CHAPTER 4

CONCLUSIONS

The main purpose of this research was to synthesize solid supported base reagent using magnetic nanoparticles (Fe_3O_4) and silica as solid supported for comparison the effiency of solid support. In preliminary study, Fe_3O_4-DIPA and Fe_3O_4-DIPAP was synthesized from surface modification of the Fe_3O_4 magnetic nanoparticles. The efficiency of this magnetic base was evaluated in substitution reaction between benzylamine and benzenesulfonyl chloride in comparison with nonmagnetic silica supported DIPA (Si-DIPA and Si-DIPAP) and the commercial DIPEA base. Results showed that high percentage yield of product could be obtained without further purification when using Fe_3O_4-DIPA (98%) or Si-DIPA (94%) as bases. The magnetic property of Fe_3O_4-DIPA also provides additional advantage over Si-DIPA in which magnetic separation can be used to facilitate in the ease of its isolation and recovery via external magnetic field.

In further studies, four amine bases including diethylamine, diisopropylamine, morpholine and piperidine were immobilized onto the surface of the modified Fe_3O_4 magnetic nanoparticles to give magnetic silica nanoparticle bases which are Fe_3O_4-Si-TEA, Fe_3O_4-Si-DIPA, Fe_3O_4-Si-NMM and Fe_3O_4-Si-MNP. These bases were successfully applied in the synthesis of *N*-benzyl-benzenesulfonamide. Comparable high percent yield of product can be obtained when using Fe_3O_4-Si-base while the highest yield was obtained when using Fe_3O_4-Si-DIPA (98%). Due to high basicity and steric shielding of alkyl group around N-atom of DIPA base found to be high an

efficient base for this study. Thus the resulted Fe_3O_4-Si-DIPA was selected to apply as base reagent in further study. Thus, the applicability of Fe_3O_4-Si-DIPA was demonstrated in sulfonation, acylation and akylation reactions. All reactions gave high yields of products without need for further purification. Fe_3O_4-Si-DIPA offer several advantages including ease of product isolation, short reaction times under mild and neutral conditions.

In summary Fe_3O_4-Si-DIPA was successfully prepared from inexpensive starting materials and developed for use as base reagent in organic synthesis. The advantages of this base include high yields of products, simple experimental procedure, avoiding poisonous solvent, no need for purification step and the high percent recovery of product. Moreover, this method is less time-consuming and require less amount of organic solvent for completed reaction, therefore this method can be used for preparation of diverse libraries of compounds.

In continuation of the work described in this study, the following suggestions for further work are made:

1.) The case of the amine base solution modified on surface magnetic silica (Fe_3O_4-**3**) in substitution reaction between chloride group of Fe_3O_4-**3** and amine base solution, this method may be improved to allow N-alkylation of amine base solution onto surface silica magnetic surface leading to high loading of required functional groups.

2.) The loading of diisopropylaminoacetamide (DIPA) modified on surface of magnetic silica should be investigated.