Synthesis, immobilization and characterization of palladium complexes for Suzuki-Miyaura coupling reactions

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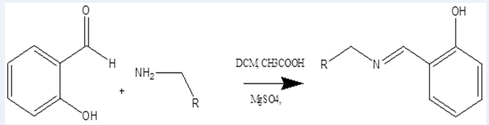
INTRODUCTION

The Palladium catalyzed Suzuki-Miyaura cross-coupling reaction is one of the most reliable methods for the construction of carbon-carbon bonds in solution. Herein, the synthesis and characterization of palladium complexes containing salicylaldimine and iminophosphine ligands are reported. The ligands are prepared conveniently in high yield via Schiff base condensation reactions between salicyaldehyde with appropriate primary amines for salicylaldimines and 2-diphenylphosphinobenzaldehyde and the same amines used for the iminophosphine ligands. The ligands were subsequently used to synthesize the palladium complexes of the type [Pd(P^N) Cl_2] and [Pd(O^N) Cl_2] from metal precursor [Pd(cod)Cl_2]. The complexes were isolated as air stable solids. The complexes were immobilized onto magnetite (Fe_3O_4) coated chicken feathers giving [Pd(P^N) Cl_2]-CF@ Fe_3O_4 and [Pd(O^N) Cl_2] -CF@ Fe_3O_4 complexes, which will behave as heterogenous catalysts.

OBJECTIVES

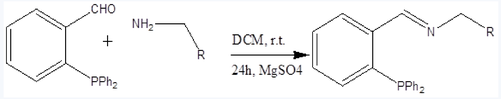
- To synthesize and characterize iminophosphine and salicyladimine Palladium complexes
- To immobilize the synthesized complexes
- To test the catalysts' activity in Suzuki-Miyaura coupling reactions
- To determine recyclability and reusability of the catalysts

EXPERIMENTAL APPROACH

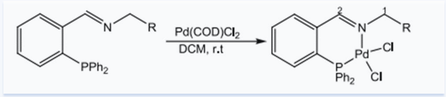


Scheme 1: synthesis of salicylaldimine ligands

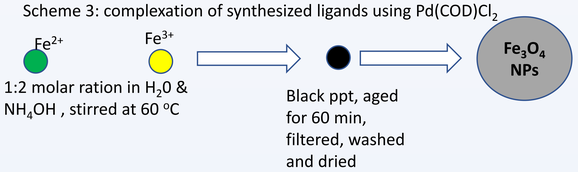
EXPERIMENTAL APPROACH (Cont…)



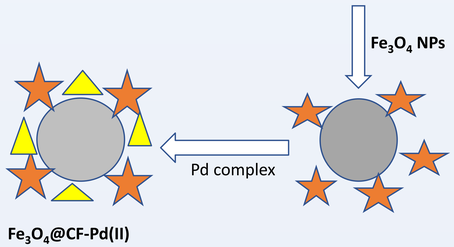
Scheme 2: Synthesis of iminophosphine ligands



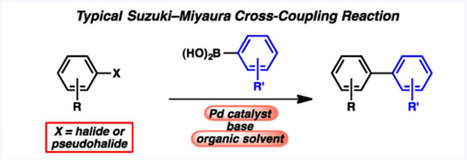
Scheme 3: complexation of synthesized ligands using Pd(COD)Cl_2



Scheme 4: Synthesis of magnetite nanoparticles



Scheme 5: immobilization process of synthesized complexes onto chicken feather coated magnetite



Scheme 7: catalysis – Suzuki-Miyaura coupling reaction

RESULTS

Table 1: List of primary amines used for both salicylaldimine and iminophosphine ligands

Primary amine
Ethylenediamine
2-Thiophenemethylamine
2-Picolylamine
4-Aminobenzoic acid
Furfurylamine

Figure 1: some of the ligands synthesized

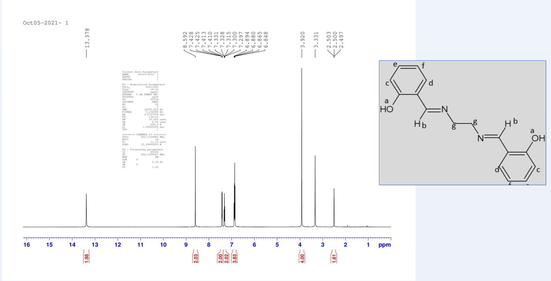


Figure 2: H-NMR spectrum of salen ligand

CONCLUSION & FUTURE WORK

5 salicylaldimine ligands and their complexes were synthesized and immobilized onto magnetite coated chicken feathers

2 iminophosphine ligands and their complexes were synthesized and coated onto magnetite coated chicken feathers

Still awaiting characterization results – NMR, IR, XRD, BET of the samples from N.W.U

Catalytic tests run but awaiting GC results from N.W.U

Further work needed on iminophsophine ligands and complexes, catalytic studies to have a constructive comparable results and test the recyclability and re-usability of the catalysts.

Analysis and outlie the impact of magnetite and chicken feathers on the complexes, and ligands using Secondary Coordination Sphere analogy.

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