
Chapter 6

Summary & Conclusions

Synthesis of 1,2,3-triazoles has received wide spread attention over the past decade due to its variety of applications. This scaffold can easily be assembled through regioselective [3+2] cycloaddition of alkyne and azide which provides access to a variety of functional groups on the triazole ring. Developing novel synthetic protocols having high yield, room temperature, short reaction time and use of benign solvent are some of the major objectives in any synthesis. Satisfying these objectives, this thesis describes the design and synthesis of new catalysts for an environmental friendly synthesis of 1,2,3-triazoles. Besides this, it also highlighted the palladation of some of the triazoles or triazolylidene counterpart leading to complexes having different bonding pattern. We have also demonstrated that the palladium complexes have potential applications as catalyst in Suzuki coupling reaction and as antitumor agent.

A comprehensive review on the synthesis and applications of 1,2,3-triazoles has been depicted in chapter 2. Much effort has been made to high light the recent developments in green protocols and use of heterogeneous catalysts in click reaction.

Chapter 3 discusses the use of polymer metal complex as a catalyst and the easiness of using it as a heterogeneous catalyst for click reactions. A mini review on the synthesis, characterization and application of polymer metal complexes as a catalyst has been discussed in the first part of the chapter. It provides a thorough knowledge of the factors affecting the catalyst's performance. However, the micro environment and use of polymer-metal complexes as catalysts in general and copper catalyzed azide-alkyne cycloaddition reaction (CuAAC) has been studied to a lesser extent. The development of such polymer supported metal catalysts for CuAAC reaction would be sensible. The polymer support has been synthesized by suspension polymerization of *N*-vinyl pyrrolidone and *N,N'*-methylenebisacrylamide as monomers and AIBN as initiator. The polymer support was used for the *in situ* generation of Cu₂O by the reduction of copper sulphate and it protects the copper (I) within in the polymer matrix from further reduction. The

Copper (I) stabilized polymer has been well characterized using FTIR, FEG-SEM, EDS, XRD, XPS analysis. The polymer-metal complex was successfully utilized as a catalyst for 1,3-dipolar cycloaddition between azide and alkyne in water as solvent. Effect of solvent, temperature and catalyst loading were studied as a part of optimization study. The product 1,4-disubstituted 1,2,3-triazole formed was confirmed by ^1H NMR, ^{13}C NMR and GCMS analysis. We have also demonstrated that the catalyst can be recovered and recycled for at least five runs. Surface morphology of the catalyst before and after the reactions was examined by SEM and EDS analysis and observed any surface damage or leaching on recycling.

Biosynthesis of nanoparticle and its application in catalysis and biological studies has been discussed in chapter 4. It starts with a mini review on synthesis of nanoparticles using biogenic methods. The use of biomaterials for the synthesis of nanoparticle sounds more effective than other methods because of the easiness in carrying out the process in water as solvent and readily availability of starting materials. Due to these reasons, the biosynthesis of nanoparticels is termed as a “green method”. We explored the effect of *Myristica fragrance* fruit extract for the synthesis of nanoparticles and no previous effort that utilizes the fruit as a biomaterial for nanoparticle synthesis has been reported. Commonly, the pericarp i.e outer layer of fruit is discarded as waste material as it doesn't have any commercial value. For the synthesis of nanoparticles, we used this fruit extract as the biomaterial. The fruit extract was directly used for the synthesis of copper oxide and silver nanoparticles without adding any other stabilizing agents. Among the strategies attempted for the synthesis of copper nanoparticle, microwave irradiation was found to be the fastest route. Nanosize and spherical nature of CuO was identified by HR-TEM analysis. Elemental analysis confirms the presence of copper and oxygen in the sample. The catalytic activity of biosynthesized CuO nanoparticle was screened for CuAAC reaction. Optimization of the reaction conditions in terms of catalyst loading, solvent, temperature and time was studied and the catalyst was found to be effective for the click reaction in water medium. Recovery and reusability of catalyst became easier as it is insoluble in water. The catalyst can be reused for the successive 4 cycles with slight decrease in the yield of each successive step. We have also carried out the synthesis of silver nanoparticles using fruit extract. Here use of sunlight was found to be effective in

the rapid formation of the nanoparticles. Formation of silver nanoparticle can be identified by the color change from yellow to brown within 2 minutes. Silver nanoparticle was found to have lower catalytic activity compared to CuO in Azide-Alkyne cycloaddition reactions. It provided a mixture of both 1,4- and 1,5-disubstituted 1,2,3-triazole. Antibacterial and antifungal activity of silver nanoparticle was also examined and showed promising antibacterial activity but minimum antifungal activity.

A study on the synthesis, characterization and application of palladium complexes with carbene as a labile ligand has been discussed in chapter 5. A brief review on palladium *N*-heterocyclic carbene complexes and its application in both catalysis and anticancer agent has been made in the first part of the chapter. Synthesis of 1,2,3-triazole ligand and its dehydrohalogenation and subsequent alkylation afforded the ligand precursor. Initial attempts to synthesize NHC palladium complex with a labile triazole ligand led to the formation of a mixture of complexes having C-Pd-C and C-Pd-N bonding pattern in very low yields. These complexes were separated by column chromatography and characterized using ^1H NMR, ^{13}C NMR and elemental analysis. To synthesis the palladium complex having C-Pd-N bonding pattern, we used ligand substitution reaction on an initially prepared N-Pd-N complex. Palladium compounds are well known catalysts for C-C bond forming cross-coupling reactions. The catalytic activity of C-Pd-N complex towards Suzuki coupling reaction was studied in water. It provided promising results compared to other synthesized complexes with C-Pd-C and N-Pd-N bonding pattern. We have studied the cytotoxic activity of these palladium complexes. Compared to the complexes with N-Pd-N bonding pattern, C-Pd-N complexes show better cytotoxicity.