Summary of thesis entitled

Synthesis of some metallopolymers and nanoreactors for catalyzing solitary and cascade organic transformations

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Submitted by

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The thesis is divided into 5 chapters

Chapter 1: Introduction

Catalysis is one of the important areas in science because various catalytic reactions used in different fields such as environment, health, pharmacy and chemical industry involves at least one catalytic step. Nanocatalysis, a branch of nanoscience, in which metal nanoparticles are utilized as catalytically active materials, has gained enormous attention during the past few decades.

Metal nanoparticles (MNPs) are defined as nanosized metals with at least one of the dimensions within 1 to 100 nm. MNPs play important role in heterogenous catalysis due to superior surface-to-volume ratio, abundant rich active surface atoms, and distinctive electronic structures as compared with their bulk counterparts.

Conventionally, MNPs possess high surface energy due to which they are thermodynamically unstable, prone to migration and agglomeration during catalysis, especially at high reaction temperatures. These structural alterations are accompanied by a dramatic decrease in catalytic activity and selectivity.

Overcoming these limitations has been the focus of the researchers working in this field by devising various strategies to heterogenize MNPs for catalytic application. It is anticipated that stabilizing MNPs by capping agents or encapsulating them within suitable materials will help to physically isolate them, halt their migration, and prevent them from coalescence which can improve their catalytic stability.

Metallopolymers are unique class of functional materials in which metal centers are incorporated in the polymers. With suitable functionalities, these polymers can support MNPs resulting in formation of metallopolymers that can aid to perform reactions under relatively mild conditions. Metallopolymers offer advantage of easy separation and regeneration from the reaction mixture due to immobilization of metal catalyst in the heterogenous polymer matrix. MNPs are stabilized in the polymer matrix via ionic, electrostatic, hydrophobic and covalent interactions. The structure and catalytic activity of MNPs/polymer composites are influenced by structure, morphology and functionality of the polymer. To serve this purpose, several porous, conductive, branched polymers along with biopolymers have been utilized to design MNPs-polymer nanocomposites catalysts for various catalytic reactions.

A switch from carrying out the reaction in "organic media" to other greener options such as water or no solvent, can advance organic chemistry sustainably and aid in pressing the rigorous environmental concerns**.** According to green chemistry principles, water is an acceptable choice of solvent due to its ubiquitous, non-toxic and non-inflammable nature. Water is attractive from both environmental and economic perspectives. However, its utility is limited in organic chemistry due to the poor solubility of organic compounds and catalysts in water as well as low E-factor of the chemical processes. The catalytic reactions in the confined hydrophobic core of micelle and vesicles are an emerging research area and an effective strategy to address the above-mentioned issues. Polymer micelles and vesicles, self-assembling aggregates, can be used to design robust and recyclable functional materials such as catalytic nanoreactors through molecular manipulation and control.

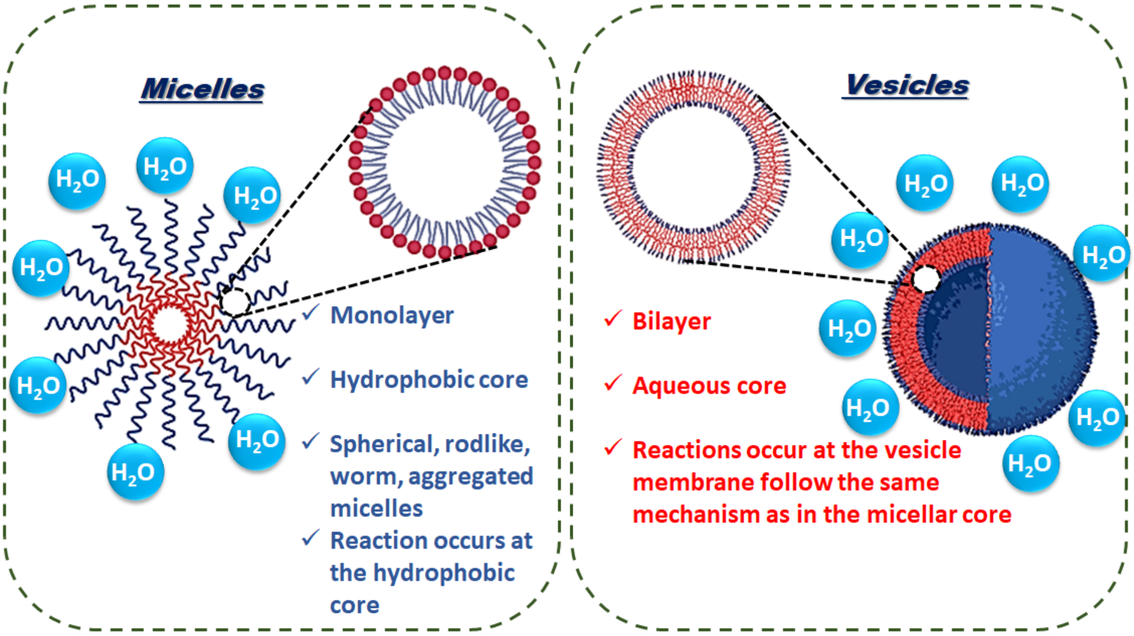


Figure 1: Schematic representation of structural difference of micelles and vesicles

Thus, the fabrication of metallopolymers and nanoreactors with appropriate rationale can turn out to bring a new advent in sustainable chemistry due to their ubiquitous assistance in the synthesis of commercially important compounds. The work was executed in the following steps:

1. Development of crosslinked polymers stabilized zerovalent iron nanoparticles

β-Cyclodextrin, a well-known host for hydrophobic substrates with multiple hydroxy groups can aid in capping the metal nanoparticles to act as catalyst. β-CD derived crosslinked polymers can be ideal stabilizing agents due to stable, biodegradable and amphiphilic nature.

β-CD was selected as a precursor and was covalently conjugated to α-hydroxy acids Nanocatalysts derived from two cyclodextrin crosslinked polymers capped zerovalent iron nanoparticles (nZVI) were synthesized via ultrasonication method. The synthesized nanocatalysts were assesed for their ability to catalyze reduction of nitroaromatics via ultrasonication.

2. Development of covalent organic frameworks encapsulated palladium nanoparticles

Covalent Organic Frameworks (COFs) exhibit many properties that are advantageous for heterogeneous catalysis. They can act as hosts for catalytically active metallic nanoparticles and minimizes their aggregation. Urethane linked COF was further functionalized with cysteamine molecules. Palladium nanoparticles was stabilized due to strong affinity towards the sulfur group. These were subsequently reduced to palladium nanoparticles. They were assesed for their ability to catalyse Suzuki Miyuara Cross Coupling reaction.

3. Development of polymer vesicles encapsulated copper nanoparticles

An amphiphilic co-polymer with carboxyl functionalities was rationally designed via urethane linkages. The free carboxyl group helps in stabilization of copper nanoparticles. The rational to choose urethane linked amphiphilic co-polymer was the intrachain H-bonding assisted in self-assembly into vesicles. This nanoreactors were assessed for their ability to catalyze synthesis of benzimidazole, an industrially important compound via cascade route.

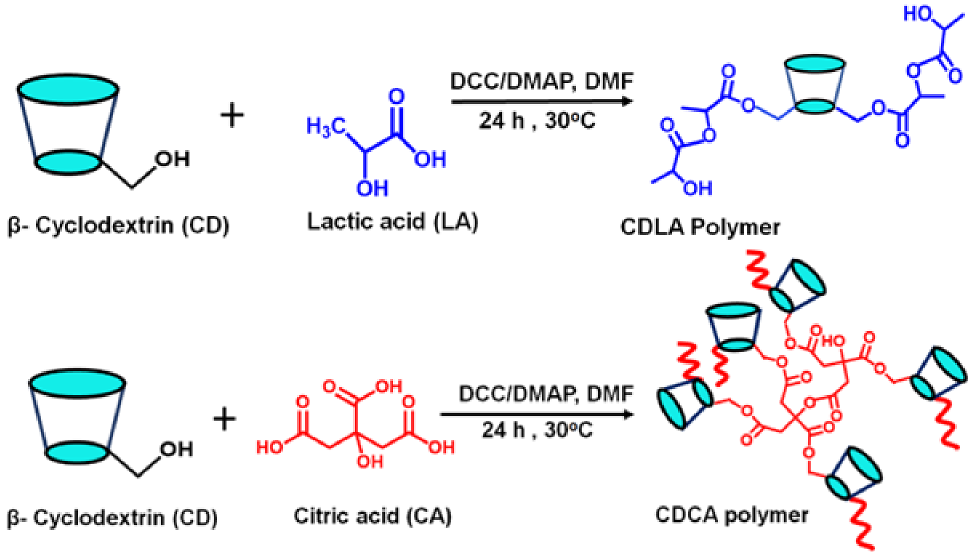
4. Development of polymer micelles loaded *Candida antarctica* Lipase B and zerovalent iron nanoparticles

For the development of biobased polymer micelles, tyrosine is used as a backbone of the molecular architecture with polyethylene glycol as the hydrophilic component and a long aliphatic acid chain as a hydrophobic component. These polymer micelles were loaded with CALB and iron nanoparticles to behave as nanoreactors, further ability to catalyze chemoenzymatic cascade synthesis of p-aminophenol from the p-nitrophenyl ester was evaluated.

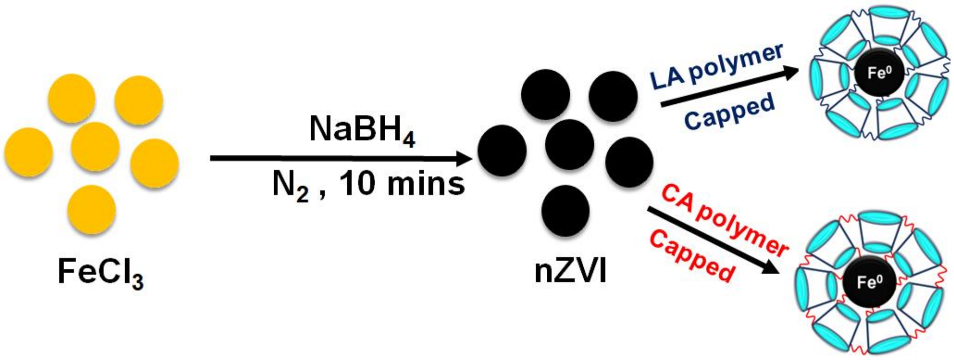
Chapter 2:

α-Hydroxy acids modified β-cyclodextrin capped iron nanocatalyst for rapid reduction of nitroaromatics: A sonochemical approach

This chapter discusses the synthesis of sustainable citric acid and lactic acid crosslinked cyclodextrin polymers via a simple one-pot esterification reaction for capping for zerovalent iron nanoparticles via the ultrasonication method (s**hown in Scheme 1 & Scheme 2**).



Scheme 1: Synthetic route for preparing lactic acid modified β-Cyclodextrin and citric acid modified β-Cyclodextrin derived crosslinked polymers.



Scheme 2: Synthetic route for preparing β-Cyclodextrin derived cross-linked polymers capped zerovalent iron nanoparticles via ultrasonication method.

The sonochemical approach imparted good control over the morphology, nano size, and dispersibility of the prepared FeNPs. The synthesized polymers and the nanocatalysts were characterized by various techniques shown in **Table 1.**

Table 1: Various properties of crosslinked polymers and nanocatalysts were assesed by various techniques.

Properties	Technique/ Instruments
Structural Elucidation	1H NMR, ^{13}C NMR, 2D NMR, FTIR, EDAX
Morphology and size	HR-TEM, FE-SEM, DLS
Optical	UV-Vis Spectroscopy
Thermal	TGA
Magnetic	VSM
Metal loading	ICP-AES, EDAX
Oxidation state of metal	XPS

The synthesized polymer capped nZVI catalyzed the sonochemical conversion of 4-nitrophenol to 4-aminophenol in a very short time in an aqueous medium. Application of the ultrasonication to the reaction system increases the temperature and reduced the reaction time. In addition, the surface area of nZVI was increased. Several other nitroaromatic compounds could be reduced with excellent isolated yield (89–95%), as well as high TON and TOF. The recycling studies especially revealed the robust nature of the polymer-capped nZVI.

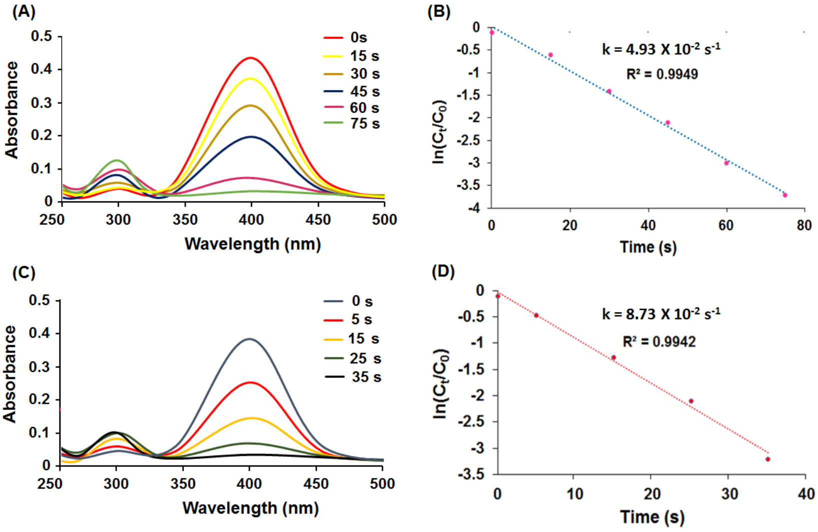
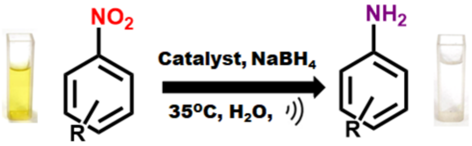
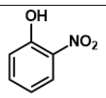
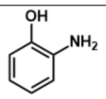
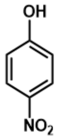
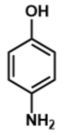
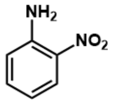
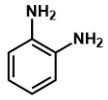
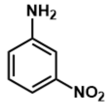
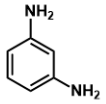
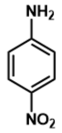
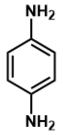
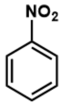
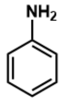


Figure 2: The time dependent UV-vis absorption spectra of 4-NP reduction and the dependence of ln(C/Co) versus time plot for the pseudo-first-order reaction kinetics in the presence of (A, B) CDCA@nZVI and (C, D) CDLA@nZVI at 35°C.

Table 2: Reduction of some nitro aromatic compounds to corresponding aromatic amines using LA@nZVI and CA@nZVI at optimized conditions



Sr. No.	Substrate	Product	Time [a] (s)	Time [b] (s)
1.			50	165
2.			70	210
3.			35	89
4.			14	50
5.			61	114
6.			35	100

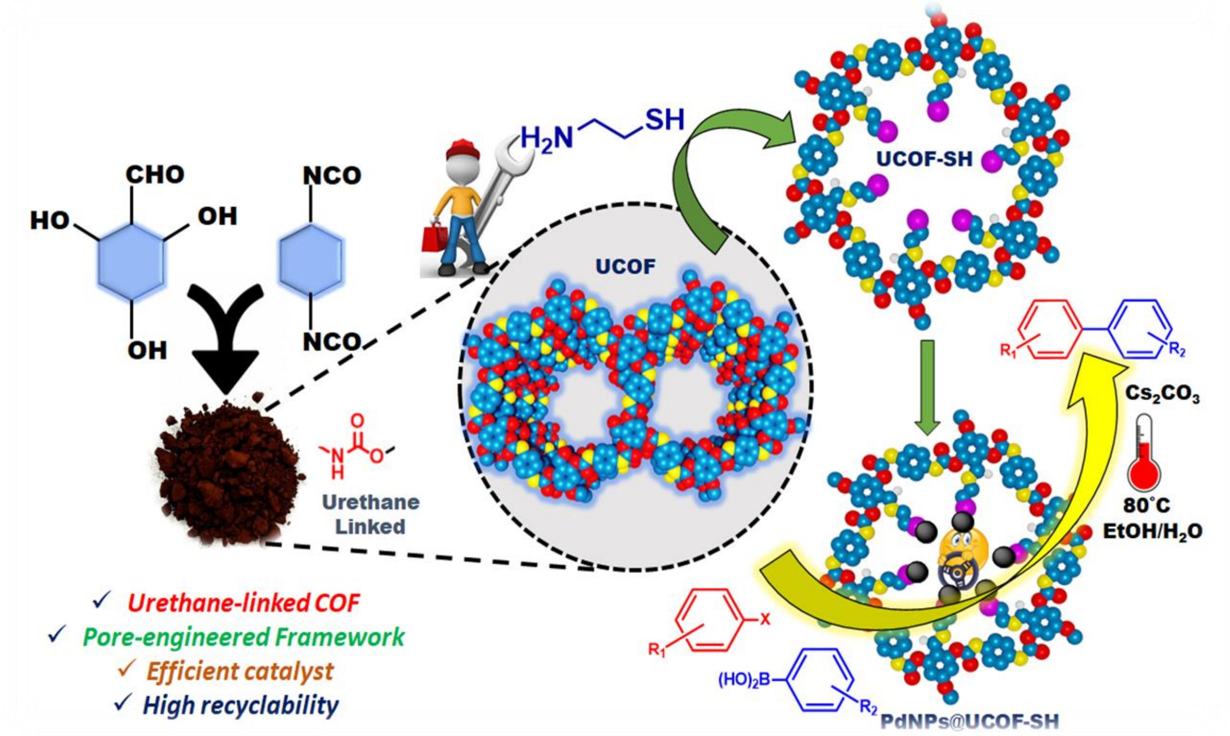
Reaction conditions: All reactions carried out at room temperature (35°C),1.5 mL of substrate (0.24 mM), catalyst 10 mg, 1.5 mL $NaBH_4$ (2.16 mM).

According to proposed mechanism, the synergistic effect of substrate captured in cyclodextrin cavity, the subsequent electron donation from metallic core and sonochemical effect facilitates a rapid conversion of the reactants in aqueous media. The recycling studies especially revealed the robust nature of the polymer capped nZVI. The rate constant values obtained from kinetic studies suggested that the catalytic activity of nZVI is comparable to the commonly used noble metal catalysts.

Chapter 3:

Palladium nanoparticles-confined pore-engineered urethane-linked thiol-functionalized covalent organic frameworks: a high-performance catalyst for the Suzuki Miyaura cross-coupling reaction

Catalyst with porosity and stability are demanding. This chapter discusses the synthesis and functionalization of a novel urethane-linked covalent organic framework for the first time. The pendant formyl group of the UCOF was rationally functionalized using cysteamine to obtain a thiol-decorated COF which can assist in the size-controlled synthesis of stable and highly dispersed ultrafine palladium NPs.



Scheme 3: Synthetic representation of palladium nanoparticles loaded thiol functionalized urethane linked covalent organic framework for Suzuki Miyuara Coupling reaction.

Beside synthesis, we also made an effort to thoroughly characterize COFs and Pd catalyst using various analytical techniques shown in **Table 3.**

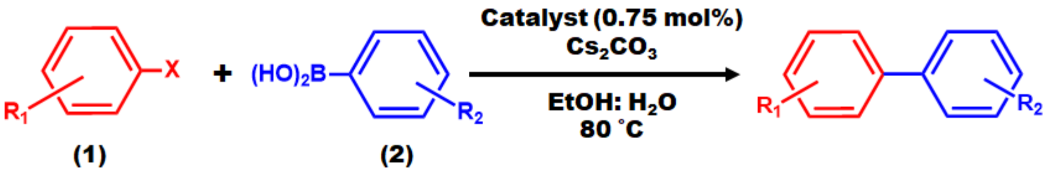
Table 3: Various properties of COFs assessed using various techniques.

Properties	Technique/ Instruments
Structural Elucidation	^{1}H NMR, CP/MAS-NMR, FTIR, EDAX
Morphology and size	HR-TEM, FE-SEM
Confirm framework formation	PXRD
Thermal	TGA
Surface area and Porosity	BET
Metal loading	ICP-AES, EDAX
Oxidation state of metal	XPS
Product formation	GCMS

With the assistance of equally spaced thiol groups in the ordered framework, Pd NPs of small size (4–6 nm) dispersion were acquired successfully. The UCOF-SH stabilized Pd NPs showed excellent catalytic activity in terms of low catalyst loading, high yield, and milder reaction conditions toward the Suzuki cross-coupling reaction.

The reaction parameters such as base additives, temperature, solvents, catalyst dosage, and solvent ratio were studied. The optimized reaction conditions were cesium carbonate as a base additive, 10 mg catalyst, 80 ˚C, and Water: Ethanol (5:5) mixture as solvent. The scope was extended to various aryl boronic acids and aryl halides (I, Br, and Cl). The halo-substituted and non-halo biaryl derivatives were obtained in good to excellent yields, within a shorter reaction time, avoiding the homocoupling of aryl boronic acid.

Table 4: Optimization of reaction conditions for Suzuki Miyuara Cross-Coupling reaction



Sr. No.	**Aryl halide (1)**	**Aryl halide**	**Aryl boronic acid (2)**	**Time** (h)	**%Yield**
1	R1=H	X=I	R2=H	**1**	**99**
2	R1=H	X=I	R2=OCH_3	**0.5**	**98**
3	R1=H	X=Br	R2=H	**2**	**99**
4	R1=OCH_3	X=Br	R2=H	**3**	**97**
5	R1=OCH_3	X=Br	R2=OCH_3	**2**	**97**
6	R1=$COCH_3$	X=Br	R2=H	**5**	**89**
7	R1=PhCN	X=Br	R2=F	**6**	**50**
8	R1=(NO_2)	X=Br, F	R2=F	**8**	**68**
9	R1=CH_3	X=Cl	R2=H	**7**	**80**
10	R1=CH_3	X=Cl	R2=F	**10**	**72**

Reaction Conditions: Aryl halide (0.5 mmol), arylboronic acid (0.75 mmol), 1.0 mmol of Cs_2CO_3, 0.75 mol % of PdNPs@UCOF-SH, and solvent EtOH: H_2O (1:1) 5 mL. [a]Isolated Yield.

Additionally, the catalyst is extremely stable, easily recyclable, and reusable. The pore-engineered COF-derived catalyst is selective and easily recycled up to 10 runs without significant loss of catalytic activity.

This reveals the robust nature of the PdNPs@UCOF-SH catalyst and the sustainability of the process which opens a new frontier for several catalytic applications.

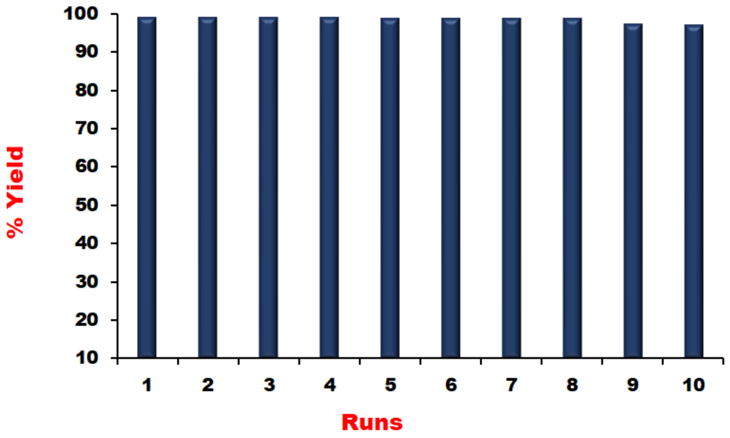
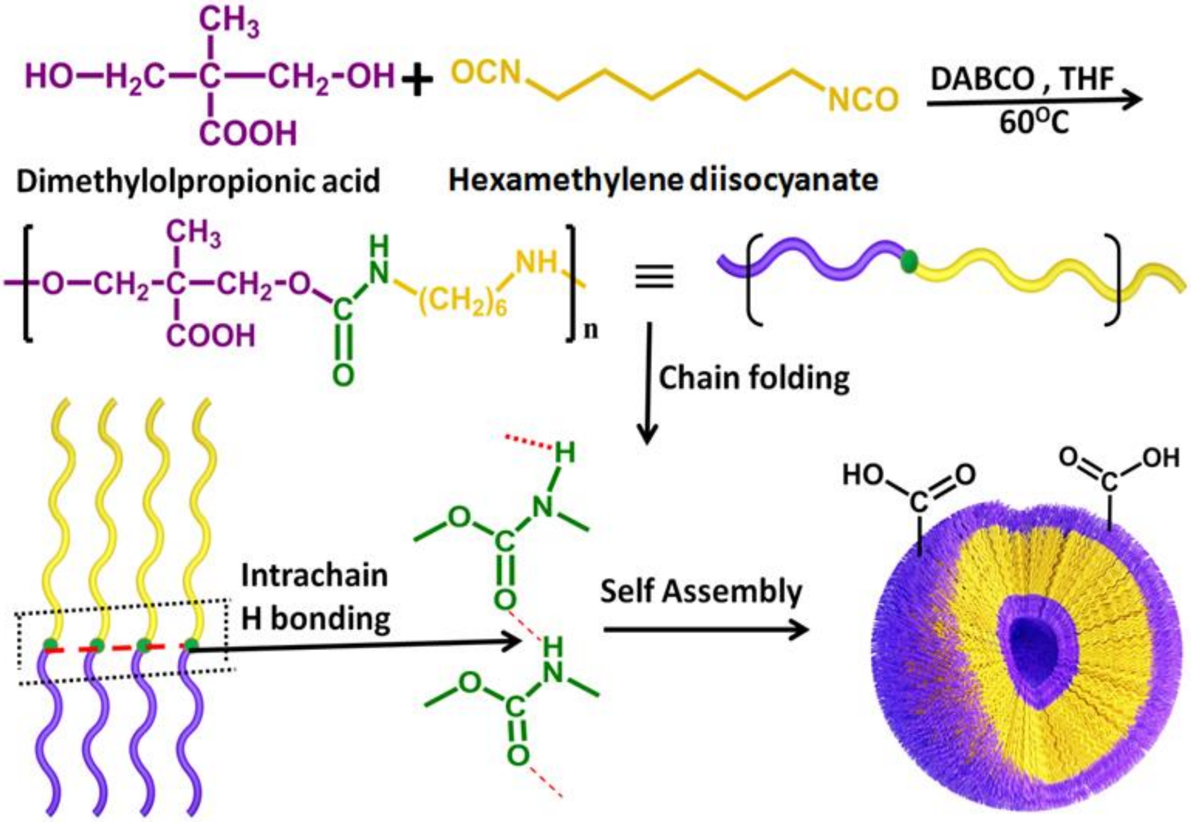


Figure 3.11: Recycling study (Reaction Conditions: Bromobenzene (0.5 mmol), phenylboronic acid (0.75 mmol), 1.0 mmol of Cs_2CO_3, 0.75 mol % of PdNPs@UCOF-SH, and solvent EtOH: H_2O 5 mL, Isolated Yield).

Chapter 4:

Copper nanoparticles loaded polymer vesicles as environmentally amicable nanoreactors: A sustainable approach for cascading synthesis of benzimidazole

Reactions in aquoues medium are desirable from environment perspective. The quest for newer nanocatalysts that perform in aquoues medium lead us towards micellar systems. This chapters discusses the synthesis of synthesis and characterization of Cu nanoparticle-loaded, surfactant-free metallovesicles (CuNPs@vesicles) as nanoreactors to produce benzimidazoles via cascade reaction.



Scheme 4: Synthesis of amphiphilic DMPA-HMDI co-polymer and subsequent self-assembly into vesicle.

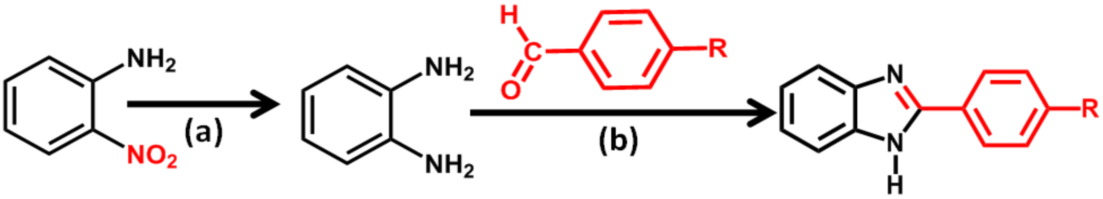
The vesicles exhibited uniform size distribution, spherical morphology, excellent stability, and efficient Cu loading. After synthesizing the polymer vesicles our efforts were directed towards loaded copper metal into polymer vesicles and subsequently reduce them inside the vesicles to metal nanoparticles to act as metallovesicle. The prepared metallovesicle was utilized as catalyst for the cascade reaction.

Scheme 5: Visual changes observed in the process of CuNPs@vesicles formation(i) bare vesicles (ii) binding of Cu^{2+} ions with vesicles (iii) Color change upon pH adjustment of the metal encapsulated vesicles (iv) reduction and formation of Cu Nanoparticles.

Table 5: Various properties of polymer vesicles and metallovesicles were assesed by various techniques.

Properties	**Technique/ Instruments**
Structural Elucidation	^{1}H NMR, FTIR, EDAX
Morphology and size	HR-TEM, FE-SEM, DLS, AFM
Surface charge	Zeta potential
Thermal	TGA
Optical	UV-Vis & Fluorescence Spectroscopy
Self-assembly	**Vesicles:** FTIR, self-quenching, fluorescence spectroscopy & microscopy
Metal loading	ICP-AES, AAS
Oxidation state of metal	XPS
Magnetic	VSM

This strategy is unique for the fact that environmental contaminants like nitroaniline have been utilized as precursors and converted into fine chemicals of commercial significance via non-toxic intermediates. CuNPs@vesicles reduces 2-nitroaniline to o-phenylenediamine which further acts as a precursor for benzimidazole synthesis.



Scheme 6: CuNPs@vesicles catalyzed reactions for synthesis of benzimidazoles via cascading pathway. [Reaction conditions (a) catalyst 5 ml, NaBH$_4$, H$_2$O, 30 ^{0}C (b) catalyst 5 ml, H$_2$O, 30 ^{0}C]

Table 3: Substrate scope of reaction as per scheme 3 by using o-PDA and substituted aromatic aldehydes via reduction of 2-Nitroaniline

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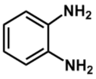
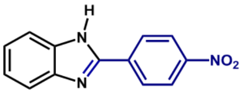
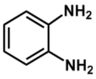
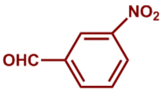
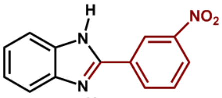
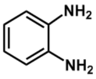
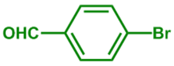
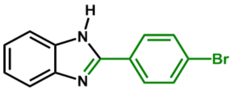
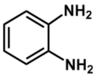
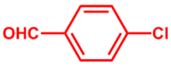
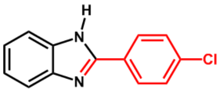
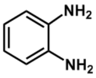
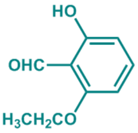
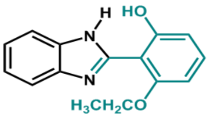
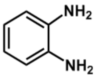
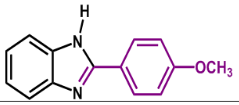
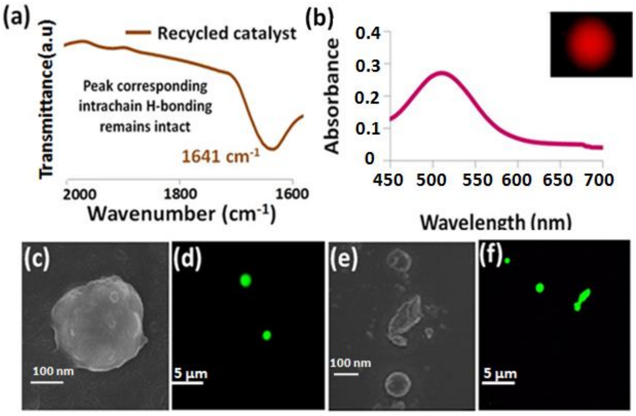
1				5	30	90	0.11
2				5	40	86	0.16
3				5	60	88	0.14
4				5	45	85	0.18
5				5	70	84	0.18
6				5	60	84	0.19

Reaction conditions: o-phenylenediamine (1.0 mmol), aromatic aldehyde (1.0 mmol), CuNPs@vesicles (5 ml, 1440 ppm), water (15 ml), room temperature (30 ^{0}C). ^{a} Isolated yield.

Thus, the approach is environmentally benign, as the entire cascade of reactions proceeds in aqueous medium at ambient conditions.

Thus, the reaction occurs via a two-step cascade pathway comprising reduction and cross-coupling reactions in water. The preliminary studies suggest encouraging results for performing dehydrogenative coupling under relatively mild conditions using CuNPs@vesicles as a catalyst. All the products are obtained in good to excellent yield with facile catalyst regeneration and recyclability up to 5 cascading cycles.



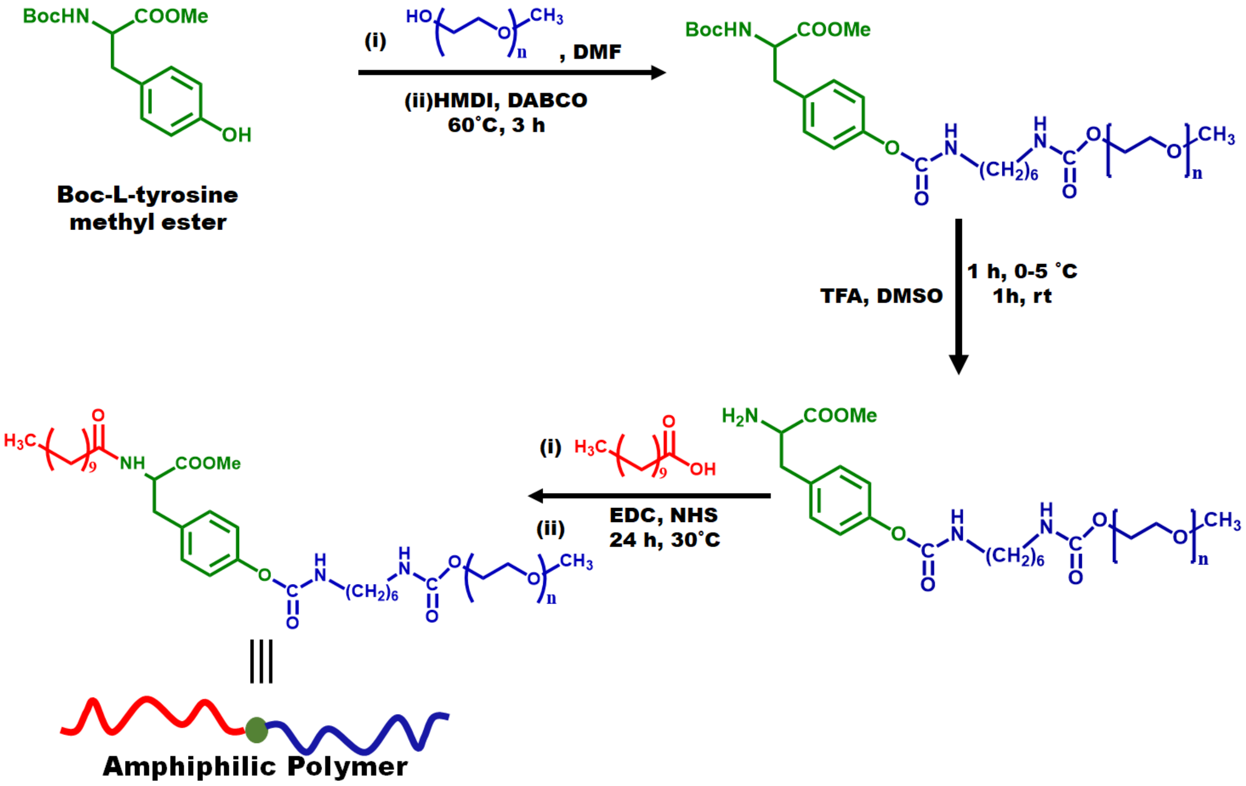
Scheme 7: Characterization of the recycled catalyst (A) FT-IR, (B) UV-Visible, (C & E) FEG-SEM of 1st and 5th cycle, (D& F) florescence imaging of 1st and 5th cycle

The important feature of regeneration and recyclability (upto 5 cascade cycles) can be attributed to the robust nature and stability of vesicular nanostructures.

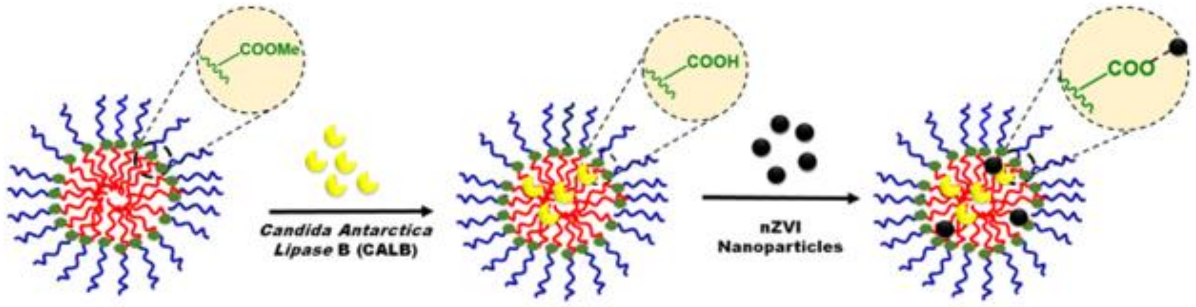
Chapter 5:

Candida antarctica Lipase B and nZVI co-loaded bioinspired nanomicelles: A sustainable catalytic nanoreactor for cascade synthesis of p-aminophenol

This work reports a synthesis of a novel amphiphilic polymer by functionalizing tyrosine with polyethylene glycol and undecanoic acid. For the development of polymer micelles, biomolecule tyrosine is used as a backbone of the molecular architecture with polyethylene glycol as the hydrophilic component and a long aliphatic acid chain as a hydrophobic component. The tyrosine-derived amphiphilic polymer self-assembles to nanomicelles. The nanomicelles resemble surfactant micelles, they possess a hydrophobic core that facilitates the organic transformation in an aqueous medium. In addition, they possess a hydrophilic shell and hydrophobic core. Nanomicelles enhance the stability of the encapsulated metal catalyst, solubility of the hydrophobic reactants, and bring the catalyst and the reactant in close proximity.



The latter successfully self-assembled to nanomicelles with a low CMC value of 0.97 mg/L, which indicated the high stability of nanomicelles. These nanomicelles were employed for stabilizing the CALB enzyme and nZVI nanoparticles at a single platform in the hydrophobic core, to obtain catalytic nanoreactors.



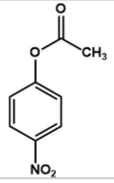
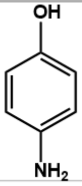
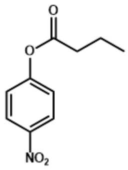
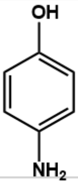
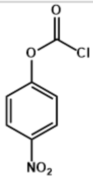
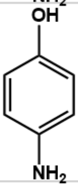
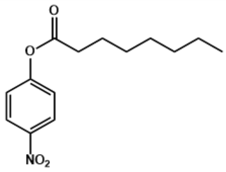
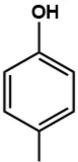
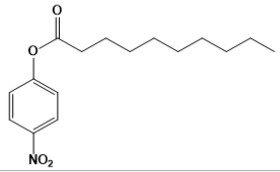
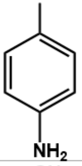
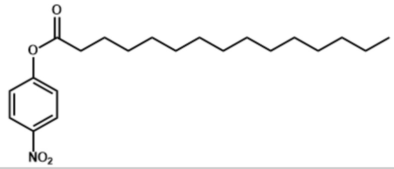
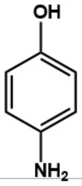
Scheme 8: Schematic representation of preparation of catalytic nanoreactors by loaded the *Candida antarctica* Lipase B enzyme and zerovalent iron nanoparticles in the nanomicelles derived from tyrosine derives amphiphilic polymer.

Properties	**Technique/ Instruments**
Structural Elucidation	^{1}H NMR, FTIR, EDAX
Morphology and size	HR-TEM, FE-SEM, DLS, AFM
Surface charge	Zeta potential
Thermal	TGA
Optical	UV-Vis & Fluorescence Spectroscopy
Self-assembly	**Micelles:** CMC determination by pyrene encapsulation, self-quenching, fluorescence spectroscopy & microscopy
Metal loading	ICP-AES, AAS
Oxidation state of metal	XPS
Magnetic	VSM

Inspired by the nanobiohybrid concept by Palomo *et.al*, we have prepared catalytic nanoreactors for the chemoenzymatic cascade reaction of p-nitrophenylesters to p-aminophenol using the rationally designed catalytic nanoreactors in water at ambient reaction conditions. To demonstrate the robust nature of the nanoreactors a series of p-nitrophenyl ester derivatives were employed as reactants for the cascade reactions. The corresponding p-aminophenol was obtained as a desired product under mild reaction conditions in reasonable reaction time.

Finally, the operational recyclability showed no significant loss in activity up to 5 consecutive cascade cycles of use at 35˚C. The easy magnetic recovery and reusability of the catalytic nanoreactors is critical for their application in scale-up processes.

Table 4: Substrate scope of the reaction under the optimized condition using nZVI-CALB@NM.

Sr.No.	Substrate	Product	Time (min)	%Conversion
1.			**20**	**98**
2.			**22**	**96**
3.			**35**	**95**
4.			**43**	**96**
5.			**45**	**98**
6.			**50**	**99**

Reaction conditions: Substrates (1 mM) Catalyst (2 mL, contains nZVI (18 wt%)), $NaBH_4$ (1.4 mM) at 35˚, % Conversion was calculated for the hydrogenation step using UV-Visible Spectrophotometry.

From this work, we envisage that the enzymatic and metallic catalysis can be combined in nanomicelles to create novel cascade industrial manufacturing processes. These findings also imply that similar results can be anticipated for other enzymatic processes and sequential cascade catalysis.

Conclusion

To summarize, this work aims towards addressing the various challenges in sustainable catalysis, these include, stabilization of metal nanoparticle for better catalytic activity, avoiding the use of expensive and hazardous metals, toxic organic solvents, harsh reaction conditions, control byproduct formation and offer recyclability. Diverse polymer architectures such as cross-linked polymers, covalent organic frameworks, polymer vesicles and polymer micelles were taken into account for the metal nanoparticles stabilization. Different metal nanoparticles such as iron, palladium and copper were stabilized using these polymers to obtain metallopolymers some of which function as nanoreactors.

The synthesized metallopolymers and nanoreactors were assessed for their ability to catalyze solitary (reduction of nitroaromatics and Suzuki Miyuara Cross Coupling reaction) and cascade (Benzimidazole synthesis and chemoenzymatic synthesis) reactions. The reaction parameters such as temperature, solvent, base, etc., were investigated to optimize the reaction conditions. The kinetics of the reduction reaction was studied. The substrates scope studies and recycling experiments were undertaken. The recycled catalysts were characterized and the plausible mechanisms were proposed for all the systems. The catalysts developed in this study are promising candidates for commercial application and patents.

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List of Publications

1. **F. Shukla,** T. Kikani, A. Khan, S. Thakore. α-Hydroxy acids modified β-cyclodextrin capped Iron Nanocatalyst for rapid reduction of nitroaromatics: a sonochemical approach. ***International Journal of Biological Macromolecule*, 2022,** *209*, 1504-1515.
2. **F. Shukla,** M. Patel, Q. Gulamnabi, S. Thakore. Palladium nanoparticles confined pore engineered urethane linked thiol functionalized covalent organic framework: A high-performance catalyst for Suzuki Miyuara cross-coupling reaction. ***Dalton Transactions*, 2023,** 52, 2518-2532.
3. **F. Shukla**, Manita Das, S. Thakore. Copper nanoparticles loaded polymeric vesicles as environmentally amicable nanoreactors: A sustainable approach for cascading synthesis of benzimidazole; ***Journal of Molecular Liquids*, 2021,** *336*, 116217.
4. **F. Shukla**, D. Singh, S. Thakore. *Candida antarctica* Lipase B and nZVI co-loaded bioinspired nanomicelles: A sustainable catalytic nanoreactor for multistep synthesis of acetaminophen **(Under revision)**

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