CHAPTER V

CONCLUSION

Cu-pillared bentonite was synthesized by the intercalation method followed by calcination at 300°C for 4 h. The 0.5 M aqueous solution of $Cu(CH_3CO_2)_2$ is used as a pillaring agent. The synthesized product was characterized using XRD, N_2 adsorption-desorption, and inductively coupled plasma–atomic emission spectroscope (ICP-AES). The XRD patterns of the Cu-pillared clays indicate that the layered structure of clay is sustained. The d_{001} spacing of Cu-pillared bentonite is reported for 15.23 Å. The nitrogen adsorption-desorption isotherms of Cu-pillared clays suggested the distorted reversible type IV isotherm indicating that CuO in calcined samples converting clay-layered structure to mesoporous structure. Therefore, the BET specific surface areas of Cu-pillared clays are higher than those of pure clays. The copper content in samples was determined by ICP-AES method, copper contents of Cu-pillared clays is higher than pure clays. Cu-pillared bentonite exhibited the highest copper content (15.59% CuO form).

The Cu-pillared bentonite is then used for olefination reaction. The optimum condition for olefination of carbonyl compounds is 30 wt% Cu-pillared bentonite to aldehydes. The exploration of halogenated reagents for the synthesis of alkenes was also examined. The optimum conditions were showed the suitable aldehyde is 4-chlorobenzaldehyde. The ratio of aldehyde and CCl_4 is 1:5 and the reaction was carried out under refluxing 3 h for step 1 and step 2 carried out under room temperature DMSO for approximately 4 h or followed by TLC. The olefination study is also applied to other halogenated reagent, such as CCl_3Br, CBr_4, CHI_3, CCl_3F,

CH_3I, Cl_3CCONH_2 and CCl_3CN. Polyhaloalkenes were obtained at room temperature for 24 h in step 2.

Suggestion for the future work

This research focuses on the methodology of olefination reaction with Cu-pillared clay catalyst. Other metals such as Al and Cu/Al may be used instead of copper in pillared clay catalyst to improve percentage yield and selectivity of the reaction. The Al complexs are commonly used to pillar the clay structure, since they are large and relatively stable. The aluminium oxide-pillared clays usually have large d-spacing between clay layers and high surface area. Copper can be doped onto aluminium oxide pillared clays to modify its acidity, selectivity of this reaction. Other aldehydes and halogenated reagents can be tested to expand the scope of the reaction.