet.in / dhruba2k3@gmail.com
Web: http://www.tezu.ernet.in/appsc/

LIST OF ABBREVIATIONS

BIBB	2-Bromoisobutyryl bromide
B3LYP	Becke 3-parameter lee yang parr
BNTN	Bentonite
BPB	2-Bromopropionyl bromide
CS	Chitosan
DMF	Dimethylformamide
DSC	Differential scanning calorimetry
FT-IR	Fourier transform infrared
HOMO	Highest occupied molecular orbital
IRC	Intrinsic reaction coordinates
KAO	Kaolin
LAF	Laminar air flow
LUMO	Lowest unoccupied molecular orbital
MCS	Modified chitosan
MEP	Molecular electrostatic potential
MIC	Minimal inhibitory concentration
NMR	Nuclear magnetic resonance
SEM	Scanning electron microscope
SIO	Silica
TEA	Triethylamine
TGA	Thermogravimetric analysis
THF	Tetrahydrofuran
TS	Transition state
UTM	Universal testing machine
UV/Vis	Ultraviolet/Visible
XRD	X-ray diffraction

LIST OF SYMBOLS

Å	Angstrom
cm	Centimeter
°C	Degree Celsius
g	Gram
h	Hours
kg	Kilogram
kV	Kilovolts
mA	Milliampere
MHz	Megahertz
min	Minute
mL	Millilitre
mm	Millimeter
mmol	Millimole
mol	Mole
MPa	Megapascal
µg	Microgram
µL	Microlitre
N	Newton
nm	Nanometer
rpm	Revolutions per minute
T_g	Glass transition temperature
T_{max}	Temperature at which maximum mass loss occurs
T_{onset}	Onset temperature of the degradation process
ppm	Parts per million
%	Percentage
λ	Wavelength

LIST OF TABLES

LIST OF FIGURES

Figure 5.8. Antimicrobial activities showing zone of inhibition by CS/clay biocomposite films against (a) *Escherichia coli* and (b) *Bacillus subtilis*.

Figure 5.9. Plots of swelling test of various CS/clay biocomposite films.