CHAPTER V

CONCLUSION

Iron oxide waste can be prepared by precipitation with ammonia solution. The resulting iron oxide has high iron content up to 95%. Fe-MCM-41 can be synthesized using the two-step crystallization. The XRD patterns of Fe-MCM-41, which was synthesized using ferric nitrate as the iron source and had the Si/Fe ratios of 50 and 100, have shown the characteristic peaks of hexagonal pore structure. But the crystallinity is decreased with increasing the iron content of the sample, e.g. Fe-MCM-41 with the Si/Fe ratio in gel of 30 and 40. Moreover, as-synthesized and calcined samples of Fe-MCM-41 synthesized using iron oxide waste with the Si/Fe ratio of 50 also exhibited four characteristic peaks, which showed the thermal stability of the sample. For identification of iron local sites in the framework, the ESR spectra of as-synthesized and calcined samples have displayed two signals at $g = 2.0$ and 5.0 which can be attributed to octahedral and tetrahedral coordination of Fe^{3+} in the framework. N_2 adsorption-desorption isotherms have also manifested the type IV isotherm of mesoporous materials with pore size range of 2.9-3.1 nm and high surface area up to 830 m^2/g.

Both of Fe-MCM-41 catalysts synthesized using difference iron sources have shown good catalytic activity for arylation of benzene with benzyl chloride and alkylation of benzene with 2-chlobutane to produce diphenylmethane and *sec*-butylbennzene, respectively. The reaction conversion is increased with increasing reaction time and temperature. For alkylation of benzene with benzyl chloride, the conversion is greater than 99% when the reaction condition is 60 °C and

2 hours. However, the selectivity of main product is decreased when the reaction time is expanded, which is due to the parallel-polyalkylation reaction of benzene to produce polyalkylbenzene molecules. Therefore, the appropriate reaction temperature and time for arylation of benzene with benzyl chloride to get high selectivity to main product in this study is 100 °C for 1 hour, and for alkylation of benzene with 2-chlorobutane is 100 °C for 2 hours.

From this work, it has been concluded that iron waste can be recycled and used as the iron oxide source for Fe-MCM-41 catalysts. The efficiency and performance of these catalysts are excellent in compared with Fe-MCM-41 synthesized using commercial ferric nitrate.