Dedicated to...

My Parents

Declaration

"I declare that this written submission represents my ideas in my own words and where other's ideas or words have been included, I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in my submission. I understand that any violation of the above will be cause for disciplinary action as per the rules and regulations of the Institute."

Date: 12-06-2023 **(Dipika Konwar)**

Place: Tezpur University Registration No. TZ201063 of 2019

TEZPUR UNIVERSITY

(A Central University established by an Act of Parliament)

Dr. Utpal Bora
Associate Professor

Email: ubora@tezu.ernet.in
Ph : +91 (3712) 275067 (O)
+91 9435699636 (Mob)
Fax: +91 (3712) 267005/6

Certificate from Supervisor

This is to certify that the thesis entitled "***Strategic Functionalization of Indoles and Oxidative Coupling Reactions***" submitted to the School of Sciences, Tezpur University in partial fulfillment for the award of the degree of Doctor of Philosophy in Chemical Sciences is a record of research work carried out by **Ms. Dipika Konwar** under my supervision and guidance. She has been duly registered (Registration No. TZ201063 of 2019) and the thesis presented is worthy of being considered for Ph.D. Degree.

All help received by her from various sources have been duly acknowledged.

No part of this thesis has been submitted elsewhere for award of any other degree.

Date: 12-06-2023

Place: Tezpur University

(Dr. Utpal Bora)

Supervisor

TEZPUR UNIVERSITY

(A Central University established by an Act of Parliament)
Napaam, Tezpur–784028, District–Sonitpur, Assam, India

CERTIFICATE OF THE EXTERNAL EXAMINER AND ODEC

This is to certify that the thesis entitled "***Strategic Functionalization of Indoles and Oxidative Coupling Reactions***" submitted by **Ms. Dipika Konwar** to the School of Sciences, Tezpur University in partial fulfillment for the award of the degree of Doctor of Philosophy in the Department of Chemical Sciences has been examined by us on ……12/06/2023…..and found to be satisfactory.

The committee recommends for the award of the Degree of Doctor of Philosophy.

Signature of:

Supervisor

Date: 12-06-2023

External Examiner

Date: 12-06-2023

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Dipika Konwar

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General Experimental Information

All the chemicals were purchased commercially and used directly without any purification. For analytical thin-layer chromatography (TLC) Merck silica gel 60F254 plates were used and analysis was done using short-wave UV light (254 nm). Column Chromatographic separations were done by distilled solvents (hexane:ethyl acetate) over silica gel (60-120 or 100-200 mesh). ^{1}H and ^{13}C NMR spectra were recorded on a JEOL JNM ECS NMR spectrometer using $CDCl_3$ and DMSO-d_6 as solvents and TMS as an internal standard. Chemical shifts (δ) were reported in parts-per-million (ppm) and NMR spectra were plotted in MestReNova software. UV-visible spectra were recorded in a UV-visible spectrophotometer (Shimadzu Corporation, UV-2550). HRMS data were obtained from the electron spray ionization technique on a Q-TOF mass analyzer.

The characterization of the synthesized catalysts was done by using FT-IR, Powder XRD, SEM-EDX, TEM, BET, and XPS analyses. FT-IR spectra were recorded on a PerkinElmer Frontier MIR FT-IR spectrometer. Powder X-ray diffraction studies were carried out using a Rigaku Miniflex X-ray diffractometer (D8 FOCUS and MINIFLEX, BRUKER AXS, Germany and Rigaku Corporation, Japan), equipped with CuKα radiation (λ = 0.1542 nm, scanning rate = 0.05 s^{-1}) at 30 kV and 15 mA, where the data obtained was in the 2θ range of 10° to 100°. Transmission Electron Microscope (TEM) (JEM-2100, Jeol, Japan), Scanning Electron Microscope (SEM, JEOL-JSM-6390LV, Japan), and elemental dispersive X-ray analysis techniques (JEOL-JSM-6390LV, Japan) were employed for morphological and elemental analyses. The SEM and TEM analyses were carried out using ImageJ software. The BET analysis was done in Quantachrome NOVA 2200 analyzer at 77 K where degassing of the samples were carried out at 120 °C for 5 hours under N_2 atmosphere. The amount of metal incorporation was determined by ICP-OES analysis (Perkin Elmer Optima 5300 DV). The elemental composite and chemical bonding information were analyzed by high resolution X-ray photoelectron spectroscopy (XPS) measurements (Thermo-Scientific ESCALAB Xi+ spectrometer) with a monochromatic Al Kα X-ray source (1486.6 eV) and a spherical energy analyzer that operates in the CAE (constant analyzer energy) mode. The CAE for high-resolution spectra is recorded at 50 eV.

Abbreviations and Symbols

%	percentage
δ	Chemical shift
J	Coupling constant
λ	Wavelength
Ar	Aryl
Ac	Acetyl
BET	Brunauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
°C	degree Centrigrade
DMA	Dimethylacetamide
DMF	*N*,*N*-dimethylformamide
DMSO	Dimethylsulfoxide
DTBP	Di-*tert*-butylperoxide
EDX	Energy Dispersive X-ray
EtOH	Ethanol
equiv.	equivalent
eV	electron volt
ESI-MS	Electron Spray Ionization-Mass Spectrometry
FT-IR	Fourier transformed infra-red spectroscopy
g	gram
HRMS	High Resolution Mass Spectrometry
h	hour
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
*i*PrOH	Isopropanol
JCPDS	Joint Committee on Powder Diffraction Standards
kV	kilovolt
mA	milliampere
MeOH	Methanol
MHz	Mega-Hertz
mmol	milli mole
mg	milligram

mL	milli Liter
m	multiplet
2-MeTHF	2-Methyltetrahydrofuran
m/z	Atomic mass units per charge
NHC	*N*-heterocyclic carbene
NMP	*N*-Methyl-2-pyrrolidone
nm	nanometer
NMR	Nuclear Magnetic Resonance
NP	Nanoparticle
PEG	Polyethylene glycol
ppm	parts-per-million
p-XRD	Powder X-ray diffraction analysis
rt	room temperature
rGO	Reduced Graphene Oxide
SAED	Selected Area Electron Diffraction
SEM	Scanning Electron Microscope
*t*Bu	*tert*-butyl
TBHP	*tert*-butylhydroperoxide
TBP	Tributyl phosphate
TEM	Transmission Electron Microscope
TEMPO	2,2,6,6-tetramethylpiperidine-*N*-oxyl
TFA	Trifluoroacetic acid
THF	Tetrahydrofuran
TLC	Thin Layer Chromatography
TMS	Tetramethylsilane
TosMIC	Toluenesulfonylmethyl isocyanide
UV-Vis	Ultra violet-visible
XPS	X-ray Photoelectron Spectroscopy