



## CHAPTER III

### EXPERIMENT

The preparations of the experiment and analytical methods are described in this chapter. It is divided into three sections. The first section concerns with the materials. The second part describes the equipment of this experiment and the third section describes the method of the transesterification of vegetable oils.

#### 3.1 Materials

##### 3.1.1 Palm olein oil

Vegetable oil used in this study was palm olein oil. The palm olein oil was a commercial grade available in supermarket: Oleen brand, Palm olein CO, Ltd.

##### 3.1.2 Coconut oil

Vegetable oil used in this study was coconut oil. The coconut oil was a commercial grade available in supermarket: Bua brand.

##### 3.1.3 Chemicals

All chemicals used in the experiment are shown in Table 3.1. They are used without further purification.

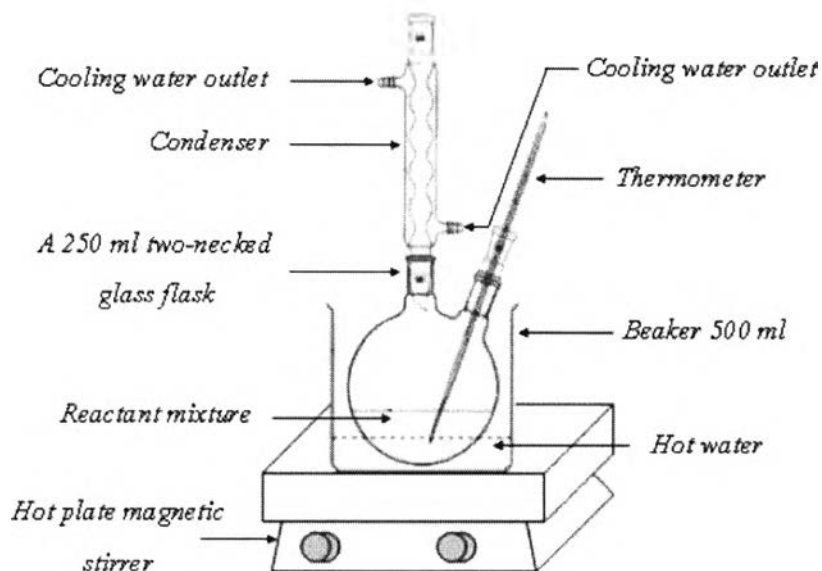
**Table 3.1** Chemicals used in the research of palm olein oil and coconut oil with methanol.

Name	Source	Purity
Methanol	Fisher Scientific	98% (industrial grade)
Tetrahydrofuran	Fisher Scientific	99.99% (analytical reagent grade)
Calcium oxide	Ajax Chemicals	96% (analytical reagent grade)
Magnesium oxide	Sigma-Aldrich	99% (analytical reagent grade)
Potassium carbonate	Ajax Finechem	99.00% (laboratory reagent)
Ammonium carbonate	Ajax Finechem	98.00% (analytical reagent grade)
Heptane	QREC	99% (analytical reagent grade)
Methyl Decanoate	ALDRICH	99% (analytical reagent grade)
Mix Fatty Acids Methyl Ester	RESTEK	99.5%+ (laboratory reagent)

## 3.2 Equipment

### 3.2.1 Transesterification of palm olein oil and coconut oil

All experiments were carried out in a 500 ml round-bottom flask. The flask was connected to a reflux condenser and was heated in a water bath as shown in Figure. 3.1. The reaction was carried out at a temperature of 60°C, oil/methanol molar ratio of 6:1 and reaction time of 2 h.



**Figure 3.1** The experimental set up for transesterification reaction of palm olein oil and coconut oil.

### 3.3 Methodology

#### 3.3.1 Characterization of palm olein oil and coconut oil.

Palm olein oil and coconut oil are analyzed based on free fatty acid content, acid value, Iodine value and saponification value (AOCS Ca 5a-40).

#### 3.3.2 Catalysts preparation

Fresh CaO, calcined CaO, Fresh MgO, calcined MgO, calcined  $K_2CO_3/CaO$ , calcined  $(NH_4)_2CO_3/CaO$ , calcined  $K_2CO_3/MgO$ , calcined  $(NH_4)_2CO_3/MgO$  are used as catalysts in this study. Fresh CaO and MgO were calcined in air at a temperature of  $600^\circ C$  for 3 h. Fresh CaO and MgO were also dipped into  $(NH_4)_2CO_3$  solution and  $K_2CO_3$  solution and stirred for 30 minutes. Solid parts were separated from the solution, dried in an oven at  $110^\circ C$  for 24 h and calcined at  $600^\circ C$  for 3 h. All catalysts were kept in a desiccator.

### **3.3.3 Characterization of Catalysts.**

#### **3.3.3.1 N<sub>2</sub> Physisorption.**

The BET surface area, pore size and pore volume were examined using Autosorb-1 AS1W in, version 1.24 (Quantachrome instruments, USA). N<sub>2</sub> gas was used for measuring the surface area, pore size and pore volume of sample. A 0.3 g of sample was degassed at 250°C for 12 h under vacuum. After degassing the sample was run at -196°C for 6 h.

#### **3.3.3.2 XRD (X-ray diffraction)**

The phases of catalyst were examined using the Bruker AXS pert powder Xray diffractometer with the Cu- $\alpha$  radiation at 40 kV and a current of 30 mA. Data was collected over a  $2\theta$  range of 5-90<sup>0</sup> with a step size of 0.02<sup>0</sup>/step. The data was evaluated with the diffracplus software. The phases were detected by a proportion ZnS (Zinc sulfide) detector. A 3 g of each sample was crushed and pressed into an calcium oxide sample holder then put it into the X-ray diffractometer. Each sample was run for 1 h.

### **3.3.4 Transesterification of palm olein oil and coconut oil using CaO and MgO catalysts**

1. CaO and MgO catalysts are calcined at temperature of 600°C for 3 hours.

2. Commercial grade palm olein oil and coconut oil are refined again to reduce water. This can be accomplished by heating to above 100°C to boil off the water.

3. A 250 ml one-necked glass flask with a reflux condenser is used in transesterification of palm olein and coconut oil to fatty acid methyl esters. A 100 g of palm olein oil and coconut oil, ratio of methanol 6:1, amount of

catalysts 5 %wt and the addition of THF in purpose of eliminating the interface and the mass transfer barrier to form a single phase in which methanol, oil and THF are mutually soluble.

4. The reactions are carried out at temperature of 65°C under stirring at constant for 2 hours.

5. Samples are taken out from the reaction.

6. The samples are separated by mean centrifuge and purified by distilling the residual methanol and THF at 67°C.

7. The remaining catalyst is extracted by successive rinses with water.

8. The water presence is eliminated by heating to above 100°C to boil off water.

### **3.3.5 Transesterification of palm olein oil and coconut oil using CaO and MgO catalysts modified with ammonium and potassium compounds.**

1. Preparation of  $K_2CO_3/CaO$ ,  $(NH_4)_2CO_3/CaO$ ,  $K_2CO_3/MgO$  and  $(NH_4)_2CO_3/MgO$  catalysts are given as follows 3.3.2.

2. Commercial grade palm olein oil and coconut oil are refined again to reduce water. This can be accomplished by heating to above 100°C to boil off the water.

3. A 250 ml one-necked glass flask with a reflux condenser is used in transesterification of palm olein and coconut oil to fatty acid methyl esters. A 100 g of palm olein oil and coconut oil ratio of methanol 6:1, amount of catalysts 5 %wt and the addition of THF in purpose of eliminating the interface and the mass transfer barrier to form a single phase in which methanol, oil and THF are mutually soluble.

4. The reactions are carried out at temperature of 60°C under stirring at constant for 2 hours.

5. Samples are taken out from the reaction.

6. The samples are separated by mean centrifuge and purified by distilling the residual methanol and THF at 67°C.
7. The remaining catalyst is extracted by successive rinses with water.
8. The water presence is eliminated by heating to above 100°C to boil off water.

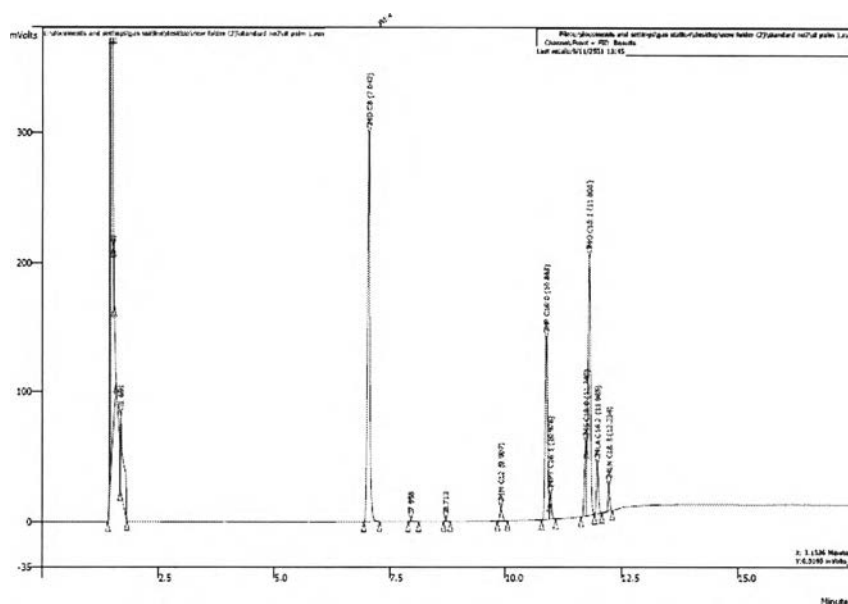
### **3.4 Analysis of methyl esters**

#### **3.4.1 Free fatty acids content (AOCS Ca 5A-40)**

The first step of the process is to reduce FFA content in palm oil by esterification with methanol. Free fatty acids content must be less than 2 %wt of oil can check by titration follow standard AOCS Ca 5A-20

#### **3.4.2 Methyl esters analysis**

The analysis of standards and the products formed were carried out on Variance 8700 gas chromatography (GC) at Hazardous Laboratory, Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University. A non-polar capillary column (Carbowax M20) with an internal diameter of 0.32 mm, length of 30 m and film thickness of 0.25 m and flame ionization detector (FID) was used. Helium was used as a carrier gas with pressure 10 psig. The injector and detector were set at 300 °C, respectively. The column temperature was programmed with an initial temperature 80°C for 5 min, heating at 20°C /min to 230°C, then holding on 5 min to a final temperature of 230°C. Methyl decanoate was chosen as an internal standard and heptane was solvent. Therefore, the type of methyl esters was compared retention time of each methyl esters with methyl esters standard in Figure 3.3 and Table 3.2.



**Figure 3.2** GC chromatogram of methyl esters

**Table 3.2** Retention time of methyl esters in GC chromatogram.

Retention time (min)	Peak of sample
1.479	N-Heptane
6.911	Methyl Decanoate
9.787	Methyl Myristate
10.788	Methyl Palmitate
10.860	Methyl Palmitoleate
11.625	Methyl Stearate
11.704	Methyl Oleate
11.860	Methyl Linoleate
11.088	Methyl Linolenate

The concentration of methyl esters and fatty acid is defined as a ratio of weight of methyl esters or fatty acid, which was determined by using gas chromatography (GC) to weight of sample as shown in Equation 3.1.

$$\% \text{Methyl Esters} = \frac{\text{Weight of Methyl Esters}}{\text{Weight of Sample}} \times 100 \quad (3.1)$$

By; weight of methyl esters calculate from response factor determined by GC