

## CHAPTER IV

### RESULTS AND DISCUSSIONS

This chapter presents experimental results and discussions on demulsification of palm oil in oil-in-water emulsion. Experiments conducted in this study are categorized into five sections:

- Section 4.1: Gravity separation of palm oil in oil-in-water emulsion
- Section 4.2: Relative wettability of each medium
- Section 4.3: Effect of coalescing media on demulsification of palm oil
- Section 4.4: Effect of flow velocity on demulsification of palm oil
- Section 4.5: Effect of temperature on demulsification of palm oil

#### 4.1 Gravity Separation of Palm Oil in Oil-in-Water Emulsion

A set of experiments was setup to study the gravity separation of palm oil in oil-in-water emulsion and was conducted at temperatures of 60°C, 70°C, and 80°C. Palm oil in oil-in-water emulsion having oil concentration of 1 wt% was placed in a beaker which was submerged in an oil bath used for temperature control. Samples were consecutively collected every 20 minutes during the period of 100 minutes.

Experimental results are tabulated in Table A1 in Appendix A. The results are averaged and presented in Table 4.1

Table 4.1 Average palm oil content in oil-in-water emulsion from gravity separation study

Time (min)	Oil Content (wt%)		
	60 Celsius	70 Celsius	80 Celsius
0	0.821	0.807	0.816
20	0.777	0.785	0.784
40	0.760	0.736	0.743
60	0.743	0.720	0.733
80	0.728	0.704	0.696
100	0.713	0.694	0.686

It was observed during these experiments that small oil droplets float on the surface of the emulsion in the beaker. These observations indicate that palm oil can separate itself from oil-in-water emulsion. The analysis results in Table 4.1 are plotted and are shown in Figure 4.1. It is clearly seen that amount of palm oil in the emulsion decreases with time. Small droplets of palm oil in the emulsion collide and form larger droplets and separate themselves from the emulsion.

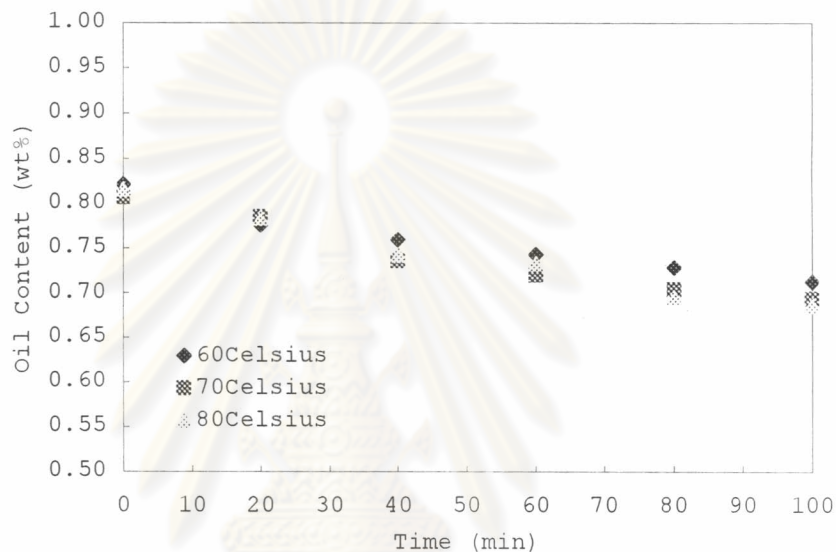


Figure 4.1 Oil content in oil-in-water emulsion

Dispersion of emulsion is stable because of a small droplet size and a presence of an interfacial film on droplets in emulsions. The suspended droplets do not settle out or float rapidly and the droplets do not coalesce quickly. Normally, oil droplets can come together in three different processes: creaming (sedimentation), aggregation, and coalescence. Creaming is the opposite of sedimentation and results from a density difference between the two liquid phases. In aggregation, two or more droplets clump together, touching only at certain points and with virtually no change in total surface area. In coalescence, two or more droplets fuse together to form a single larger unit with a reduced total surface area. The original species lose their identity and become part of new species. Oil droplets in an emulsion will

have some tendency to settle according to Stokes's law shown in Equation 4.1. Uncharged spherical droplets in a fluid will sediment if its density is greater than that of the fluid the driving force is that gravity; the resisting force is viscous and is approximately proportional to the droplet velocity, when the forces are match.

$$v = \frac{2r^2(\rho_2 - \rho_1)g}{9\eta} \quad (4.1)$$

Where

$v$	is velocity of oil droplet
$r$	is radius of oil droplet
$\rho_2$	is density of water
$\rho_1$	is density of oil
$\eta$	is viscosity of oil droplet

Oil with high viscosity has an ability to hold up more water droplets than oil with lower viscosity. The viscosity of oil can be reduced by the application of heat. Lowering the viscosity increases both the rate at which water droplets settle and the mobility of water droplets. This phenomena leads to collisions and coalescence of the droplets which interns increase the rate of separation. In general, the higher the temperature is, the greater the ability to resolve emulsion is.

However, increasing temperature may affect other factors negatively. Knut Gaaseidnes and Joseph Turbeville (1999) found that Stokes' law could not directly be used to calculate the profile of gravity separation of palm oil in oil-in-water emulsion. They indicated that other factors are interfered the separation process.

Borwankar et al. (1992) studied the kinetics of flocculation and coalescence of an emulsion in their experiments. They found that the concentration of the dispersed droplets in the emulsion decreased exponentially with time. Hartland and Vohra (1980) reported similar finding and suggested that the demulsification process proceeds as



$$\frac{C}{C_0} = \exp(-Kt) \quad (4.2)$$

Where  $C$  is the concentration of the emulsion at time  $t$ ,  $C_0$  is the initial concentration of the emulsion and  $K$  is an overall rate constant for demulsification.

Equation 4.2 is used for prediction of separation of palm oil in oil-in-water emulsion in this study. The experimental results from Table 4.1 are calculated in the form of  $C/C_0$ . The results are shown in Table 4.2 and are plotted in Figure 4.2. Equation 4.2 is used for calculation of overall rate of demulsification ( $K$ ) for each temperature. The calculated rates of demulsification ( $K$ ) are also shown in Table 4.2.

Table 4.2 Average value of oil fraction ( $C/C_0$ ) from gravity separation

Time (min)	Oil Fraction ( $C/C_0$ )		
	60 Celsius	70 Celsius	80 Celsius
0	1.000	1.000	1.000
20	0.946	0.973	0.960
40	0.926	0.912	0.910
60	0.906	0.893	0.899
80	0.888	0.873	0.852
100	0.868	0.860	0.841
$K$	0.0015	0.0017	0.0019

The rate of demulsification ( $K$ ) found in these experiments are low and are relatively close to each other. These results indicated that the demulsification of palm oil from oil-in-water emulsion by gravity is a slow process and an increase in demulsification temperature has only a slight effect on the rate of demulsification.

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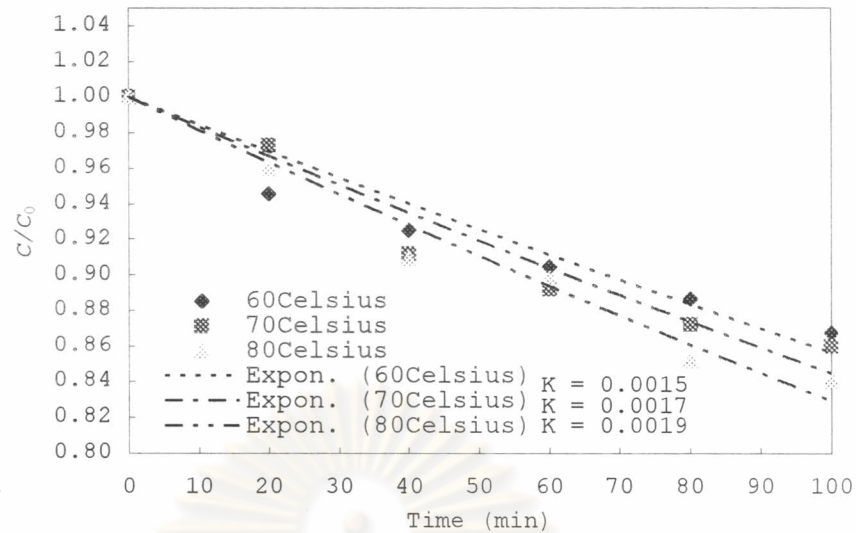


Figure 4.2 Gravity separation of palm oil in oil-in-water emulsion on the separation rate

A set of experiments was also conducted using empty column, without any type of coalescing medium, to verify an effect of empty column on demulsification of palm oil from oil-in-water emulsion at temperatures of 60, 70, and 80 °C. The results are shown in Tables A3 in Appendix A. The experimental results from Table A3 are calculated in the form of  $C/C_0$  and the results are shown in Table 4.3 along with the results from gravity separation experiments. Again Equation 4.2 is used to calculate a rate of demulsification ( $K$ ) and the results are also shown in Table 4.3 and plotted in Figures 4.3 to 4.5.

Table 4.3 Oil fraction and  $K$  value from empty column and gravity separation experiments.

Time (min)	Oil Fraction ( $C/C_0$ )					
	60 Celsius		70 Celsius		80 Celsius	
	Empty Column	Gravity Separation	Empty Column	Gravity Separation	Empty Column	Gravity Separation
0	1.000	1.000	1.000	1.000	1.000	1.000
20	0.931	0.949	0.968	0.966	0.989	0.935
40	0.917	0.939	0.967	0.958	0.987	0.908
60	0.911	0.926	0.924	0.913	0.912	0.905
80	0.897	0.905	0.919	0.886	0.869	0.886
100	0.877	0.898	0.854	0.849	0.861	0.868
$K$	0.0012	0.0015	0.0013	0.0015	0.0015	0.0016

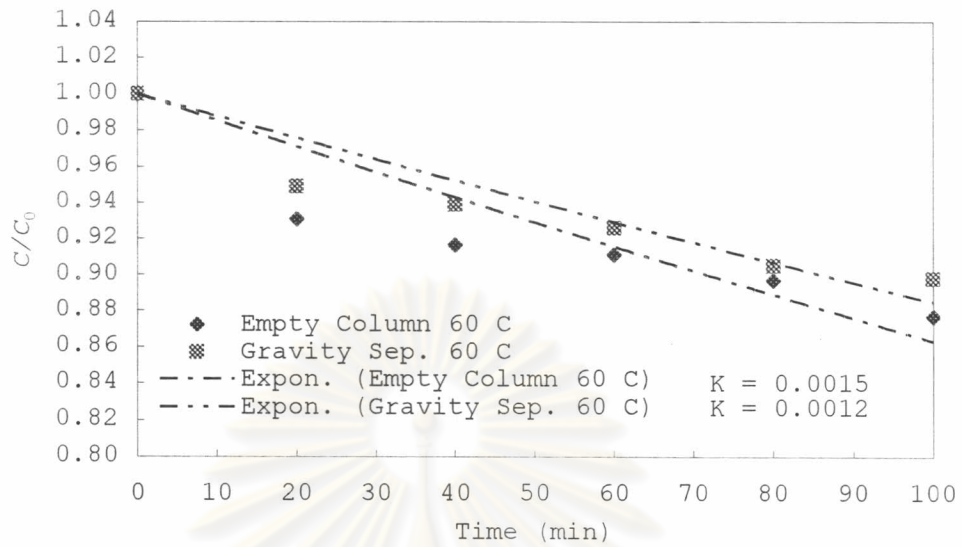


Figure 4.3 Palm oil fractions from empty column experiment and gravity separation experiment at 60 Celsius

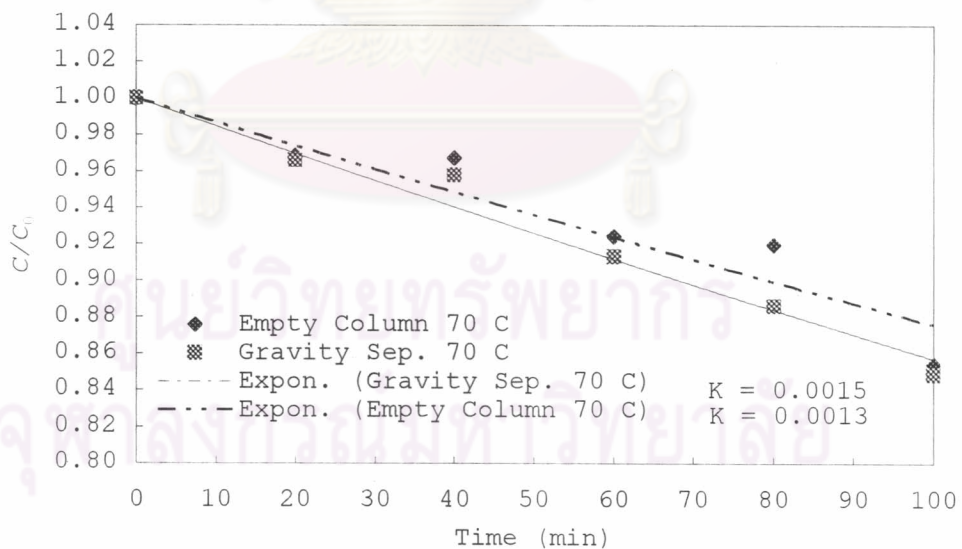


Figure 4.4 Palm oil fractions from empty column experiment and gravity separation experiment at 70 Celsius

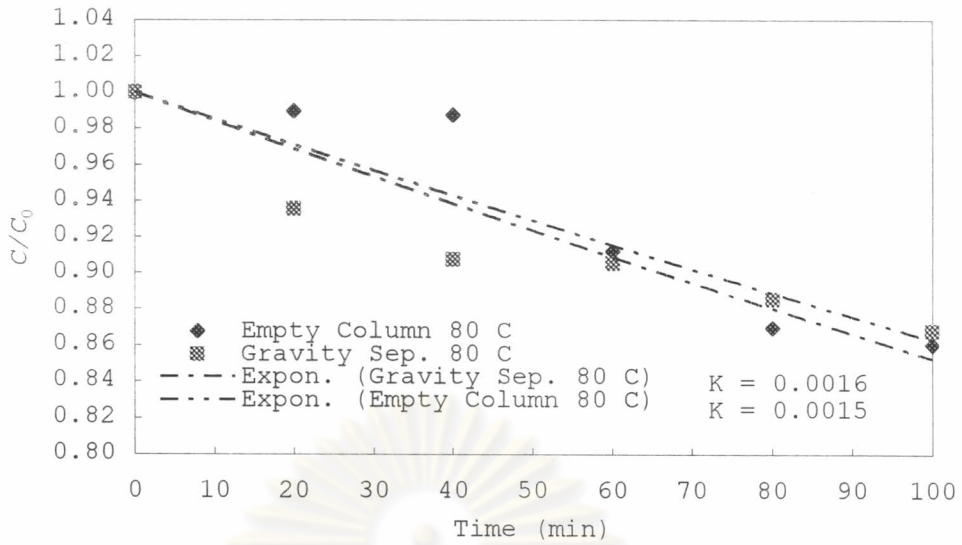


Figure 4.5 Palm oil fractions from empty column experiment and gravity separation experiment at 80Celsius

Figure 4.6 shows comparison of rate of demulsification ( $K$ ) taken from gravity separation study and rate of demulsification ( $K$ ) taken from empty column study. The results from both studies indicate that flowing palm oil emulsion through an empty column do not enhance its separation from the emulsion.

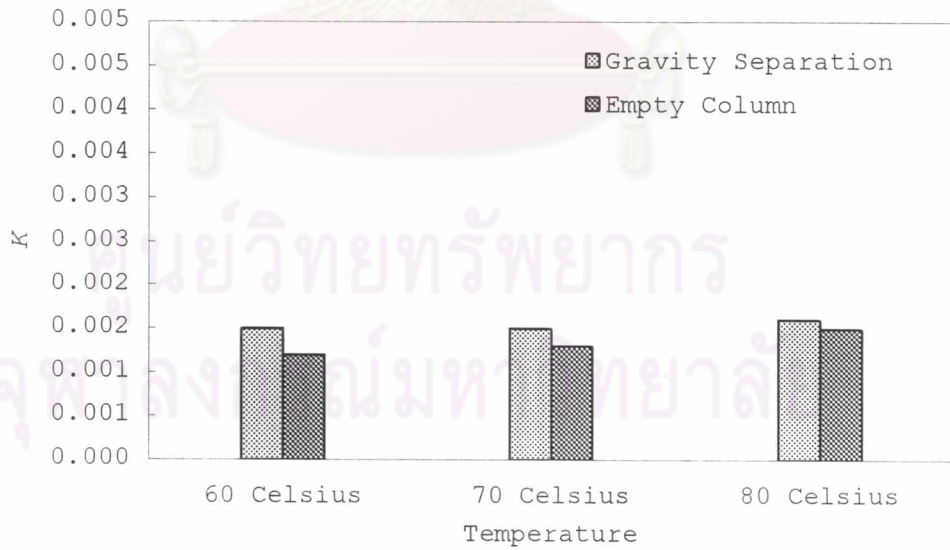


Figure 4.6 Comparison of gravity separation with rate of demulsification ( $K$ ) of empty column separation



#### 4.2 Relative Wettability of Each Media

Coalescing media used in this study are palm fiber, synthetic fiber and pumice stone. Each medium has been tested for its ability to wet water and palm oil at different temperatures. Each medium is weighed to find its dry weight and is put into a beaker containing water or palm oil at specified temperature for a period of time. The medium is then taken out of the beaker and is placed on a screen inside a temperature controlled oven to allow the liquid to separate from the medium. The medium is weighed again. The weight difference is considered to be the amount of liquid wetted on the medium. When the amount of liquid adsorbed on the medium is divided by the dry weight of each medium, the liquid content wetted on each medium at each temperature is obtained. The liquid content can be considered as amount of liquid that can wet the medium surface. The ratio between palm oil content and water content on each medium at each temperature is a relative ability of palm oil and water to wet each medium. Table 4.4 summarizes the results of this study and the relative wettability of each medium is plotted in Figure 4.7. It is observed that palm fiber has a value of relative wettability higher than 1.0 which indicates that palm oil has a higher ability to attract palm oil than water. On a contrary, pumice stone and synthetic fiber are considered to be poor palm oil attraction. This study also shows that temperature slightly affects the wettability of the medium.

Table 4.4 Relative wettability

Medium	$(W_{oil}/W_{medium}) / (W_{water}/W_{medium})$		
	60 Celsius	70 Celsius	80 Celsius
Palm Fiber	1.236	1.193	1.162
Pumice Stone	0.994	0.954	0.690
Synthetic Fiber	0.576	0.376	0.406

Where  $W_{oil}$  is the weight of palm oil on each medium  
 $W_{medium}$  is the weight of each medium  
 $W_{water}$  is the weight of water on each medium



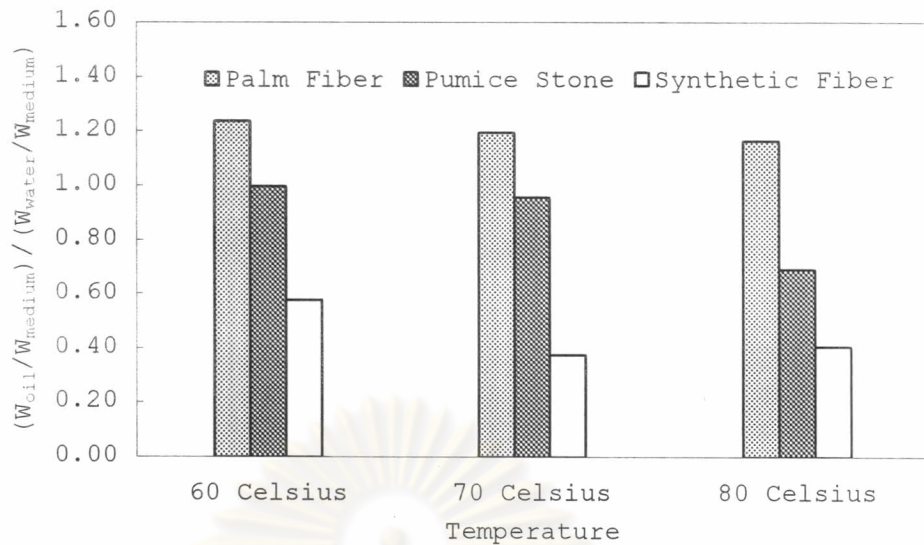


Figure 4.7 Relative wettability

Experiments on demulsification of palm oil emulsion using palm fiber, synthetic fiber and pumice stone as coalescing media were conducted at temperatures of 60 C°, 70 C° and 80 C° and liquid flow velocities of 0.12, 0.25, 0.40 and 0.50 mm/sec. Each medium was packed in a glass column to the height of 100 mm and a packing density of 0.29, 0.11 and 0.41 g/ml for palm fiber, synthetic fiber and pumice stone, respectively. Palm oil emulsion with a concentration of 1 wt% was used as initial stock. Each experiment was conducted for 100 minute and a sample was taken every 20 minutes. The samples were analyzed for palm oil content. The results are summarized in Table A2 in Appendix A.

The results are then calculated as  $C/C_0$  and are summarized in Table 4.5 and plotted in Figures 4.8 to 4.19. Equation 4.2 is again used for calculation of demulsification rate. The demulsification rates ( $K$ ) are summarized in Table 4.6.

Table 4.5 Palm oil fraction of flowing through a packed column experiments

Medium	Temperature	Time (min)	Oil Fraction ( $C/C_0$ )			
			0.12	0.25	0.40	0.50
Palm Fiber	60	0	1.0000	1.0000	1.0000	1.0000
		20	0.8973	0.9274	0.7898	0.6735
		40	0.8448	0.7998	0.5470	0.4909
		60	0.7783	0.6066	0.4495	0.4048
		80	0.6628	0.4883	0.4425	0.3946
	100	0.5683	0.4251	0.3984	0.3844	
	70	0	1.0000	1.0000	1.0000	1.0000
		20	0.7929	0.9482	0.8653	0.7733
		40	0.7835	0.8003	0.7468	0.6537
		60	0.7235	0.6498	0.6702	0.5491
		80	0.6906	0.5310	0.5296	0.4736
	100	0.6788	0.4956	0.4669	0.4786	
	80	0	1.0000	1.0000	1.0000	1.0000
		20	0.9275	0.9225	0.8079	0.8383
		40	0.8709	0.8689	0.7192	0.7408
60		0.7905	0.8308	0.7032	0.6861	
80		0.7510	0.7020	0.7020	0.6694	
100	0.7128	0.6448	0.6983	0.6278		
Synthetic Fiber	60	0	1.0000	1.0000	1.0000	1.0000
		20	0.9478	0.9015	0.9326	0.9426
		40	0.8672	0.8723	0.9196	0.8933
		60	0.8618	0.8552	0.8546	0.8581
		80	0.8161	0.7993	0.7222	0.7421
	100	0.8074	0.7944	0.7009	0.6964	
	70	0	1.0000	1.0000	1.0000	1.0000
		20	0.9825	0.9708	0.9545	0.9539
		40	0.9771	0.9668	0.9201	0.9119
		60	0.9352	0.9017	0.8857	0.8889
		80	0.8704	0.8446	0.7873	0.7590
	100	0.8259	0.8340	0.7534	0.7398	
	80	0	1.0000	1.0000	1.0000	1.0000
		20	0.9846	1.0024	0.9893	0.9582
		40	0.9601	0.9927	0.9797	0.9258
60		0.9550	0.9768	0.9487	0.9107	
80		0.9151	0.9072	0.8842	0.9072	
100	0.8932	0.8486	0.8711	0.8852		
Pumice Stone	60	0	1.0000	1.0000	1.0000	1.0000
		20	0.9472	0.9363	0.9322	0.9718
		40	0.8930	0.8848	0.8725	0.9559
		60	0.8362	0.8358	0.8585	0.8848
		80	0.7305	0.7843	0.8047	0.8297
	100	0.6909	0.7218	0.7848	0.8113	
	70	0	1.0000	1.0000	1.0000	1.0000
		20	0.9324	0.9411	0.9626	0.9836
		40	0.8297	0.8533	0.9185	0.9813
		60	0.7886	0.8044	0.8652	0.9416
		80	0.7669	0.7878	0.8437	0.8575
	100	0.7452	0.7522	0.8154	0.8435	
	80	0	1.0000	1.0000	1.0000	1.0000
		20	0.8772	0.9564	0.9206	0.9534
		40	0.8169	0.9177	0.9042	0.9436
60		0.7741	0.8717	0.8785	0.8909	
80		0.7654	0.8232	0.8715	0.8860	
100	0.7599	0.7990	0.8657	0.8725		

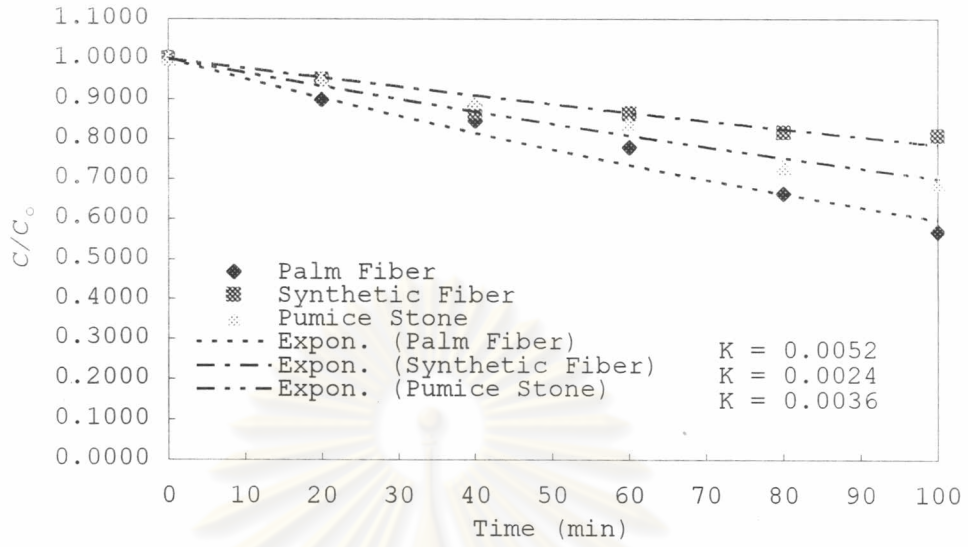


Figure 4.8  $C/C_0$  of Flow velocity 0.12 mm/sec and Temperature 60 °C

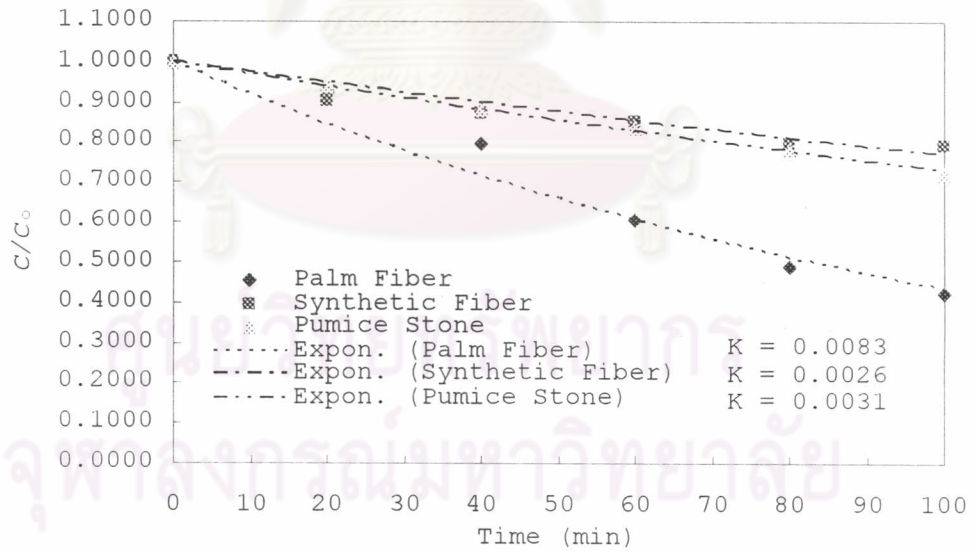


Figure 4.9  $C/C_0$  of Floe Velocity 0.25 mm/sec and temperature 60 °C

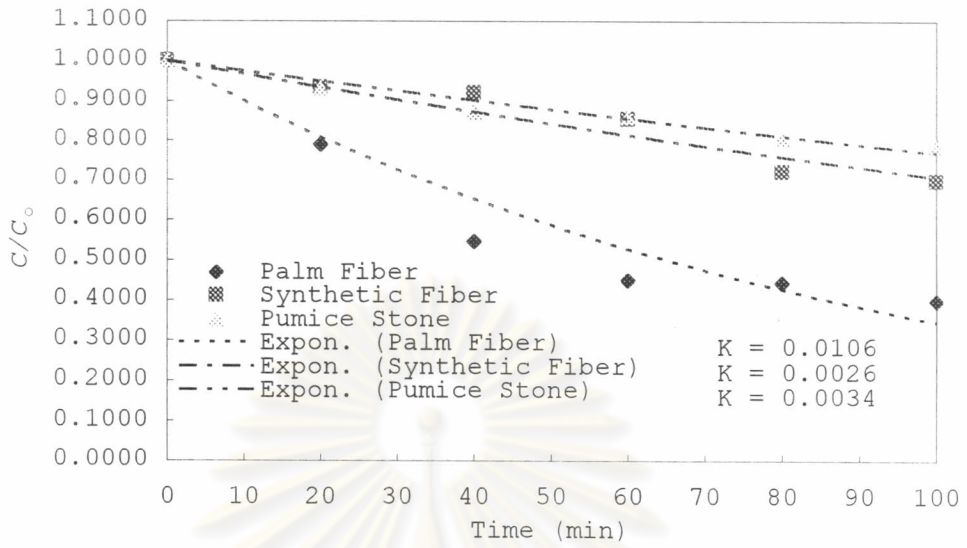


Figure 4.10  $C/C_0$  of Flow Velocity 0.40 mm/sec and Temperature 60 °C

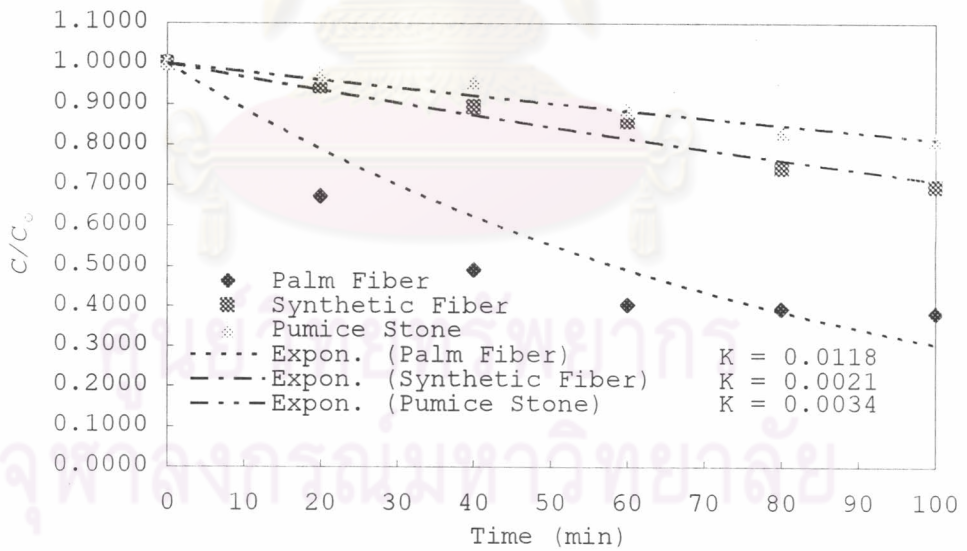


Figure 4.11  $C/C_0$  of Flow Velocity 0.50 mm/sec and Temperature 60 °C



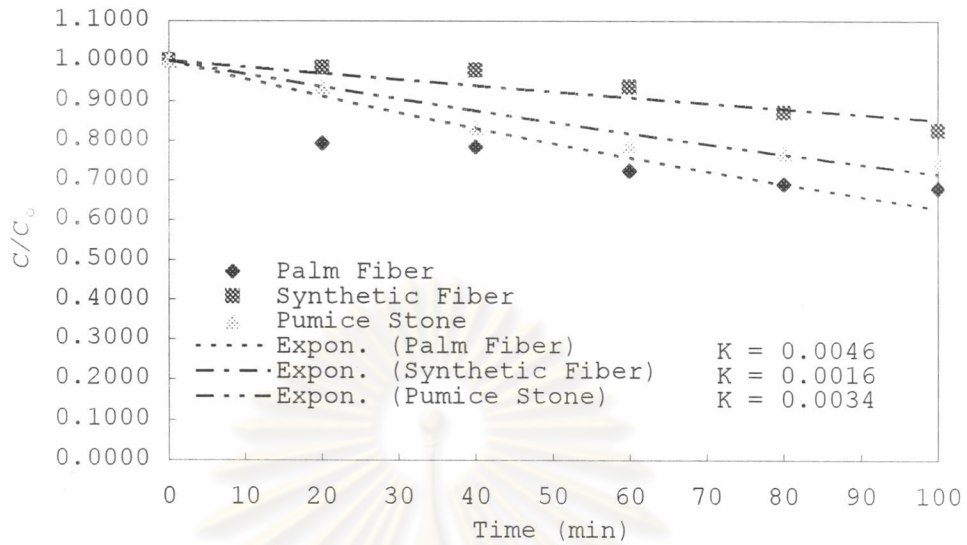


Figure 4.12  $C/C_0$  of Flow Velocity 0.12 mm/sec and Temperature 70 °C

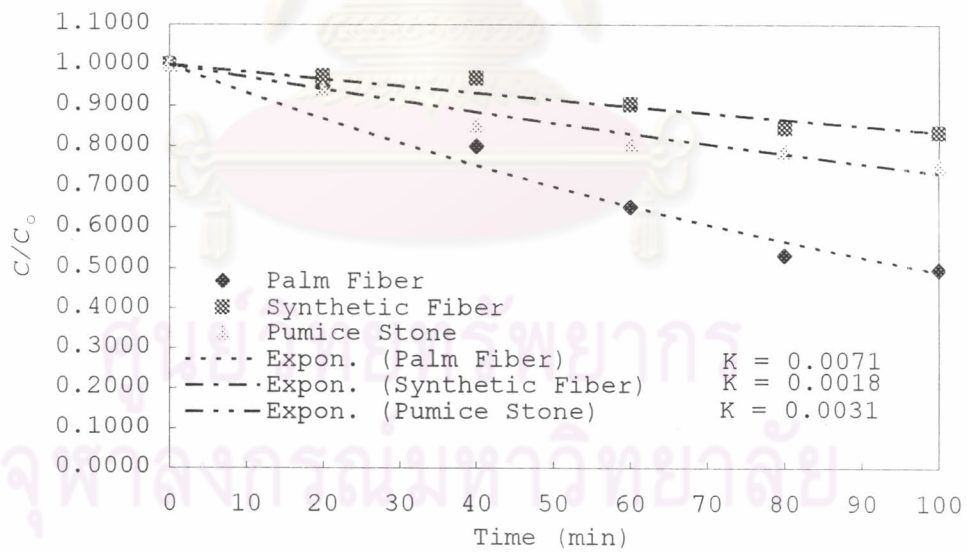


Figure 4.13  $C/C_0$  of Flow velocity 0.25 mm/sec and Temperature 70 °C

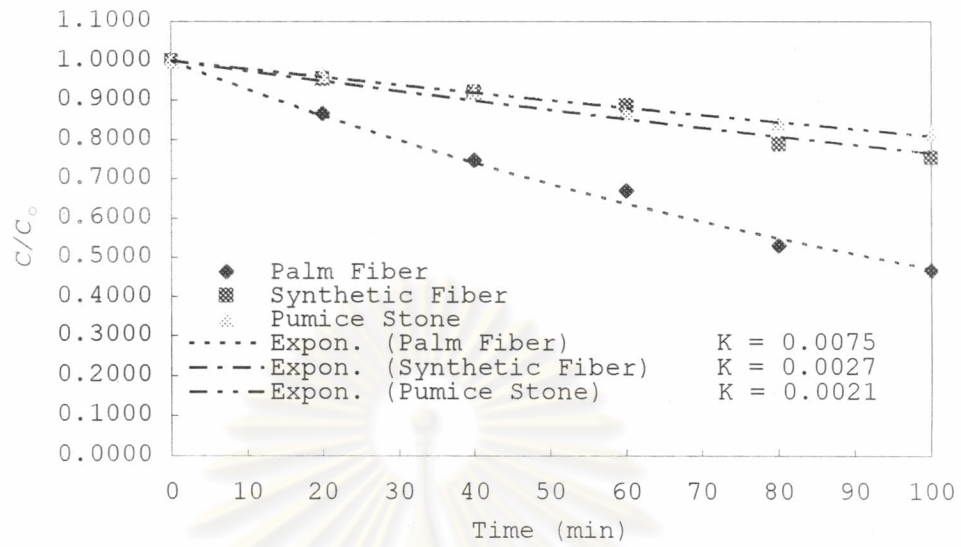


Figure 4.14  $C/C_0$  of Flow velocity 0.40 mm/sec and Temperature 70 °C

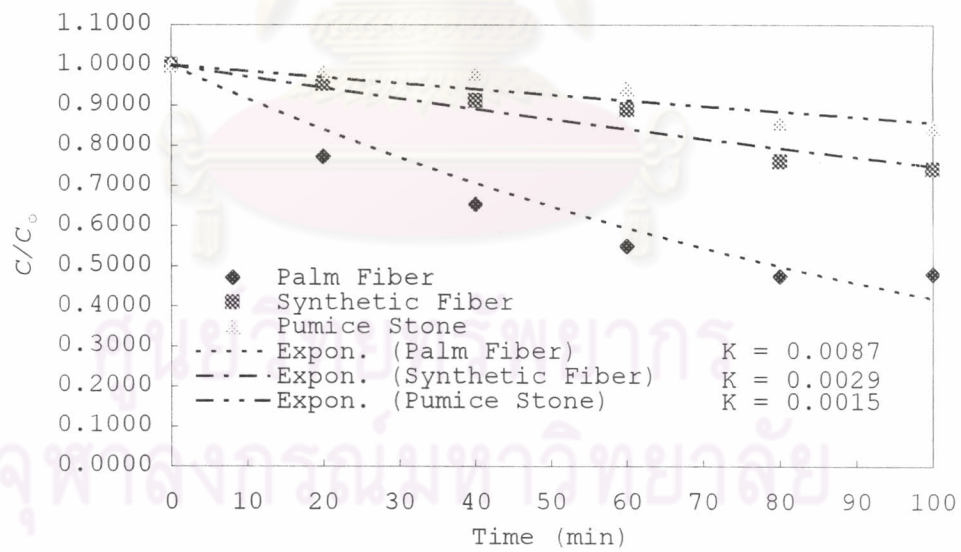


Figure 4.15  $C/C_0$  of Flow velocity 0.50 mm/sec and Temperature 70 °C

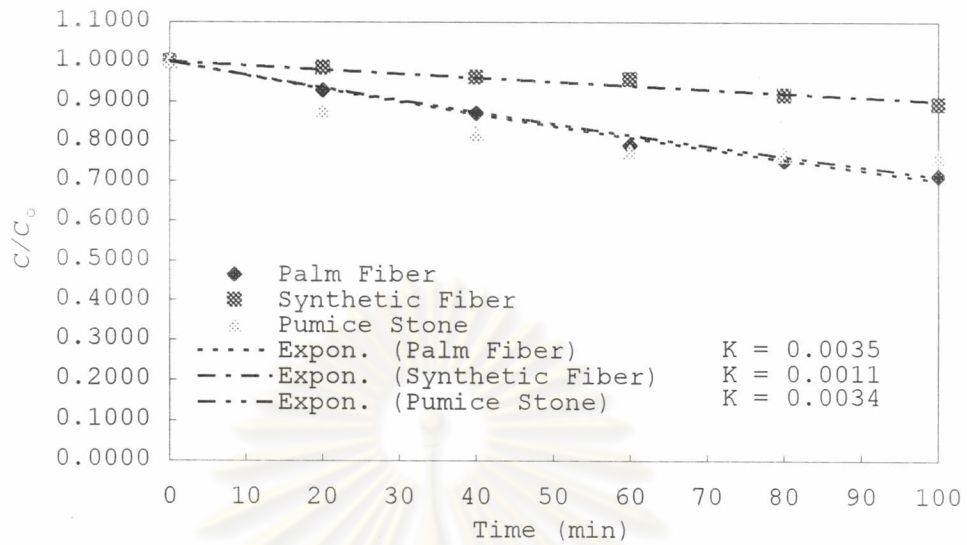


Figure 4.16  $C/C_0$  of Flow velocity 0.12 mm/sec and Temperature 80 °C

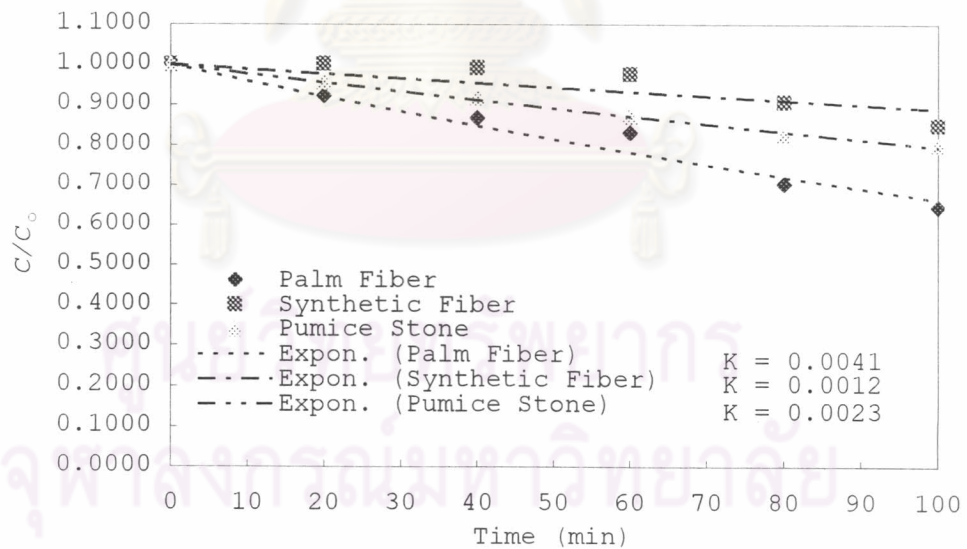


Figure 4.17  $C/C_0$  of Flow velocity 0.25 mm/sec and Temperature 80 °C

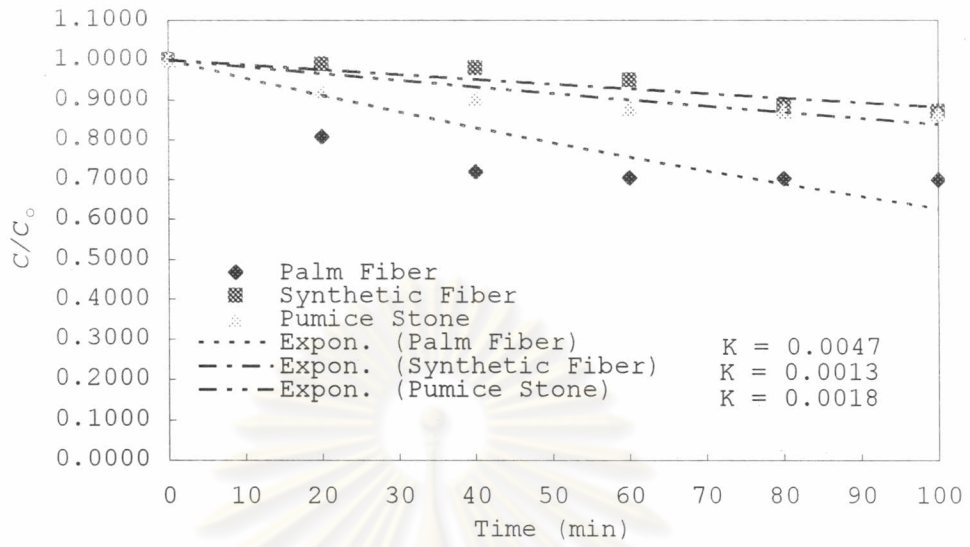


Figure 4.18  $C/C_0$  of Flow velocity 0.40 mm/sec and Temperature 80 °C

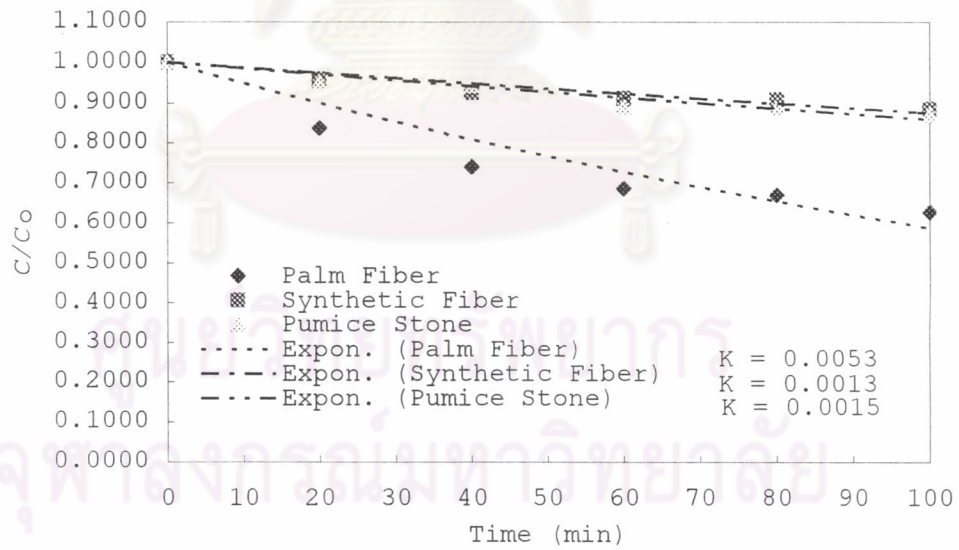


Figure 4.19  $C/C_0$  of Flow velocity 0.50 mm/sec and Temperature 80 °C



Table 4.6 Demulsification rates of flowing through a packed column experiments.

Media	Temperature	Demulsification rate (K)				Gravity Separation
		Flow velocity (mm/sec)				
		0.12	0.25	0.40	0.50	
Palm Fiber	60 °C	0.0052	0.0083	0.0106	0.0118	0.0015
	70 °C	0.0046	0.0071	0.0075	0.0087	0.0015
	80 °C	0.0035	0.0041	0.0047	0.0053	0.0016
Synthetic Fiber	60 °C	0.0024	0.0026	0.0033	0.0035	0.0015
	70 °C	0.0016	0.0018	0.0027	0.0029	0.0015
	80 °C	0.0011	0.0012	0.0013	0.0013	0.0016
Pumice Stone	60 °C	0.0036	0.0031	0.0026	0.0021	0.0015
	70 °C	0.0034	0.0031	0.0021	0.0015	0.0015
	80 °C	0.0034	0.0023	0.0018	0.0015	0.0016

### 4.3 Effect of Coalescing Media on Demulsification of Palm Oil

Figures 4.20 to 4.22 show the comparisons of each coalescing medium against flow velocity at different temperatures. It is clearly observed that palm fiber is a good coalescing medium for demulsification of palm oil. The rate of demulsification of palm fiber is higher than that of gravity demulsification by factors of 2.2 to 7.9. Synthetic fiber and pumice stone show only a slight improvement in rates of demulsification than gravity separation and their rates are many times lower than the rate of palm fiber. The results from previous section also indicate that palm fiber has higher relative wettability than other media.

Media et al. (1976) studied the influence of packing wettability on coalescence by granular packed bed coalescer. They reported that oil removal ability increased with increasing oil wettability of bed material.

Magiera and Blass (1997) conducted experiments to compare many types of fiber in a flow through system. They concluded that highly energetic surface fiber showed higher separation efficiency. Short fiber and closely packed bed provided better separation results than larger fiber and lower packing density bed. Unfortunately, there is no comparison between granular packing bed and fiber packing bed.

In this study, palm fiber is the packing medium which shows very high wettability of palm oil over water. When palm oil-in-water emulsion flows through the packed bed of palm fiber, small droplets of palm oil would easily attached on the surface of the fiber. The attached droplets would then wet from film on the surface of fiber and this film start to grow. A larger droplet resulting from the growth of this film would detach from the surface due to its destructive of the main emulsion flow.

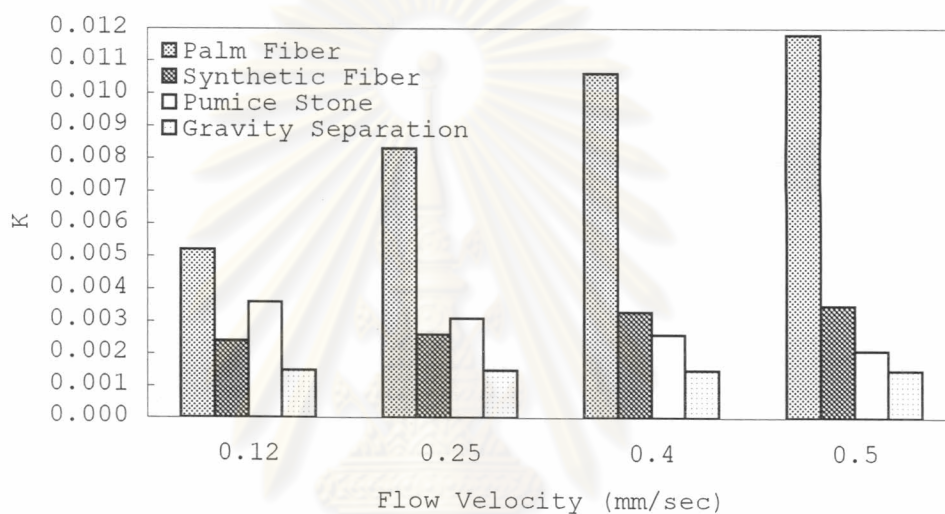


Figure 4.20 Comparisons of each coalescing medium against flow velocity at 60 °C

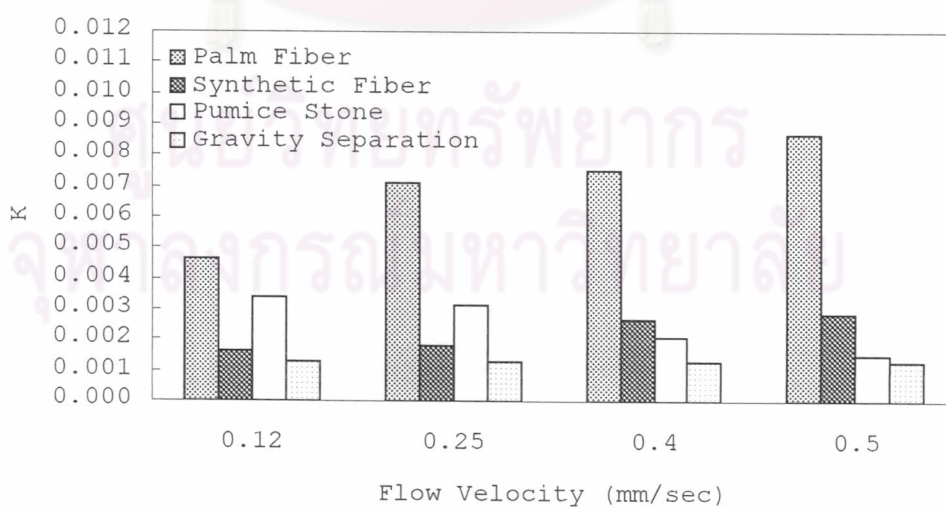


Figure 4.21 Comparisons of each coalescing medium against flow velocity at 70 °C

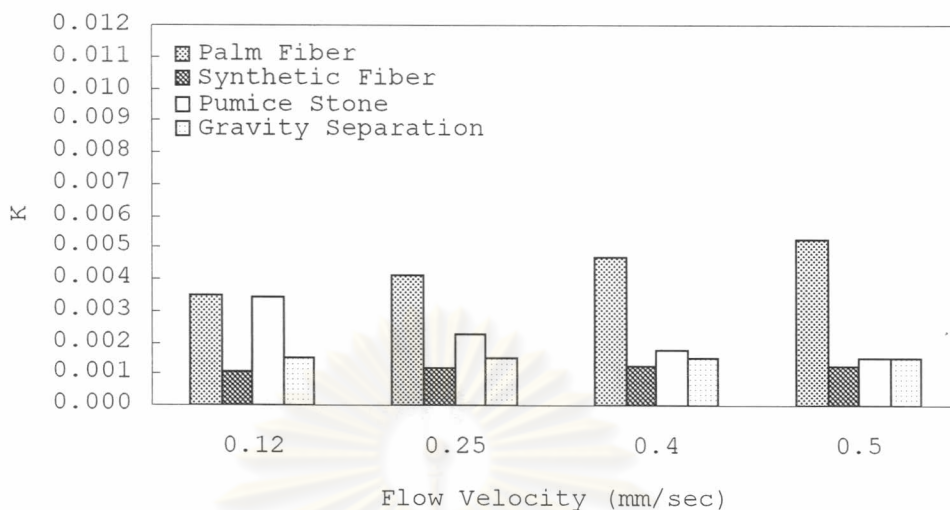


Figure 4.22 Comparisons of each coalescing medium against flow velocity at 80 °C

#### 4.4 Effect of Flow Velocities on Demulsification of Palm Oil

Figures 4.23 to 4.25 show comparison of flow velocity against coalescing media at different temperatures. It is observed that increasing flow velocity of emulsion through palm fiber packed bed increases the rate of demulsification. The improvement is more pronounced at low operating temperature. Increasing flow velocity of emulsion through synthetic fiber packed bed slightly increases the rate of demulsification while a reverse effect is observed on pumice stone.

Hazlett (1969) studied the coalescence phenomena in a fibrous filter bed system. He suggested that the coalescence of water droplets proceeds through three steps: approach of droplets to a fiber, attachment of the droplets and release of enlarged droplets to the downstream side of the filter. Increasing of flow velocity of emulsion through fibrous bed would increase the amount of droplets approaching the fiber which in turn increases the amount of droplets attached on the fiber. The roughness of palm fiber surface helps increasing ability of the droplet to easily attach and collide while the smooth surface of synthetic fiber slightly affects this ability.



A reverse effect observed on pumice stone can be explained using the work conducted by Li and Gu (2003) studied coalescence of oil droplets in oil-in-water emulsions through fibrous and granular beds. They found that the coalescence efficiency is not a monotonous function of flow rate in the granular beds. The coalescence phenomena for the granular beds may not be adequately assessed by using the coalescence phenomena in a fibrous filter bed system.

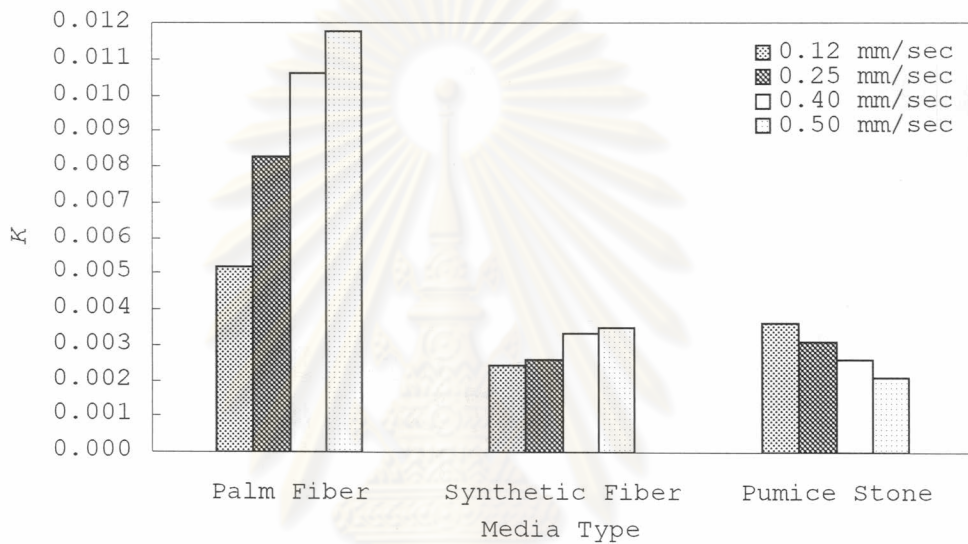


Figure 4.23 Comparisons of each flow velocity against coalescing media at 60 °C

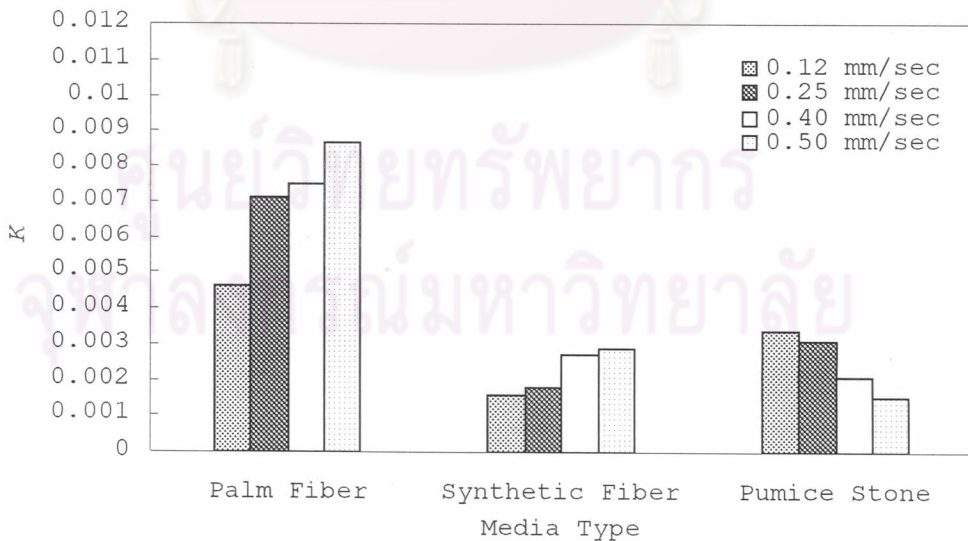


Figure 4.24 Comparisons of each flow velocity against coalescing media at 70 °C



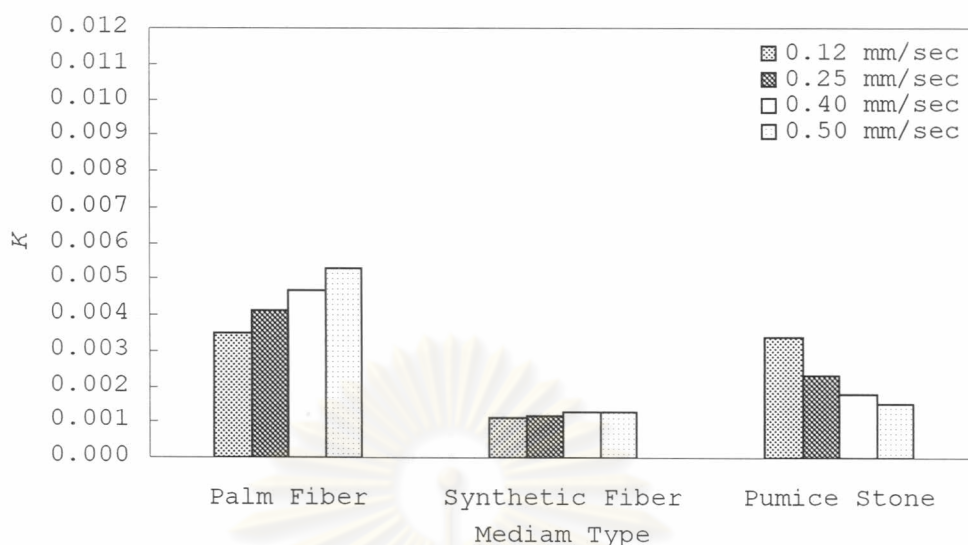


Figure 4.25 Comparisons of each flow velocity against coalescing media at 80 °C

#### 4.5 Effect of Temperatures on Demulsification of Palm Oil

Figures 4.26 to 4.29 show the comparisons of temperature against coalescing media at different flow velocities. It is observed that the rate of demulsification of each coalescing medium decreases with increasing of temperature.

The phenomena can also be explained using Hazlett (1969)'s concept of coalescence. An increase in temperature of the solution would decrease the viscosity and density of droplets on the surface of the media. A decrease in attachment of the droplets would reduce an amount of the enlarge droplet on the surface of the media result in a high number of small droplets remaining in the emulsion. Limited number of researcher are conducted on temperature effect in coalescence of emulsion and the phenomena is still inconclusive.

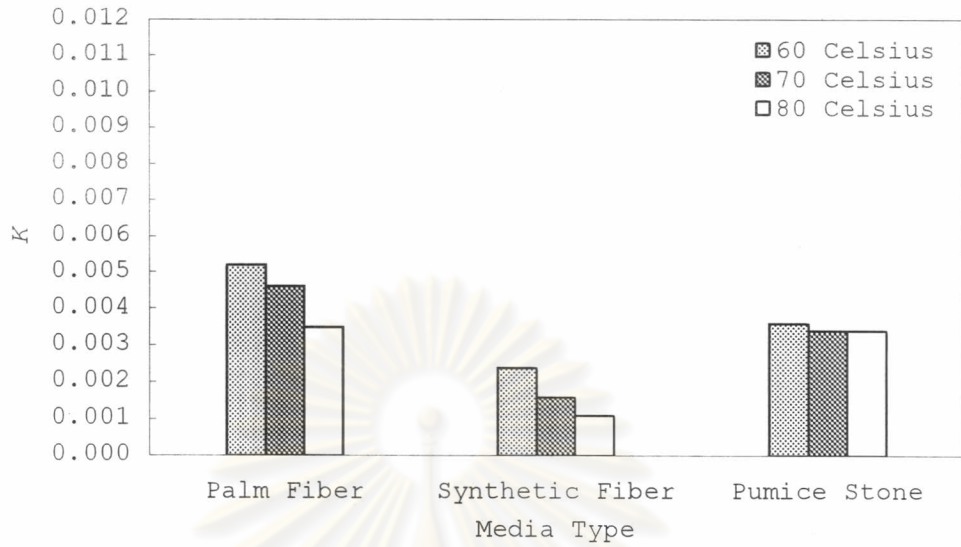


Figure 4.26 Comparisons of each temperature against coalescing media at flow velocity of 0.12 mm/sec

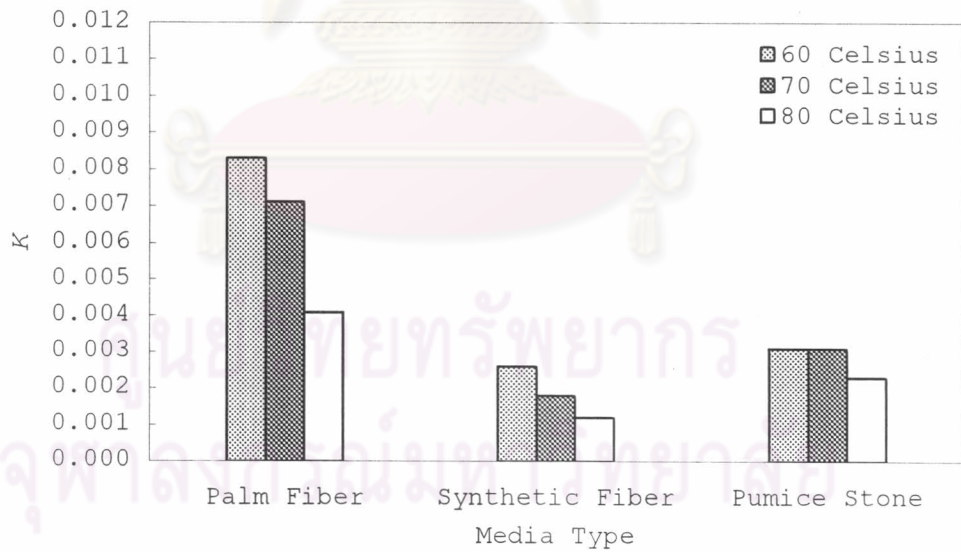


Figure 4.27 Comparisons of each temperature against coalescing media at flow velocity of 0.25 mm/sec

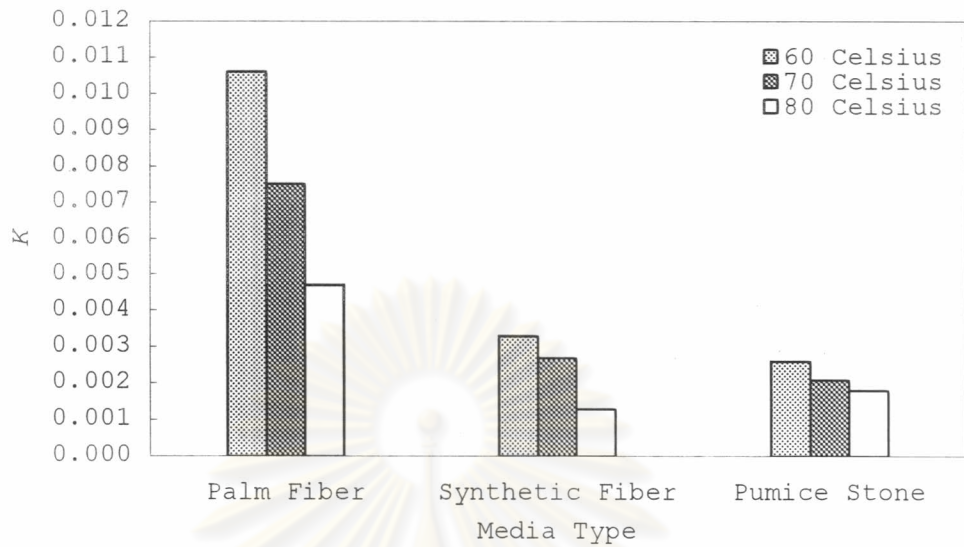


Figure 4.28 Comparisons of each temperature against coalescing media at flow velocity of 0.40 mm/sec

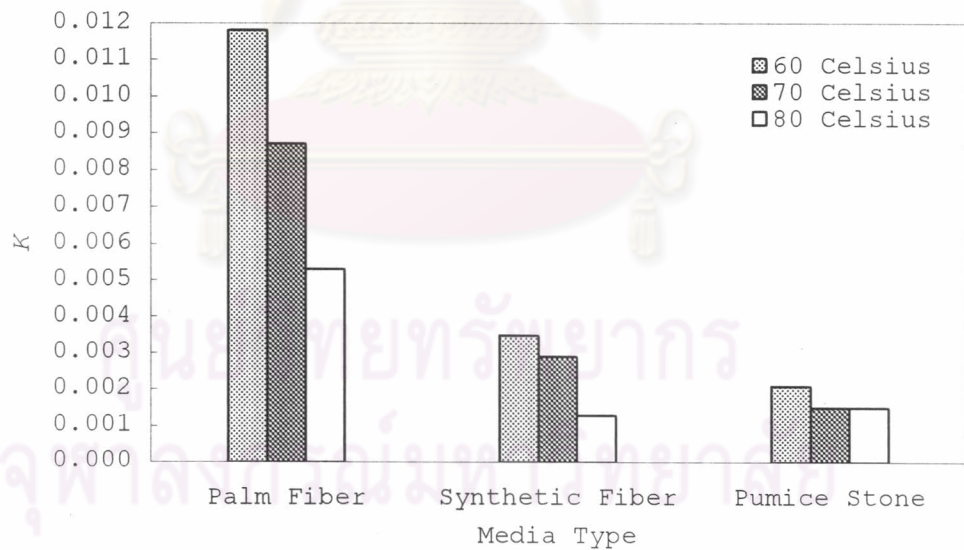


Figure 4.29 Comparisons of each temperature against coalescing media at flow velocity of 0.50 mm/sec