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## DECLARATION

I declare that the thesis entitled “**PREPARATION, CHARACTERIZATION OF BIMETALLIC NANOPARTICLES SOAKED ON POLY-IONIC RESINS AND THEIR CATALYTIC APPLICATIONS**” has been prepared by me under the guidance of Dr. Basudeb Basu, Professor of Chemistry, University of North Bengal. No part of this thesis has formed the basis for the award of any degree or fellowship previously.

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## CERTIFICATE

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## **PREFACE**

Ion-exchange materials are insoluble substances containing loosely held ions which are able to be exchanged with other ions in solutions which come in contact with them and normally obtained as beads of 1-2 mm diameter. The most typical ion-exchange resins are based on cross-linked polystyrene. Ion-exchange resins are widely used in different separation, purification and decontamination process. This heterogeneous material has been utilized in different organic transformations too. On the other hand, graphene belongs to a new class of carbon nanomaterials, and graphene oxide (GO) possesses a range of reactive oxygen functional groups that render it to be a good candidate for use in the several organic transformations.

The present research work represents the multidisciplinary branches of material chemistry, nanocatalysis and green chemistry. This dissertation begins with a brief review on heterogeneous catalysis by metal nanocomposites followed by the application of poly-ionic resin hydroxide in disulfide bond-forming reactions. The next chapters of the thesis deal with poly-ionic resin-supported mono- and bi-metallic nanocatalysts preparation, characterization and catalytic applications in diverse cross-coupling reactions. The last three chapters describe applications of graphene oxide (GO) as the 'carbocatalyst' in different organic transformations, and finally a Ni/RGO nanocomposite has been shown to be a highly efficient heterogeneous catalyst for the Kumada-Corriu and C-S cross-coupling reactions.

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## **APPENDIX A:**

### **List of Publications**

1. “An efficient metal-free synthesis of organic disulfides from thiocyanates using poly-ionic resin hydroxide in aqueous medium” **Debasish Sengupta** and Basudeb Basu, *Tetrahedron Lett.*, **2013**, *18*, 2277–2281.
2. “An efficient heterogeneous catalyst (CuO@ARF) for on–water C–S coupling reaction: an application to the synthesis of phenothiazine structural scaffold” **Debasish Sengupta** and Basudeb Basu, *Organic and Medicinal Chemistry Letters*, **Accepted**.
3. “Pd/Cu bimetallic nanoparticles embedded in macroporous ion-exchange resins: an excellent heterogeneous catalyst for the Sonogashira reaction” **Debasish Sengupta**, Jony Saha, Goutam De and Basudeb Basu, *J. Mater. Chem. A*, **2014**, *2*, 3986–3992.
4. “Graphene oxide as a carbocatalyst: the first example of a one-pot sequential dehydration–hydrothiolation of secondary aryl alcohols” Basudeb Basu, Samir Kundu and **Debasish Sengupta**, *RSC Adv.*, **2013**, *3*, 22130–22134.
5. “Graphene Oxide (GO)–catalyzed chemoselective thioacetalization of aldehydes under solvent-free conditions” Babli Roy, **Debasish Sengupta** and Basudeb Basu, *Tetrahedron Lett.*, **2014**, *55*, 6596–6600.
6. “Reduced graphene oxide supported Ni nanoparticles: a high performance reusable heterogeneous catalyst for Kumada–Corriu cross-coupling reactions” Koushik Bhowmik, **Debasish Sengupta**, Basudeb Basu and Goutam De, *RSC Adv.*, **2014**, *4*, 35442–35448.
7. “In Quest of “Stereoselective Switch” for On-Water Hydrothiolation of Terminal Alkynes Using Different Additives and Green Synthesis of Vicinal Dithioethers” Basudeb Basu, Kinkar Biswas, Samir Kundu and **Debasish Sengupta**, *Org. Chem. Int.*, doi:10.1155/2014/358932.

## **APPENDIX B:**

- **Oral Presentation** in the One-day Seminar in Chemistry organized by the **Chemical Research Society of India** NBU Local Chapter in collaboration with Department of Chemistry, University of North Bengal, held at of University of North Bengal on September 12, 2014.

### **Poster Presentation**

- Pd/Cu bimetallic nanoparticles soaked on poly-ionic resins: an excellent heterogeneous catalyst for Suzuki-Miyaura and Heck coupling reactions, **Debasish Sengupta**, Babli Roy, Samir Kundu and Basudeb Basu, *National Seminar on Frontiers in Chemistry 2014* held at University of North Bengal, Darjeeling, March 11, 2014.
- Heterogeneous Bimetallic Nano Particles (NPS): Preparation Characterization and Application in catalysis, **Debasish Sengupta**, Samir Kundu, Susmita Paul and Basudeb Basu\*, *National Seminar on Frontiers in Chemistry 2013* held at University of North Bengal, Darjeeling, February 28, 2013.

## ABBREVIATION

ARF	Amberlite resin formate	NMP	<i>N</i> -Methylpyrrolidone
Bu	Butyl	nm	nanometer
CNT	Carbon nanotube	NMR	Nuclear Magnetic Resonance
°C	Degree Celsius	PEG	Polyethylene glycol
d	Doublets	ppm	parts per million
dd	Doublet of a doublet	PVP	Polyvinylpyrrolidone
DVB	Divinylbenzene	PS	Polystyrene
DMF	Dimethyl formamide	RT	Room temperature
DMSO	Dimethyl sulfoxide	SAED	Selected area electron diffraction
EDX	Energy Dispersive X-ray	SBA	Santa Barbara Airport
GO	Graphene oxide	SDS	Sodium dodecyl sulfate
HMT	Hexamethylenetetramine	SEM	Scanning Electron Microscopy
HRTEM	High Resolution Transmission Electron Microscopy	s	singlet
h	Hours	t	triplate
LDHs	Layered Double Hydroxides	TBAF	Tetrabutylammonium fluoride
MCM	Mobil Company of Matter	TBAB	Tetrabutylammonium bromide
min	minute	TEG	Tetraethylene glycol
mol%	mol percent	TEM	Transmission Electron Microscopy
mg	milligram	THF	Tetrahydrofuran
MHz	mega hertz	XRD	X-ray Diffraction
NPs	Nanoparticles		