Chapter 8

Summary and Future Scope

8.1 Introduction

Non-magnetic metal or metal oxide NPs of different sizes and shapes have been prepared and characterized well with the help of SEM, FESEM, BET, TEM, FT-IR spectroscopy etc. We have studied catalytic property of those NPs to synthesize some homo-/hetero-biphenyl compounds through some easier pathway. The lab synthesized NPs are very much effective in water purification. The materials are water insoluble and reusable, suitable for environmental contamination remediation.

This chapter summarizes the conclusions based on the work on "DEVELOPMENT OF 'NANOPARTICLE CATALYSED ORGANIC SYNTHESIS ENHANCEMENT' (NOSE) APPROACH AND DECONTAMINATION OF WATER BY INORGANIC NANOPARTICLES". In addition, the contribution of the present thesis including the future scope has also been highlighted.

8.2 Contribution of present work

The general conclusions inferred from the individual chapters in the present thesis are as follows:

Chapter 1: This chapter deals with a general introduction on NPs of different metal oxides followed by various type of synthesis procedure. In addition, literature survey dealing with the synthesis, properties and applications of the NPs is also provided. Finally, the aim and objectives of the research work in the present thesis have been highlighted.

Chapter 2: This chapter describes the various instruments/techniques used in the characterization of the NPs and synthesized compounds along with very brief description of their working principle.

Chapter 3: CuO NPs have been efficiently synthesized using wet chemical procedure and it has been found that the NPs are of rod shaped with less than 50 nm in dimension. A simple and efficient rod shaped CuO NPs catalyzed methodology for the homocoupling of arylboronic acids to symmetrical biaryls has been developed. The reaction proceeds in green, economical, cheapest and easily available solvent water under extremely mild conditions: in air, at room temperature in presence of a very mild base. The synthesized CuO NPs remain in same condition even after performing the catalytic reaction. The catalyst is efficient, easily recoverable and reusable up to another three to four times without losing its activity appreciably. This protocol is relatively inexpensive and environmentally friendly manner. The good selectivity of homocoupling in the Suzuki cross-coupling shows potential application in the synthesis of biphenyls.

Chapter 4: Substituted arylboronic acids and arylsulfonyl chlorides are coupled under microwave irradiation (MWI) to produce cross biphenyls in high yields. The principal advantage of this reaction is that cross biphenyl formed under MWI in less time with desulfurization of arylsulfonyl chloride, fact is supporting to inhibit environmental pollution caused by sulphur containing organic substances. *In-situ* generated Pd NPs are able to transform boronic acid and sulfonyl chlorides to un-symmetrical biaryls which is an example of *i*-NOSE approach for biphenyl synthesis. The cross-coupling reaction proceeds in economical, cheapest and easily available solvent under extremely mild conditions: in presence of air, very mild base and microwave irradiation. The synthesized Pd NPs remain unaffected even after performing the catalytic reaction. The catalyst is efficient, easily recoverable and reusable more than four times without losing its activity. This work reveals a new cheap and fast procedure to synthesize un-symmetrical biaryls in a relatively environmentally friendly manner. The good selectivity of cross-coupling in the Suzuki cross-coupling might pave the way for potential application in the synthesis of biphenyls.

Chapter 5: There is sufficient report evidenced that azo-aromatic compunds need at least two-step reaction to form from its nitro-analogs. Here, we report a one step synthesis of substituted azo-benzene from substituted nitrobenzene in presence of DMF-water solvent and CuO NPs catalyst. CuO NPs have been synthesized through simple wet chemical procedure and well characterized with the help of SEM, TEM, FT-IR spectroscopy etc. Azo-biphenyls are formed as major products in addition to small amount axoxy-benzene and aniline. The nanocatalyst is reusable and environmently friendly in nature. In conclusion, we have successfully developed a simple and efficient CuO NPs catalyzed methodology toward the formation of AroA compound directly from the corresponding nitroaromatic compounds. This protocol is relatively inexpensive and environmentally friendly. This reaction skips two-step synthesis of AroA compounds from nitroaromatics and open a bright synthesis procedure.

Chapter 6: Non-magnetic iron oxide hydroxide NPs have been prepared through wet chemical procedure at room temperature and is found to play as an effective adsorbent media to remove As(III) from 300 μgL^{-1} to less than 10 μgL^{-1} (WHO, Desirable limit of As) from drinking water over wide range of pH. XRD analysis, BET surface area, FT-IR, FESEM and TEM images were used to study the synthesized iron oxide hydroxide NPs. TEM image clearly reveals that the NPs have flower like morphology with average particle size less than 20 nm. The maximum sorption capacity of the sorbent is found to be 475 μgg^{-1} for arsenic at room temperature and the data fitted to different isotherm models indicates the heterogeneity of the adsorbent surface. There is no significant influence of other co-anions like hydroxide, sulphate and phosphate ions on the dearsenification capacity of the NPs except high concentration of phosphate ions. Study on adsorption kinetics shows that adsorption of arsenic onto iron oxide hydroxide nanoflower follows pseudo-second order kinetic. The material can be regenerated up to 70% using dilute hydrochloric acid and it would be utilized for de-arsenification purposes.

Chapter 7: The work described in this chapter deals with a simple *in-situ* soft chemical synthesis route towards nanoscale copper(II) oxide and its environmental application to remove lead from water. The NPs are well characterized by XRD, FESEM, TEM and BET surface area analyzer, Electron microscopy image clearly reveals rod like morphology of rhombohedral CuO, with average diameter ~5 nm and length extending up to 50 nm. BET show average surface area of the nanorods ~ 52.57 m^2g^{-1}.

Subsequently, the CuO nanorods are used as adsorbent of lead from contaminated water and adsorption studies also reveal that the Pb(II) uptake onto CuO is a fast process and >70% of the uptake occurred within the first 10 min contact time and attained >92% in 60 min. In addition, the maximum sorption capacity is found to be 18.3 mgg^{-1} for lead. The regeneration studies show up to 82.2% regenerability with dilute hydrochloric with could be further utilized for removal of lead from contaminated water. As far as practical applicability is concerned, CuO is more promising and is effective over a wide pH range compared in presence of other competing/interfering ions. Reduced sensitivity to pH makes this adsorbent more attractive for cleaning up industrial effluents, especially effluents having low pH.

Chapter 8: It summarises the contribution of the present work and its possible future applications in society. An insight of the thesis has been also delineated.

8.3 Future Scope of the Work

Based on all these findings, the future prospects of the present work may be summarized as follows:

- Copper oxide and Palladium NPs and other NPs may be checked for other organic reactions.
- Iron oxide-hydroxide NPs impregnated over some suitable materials may be checked for superior removal efficiency of other heavy metal contaminants from water.
- It is expected that the present research work might provide new dimensions in developing iron oxide NPs through a simple approach, which can be scaled up and further explored as an effective and replicable adsorbent media for dearsenification of drinking water.
- CuO NPs as adsorbent may be more attractive for cleaning up industrial effluents, especially effluents having low pH.
- Organic NPs may be explored in catalysis and decontamination of water.