

CHAPTER IV

CONCLUSION

From the preceding results and discussion, the main focus of this research is to search for the optimum conditions for epoxidation of alkenes catalyzed by six cobalt(II) calix[4]pyrroles. The study was carried out in the homogeneous catalytic system with a combination of an aldehyde and oxygen as an oxidizing agent at room temperature. Cobalt(II) calix[4]pyrrole binding with 4-methoxyphenyl ligand was disclosed to be the most effective catalyst in this developed epoxidation. Cyclohexene 5 mmol as substrate, cobalt(II) calix[4]pyrrole complex **16**, 0.05 mmol as catalyst, acetonitrile 36 mL as solvent, 2-ethylbutyraldehyde 10 mmol and oxygen and aldehyde as oxidant in the reaction period of 24 hrs was discovered to be the optimum condition. The highest yield, 85% of the corresponding epoxide was attained.

Epoxidation study was also widened to other types of alkenes. Applications based upon the developed epoxidation of aliphatic alkenes such as 1-dodecene, *trans*-2-hexen-1-ol, aromatics containing double bond such as styrene, α -methylstyrene, monoterpenes containing double bonds such as α -terpinene, γ -terpinene, *R*-(+)-limonene and *S*-(-)-limonene, and 4-vinylcyclohexene was achieved and gave the desired products in high yield with excellent selectivity.

In terms of kinetic study the results displayed that the half-life for cyclohexene epoxidation under this system was approximately 7 hrs in acetonitrile. In addition, the comparative kinetic study of 1-dodecene catalyzed by cobalt(II) calix[4]pyrrole complex, **16** in the presence of acetonitrile and toluene was investigated. The reaction in toluene took place faster than that in acetonitrile, respectively. The half-life of the reaction was decreased from 10 hrs in acetonitrile to 8 hrs in toluene. Moreover, toluene was employed as an appropriate solvent for the selected alkenes. The mechanism of the alkene epoxidation was confirmed to occur *via* free radical pathway supported by both the results derived from the addition of radical scavengers and the isolation of byproduct, 2-ethylbutanoic acid. High valent cobalt(IV)

oxocalix[4]pyrrole radical intermediate was believed to be responsible for the epoxidation of alkenes.

To our best knowledge, the utilizing of cobalt(II) calix[4]pyrrole catalyst in the presence of aldehyde and oxygen has never been reported in chemical literature for alkene epoxidation.

Recommendation for the further work

The modification of this catalytic system to larger scale experiment that could be practiced in a pilot scale of petrochemical industry may be the one of important goals to accomplish. The comparative study between cobalt(II) calix[4]pyrrole catalysts and/or the catalysts such as Schiff-base complexes in alkene epoxidation should be performed. Furthermore, the variation of metal in order to form complexation of calix[4]pyrrole ligand for the epoxidation of alkenes or other related reactions should be examined. In addition, other types of alkenes: allylic alcohol or alkenes containing other functional groups should also be investigated to fulfill the scope of this developed system.



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