CHAPTER V
CONCLUSIONS

Natural Materials were tested as a catalyst for biodiesel production. There had no biodiesel yield from any runs of this set at reaction conditions; catalyst concentration; 10% by weight of oil, methanol to oil molar ratio; 6:1, reaction temperature of 65°C, and 60 minutes of reaction time. These results showed that natural materials by itself were not active for transesterification.

The next part of the experiments was conducted by using many types of metal oxides; M1O, M2O, M3O, M4O, and M5O including complex metal oxides. The concentration of the catalyst used was 3% by weight of oil. The methanol to oil molar ratio is 6:1. Reaction temperature of 65°C, and 30 minutes of reaction time were used. Normally, the higher basic strength of the metal oxide could achieve higher yield from transesterification. But, M1O and M2O could achieve yield higher than 97%. The yield obtained from M3O was less than 1%. And, M2O could form toxic solutions with methanol. Then, only M1O was selected for evaluation as the main catalyst or as a part of a mixture of catalysts. For complex metal oxides did not show any signs of conversion of canola oil to biodiesel as well as the use of metal carbonates.

The catalysts combinations were tested with the ratio of M1O-to-CS1 powder varied from 1:9, 2:8, 3:7, 4:6, 5:5, 6:4, 7:3, 8:2, and 9:1 for a total concentration of 1wt% catalyst mixture by weight of canola oil. The reaction conditions used were; methanol to oil molar ratio 6:1, 30 and 60 minutes of reaction time, and the reaction temperature of 45°C. The maximum improvement of biodiesel yield obtained using this combination of metal oxide and crustacean shell was observed when at a reaction time of 30 minutes. Based on these experimental observations, the reaction time of 30 minutes was chosen to be used for the rest of the experiments. Because the effect of the longer reaction time over took the effect of catalyst combination.

In order to optimize the reaction temperature that the catalysts combinations could increase the yield improvement, only some ratios that worked from the previous section were tested. The ratio of M1O-to-CS1 powder varied from 5:5, 6:4, 7:3, 8:2, and 9:1 for a total concentration of 1wt% catalyst mixture by weight of canola oil were investigated. The reaction conditions used were; methanol to oil molar ratio 6:1, 30 minutes of reaction time, and the reaction temperature varied from 45°C, 55°C, and 65°C. The maximum improvement of biodiesel yield obtained using this combination of metal oxide and crustacean shell was observed when at a reaction temperature of 45°C. The improvement from the use of M1O-to-CS1 powder that varied from 5:5, 6:4, 7:3, 8:2, and 9:1 were 1.63%, 4.33%, 3.80%, 5.66%, and 2.55% respectively. For the reaction at 55°C and 65°C, the combination slightly improved biodiesel yield. Based on these experimental observations, a reaction temperature of 45°C was chosen to be used for the rest of the experiments because the main objective of this work is the optimization of biodiesel production at mild reaction conditions aiming an energy efficient process for the production of biodiesel.

For the next part of the experiments, the same reaction conditions were used with several catalysts combinations. Animal shell, crustacean shell and mollusk shell were evaluated to formulate catalyst mixtures with M1O. These natural materials were CS2, AS, MS1 and MS2. The mixture of M1O:CS2, and M1O:AS showed the good performance as was in the case of using the mixture of MO1:CS1. These results indicated that the natural materials from the crustaceans that live in the sea which contain some traces of metal elements such as M1, M2 ,M3 ,sodium and silica, worked very well as a catalyst additive. The same result obtained from the test of AS combination which contains calcium carbonate as a major component without the other trace of metal elements like the crustacean shells. In the other hand, mollusk shells were not work as catalyst additives even it contains calcium carbonate as a main component. The possible explanation for this phenomenon is the difference in the calcium carbonate formed. However, more researches need to be done to confirm this idea.

Combinations of metal oxides were tested in the same way. M1O was used as a base catalyst combined with M3O, M4O, and M5O. The mixture of M3O:M4O improved biodiesel yield up to 4% compared to the use of M1O alone at the same reaction conditions. M3O itself work as a catalyst for biodiesel production but it needs longer reaction time and higher reaction temperature Mixing of M1O with M3O could increase the reactivity of the reaction.

The main chemical component of natural material is $CaCO_3$. Then, the combinations of M1O and many kinds of metal carbonates were investigated. Three types of carbonates; M1CA, M3CA, and M6CA were used at the same ratio as in previous tests for the natural materials. Two combinations of catalysts performed very well improving biodiesel yield. The catalyst combinations MO1: M1CA and M1O:M3CA achieved higher biodiesel yield compared to M1O alone. These results confirm that some kinds of metal carbonates which contain in the crustacean shells and animal shell, take the main roles for reaction activity improvement.

However the further steps for this research include the evaluation of the effective catalyst combinations obtained in this study with others types of feedstock such as waste cooking oils and animal fats, which contain higher free fatty acid, to study the effects of free fatty acid on the activity of these catalyst combinations. In additions, more research needs to be done in order to establish the reaction mechanisms of these catalysts during the transesterification reaction of vegetable oil as well as the feed stocks which contain high amount of free fatty acid such as waste cooking oils and animal fats.