# CHAPTER V

# CONCLUSIONS

To study the effect of basic heterogeneous catalyst on the activities of palm oil transesterification (biodiesel production), ETS-10 from two types of silica source; colloidal silica (CS) and water glass solution (WG), germanium substitute ETS-10 (ETGeS-10) and base modified ETS-10 catalysts were used. Moreover, the optimum conditions were also investigated to increase the methyl ester yield.

ETS-10 catalyst was synthesized under basic conditions by modifying the method according to Tutsumi, T., *et al.* [16] and Anderson, M. W., *et al.* [10] with the gel composition $5SiO_2$: $TiO_2$: 3NaOH: KF: 75 $H_2O$ and $5.5SiO_2$: $TiO_2$: $5.2Na_2O$: 0.5 $K_2O$: $113H_2O$, respectively. Germanium substituted ETS-10 (ETGeS-10) was the first time synthesized with Ge/Ti 0.15 to 0.5 to observe the increased basicity and catalytic activity in transesterification reaction. Moreover, basic modified ETS-10 catalyst was also prepared by two methods; ion-exchange (NaETS-10) and incipient wetness impregnation with sodium compounds (NaOAc/ETS-10 and NaOH/ETS-10) and subsequent calcination at 450°C for 6 h.

From characterization of all materials, XRD patterns of bared ETS-10 both synthesized from colloidal silica and water glass solution exhibit ETS-10 structure together with co-crystallized anatase at 2-theta about 25.3. ET(Ge)$_x$S-10 (x is 0.15 and 0.5) still exhibit the ETS-10 structure. Upon increasing the germanium loading, the characteristic peaks of ETS-10 decrease in intensity along with the increase in peak intensities of quartz. When x equals to 1.0 and 1.5, the characteristic peaks of ETS-10 structure disappear completely. All of Na-modified materials also maintain characteristic peaks of the ETS-10 structure with lower crystallinity. From SEM technique, all of catalysts were quasi-cubic shape and the particle size was in the range of 300-700 nm. In addition, all of catalysts exhibit $N_2$ adsorption isotherm of type I, characteristic of micropores. Surface area of germanium substituted and sodium loading ETS-10 reduce compared with ETS-10 sample but the pore size was nearly the same. DR-UV spectra can identify titanium form that occupied in catalyst including pentahedral, octahedral form and anatase. Germanium content was quantitative analyzed by SEM-EDX technique and Si/Ti, Na/Ti and K/Ti mole ratio of all catalysts were determined by ICP-AES and AAS.

In transesterification reaction of palm oil, all of catalysts were applied to study the catalytic activity. ETS-10 (CS) exhibits higher activity than ETS-10 (WG) due to the smaller particle leads to higher surface area and higher crystallinity. Moreover, it can be concluded that the optimum conditions of ETS-10 (CS) were 10 wt% catalyst to reactant mixture, 9:1 methanol to oil mol ratio at 120°C for 24 h. The increasing of germanium content resulted in increasing the methyl ester yield. At low germanium contents in $ET(Ge)_xS$-10 catalysts (x = 0.15 and 0.5), methyl ester was produced at the yield of 3.22 and 13.30 wt%, respectively. At higher germanium loadings where x equals to 1.0 and 1.5, the methyl ester yield decreases due to the collapsed structure. In addition, Na-loaded ETS-10 from ion-exchange and impregnation method displays more catalytic activity than pure sample. The highest methyl ester yield was 77.57 and 79.80% that was obtained when NaETS-10 and NaOH/ETS-10 were used, respectively. The catalytic activities of regenerated catalyst were decreased while after reloaded with NaOH by either ion-exchange or impregnation, the methyl ester yield was reached almost the same yield as fresh one.

**The suggestion for future work**

The catalyst in transesterification reaction can be adapted to improve the product yield. Aluminium and gallium substituted ETS-10 was interesting as basic catalyst to improve the methyl ester yield. Base-modified ETS-10 by ion-exchange with higher electronegativity species such as K, Rb and Cs was also an attractive point. Moreover, triglyceride sources may be changed to other natural oils or wasted oil to save the capital cost.