CHAPTER IV

RESULTS AND DISCUSSIONS

Experimental results and discussions of waste from biodiesel production process by transesterrification of palm oil with methanol using acid catalyst and basic catalyst are presented in this chapter. Details of each experiment were presented in Table C-1 to Table C-4, Appendix C. Procedure of each experiment was explained in section 3.3, CHAPTER III. In each experiment, transesterification reaction of palm olein, palm stearin, and used palm olein with methanol were performed in a batch reactor using potassium hydroxide and sulfuric acid as catalysts. Amount of catalysts are 0.5 and 1.0% calculated by weight of oil. The reaction was carried out with 6.1 molar ratio of methanol to oil at 60-65°C of reaction temperature and two hours of the reaction time.

After the end of each experiment, reaction products were transferred into a separating funnel then left for two hours for separation of biodiesel and glycerol phase. Samples were taken from both phases and were kept for analysis to determine the amount of residue catalysts, soaps, and methanol. Biodiesel phase was further washed with water to remove its contamination. Washed water is also analyzed. The contamination as residue catalysts and soaps in each phase of reaction products and amount of washed water were defined as the waste from process. The results of each experiment are shown in Table C-13 to Table C-16, Appendix C. Results and discussions are categorized into five parts as follows:

4.1 Characterization of feedstocks

4.2 Distribution of unreacted methanol and percentage of methyl esters

4.3 Effect of type and amount of catalysts in percentage of residue catalysts and soaps in reaction products and amount of waste water

4.4 Effect of types of palm oils in percentage of residue catalysts and soaps in reaction products

4.5 Comparison percentage of residue catalysts and soaps in reaction products from industrial scale and reaction products from laboratory scale

4.1 Characterization of feedstocks.

Palm oil feedstocks were used in this study received from the supermarket, biodiesel industry, and frying shop respectively. They are analyzed the properties and the analysis results are shown in Table 4.1.

Table 4.1 Properties of feedstocks

Properties	Palm olein	Palm stearin	Used palm olein
Acid Value, (mg KOH/g)	0.86-0.91	0.71-0.74	2.05
Saponification Value, (mg KOH/g)	196.47	193.35	200.76
% Free Fatty Acid as Palmitic Acid	0.30-0.32	0.24-0.26	1.29
Mean molecular Weight, (g/mole)	845.33	832.68	800.81
Color	Clear yellow	White	Dark brown

At room temperature palm olein is in liquid phase. Palm stearin is in solid phase. Used palm olein is in liquid phase and contains a solid particle in the bottom. The results show that used palm olein has a higher of acid value than palm olein and palm stearin. From the results, used palm olein contains a high of free fatty acids. Free fatty acids affected with amount of catalyst. They reduced amount of catalyst when basic catalyst was used. The calculation of amount of catalysts must be calculated amount of a catalyst used for neutralized free fatty acids. The calculation value is 1.8 grams of potassium hydroxide needed to neutralize free fatty acids in used palm olein. Amount of basic catalysts was equal 1.8 grams plus 0.5% of input catalyst by weight of oil when using used palm olein as raw material.

4.2 Distribution of unreacted methanol and the percentage of methyl esters

4.2.1 Distribution of unreacted methanol

Product mixtures of crude biodiesel and crude glycerol were transferred into a separating funnel then left for two hours for separation of crude biodiesel and crude glycerol phase. After phase separation, unreacted methanol in each phase of reaction products were removed by vacuum distillation, then weighing amount of methanol and amount of products from both phases. Amount reaction products and percentage of unreacted methanol are shown in Table 4.2 and Table C-5 to Table C-12 in Appendix C.

Table 4.2 Amount of reaction products and percentage of unreacted methanol in each phase of reaction products

Feedstock	Catalyst	Amount of catalyst (g)	Amount of reaction products and percentage of unreacted methanol in reaction products			
			Crude glycerol phase		Crude biodiesel phase	
			CG* (g)	Methanol (%wt)	CB* (g)	Methanol (%wt)
Palm Olein	KOH	2.527	64.7	26.58	496	7.52
		5.032	73.2	26.95	486	7.15
	H_2SO_4	2.502	19.2	36.61	502	8.22
		5.057	25.6	33.25	499	8.41
Palm Stearin	KOH	2.521	64.2	26.62	495	8.67
		5.045	72.7	26.67	487	7.08
	H_2SO_4	2.511	18.8	36.18	502	7.35
		5.032	25.6	32.41	499	8.39
Used Palm Olein	KOH	4.302	79.7	26.62	488	7.93
		6.810	91.2	25.84	475	7.71
	H_2SO_4	2.515	26.1	41.25	491	9.32
		5.045	37.8	32.20	488	9.01

*"*CG = Crude glycerol, *CB = Crude biodiesel"*

Experimental results show that the unreacted methanol dissolved in both phases of reaction products. The percentage of unreacted methanol in each phase of reaction products was shown in Table C-9 to Table C-12 in Appendix C. The percentage of unreacted methanol was calculated by weight of input methanol. From the results, approximate 25-40% of excess methanol dissolved in crude glycerol phase and 7-9% dissolved in crude biodiesel phase. The polarity of chemical compounds can explain this result. Methanol and glycerol are the polar chemical compounds but biodiesel is non polar. The polar chemical compounds are good dissolved in the polar chemical compounds together. This indicates that most of methanol dissolved in crude glycerol phase.

Samples of crude biodiesel and crude glycerol were taken to determine for their contamination by titration technique. Crude biodiesel was washed with water to remove its contamination and washed water is also analyzed. Amount of residue catalysts and soaps in each phase of reaction products and washed water are shown in Table C-13 to Table C-16 in Appendix C.

4.2.2 Percentage of methyl esters

Samples of washed biodiesel were taken and kept to analyze the percentage of biodiesel (methyl eaters) by gas chromatography technique. This can be confirmed that the reaction in the experiment produced the product of biodiesel. The results are shown in Table 4.3 and Table 4.4.

Table 4.3 Percentage of biodiesel in basic catalyzed process

Feedstock	Catalyst	Amount of catalyst (g)	Percentage of biodiesel (% wt)
Palm Olein	KOH	2.527	73.53
		5.032	78.34
Palm Stearin	KOH	2.521	73.41
		5.045	77.95
Used Palm Olein	KOH	4.302	70.53
		6.810	75.45

Experimental results show that percentages of methyl esters are in the range of 70-80 %wt. These results are similar to those found in the literature. Antolin et al. (2002) and Vicente et al. (1998) studied the transesterification process of sunflower oil using sodium hydroxide and potassium hydroxide as catalysts. The reaction carried out with 6:1 molar ratio of methanol to oil. They concluded that transesterification of sunflower oil using potassium hydroxide and sodium hydroxide as catalysts gave high yield of methyl esters. Pedro (2005) studied transesterification process of used frying oil with methanol using sodium hydroxide as catalyst. 3:6 to 5:4 molar ratio of methanol to oil were used. The reaction carried out with 0.2 to 1.0% of catalyst. They concluded that transesterification of used frying oil with methanol using 0.6% of catalyst and 4:8 molar ratio of methanol to oil gave a high yield of methyl esters. The literature indicates that potassium hydroxide and sodium hydroxide are good catalysts because they gave a high conversion or high yield of biodiesel.

Table 4.4 Percentage of biodiesel in acid catalyzed process

Feedstock	Catalyst	Amount of catalyst (g)	Percentage of biodiesel (% wt)
Palm Olein	H_2SO_4	2.502	7.33
		5.057	9.05
Palm Stearin	H_2SO_4	2.511	7.35
		5.032	9.04
Used Palm Olein	H_2SO_4	2.515	9.65
		5.045	10.54

Experimental results show that percentages of methyl esters are in the range of 7-10 %wt. The literature presents several studies relating to this process to reduced high free fatty acid in feedstock. Canaki et al. (1999) studied the affected of molar ratio, reaction rates, and product yield in the transmethylation of soybean oil by sulfuric acid. Five different molar ratios, from 3.3:1 to 30:1, were studied. Their results indicated that ester formation increased with increasing molar ratio, and gave 98.4% of conversion at the highest molar ratio. Crabbe et al. (2001) investigated acid catalyzed (sulfuric acid) production of methyl ester (biodiesel) from crude palm oil. The reactions were carried out at 95°C. They determined the affect of molar ratio of methanol to oil from 3:1to 40:1, the effect of amount of catalysts at 1 to 5% H_2SO_4 (vol/wt%). They concluded that the optimized variables, 40:1 methanol to oil (mol/mol) with 5% H_2SO_4 (vol/wt%) reacted at 95 °C for 9 hour, gave a maximum ester yield of 97%.

Commercial biodiesel production processes do not use acid catalyst in direct process. They usually use acid catalyst in case of high free fatty acids in feedstock.

The acid catalyst reduces free fatty acids by converting them to ester. This reaction is called "esterification reaction". Detail of the reaction was presented in section 2.3, CHAPTER II. The process using acid catalyst to reduce free fatty acid content also called "acid catalysis pre-treatment". Some process both acid and basic catalyst was used. This process is call "two-step transesterification process". Acid catalyst was used in the first step and then basic catalyst was used in the second step.

4.3 Effect of type and amount of catalysts in percentage of residue catalysts and soaps in reaction products and amount of waste water

4.3.1 Effect of type of catalysts

Effect of type of catalysts on the waste from transesterification process was studied in batch system. Potassium hydroxide and sulfuric acid were used as basic and acid catalysts. Amount of catalysts is 0.5% calculated by weight of oil. The reaction was carried out with 6:1 molar ratio of methanol to oil at 60-65°C of reaction temperature and two hours of the reaction time. The results are shown Table 4.5 and Table 4.6.

Table 4.5 Percentage of residue catalysts in each phase of reaction products of basic and acid catalyzed transesterification experiments

Feedstock	Catalyst	Amount of catalyst (g)	Percent of residue catalysts by weight of product in each phase (%wt)			
			CG*	CB*	WW*	RB*
Palm Olein	KOH	2.527	2.13	0.074	0.028	-
	H_2SO_4	2.502	5.30	0.098	0.075	-
Palm Stearin	KOH	2.521	2.12	0.073	0.028	-
	H_2SO_4	2.511	5.24	0.098	0.075	-
Used Palm Olein	KOH	4.302	2.43	0.127	0.032	-
	H_2SO_4	2.515	3.35	0.098	0.075	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Experimental results show that the distribution of residue catalysts in each phase of reaction products was similar when using basic and acid catalysts. The percentage of residue catalysts was calculated by weight percent of product in each phase. It is found that residue catalysts dissolved in each phase of reaction products. Amounts of residue catalysts in crude glycerol phase were higher than in crude biodiesel phase. Residue catalysts are good dissolved in the polar chemical compounds. It can be explained that crude glycerol phase contains the polar chemical compounds. Methanol and glycerol are the polar chemical compounds but biodiesel is non polar. The percentage of methanol was shown in Table C-5 to Table C-8 in Appendix C. Amount of residue catalysts were not presented in refined biodiesel because they were eliminated by washing with water.

Table 4.6 Percentage of soaps in each phase of reaction products of basic and acid catalyzed transesterification experiments

Feedstock	Catalyst	Amount of catalyst (g)	Percent of soaps by weight of product in each phase (%wt)			
			CG*	CB*	WW*	RB*
Palm Olein	KOH	2.527	5.34	0.06	0.098	-
	H_2SO_4	2.502	-	-	-	-
Palm Stearin	KOH	2.521	5.34	0.06	0.098	-
	H_2SO_4	2.511	-	-	-	-
Used Palm Olein	KOH	4.302	8.90	0.09	0.106	-
	H_2SO_4	2.515	-	-	-	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Experimental results show that basic and acid catalysts give the different results on product of soaps. Soaps were dissolved in each phase of reaction products. Amounts of soaps in crude glycerol phase were higher than in crude biodiesel phase. The percentage of soaps was calculated by weight percent of product in each phase. Soaps were produced from the sponification reaction. This is the reaction of free fatty acids and potassium hydroxide converted to product of salt of carboxylic acid or soap. The details of saponification reaction or reaction of soaps were presented in section 2.4 in CHAPTER II. Soaps were not presented in acid catalyzed process. The distribution of soaps and residue catalysts in reaction products was similar. Amount of soaps was not presented in refined biodiesel because they were eliminated by washing with water.

4.3.2 Effect of amount of basic catalyst

Effect of amount of basic catalysts on the waste from transesterification process was studied in batch system. Potassium hydroxide was used as basic catalysts. Amount of catalysts are 0.5 and 1.0% calculated by weight of oil. The reaction was carried out with 6:1 molar ratio of methanol to oil at 60-65°C of reaction temperature and two hours of the reaction time. The results are shown Table 4.7 to Table 4.9.

Table 4.7 Percentage of residue catalysts in each phase of reaction products of basic catalyzed transesterification experiments

Feedstock	Catalyst	Amount of catalyst (g)	Percent of residue catalysts by weight of product in each phase (%wt)			
			CG*	CB*	WW*	RB*
Palm Olein	KOH	2.527	2.13	0.074	0.028	-
		5.032	3.51	0.153	0.057	-
Palm Stearin	KOH	2.521	2.12	0.073	0.028	-
		5.045	3.51	0.152	0.056	-
Used Palm Olein	KOH	4.302	2.43	0.127	0.032	-
		6.810	3.75	0.202	0.073	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Experimental results show that residue catalysts are dissolved in each phase of reaction products. The percentage of residue catalysts was calculated by weight percent of product in each phase. Amounts of residue catalysts increase with an increase in amount of catalysts. This indicates that amount of catalyst affected with amount of residue catalysts. It is found that amounts of residue catalysts in crude glycerol phase higher than in crude biodiesel phase because residue catalysts are good dissolved in the polar compounds. It can be explained that crude glycerol phase contains the polar chemical compounds. Methanol and glycerol are the polar chemical compounds but biodiesel is non polar. The percentage of methanol was shown in Table C-5 to Table C-8 in Appendix C. Amount of residue catalysts were not presented in refined biodiesel because they were eliminated by washing with water.

Table 4.8 Percentage of soaps in each phase of reaction products of basic catalyzed transesterification experiments

Feedstock	Catalyst	Amount of catalyst (g)	Percent of soaps by weight of product in each phase (%wt)			
			CG*	CB*	WW*	RB*
Palm Olein	KOH	2.527	5.34	0.060	0.098	-
		5.032	5.54	0.059	0.105	-
Palm Stearin	KOH	2.521	5.34	0.060	0.098	-
		5.045	5.52	0.060	0.103	-
Used Palm Olein	KOH	4.302	8.90	0.089	0.106	-
		6.810	9.28	0.089	0.105	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Experimental results show that soaps were dissolved in each phase of reaction products. Amounts of soaps in crude glycerol phase were higher than in crude biodiesel phase. The percentage of soaps was calculated by weight percent of product in each phase. Soaps are intermediate products. They were produced from the sponification reaction. This is the reaction of free fatty acids in feedstock and potassium hydroxide as catalyst converted to product of salt of carboxylic acid but they usually called soaps. The details of saponification reaction or reaction of soaps were presented in section 2.4 in CHAPTER II. Soaps were not presented in acid catalyzed process. The distribution of soaps and residue catalysts in reaction products was similar. Amount of soaps was not presented in refined biodiesel because they were eliminated by washing with water.

Table 4.9 Amount of waste water of basic catalyzed transesterification experiments

Feedstock	Catalyst	Amount of catalyst (g)	Amount of waste water (g)
Palm Olein	KOH	2.527	143.4
		5.032	284.0
Palm Stearin	KOH	2.521	142.5
		5.045	284.6
Used Palm Olein	KOH	4.302	141.2
		6.810	275.8

Waste water was determined from amount of output washed water in purification step of biodiesel production process. Amount of waste water are shown in Table 4.9. The water was added in the purification of crude biodiesel to reduce the contamination in crude biodiesel as residue catalysts and soaps. Increasing of input catalysts tended to increase of waste in reaction products. Biodiesel was washed until 6-7 of pH was presented. The pH of biodiesel was not equal 6-7 in the first time of washing when 1% of catalyst is used. Amount of washed water or waste water is shown in Table 4.9 and Table C-9 to Table C-12 in Appendix C.

4.3.3 Effect of amount of acid catalyst

Effect of amount of acid catalysts on the waste from transesterification process was studied in batch system. Sulfuric acid was used as acid catalyst. Amount of catalysts are 0.5 and 1.0% calculated by weight of oil. The reaction was carried out with 6:1 molar ratio of methanol to oil at 60-65°C of reaction temperature and two hours of the reaction time. The results are shown Table 4.10 and Table 4.11.

Table 4.10 Percentage of residue catalysts in each phase of reaction products of acid catalyzed transesterification experiments

Feedstock	Catalyst	Amount of catalyst (g)	Percent of residue catalysts by weight of product in each phase (%wt)			
			CG*	CB*	WW*	RB*
Palm Olein	H_2SO_4	2.502	5.30	0.098	0.075	-
		5.057	8.85	0.210	0.155	-
Palm Stearin	H_2SO_4	2.511	5.24	0.098	0.075	-
		5.032	8.90	0.215	0.153	-
Used Palm Olein	H_2SO_4	2.515	3.35	0.098	0.075	-
		5.045	5.23	0.213	0.154	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Experimental results show that residue catalysts were dissolved in each phase of reaction products. Percentage of residue catalysts was calculated by weight percent of product in each phase. Amounts of residue catalysts increase with an increase in amount of catalysts. This indicates that amount of catalyst has affected with amount of residue catalysts. The results show that amounts of residue catalysts in crude glycerol phase were higher than in crude biodiesel phase because residue catalysts are good dissolved in the polar chemical compounds. It can be explained that crude glycerol phase contains the polar chemical compounds. Methanol and glycerol are the polar chemical compounds but biodiesel is non polar. The percentage of methanol was shown in Table C-5 to Table C-8 in Appendix C. Amount of residue catalysts were not presented in refined biodiesel because they were eliminated by washing with water. Basic and acid catalyzed process give the same results on the distribution of residue catalysts in both phases of reaction products.

Table 4.11 Amount of waste water of acid catalyzed transesterification experiments

Feedstock	Catalyst	Amount of catalyst (g)	Amount of waste water (g)
Palm Olein	H_2SO_4	2.502	149.5
		5.057	299.6
Palm Stearin	H_2SO_4	2.511	150.8
		5.032	297.6
Used Palm Olein	H_2SO_4	2.515	145.1
		5.045	293.7

Waste water was determined from amount of output washed water in purification step of biodiesel production process. Amount of waste water are shown in Table 4.11. The water was added in the purification of crude biodiesel to reduce the contamination in crude biodiesel as residue catalysts and soaps. Increasing of input catalysts tended to increase of waste in products. Biodiesel was washed until 6-7 of pH was presented. The pH of biodiesel was not equal 6-7 in the first time of washing when 1% of catalyst is used. Amount of washed water or waste water is shown in Table 4.11 and Table C-9 to Table C-12 in Appendix C.

The results of this section can concluded that types of catalysts and amount of catalysts affected with amount of waste from transesterification process. Soaps are intermediate product from basic catalyzed process and defined as waste from process. They were not presented in acid catalyzed process. The saponification reaction can explained this result. The distribution of waste in each phase of reaction products of both catalysts was similar. It is found that most of waste dissolved in crude glycerol phase more than in crude biodiesel phase.

4.4 Effect of types of palm oils in percentage of residue catalysts and soaps in reaction products

Transesterification reaction of palm olein, palm stearin, and used palm olein with methanol was conducted to study effect of type of feedstocks on the concentration of waste. Detail of set of experiment was indicated in Table C-1 to Table C-4 in Appendix C. The experiments were conducted in a batch system at atmospheric pressure. Reaction temperature of 60-65°C and two hours of reaction time were used for each experiment. The results are shown in Table 4.12 and Table 4.13.

Table 4.12 Percentage of residue catalysts in each phase of reaction products of several types of palm oils transesterification experiments

Catalyst	Type of feedstock	Percent of residue catalysts by weight of product in each phase (%wt)			
		CG*	CB*	WW*	RB*
KOH	Palm Olein	2.13	0.074	0.028	-
	Palm Stearin	2.12	0.073	0.028	-
	Used Palm Olein	2.43	0.127	0.032	-
H_2SO_4	Palm Olein	5.30	0.098	0.075	-
	Palm Stearin	5.24	0.098	0.075	-
	Used Palm Olein	3.35	0.098	0.075	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Experimental results show that the distribution of residue catalysts in each phase of reaction products was similar when using several types of palm oils feedstocks. Percentage of residue catalysts was calculated by weight percent of product in each phase. Residue catalysts were dissolved in each phase of reaction products. It is found that amounts of residue catalysts in crude glycerol phase higher than in crude biodiesel phase because residue catalysts are good dissolved in the polar chemical compounds. It can be explained that crude glycerol phase contains the polar chemical compounds. Methanol and glycerol are the polar chemical compounds but biodiesel is non polar. The percentage of methanol was shown in Table C-5 to Table C-8 in Appendix C. Amount of residue catalysts were not presented in refined biodiesel because they were eliminated by washing with water.

Table 4.13 Percentage of soaps in each phase of reaction products of several types of palm oils experiments

Catalyst	Type of feedstock	Percent of soaps by weight of product in each phase (%wt)			
		CG*	CB*	WW*	RB*
KOH	Palm Olein	5.34	0.060	0.098	-
	Palm Stearin	5.34	0.060	0.098	-
	Used Palm Olein	8.90	0.089	0.106	-
H_2SO_4	Palm Olein	-	-	-	-
	Palm Stearin	-	-	-	-
	Used Palm Olein	-	-	-	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Experimental results show that soaps were dissolved in each phase of reaction products. Percentage of soaps was calculated by weight percent of product in each phase. The results show that amount of soaps in reaction products when using used palm olein are higher than other type of palm oils. This indicates that used palm olein contains a high value of free fatty acids. Soaps were produced from the sponification reaction. Section 2.4 in CHAPTER II which presents details of saponification reaction, the reaction of soap from free fatty acids was showed in Equation 2.3. Free fatty acids were reacted with basic catalyst converted to soap and water. If water presents, the mixture of ester reacted with basic catalyst converted to soap as Equation 2.4. Soaps of saturated fatty acids tended to solidify at ambient temperatures so a reaction. This indicates that the types of feedstocks affected with amount of soap because different type of feedstocks have different amount of free fatty acids. Soaps were not presented in acid catalyzed process. It is found that amounts of soaps in crude glycerol phase higher than in crude biodiesel phase. The distribution of soaps and residue catalysts in reaction products was similar. Amount of soaps was not presented in refined biodiesel because they were eliminated by washing with water

The results of this section can concluded that type of feedstocks affected with amount of soaps. Amount of soaps of used palm olein feedstock were higher than amount of soaps from other type of feedstocks. Several types of feedstock give the same results on the amount of residue catalysts. Most of residue catalysts and soaps were dissolved in crude glycerol phase.

4.5 Comparison percentage of residue catalysts and soaps in reaction products from industrial scale and reaction products from laboratory scale

This section is conducted to study the comparison of waste of process from industrial scale and process from laboratory scale. The product samples were received from biodiesel industry. The samples from industry were investigated as same as the sample from laboratory experiments. Samples were taken from both phases and were kept for analysis to determine the amount of residue catalysts, soaps, and methanol. Biodiesel phase was further washed with water to remove its contamination. Washed water was also analyzed. The results of samples from industry were comparison with result of the laboratory experiments. This section is divided into two groups following by source of industrial sample.

4.5.1 Sample of "Verasuwan" biodiesel industry

The first group is conducted to study the comparison of waste of product samples from "Verasuwan" biodiesel industry and product samples of laboratory experiments. The process was used the transesterification reaction of palm stearin with methanol using sodium hydroxide as a catalyst. The amount of catalyst was approximate at 0.35% by weight of oil. The reaction was operated at 65°C of reaction temperature and one hour of the reaction time. 4.5:1 molar ratio of methanol to oil was used. The laboratory experiments were described at the same conditions. Samples of product from industrial process were taken at 500 grams of crude biodiesel and 100 grams of crude glycerol. The results are shown in Table 4.14 and Table 4.15.

Table 4.14 Comparison percentage of residue catalysts in reaction products from industrial scale process and laboratory scale process

Scale of process	Percent of residue catalysts by weight of product in each phase (%wt)			
	CG*	CB*	WW*	RB*
Industrial scale	1.85	0.071	0.029	-
Industrial scale	1.82	0.070	0.029	-
Industrial scale	1.83	0.070	0.029	-
Lab. scale	1.71	0.069	0.029	-
Lab. Scale (repeat1)	1.72	0.069	0.029	-
Lab. Scale (repeat2)	1.72	0.068	0.029	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Table 4.15 Comparison percentage of soaps in reaction products from industrial scale process and laboratory scale process

Scale of process	Percent of soaps by weight of product in each phase (%wt)			
	CG*	CB*	WW*	RB*
Industrial scale	5.14	0.057	0.077	-
Industrial scale	5.14	0.057	0.077	-
Industrial scale	5.14	0.057	0.077	-
Lab. scale	4.57	0.056	0.076	-
Lab. Scale (repeat1)	4.57	0.056	0.075	-
Lab. Scale (repeat2)	4.57	0.056	0.075	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Table 4.14 and Table 4.15 show the concentration of residue catalysts and soaps in each phase of reaction products of product samples from biodiesel industry and products of laboratory experiments. The results show that residue catalysts and soaps were dissolved in each phase of reaction products. Percentage of residue catalysts and soaps was calculated by weight percent of product samples in each phase. Percentage of residue catalysts and soaps in each phase of products from industrial scale and laboratory scale was similar. This indicates that the scale of process does not have effect on the distribution of waste. Example, the percentage of waste from laboratory experiment were about 10% when scale up the process into industrial scale the percentage of waste were about 10% as same as the laboratory experiment.

4.5.2 Sample of "Patum Vegetable Oil Co, Ltd" biodiesel industry

The second group is conducted to study the comparison of waste of product samples from "Patum Vegetable Oil Co, Ltd" biodiesel industry and product sample of laboratory experiment. Blended of palm olein and palm stearin and methanol were used as raw material. Sodium methoxide was used as a catalyst. The operating conditions of process from the industry were not known. So the operating conditions of laboratory experiments were assumed as follows. The reaction was operated at 60-65°C of reaction temperature and two hours of reaction time. The molar ratio of methanol to oil of 6:1 was used and amount of catalyst is 0.5% and 1.0% by weight of oil. The results are shown in Table 4.16 and Table 4.17.

Table 4.16 Comparison percentage of residue catalysts in reaction products from industrial scale process and laboratory scale process

Scale of process	Amount of Catalyst (g)	Percent of residue catalysts by weight of product in each phase (%wt)			
		CG*	CB*	WW*	RB*
Industrial scale	-	2.26	0.073	0.028	-
Industrial scale	-	2.26	0.073	0.028	-
Lab. Scale	2.514	2.42	0.072	0.028	-
Lab. Scale (repeat1)	2.543	2.44	0.072	0.028	-
Lab. Scale (repeat2)	2.525	2.41	0.072	0.028	-
Lab. Scale	5.018	3.53	0.159	0.029	-
Lab. Scale (repeat1)	5.006	3.53	0.159	0.029	-
Lab. Scale (repeat2)	5.021	3.54	0.159	0.029	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Table 4.17 Comparison percentage of soaps in reaction products from industrial scale process and laboratory scale process

Scale of process	Amount of Catalyst (g)	Percent of soaps by weight of product in each phase (%wt)			
		CG*	CB*	WW*	RB*
Industrial scale	-	5.13	0.057	0.090	-
Industrial scale	-	5.13	0.057	0.090	-
Lab. Scale	2.514	5.27	0.056	0.089	-
Lab. Scale (repeat1)	2.543	5.27	0.056	0.089	-
Lab. Scale (repeat2)	2.525	5.27	0.056	0.090	-
Lab. Scale	5.018	5.71	0.057	0.089	-
Lab. Scale (repeat1)	5.006	5.71	0.057	0.089	-
Lab. Scale (repeat2)	5.021	5.71	0.057	0.089	-

*"*CG = Crude Glycerol, *CB = Crude Biodiesel, *WW = Washed Water, *RB = Refined Biodiesel"*

Table 4.16 and Table 4.17 show the concentration of residue catalysts and soaps in each phase of reaction products on transesterification process of blended of palm olein and palm stearin. The results show that the concentration of residue catalysts and soaps in each phase of product samples from industrial process is similar to the concentration of residue catalysts and soaps in laboratory experiment when 0.5% of catalyst is used. The results of this group and the previous group are similar on the distribution of waste in each phase of reaction products. The results of the distribution of waste were explained in the section 4.2 and section 4.3.

The results of this section show that the scale of process does not have effect on amount and the distribution of waste. The steps in production should be affected with amount of waste more than the scale of process as the biodiesel purification step. Some process was used the purification of product by wash with water while some process using the distillation to purification of products. This indicates that the purification step in process has effect on amount of waste more than scale of process.