REMOVAL OF COLOURING MATERIALS AND IMPURITIES IN PALM OIL BY USING BENTONITE CLAY

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Abstract

Bleaching or purification is an important step in the refining of fats and vegetable / animal oils for industrial applications. Bleaching clay primarily removes colouring pigments such as chlorophylls and carotenes but peroxide and other impurities (e.g., soaps, trace metals and phosphatides) are also important target of the bleaching process. In the research, palm oil sample was collected to carry out the adsorption of colouring materials and impurities by using raw bentonite, 4 M and 8 M HCl activated bentonite and foreign bleaching clay. In this study, raw bentonite clay was treated with 4 M HCl and 8 M HCl solutions to get better adsorption properties. Characterizations of raw bentonite clay, 4 M HCl and 8 M HCl activated bentonite clay samples were carried out by using FT IR, XRD and chemical analyses. Before bleaching processes, some properties (moisture and impurities, iodine value, peroxide value, free fatty acid content, saponification value, unsaponifiable matters, refractive index and specific gravity) were determined by AOAC and AOCS methods. After treatment with raw and acid activated bentonite clay sample, iodine value and saponification value of palm oil increased, while free fatty acids content, peroxide values, unsaponifiable matters, moisture and impurities of palm oil sample decreased. In this research, bleaching efficiencies of the bentonite clay on plam oil were determined spectrophotometrically. In this research, bleaching efficiency of the raw bentonite clay, 4 M HCl activated and 8 M HCl activated bentonite clay samples were determined at 65, 80 and 90 °C for different contact times. The bleaching efficiencies of raw bentonite clay samples, 4 M HCl activated bentonite clay, 8 M HCl activated bentonite clay samples and foreign bleaching clay were 14.3, 42.8, 49.9 and 26.6 %, respectively, for 100 min contact time. Therefore, 8 M HCl activated bentonite clay sample give the highest bleaching efficiencies. This may concern with the structure of 8 M HCl activated bentonite clay sample. According to XRD data, 8 M HCl activated bentonite clay sample showed the amorphous nature due to the clay structure collapsing, whereas raw and 4 M HCl activated bentonite clay samples were crystalline nature. The amorphous nature of the 8 M HCl activated bentonite clay sample has more sorption ability to carotene in oil sample.

Keywords: Bentonite clay, acid activated bentonite clay, palm oil, physicochemical properties

Introduction

Palm oil originates from the fruit of *Elacis guineensis*. It is originally a tall stemmed tree belonging to the Palm family. Palm oil is a major dietary component and plays important nutritional role. Palm oil is a mixture of triglycerides of saturated and unsaturated fatty acids. Palm oil may be used in a variety of ways (Abdullah, 1994).

Bentonite is a native, colloidal, hydrated and mineral of the dioctahedral smectite group that is primarily composed of the mineral montmorillonite. The colour of bentonite is varied from white to gray, yellow, green, blue and black. Bentonites have a characteristic of waxy appearance when freshly dug and are generally soapy to touch. Hence, it is also called "mineral soap" or "soap clay". Bentonite is spread around the world including the types of locality. Bentonite occurs in France, Greece, Hungary, Poland, Romania, India, Japan and Australia. Italy is one of

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the leading European bentonite producers. In Myanmar, Bentonite occurs in several places around the Kyauktaga area in Kyaukpadaung Township, Mandalay Region (Economic and Social Commission for Asia and the Pacific, 1996).

Acid activation of bentonites is one of the most important processes that have been performed to achieve desirable structural condition in edibile oil bleaching applications. Activated bentonites are removing chlorophyll, carotenoids, phospholipids, metals and oxidation products from oils (Makhoukhi *et al.*, 2009).

Bleaching is an important step in the refining of vegetable oils producing light colour oil with simultaneous removal of trace metal, soaps and peroxides. This process has drawn attention to the processors of oils and technologists since it improves the appearance of oil and removes certain materials which degrades the quality of oil with time.

Among different bleaching methods, adsorptive bleaching is most commonly practiced because of its non-destructive nature and can simultaneously remove the undesired components (Bijay and Jignesh, 2009).

The removal of pigment and other trace constituents by adsorption process (bleaching) is one of the most important steps in the vegetable oil refining and this process removes the carotenes, chlorophyll and other pigments as impurities (Salawudeen *et al.*, 2007).

The process makes the oil more appealing and convenient for use. Despite the health benefits derived from palm oil it must be refined to improve the purity characteristic desirable in edible oil (Barison, 1996).

The purpose of this research is to study the efficiency in removal of colouring material and impurities from crude oil by using clay mineral. Bentonite clay was used for the removal of colouring materials and impurities in palm oil sample. At first physicochemical properties (moisture and impurities, iodine value, peroxide value, free fatty acid content, saponification value, unsaponifiable matter, refractive index and specific gravity) of palm oil was determined by using appropriate methods.

Acid activation of bentonite clay samples were carried by using 4 M and 8 M HCl solution for better adsorption properties and characterized by using FT IR, TG-DTA, XRD and chemical analysis.

In this study, the bleaching efficiencies of raw and acid activated bentonite clay on palm oil sample were studied by using the various contact time and different temperatures.

Materials and Methods

All chemicals used in the research were from British Drug House Chemical Limited, Poole, England. The chemicals had been used as it was received unless otherwise stated. Apart from the glassware and other supporting equipment are balance (E Mettler, AE 160), Thermostatic shaker (Yamaha), XRD, (Regaku; D/Max 2200 Japan), FT IR (Perkin Elmer 1600) and TG-DTA (Hi-TGA 2950 Thermo Gravimetric, Shimadzu Analyzer).

Sample Collection and Characterization

The grey coloured bentonite clay sample was obtained locally from Kyaukpadaung Township, Mandalay Region. The crude palm oil used for this study was obtained from Myeik Township, Tanintharyi Region. Petroleum ether and the hydrochloric acid used were of analytical grade. The physicochemical properties of the palm oil were determined according to the AOAC and AOCS methods.

The raw bentonite clay sample was characterized by using FT IR and XRD techniques. Some chemical properties of raw bentonite clay sample were determined. The chemical properties (loss on ignition, SiO₂, Al₂O₃, CaO, MgO and Na₂O + K₂O percent) were determined according to standard methods of analysis of ceramic materials (Hikichi, 1998).

Preparation of Acid Activation of Bentonite Clay

The local bentonite clay sample was ground and sieved to 200 meshes. The sample was rinsed with water several times. The washed bentonite clay was dried at 105-110 °C for four hours. Then the samples were ground and stored for further use. A 20 g of clay sample was weight into a beaker. A 200 mL of 4 M hydrochloric acid was added and stirred. Acid treatment was carried out at 90 °C for 2 h under reflux and the clay was separated by filtration. The clay residue was rinsed with water several times to remove remaining chloride ion (checked with a 1 % AgNO₃ solution). The washed clay material was dried at 55 °C and again crushed and passed to a 200 mesh sieve. Then, the acid activated clay was obtained. Similarly, the 8 M HCl acid activated clay was carried out using the above procedure. Acid activation was done according to the method described by Foletto *et al.* (2006).

Characterization of Bentonite Clays and Foreign Bleaching Clay

The acid activated bentonite clays and foreign bleaching clay sample were characterized by using FT IR and XRD techniques. Some chemical properties of acid activated bentonite clay samples were determined. The chemical properties (loss on ignition, SiO₂, Al₂O₃, CaO, MgO and Na₂O + K₂O percent) were determined according to standard methods of analysis of ceramic materials (Hikichi, 1998).

Bleaching Process

Effect of contact time and temperature on adsorption of palm oil sample

The adsorption treatment of palm oil with bentonite clay samples (raw, 4 M, 8 M HCl acid activated bentonite and foreign bleaching clay) were carried out for different contact times (5, 15, 20, 30, 40, 50, 60, 70, 80, 90 and 100 min) at different treatment temperatures (65, 80 and 90 °C). A 5.0 g of crude palm oil was placed in a glass stopper conical flask held in a shaking water bath thermostated at a temperature that is 65 °C \pm 1. When the temperature reached at 65 °C, 0.2 g of clay sample was added to the hot oil, which was then heated in the shake for different contact times (5 to 100 min) at 65 °C. After 5 min, the oil was removed from clay sample by filtration. Similarly, the oil was removed from respective conical flask for different contact times (15 to 100 min) at 65 °C. Similarly, the effect of temperature was studied at 65, 80 and 90 °C.

Bleaching efficiencies (% BE)

The absorbance of the palm oil was measured by using a UV Spectrophotometer. The bleaching efficiencies of bentonite clay samples were determined for different contact times (5, 15, 20, 30, 40, 50, 60, 70, 80, 90 and 100 min) at different treatment temperatures (65, 80 and 90 °C). A 0.1 g of palm oil sample after bleaching with bentonite clay for 5 min contact time at 65 °C was put into a conical flask and diluted by dissolving in 7.5 mL of petroleum ether (analytical grade). The mixture was poured in the cuvette and the value of the absorbance was read at 445 nm wavelength using UV-visible spectrophotometer (Kamga *et al.*, 2007).

The percentage bleaching efficiency (% BE) was determined by the equation;

$$\% BE = \frac{A_0 - A_t}{A_0} \times 100$$

Where, $A_0 =$ absorbance of unbleached palm oil and

 A_t = absorbance of bleached palm oil at time, t.

Similarly, the bleaching efficiencies of palm oils after teaching with bentonite clay samples for different contact times (5, 15, 20, 30, 40, 50, 60, 70, 80, 90 and 100 min) at treatment temperature 80 and 90 °C were determined.

Results and Discussion

In this research, raw bentonite clay sample was used as adsorbent to remove the colouring materials and impurities in crude oils. Natural occurring bentonites may show very little activity for bleaching oils and fats. For this reason, bentonite clay mineral was treated with HCl (4 M and 8 M) for two hours in order to evaluate important structural properties modifications that may affect oil bleaching. Figure 1 shows raw, 4 M and 8 M acid activated bentonite clay and foreign bleaching clay.

Characteristics of Bentonite Clay Samples

FT IR analysis

Figures 2, 3, 4 and 5 show respectively the FT IR spectra of raw, 4 M and 8 M acid activated bentonite clay samples and foreign bleaching clay sample. The data of absorption bands and possible assignments of raw, 4 M and 8 M acid activated bentonite and foreign bleaching clay are shown in Table 1. It can be observed that the hydroxyl, Si-OH, Si-H and Si-O-Si groups were presented in these spectra of bentonite clay samples.

In these spectra of the clay samples showed absorption bands between 681 and 820 cm⁻¹ due to Si-O-Si stretching. The band at 800 - 950 cm⁻¹ showed Si-H bending and the band at 3200 - 3700 cm⁻¹ showed OH stretching vibration. The absorption bands at 1639 - 1643cm⁻¹ appeared due to OH bending vibration.

XRD analysis

XRD measurement was carried on all bentonite clay samples and foreign bleaching clay sample. Figures 6, 7, 8 and 9 showed XRD measurements of raw bentonite, 4 M and 8 M acid activated bentonite clays and foreign bleaching clay, respectively. The presence of smectite (SiO₂), feldspas and quartz compounds in bentonite clay was found in the XRD diffractogram.

According to XRD data, 8 M HCl activated clay sample showed the amorphous nature, whereas the raw and 4 M HCl activated clay samples were crystalline nature.

Chemical analysis

Chemical constituents (SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, K₂O + Na₂O and loss on ignition) of the raw and acid activated bentonite clay samples were determined according to the standard method of analysis of ceramic materials. Determination of silica percent was carried out by using alkaline fusion. Table 2 shows chemical composition of clays samples.

The chemical analysis of clay samples showed a high silica content which suggests its high potential for the production of floor tiles. After treating the raw bentonite clay with acid solution was found on increase in SiO_2 concentration.

Physicochemical properties of palm oil

The physicochemical properties of crude palm oil were determined and then it was treated with various adsorbents (raw bentonite, 4 M and 8 M acid activated bentonite clays). The physicochemical properties of bleached palm oils after treating with various adsorbents were determined and shown in Table 3. The percentages of free fatty acid in palm oil sample before and after treating with various adsorbents were 10.90 % (raw), 7.24 % (4 M) and 6.61 % (8 M), respectively. Thus, it was found that free fatty acid in oil samples treated with 8 M acid activated clay was less than that treated with other two bentonite clays. The free fatty acid is among the undesirable constituent to be removed and thus its low percentage enhances the efficiency of the refining process.

It can be seen that the iodine value of palm oil after treating with 8 M activated bentonite clay was larger than that after treating with the other adsorbents. When iodine value in oil sample is greater, the unsaturated fatty acid in oil sample is greater. The saponification value increased more after treating with the 8 M activated bentonite clay. High saponification value indicates the presence of low molecular weight fatty acid. High value of saponification suggests that the palm oil can be used for soap production.

Unsaponifiable matter means hydrocarbon, alcohols and sterols. Unsaponifiable matter in palm oil samples were 0.179 % (crude), 0.177 % (raw), 0.073 % (4 M) and 0.057 % (8 M), respectively. After treating with 8 M activated bentonite clay, unsaponifiable matter in palm oil samples was found to decrease significantly.

Adsorption of carotene in palm oil

The evaluation of the amount of pigment removed was made by UV-visible spectroscopy. Adsorption of carotene in palm oil on various adsorbents (raw, 4 M and 8 M activated bentonite and foreign bleaching clay) was determined at 65, 80 and 90 °C for various contact time (5, 15, 20, 30, 40, 50, 60, 70, 80, 90 and 100 min) and the absorbance of final product read at 445 nm. Table 4 mentions the absorbance of carotene in palm oil at different temperatures and different contact times for various adsorbents. It is evident from these results that the time required to reach the adsorption equilibrium decrease as the temperature increases. The absorbance decreases as increasing the temperature and the contact time. It can be seen from the results that the absorbance value (at 445 nm) for 100 min contact time for 8 M HCl activated bentonite are 1.025 at 65 °C, 0.922 at 80 °C and 0.845 at 90 °C, respectively. The dark colour of crude oil converts to a light colour as the absorbance value decrease.

Bleaching efficiencies (% BE)

In this research, the kinetics of adsorption of palm oil carotenes at three different temperatures (65, 80 and 90 °C) and various contact times by various adsorbents (raw, 4 M HCl and 8 M HCl activated bentonite clays and foreign bleaching clay) were determined. The values of bleaching efficiencies (% BE) of various adsorbent clays samples are mentioned in Tables 5, 6, 7 and 8 for the bleaching process at contact times of 5, 15, 20, 30, 40, 50, 60, 70, 80, 90 and 100 min and three different temperatures.

As seen in these tables, the bleaching efficiency increases as the temperature and contact time increase. It was observed that the percent of bleaching efficiencies for 100 min contact time are 49.9 at 65 °C, 54.9 % at 80 °C and 58.7 at 90 °C for 8 M HCl activated clay. Figures 10, 11, 12 and 13 shows the plot of bleaching efficiencies of various adsorbents clays as a function of contact time at 65, 80 and 90 °C. Preliminary tests showed that the raw clays were effective in the adsorption of palm oil pigment, 8 M HCl activated bentonite clay had a better performance compared to other clays, this can be explained by the increase of acid concentration of the acid solution used for clay leaching.



(a) (b) (c) (d) Figure 1 Various type of bentonite clay (a) Raw bentonite (b) 4 M HCl acid activated bentonite (c) 8 M HCl acid activated bentonite (d) Foreign bleaching clay



Figure 2 FT IR spectrum of raw bentonite



Figure 3 FT IR spectrum of 4 M HCl acid activated bentonite



Figure 4 FT IR spectrum of 8 M HCl acid activated bentonite



Figure 5 FT IR spectrum of foreign bleaching clay

Table 1	FT IR Data of Raw Bentonite, 4 M HCl and 8 M HCl Acid Activated Bentonite
	Clays and Foreign Bleaching Clay

	Wave number (cm ⁻¹)							
Sr No.	Raw bentonite clay	4M HCl acid activated bentonite clay	8M HCl acid activated bentonite clay	Foreign bleaching clay	Literature* Wave number	Related functional group		
1	3618	3695-3406	3695- 3626	3695-3232	3700-3200	υ _{O-H}		
2	1643	1639	1643	1627	1639-1643	$\delta_{\text{O-H}}$		
3	1033	1033	1033-1103	1010-111	830-1110	υ _{si-O}		
4	925	914	910	918	800-950	$\delta_{\text{Si-H}}$		
5	694-786	794,756	-	694,786	681-820	υ _{si-O-Si}		

* Silverstein and Terence. (1991)



Figure 6 XRD diffractogram of raw bentonite Figure 7 XRD diffractogram of 4 M HCl acid activated bentonite



Figure 8 XRD diffractogram of 8 M HCl acid activated bentonite



Figure 9 XRD diffractogram of foreign bleaching clay

Sr No.	Compounds	Raw Bentonite clay (%)	4 M HCl acid activated Bentonite clay (%)	8 M HCl acid activated Bentonite clay (%)
1	SiO ₂	52.54	54.68	55.94
2	Al_2O_3	30.28	29.44	29.04
3	Fe ₂ O ₃	3.36	3.52	3.50
4	CaO	0.80	0.40	0.18
5	MgO	2.50	0.80	0.71
6	$K_2O + Na_2O$	1.55	0.30	0.21
7	Loss on ignition	10.61	10.80	10.96

 Table 2
 Chemical Composition of Bentonite Clay Samples

 Table 3 Physicochemical Properties of Raw and Acid Activated Palm Oil Samples

		Palm oil					
Sr No.	Properties	Crude oil	After bleaching with raw bentonite	After gbleaching with 4 M HCl acid activated bentonite	After bleaching with 8 M HCl acid activated bentonite		
1	Specific gravity (20 ° C)	0.907	0.914	0.912	0.897		
2	Iodine value (%)	46.380	48.650	50.700	52.170		
3	Peroxide value (meq/kg)	1.420	2.010	1.860	1.420		
4	Saponification value (mg KOH/g)	201.290	192.720	193.610	195.400		
5	Unsaponifiable matter (%)	0.179	0.177	0.073	0.057		
6	Free fatty acid (%) (as oleic)	11.800	10.900	7.240	6.610		
7	Moisture and Impurities (%)	0.129	0.102	0.078	0.034		

* obtained from Myeik Township, Tanintharyi Region

C	Contact					Absor	bance ((at 445)	nm)				
Sr No	time		65	°C			80	°C			90 °	C	
INU.	(min)	Raw	4 M	8 M	F	Raw	4 M	8 M	F	Raw	4 M	8 M	F
1	5	1.929	1.833	1.598	1.699	1.913	1.679	1.432	1.552	1.752	1.514	1.403	1.389
2	15	1.875	1.719	1.462	1.681	1.848	1.620	1.362	1.506	1.747	1.490	1.293	1.373
3	20	1.871	1.630	1.427	1.662	1.786	1.525	1.270	1.466	1.695	1.379	1.230	1.354
4	30	1.869	1.551	1.390	1.647	1.767	1.425	1.214	1.418	1.643	1.328	1.146	1.306
5	40	1.864	1.519	1.381	1.613	1.702	1.377	1.189	1.386	1.557	1.292	1.113	1.248
6	50	1.854	1.349	1.226	1.598	1.656	1.336	1.106	1.288	1.530	1.231	1.071	1.200
7	60	1.839	1.357	1.149	1.575	1.647	1.259	1.071	1.254	1.507	1.202	1.010	1.186
8	70	1.934	1.309	1.180	1.562	1.635	1.229	1.034	1.214	1.480	1.160	0.963	1.166
9	80	1.814	1.190	1.082	1.540	1.626	1.154	0.978	1.168	1.452	1.125	0.921	1.148
10	90	1.801	1.185	1.055	1.512	1.553	1.118	0.929	1.144	1.442	1.085	0.859	1.136
11	100	1.753	1.170	1.025	1.501	1.516	1.104	0.922	1.134	1.435	1.068	0.845	1.118
Raw	= Raw	bentonit	wentonite clay sample $4 M = 4 M HCl$ acid activated clay sample										
8 M	= 8 M I	HCl acid	activate	d clay sa	mple		F =	Foreig	n bleach	ing clay	sample		

Table 4 Absorbance (445 nm) of Carotene in Palm Oil at Different Temperatures and
Different Contact Times on Bentonite Clay Samples

Table 5Bleaching efficiencies (% BE) of Raw Bentonite Clay Sample for Different
Contact Times at 65 °C, 80 °C and 90 °C

Sr	Contact time (min) -	Bleaching Efficiencies (% BE)						
No.	Contact time (mm)	65 °C	80 °C	90 °C				
1	5	5.7	6.3	14.3				
2	15	8.3	9.6	14.8				
3	20	8.5	12.7	17.1				
4	30	8.6	13.6	19.7				
5	40	8.8	16.8	23.9				
6	50	9.3	19.0	25.2				
7	60	10.1	19.5	26.3				
8	70	10.3	20.0	27.6				
9	80	11.3	20.5	29.0				
10	90	12.0	24.1	29.5				
11	100	14.3	25.9	29.8				

Table 6Bleaching Efficiencies (%.BE) of 4 M HCl Activated Bentonite Clay Sample for
Different Contact Times at 65 °C, 80 °C and 90°C

Cr No	Contact time (min) -	Bleaching Efficiencies (% BE)				
Sr 110.		65 °C	80 °C	90 °C		
1	5	10.4	17.9	26.0		
2	15	16.0	20.8	27.1		
3	20	20.3	25.5	32.6		
4	30	24.2	30.4	35.1		
5	40	25.2	32.7	36.9		
6	50	26.3	34.7	39.3		
7	60	33.7	38.5	41.3		
8	70	36.7	39.9	43.4		
9	80	41.8	43.6	45.0		
10	90	42.1	45.4	47.0		
11	100	42.8	46.0	47.8		

S. No	Contact time (min) -	Bleaching Efficiencies (% BE			
Sr No.	Contact time (min)	65 °C	80 °C	90 °C	
1	5	21.9	30.0	31.4	
2	15	28.5	33.4	36.8	
3	20	30.3	37.9	39.9	
4	30	32.1	40.7	44.0	
5	40	32.5	41.9	45.6	
6	50	40.1	45.9	47.7	
7	60	41.9	47.7	50.6	
8	70	42.3	49.5	52.9	
9	80	47.1	52.2	55.0	
10	90	48.5	54.6	58.0	
11	100	49.9	54.9	58.7	

Table 7 Bleaching Efficiencies (%.BE) of 8 M HCl Acid Activated Bentonite Clay Sample for Different Contacts Times at 65 °C. 80 °C and 90°C

Table 8Bleaching Efficiencies (% BE) of Foreign Bleaching Clay (China) for Different
Contact Times at 65 °C, 80 °C and 90 °C

Sr No.	Contact time (min) -	Bleaching Efficiencies (% BE)				
		65°C	80°C	90°C		
1	5	17.0	24.1	32.1		
2	15	17.8	26.4	32.9		
3	20	18.8	28.3	33.8		
4	30	19.5	30.7	36.2		
5	40	21.2	32.3	38.1		
6	50	21.9	37.0	41.3		
7	60	23.0	38.7	42.0		
8	70	23.7	40.7	43.0		
9	80	24.7	42.9	43.9		
10	90	26.1	44.1	44.5		
11	100	26.6	44.6	45.4		





Figure 10 Plot of bleaching efficiencies of raw clay samples as a function of contact time at 65, 80 and 90 °C

Figure 11 Plot of bleaching efficiencies of 4 M HCl activated bentonite clay samples as a function of contact time at 65, 80 and 90 °C



Figure 12 Plot of bleaching efficiencies of 8 M HCl activated bentonite clay samples as a function of contact time at 65, 80 and 90 °C



Figure 13 Plot of bleaching efficiencies of foreign bleaching clay (china) as a function of contact time at 65, 80 and 90 °C

Conclusion

In this research, palm oil sample was collected to carry out the adsorption by using raw bentonite, 4 M and 8 M HCl treated bentonite and foreign bleaching clay. In this study, raw bentonite clay was treated with 4 M HCl and 8 M HCl solutions to get better adsorption properties. After treatment with raw and acid activated bentonite clay sample, iodine value and saponification value of palm oil increased, while free fatty acids, peroxide values, unsaponifiable matters, moisture and impurities of palm oil samples decreased. The bleaching efficiencies of raw, 4 M HCl activated, 8 M HCl activated clay samples and foreign bleaching clay were 14.3, 42.8, 49.9 and 26.6 %, respectively, for 100 min contact time. Therefore 8 M HCl activated clay sample give the highest bleaching efficiencies. This may concern with the structure of 8 M HCl activated clay sample. According to XRD data, 8 M HCl activated clay sample showed the amorphous nature due to the clay structure collapsing. Whereas raw and 4 M HCl activated clay sample has more sorption ability to carotene in oil sample.

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