CHAPTER IV

RESULTS AND DISCUSSIONS

This chapter presents the experimental results and discussion on continuous demulsification of palm oil from oil – in – water (O/W) emulsion by an electric field. The O/W emulsion was prepared by homogenizing mixtures of crude palm oil (CPO) and water which was contained electrolyte and sodium sulfate (Na_2SO_4). In the experiments, water was used deionize water at pH7. CPO was obtained from palm oil mill in the South of Thailand. The physical properties of CPO are shown in Table 4.1.

Table 4.1 Physical properties of crude palm oil

Physical property	Crude Palm Oil
Molecular weight, (g/mol)	848
Density at 40°C, (g/ml)	0.9155
Viscosity at 40°C, (cP)	36.70
Free fatty acids, (%wt)	7.32
Acid value, (mg KOH/g)	0.67
Saponification value, (mg KOH/g)	202.00
Solid content, (mg/ml)	0.013
pH	4
Color	Red-orange

The color of crude palm oil is dark red – orange. Crude palm oil has high contents of natural compositions of acidity, solid, fatty acid and gum. At a room temperature, generally, there are two phases of crude palm oil because a fatty acid was become a solid phase under a room temperature.

The demulsification was studied in batch and continuous systems. The emulsions were prepared at concentration of CPO in emulsion of 2 %wt and addition with sodium sulfate to a final concentration of 3 mmol/l. The vessel was made by an acrylic in a size of 3.0 cm x 8.0 cm x 2.5 cm and stainless steel plate electrodes in a size of 2.5 cm x 12 cm. The emulsion was poured into the vessel in the batch system and was flown pass through the vessel by a micro pump in the continuous system. Samples were taken at a specific time and oil content was analyzed. The results of each experiment were summarized in Appendix C. The percentage of oil content in oil – in – water emulsion in both batch and continuous systems was calculated by Equation 4.1.

$$\text{Percentage of oil content} = \frac{W_o}{W_s} \times 100 \qquad (4.1)$$

Where W_0 is weight of oil in sample

W_s is weight of sample

Experimental results and discussion in this study are categorized into six sections as follows:

4.1 Gravity separation of palm oil from oil – in – water emulsion in the batch system.

4.2 Effect of concentration on demulsification of palm oil – in – water emulsion.

4.3 Gravity separation of palm oil from oil – in – water emulsion by continuous flowing through an empty vessel.

4.4 Effect of electric field on demulsification of palm oil emulsion.

4.5 Effect of voltage on continuous demulsification of palm oil emulsion by electric field.

4.6 Effect of flow on continuous demulsification of palm oil emulsion by electric field.

4.1. Gravity separation of palm oil from oil – in – water emulsion

A set of experiments was conducted in order to study the effect of time on demulsification of CPO in the O/W emulsion by gravity force in batch system. The emulsion was poured to the vessel. Samples were taken from the bottom of the vessel every 3 minutes and oil content was analyzed. The percentage of oil content in O/W emulsion was calculated by Equation 4.1. The results of this experiment are shown in Appendix C.1, and the summary of the percentage of oil content is shown in Table 4.2.

Table 4.2 The percentage of palm oil content in the bottom of vessel in demulsification by gravity separation.

Time (min)	**% Oil Content**
0	2.000
3	1.897
6	1.910
9	1.927
12	1.909
15	1.917
18	1.906
21	1.830
24	1.685
27	1.598
30	1.497

The results show that the percentages of oil content in the bottom of vessel were decreased with the increasing time. The percentage of oil content was 0.769 at the retention time 580 minutes. Figure 4.1 presents the results from Table 4.2.

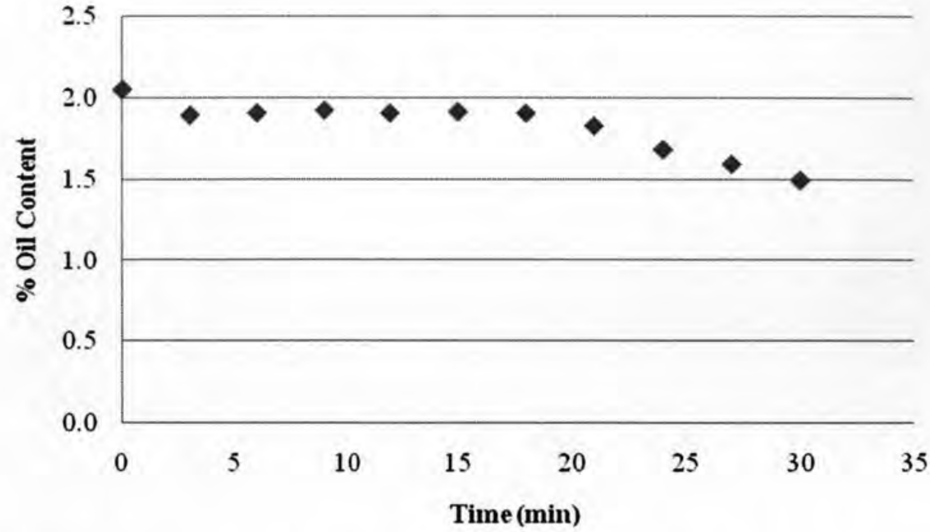

Figure 4.1 The percentage of palm oil content in demulsification oil – in – water emulsion by gravity separation in batch system.

During the experiments, small oil droplets floated to the surface of the emulsion in the vessel. The content of CPO in the emulsion in the bottom of vessel was decreased with time while the CPO content in the top of the vessel was increased with time. The CPO content did not change rapidly. The dispersion of the emulsion was quite stable because of the small sizes of droplets and the production of the interfacial film. The suspended droplets neither settled nor floated rapidly, and the droplets did not coalesce quickly.

Normally, oil droplets can come together in two processes, creaming or sedimentation and coalescence. The different of density between the two liquid phases affect creaming and sedimentation. Sedimentation is the separation process between high density of dispersed droplets and low density of external phase. The droplets will fall to the bottom of the vessel by gravity force. Creaming is the separation process between low density of dispersed droplets and high density of external phase. The droplets will lift to a surface layer by gravity force. The separation by gravity force is called the gravity separation. With regard to coalescence, two or more droplets are collided while moving through the fluid and, then, form a single larger droplet with lower total surface area. In this experiment, the emulsion was oil – in – water emulsion. Oil is in the dispersion phase and water is in the external phase. Oil droplets can come together with creaming and coalescence process.

Amaralikit (2004) studied the gravity separation of palm oil in oil – in – water emulsion. The emulsion was placed in a beaker which was submerged in an oil bath used for the temperature control. Samples were collected every 20 minutes, for the period of 100 minutes, and oil content was analyzed. The results indicated that the amount of palm oil in the emulsion gradually decreased with time. The demulsification of crude palm oil – in – water emulsion by gravity force was a slow process and increase temperature had only a slight effect on the rate of demulsification.

Wiwattanangkul (2005) studied the separation of crude palm oil emulsion by gravity force. The emulsion, 1 %wt of crude palm oil dispersed in deionized water, was put into a beaker and placed on the temperature controlled system at temperature of 60°C, 70°C and 80°C, respectively. Samples were taken every 2 minutes and oil content was analyzed. It is found that the amount of oil in the emulsion gradually decreased with time by gravity force.

Yang (2006) studied the gravity separation of motor oil in oil-in-water emulsion. The reactor was composed of an acrylic plastic with the following dimensions: 16 cm long, 6.7 cm wide and 9.2 cm deep. The effectiveness was determined by the differences in turbidity between treated and untreated emulsions. The turbidity of samples increased when oil content increased. Samples were taken every 5 minutes to determine their turbidity. It is found that turbidity slowly decreased from 1430 to 1315 FAU within 50 minutes. This indicated that the demulsification by gravity force was processed at a slow rate.

Bunyen (2006) studied the separation of CPO and palm olein in oil – in – water emulsion by gravity force. The emulsion was poured to the vessel. Samples were taken every 3 minutes, for the period of 30 minutes. The results indicated that the demulsification of crude palm oil and olein from oil – in – water emulsion by gravity force was processed at a slow rate.

The results are in accordance with that of Amaralikit, Wiwattanangkul and Bunyen. From this experiment, it can conclude that the demulsification by gravity force increased with time and was a slow process.

4.2. The effect of concentration on demulsification of palm oil – in – water emulsion

A set of experiments was conducted in order to study the effect of concentration on demulsification of CPO in the O/W emulsion by gravity force in batch system. This effect was studied by compared between the concentration 1 and 2 %wt of emulsion. The results of demulsification at concentration 1%wt were come from the experiment of Amaralikit (2004). The emulsion was poured to the vessel. Samples were taken from the bottom of the vessel every 20 minutes and oil content was analyzed. The percentage of oil content in O/W emulsion and the percentage of demulsification were calculated by Equation 4.1 and 4.2 respectively. The results of this experiment are shown in Appendix C.1, and the summary of the percentage of oil content is shown in Table 4.3.

$$\text{Percentage of Demulsification} = \frac{(C_0 - C_x)}{C_0} \times 100 \qquad (4.2)$$

Where C_0 is initial oil content and

C_x is oil content at $t = t_x$

Table 4.3 The percentage of oil content and percentage of demulsification of each concentration.

Time (min)	2 %wt of Emulsion		1 %wt of Emulsion	
	% Oil Content	% Demulsification	% Oil Content	% Demulsification
0	2.000	0.000	0.821	0.000
20	1.666	16.705	0.777	5.359
40	1.636	18.210	0.760	7.430
60	1.627	18.655	0.743	9.501
80	1.574	21.280	0.728	11.328
100	1.504	24.815	0.713	13.155

The results show that oil content in the bottom of vessel decreased with time in both concentrations. The demulsification of both concentrations was compared by percentage of demulsification. The percentage of demulsification of concentration at 2 %wt was more than that at 1 %wt. The results agreed with the study conducted by Hosny (1996) who used marine oil was chosen as the oil phase. Their found that increasing oil concentrations enhances the percentage oil removal. The enhancement in oil removal may be due to an increase in the chance of gas bubbles to attach to floating oil drops in the emulsion. The results show that for all the initial oil concentrations; the percentage removal starts to stabilize after specific time. The oil drops inside the emulsion have several sizes, once the largest drops are removed, the efficiency of the process slows down.

Bunyen (2006) studied the separation of CPO and palm olein in oil – in – water emulsion by electric field. The emulsion was poured to the vessel. Samples were taken every 3 minutes. The results indicated that the demulsification of crude palm oil and olein from oil – in – water emulsion by electric field increased when the concentration of emulsion increased.

From this experiment, it can conclude that the demulsification by gravity force increased when the concentration of emulsion increased.

4.3. Separation of palm oil from oil – in – water emulsion by flowing through empty vessel

A set of experiments was conducted in order to study the effect of flow on gravity separation. The volume of vessel was 60.0 ml. In the continuous system, when the emulsion was flown pass through the vessel, the emulsion was to be in vessel with retention time. The oil content from the continuous system should have the same value as that from the batch system with the same retention time. Therefore, the effect of flow on gravity separation was studied by compared oil content from both batch and continuous systems at the same retention time. In the continuous system, the emulsion was flown pass through the vessel by a micro pump at flow rates of 3.0, 5.4, 11.0, 16.0 and 20.0 ml/min. Samples were taken and oil content was analyzed until a steady state. In the batch system, the emulsion was poured into the vessel and samples were taken at a retention time. The oil contents from the batch and continuous systems were compared at the same periods of time. Calculated by Equation 4.3, the retention time of each flow rate is shown in Table 4.4.

$$\tau = \frac{V}{Q} \tag{4.3}$$

Where τ is retention time (min)

Q is volumetric flow rate (ml/min)

V is volume of vessel (ml)

Table 4.4 The retention time of each flow rate

Flow rate (ml/min)	Retention time (min)
3.0	20.00
5.4	11.10
11.0	5.30
16.0	3.45
20.0	3.00

The results of the experiments on the batch and continuous systems are shown in Appendix C.1 and Appendix C.2, respectively. The percentages of oil content of both systems were calculated by Equation 4.1 and the summary of the averages the percentage of oil content is shown in Table 4.5.

Table 4.5 The percentage of oil content of batch and continuous flow systems

Batch System		Continuous System	
Time (min)	% Oil Content	% Oil Content	Flow rate (ml/min)
3.00	1.904	1.869	20.0
3.45	1.896	1.867	16.0
5.30	1.894	1.834	11.0
11.10	1.828	1.685	5.4
20.00	1.774	1.474	3.0

The results show that the percentages of oil content were decreased with time in both systems. The results from both systems were compared in term of a retention time. Retention time increased while flow rates decreased. The results are plotted and shown in Figure 4.2.

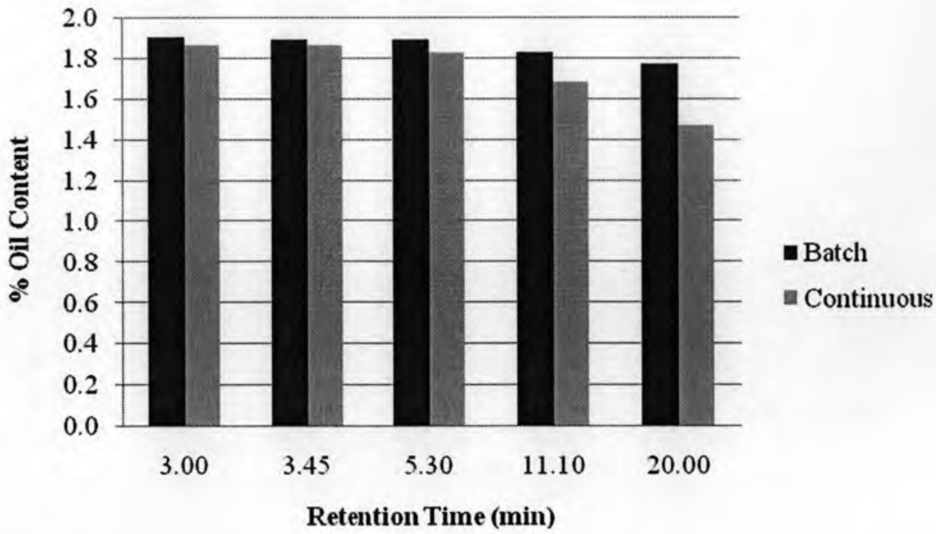

Figure 4.2 The percentage of oil content in bottom of vessel in demulsification by gravity separation of batch and continuous systems

Figure 4.2 shows the percentage of oil content by gravity force in batch and continuous flow systems as a function of time. Normally, the percentages of oil content in both batch and continuous systems should be equal at the same period of retention time. However, the experimential results from the continuous system were higher than those from the batch system at the same period of retention time. This is because there are higher possiblity of collision between droplets in the continuous system. Flows can be divided into two types, the laminar flow and the turbulence flow. In case of laminar flow, fluids tend to flow without lateral mixing and adjacent layers slide past one another at low velocities. In case of turbulence flow, fluids lead to lateral mixing at high velocities. Types of flows can classified by the Reynolds number. The Reynolds number of each flow rate was calculated by Equation 4.4 and the results were described in Table 4.6.

$$Re = \frac{\rho v D}{\mu} \qquad (4.4)$$

Where Re is Reynolds number.
ρ is density of liquid.
υ is velocity of liquid.
D is diameter of tube.
μ is viscosity of liquid.

Table 4.6 The Reynolds number of each flow rate

Flow rate (ml/min)	Reynolds number
3.0	109.01
5.4	196.23
11.0	399.72
16.0	581.41
20.0	726.76

Flow in this experiment was laminar because the Reynolds number was less than 2,100. The length of the vessel was 8.0 cm and end with plate. When the fluid reached the plate, the fluid would turn around. Therefore, possiblity of collision between droplets increased and finally coalesced.

Wiwattanangkul (2006) studied the effect of flow on the demulsification of CPO in oil – in – water emulsion, which was pumped into the column at flow velocities of 0.75, 1.50 and 2.25 mm/s and temperatures of 60, 70 and 80°C. Column heights were set at 100, 300 and 500 mm. The emulsion was allowed to flow through the column until a steady state and sample were taken. The results indicate that removal of palm oil emulsion was enchanced by flowing the emulsion through empty column.

The results from the experiment is in accordance with those from Wiwattanangkul's experinent. From this experiment, it can conclude that the demulsification by gravity force in the continuous system is much more easily than that in the batch system. The oil content decreased when the retention time increased or flow rates decreased.

4.4. Effect of electric field on demulsification of palm oil from oil – in – water emulsion.

In this section, the effect of the electric field on demulsification of CPO from O/W emulsion in both batch and continuous systems was studied. The percentages of oil content between the gravity force and the electric field were compared. The experiment of demulsification by electric field was applied the electric at intensity of electric field of 4 V/cm. The emulsion was poured to the vessel and sample was taken at 20 minutes in case of the batch system. In the continuous system, the emulsion was flown pass through the vessel at flow rate of 3 ml/min. The results of this experiment are shown in Appendix C.2, and the summary of average percentage of oil content is shown in Table 4.7.

Table 4.7 The percentage of oil content in demulsification by gravity force and electric field in batch and continuous systems

Percentage of Oil Content					
Batch System			Continuous System		
Time (min)	Gravity Force	Electric Field	Gravity Force	Electric Field	Flow Rate (ml/min)
20.00	1.774	0.243	1.474	0.133	3.00

The results show that the percentage of oil content in the bottom of the vessel in case of the electric field was less than that in the case of without electric field for both systems. Because the electric field distorts oil droplets and attracts them to anode, then, high concentration of oil droplets was found near anode. Therefore, coalescence can happen more easily and will float upwards to the top of the vessel. Oil content in the top layer of the vessel increased with time as oil droplets lifted upwards and oil content in the bottom decreased with time.

Elektorowicz et al. (2005) studied the effect of the electric field on demulsification. Emulsion was used oily sludge in petroleum refineries and petrochemical industries. The rectangular polyethylene cell and stainless steel material was used to build flat anodes and cathodes. The behaviors of emulsion under the influence of DC field were compared the case with no connection to DC current.

After the electric field was applied, the demulsification immediately took place over the space between electrodes, but not close to the electrodes. Large oil droplets generated by the coalescence of emulsion droplets went up vigorously into the oil layer, so that the emulsion appeared like boiling water during the demulsification. The application of the electric field increased the demulsification.

Yang (2006) studied the effect of electric field on demulsification of motor oil from oil – in – water emulsion. Synthetic oil – water emulsion was prepared by mixing motor oil with water, and a trace amount of sodium chloride was added to provide conductivity. Current 1 A passed through the cell and samples were taken from the cell along the treatment timeline in order to determine the turbidity. The results show that the turbidity dropped from 1380 to 160 FAU in a treatment time of 6 min.

Bunyen (2006) studied the effect of the electric field on demulsification of crude palm oil and olein from oil – in – water emulsion. The emulsion was filled to the vessel and the electric field was applied at intensity of the internal electric field 2 V/cm. Samples were taken every 3 minutes. The results show that oil was increasingly separated from the emulsion under the electric field. After the electric field was applied, the demulsification was immediately observed and it was found that the demulsification by electric field was better than that by gravity force.

It can conclude that the efficiency of the demulsification by low electric fields was higher than that by gravity force.

4.5. Effect of voltage on demulsification of palm oil emulsion by electric field

A set of experiments was conducted to study the effect of voltage on demulsification of CPO from O/W emulsion in both batch and continuous systems by the electric field. In case of the batch system, the electric field was applied to electrodes at intensity of electric field of 4, 6, 8 and 10 V/cm. Samples were taken at 20 minutes and oil content was analyzed. In case of the continuous system, the emulsion was flown pass through the vessel by a micro pump at flow rate 3 ml/min and the electric field was applied to electrodes at 2, 4, 6, 8 and 10 V/cm. Samples were taken every 7 minutes until a steady state and oil content was analyzed. The percentages of oil content in both the batch and continuous systems were calculated by Equation 4.1. The results of this experiment are shown in Appendix C.3 and the summary of average percentages of oil content is shown in Table 4.8.

Table 4.8 The percentages of oil content in batch and continuous demulsification at different voltage

Voltage Between Electrodes (V/cm.)	% Oil Content	
	Batch System	Continuous system
2	-	1.236
4	0.243	0.133
6	0.227	0.230
8	0.207	0.205
10	0.191	0.196

The results show that the percentages of oil content decreased while voltages between electrodes increased, in case of both systems. But in the continuous system, at the voltage between electrodes of 4 V/cm, oil content was the lowest value.

The results in the batch system are similar with that of Bunyen's study (2006). The effect of voltage on demulsification of crude palm oil and palm olein in oil – in – water emulsion in batch system was studied. The intensities of the electric field were between 2 and 10 V/cm. The results showed that the demulsification increased with time. The application of the electric field significantly increased the demulsification of oil from the emulsion while voltages between electrodes increased. When the electric field was applied at voltages between electrodes of more than 6 V/cm, the demulsification slightly change or was almost the same value.

The results in the continuous system are plotted and shown in Figure 4.3.

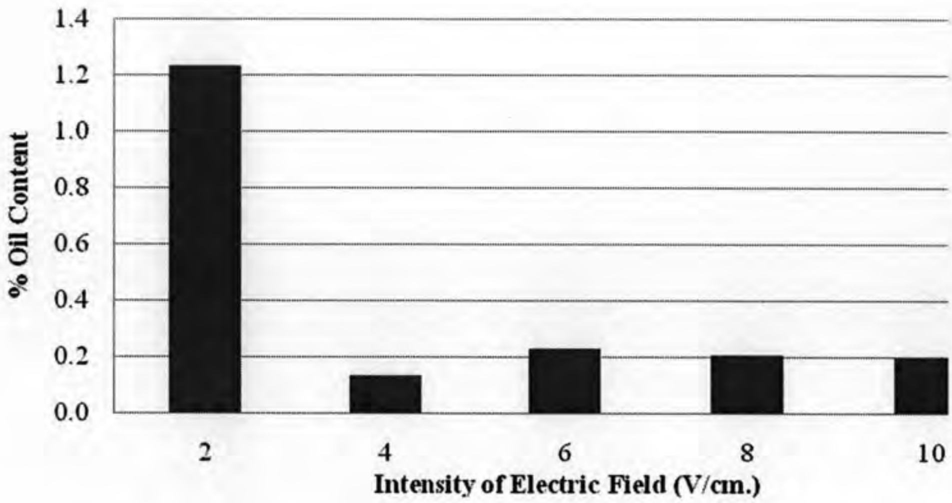

Figure 4.3 The percentage of oil content of demulsification with flow rate 3 ml/min at different voltage in continuous system

In case of the continuous system, the percentages of oil content significantly decreased when voltages between electrodes increased. The lowest percentages were at 4 V/cm. However, when voltages between electrodes were higher than 4 V/cm, the percentages of oil content were close to the same value.

The water molecules are broken down into hydrogen and oxygen gases when direct current is applied to water through a pair of electrodes. The reaction is observed through the hydrogen bubbles that evolve from the cathode and oxygen bubbles that originate from the anode. During the experiment in the event that voltages between electrodes were more than 4 V/cm, bubble floated near electrodes. The bubble was obstructed the droplets moved to anode and, then, the concentration oil droplets near anode decreased. Therefore, the possibility of collision between droplets decreased. The content in the bottom of the vessel was more than 4 V/cm and slightly changed in case of 6, 8 and 10 V/cm.

This result is similar with that of Yang's study (2006). Synthetic oily water was prepared from motor oil for studying the demulsification by the electric field. The emulsion was pumped into the reactor and the current was applied to the cell at 1 and 2 A. Samples were taken from the cell along the treatment to determine the turbidity. At a steady state, the turbidity of 1 A was closed to that of 2 A or oil contents at 1 A and 2 A were almost equal.

It can conclude that oil content decreased close to one value when voltages between electrodes increased.

4.6. Effect of flow rate on demulsification of palm oil emulsion by electric field.

In this section, the effect of flow rate on demulsification of CPO from O/W emulsion by the electric field was studied by comparing the percentages of oil content between both batch and continuous systems. The effect of flow rate in the continuous system was studied by comparing oil content at different flow rates. From the previous section, the oil content at intensity of electric field of 4 V/cm was less than 10 V/cm in the continuous system. It was not the aspect. In this section was studied in both of intensity of electric field at the different flow rate. In the batch system, the emulsion was poured to the vessel and the electric field was applied at intensity of electric field of 4 and 10 V/cm. Samples were taken at 3.45, 5.3, 11.10 and 20 minutes and oil content was analyzed. In case of the continuous system, the emulsion was passed through the vessel at flow rates of 3, 5.4, 11.0 and 16.0 ml/min and the electric field was applied at intensity of electric field of 4 and 10 V/cm. Samples were taken every 5 minutes until a steady state and oil content was analyzed. The percentages of oil content in both batch and continuous systems were calculated by Equation 4.1. The results of this experiment are shown in Appendix C.3. The summary the average percentages of oil content are shown in Table 4.9.

Table 4.9 The percentage of oil content of demulsification in batch and continuous systems at 4 V/cm and 10 V/cm.

The Percentage of Oil Content					
Batch System			Continuous System		
Retention Time (min)	4 V/cm	10 V/cm	4 V/cm	10 V/cm	Flow Rate (ml/min)
3.45	1.110	0.309	1.561	0.827	16.0
5.30	0.920	0.266	1.216	0.573	11.0
11.10	0.361	0.199	0.534	0.355	5.4
20.00	0.243	0.191	0.133	0.196	3.0

In case of the batch system, the percentages of oil content in the bottom of the vessel decreased with time in both intensity of the electric field. The results are plotted and shown in Figure 4.4.

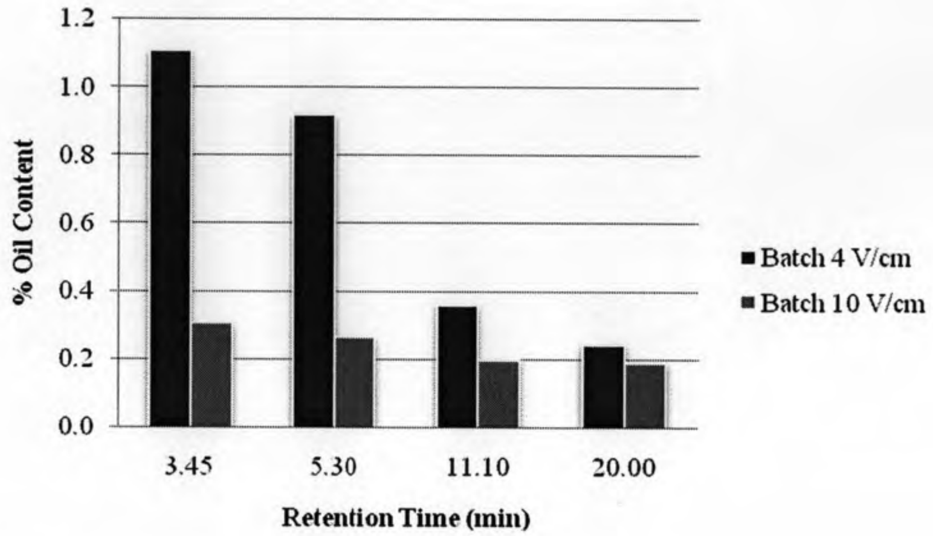

Figure 4.4 The percentage of oil content in batch system at intensity of internal electric field was 4 and 10 V/cm.

Figure 4.4 shows that the demulsification in the batch system of both intensity of the electric field as a function of time. At the same time, the percentage of oil content at 4 V/cm was less than 10 V/cm. This result is similar with the experiment in Section 4.5. The efficiency of the demulsification by the electric field increased with time.

In the continuous system, the percentages of oil content in the bottom of the vessel decreased with time in both intensity of electric field. The results are plotted and shown in Figure 4.5

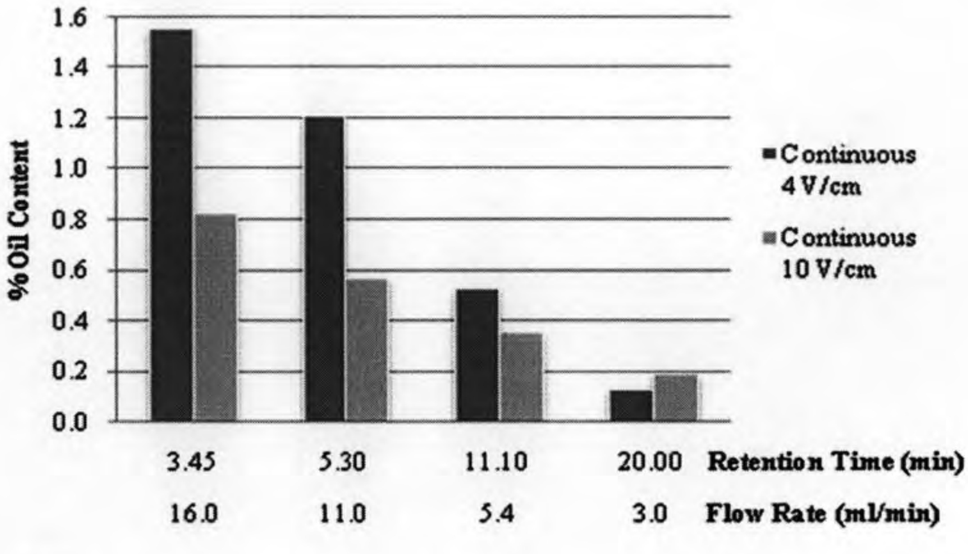

Figure 4.5 The percentage of oil content in continuous demulsification at voltage between electrodes was 4 and 10 V/cm.

Figure 4.5 shows that oil content as a function of time in both intensities. At the same flow rate or retention time, the percentage of oil content at intensity of electric field of 4 V/cm was more than 10 V/cm, except at flow rate of 3 ml/min. At 4 V/cm, oil content was less than 10 V/cm.

The percentages of oil content in both batch and continuous systems were compared at the same periods of time and with voltages between electrode at 4 and 10V/cm. as shown in Figures 4.7 and 4.8, respectively.

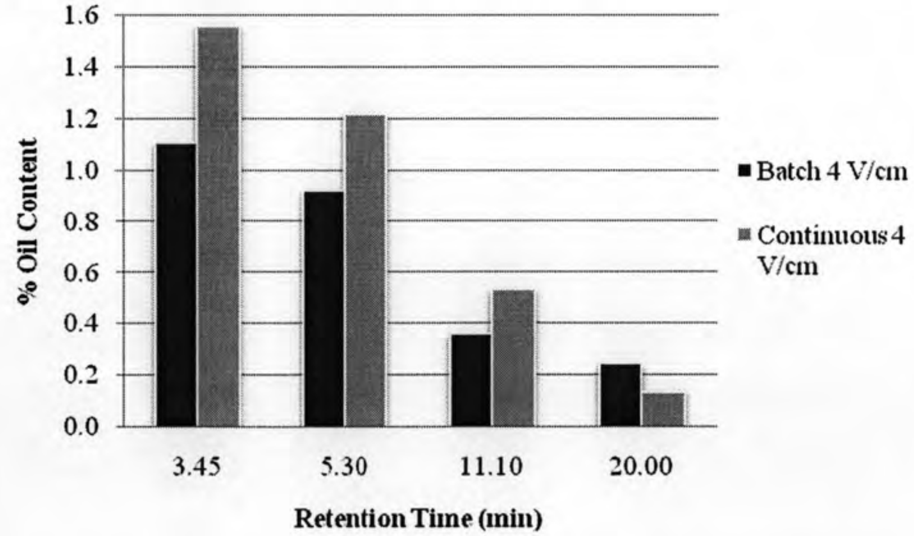

Figure 4.6 The percentage of oil content in batch and continuous system at voltage between electrodes was 4 V/cm

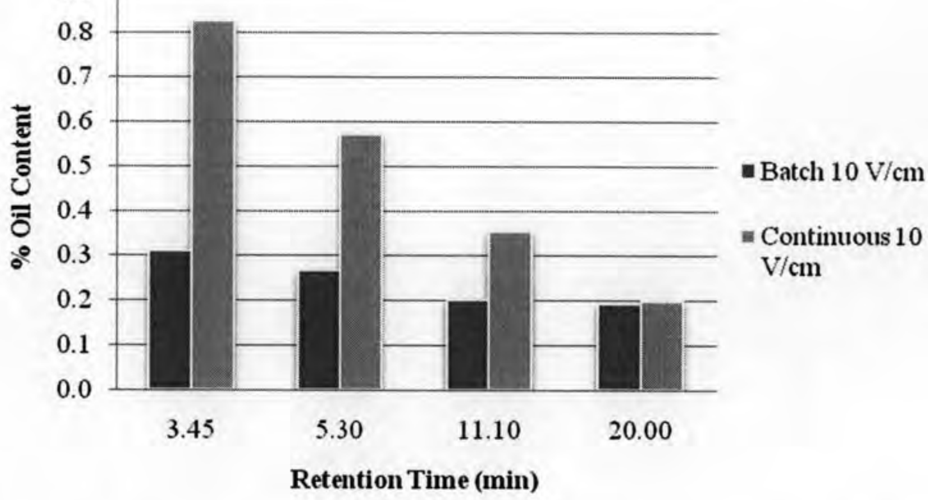

Figure 4.7 The percentage of oil content in batch and continuous system at voltage between electrodes was 10 V/cm

The results show that the percentages of oil content in the batch system were more than those in the continuous system, at the same retention time and flow rate. This result contrasted with the result occurred in Section 4.2. In the continuous system, the emulsion was flown along the vessel while the electric field was attracted the droplet move across the vessel. In my opinion, the movements of the emulsion by flow was obstructed the movements by electric fields. The concentration of oil droplets near anode was less than batch system. Therefore, coalescence can happen much more easily in case of the batch system.

With regard to the study of Elektorowicz (2006), the demulsification of the emulsion which had two movements, vertical and horizontal, was observed within the cells. The breakdown of the colloidal aggregates occurred under the influence of the electric field. This process took place in the vertical direction. A separated liquid phase took place in the horizontal direction.

From the experimental results, it can conclude that the efficiency of the demulsification by the electric field in the batch system is better than that in the continuous system. The demulsification is better when the intensity of the electric field and retention time increases.