Palm Oil Fractionation Process *

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Abstract

Palm oil is semisolid at ordinary temperatures of 20º to 27ºC because of the precipitation of saturated glycerides. These glycerides have relatively high melting points and possess only a limited solubility in the unsaturated glycerides. Fractionation was carried out for the separation of high-melting triglycerides from low-melting triglycerides by crystallization when they melt.

The purification steps of the crude palm oil sample such as filtration, degumming, neutralization and bleaching was carried out to ensure the removal of non-glycerides impurities which also affect the fractionation process. By the method of fractional crystallization after complete treatment of Crude Palm Oil (CPO), Filtered, Degummed, Neutralized and Bleached Palm Olein (FDNB Palm Olein) was produced. In this process, liquid olein was produced as the main product and solid stearin as by-product. Separation of liquid (olein) and solid (stearin) was carried out by centrifugation. Plate-and-frame filtration method (Yashino System) was also attempted.

To control the process and quality of the product, analysis of oil was carried out after every stage of processing from CPO. In this study, second fractionation of palm oil at 25ºC gave the favorable conditions in which the iodine value of super olein was raised to 65.35 and refractive index of those was also increased to 1.4655.

Keywords: saturated glycerides, fractional crystallization, CPO, FDNB palm olein, stearin

Introduction

Palm oil is derived from the fruit of *Elaeis guinensis*. The pericarp of the fruit consists of the outer exocarp or skin, the mesocarp or pulp, and the endocarp or shell. The pulp is made up of only fibres (35 to 85% of the fruit weight) containing 40 to 60% of palm oil and 35 to 45% of water. Palm oil is composed of approximately 50 per cent saturated fatty acids, primarily palmitic acid, and 50 per cent unsaturated fatty acids, mainly oleic and linoleic acid. The fatty acid compositions of palm oil are 0.5-6 myristic; 35-40 palmitic; 2-8 stearic; 40-50 oleic and 5-11 linoleic. (Gander, 1977)

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*** The Best Paper Award Winning Paper in Industrial Chemistry (2008)**

Crude Palm oil contains non-glyceride impurities such as mono- and diglycerides, free fatty acids, phospholipids, sterols, fat-soluble vitamins, fatty alcohol, trace metals, pigments (carotene, tocopherol) and oxidation products that are removed through a series of processing steps. PORIM (Palm Oil Research Institute of Malaysia) recommended standard for crude palm oil are 0.05 (max) dirt; 2.5 (max) free fatty acid; 50-55 iodine value; 5(max) acid value and 5(max) peroxide value. Low-grade palm oil, that is with a free fatty acid content in excess of 5 per cent is used in soap and candle while palm oil with even higher free fatty acid content can be used in tin plating. (Gander, 1977)

More than 45 per cent of the triglycerides in palm oil have a melting point of 34˚C or higher and these are the main constituent of the stearin fraction. The majority of the glycerides comprising the olein fraction have a melting point of 19˚C. This research was carried out to remove the stearin by using the fractionation process in three steps, namely: (1).Conditioning which consists in heating the oil, (2).Keeping the fat at a controlled temperature to allow crystallization, and (3). Separation of the oleins from the stearins.

For purposes of specification and quality control of this is generally assessed by the refractive index of the oil although the iodine value is also indicative of the reduction of the more saturated triglycerides in the oil.

Materials and Methods

Crude palm oil (CPO) sample and Refined, bleached and deodorized palm oil (RBDPO) sample were obtained from Soap Raw Material and Refined Oil Factory of Myanma Pharmaceutical Industries at Hlaing Township.

Purification of Crude Palm Oil

Sludge Filtration

At room temperature, Crude palm oil was filtered to remove only suspended or dispersed solids such as dirt and impurities, sludge, mucilage, proteinaceous substances and waxes by using perforated basket centrifuge.

Degumming

Filtered palm oil was gradually preheated to 72˚C, and 2% by volume of hot water (at 80˚C) was added to it and stirred at (200) rpm for (30)

minutes. The hydratable phosphatides or gums were precipitated and separated by mechanical centrifuge. The same experiment was carried out with varying amount of hot water, 3% by volume.

Water-degummed oil was further refined with 0.1% by volume of concentrated phosphoric acid at 90˚C with continuous stirring at 200 rpm for (45) minutes. The non-hydratable gums were converted to hydratable, after which the coagulated gums in the oil was separated by mechanical centrifuge. The experiment was simply repeated, using different percentages of concentrated phosphoric acid, such as 0.2% and 0.3%.

Neutralization

Filtered and degummed palm oil (0.2% by volume of concentrated phosphoric acid being the most suitable) was gradually preheated to 72˚C and neutralized with 22˚Be′caustic soda solution to remove free fatty acids. After (30) minutes, soap particles thus formed were removed by mechanical centrifuge. The neutralized oil was washed with brine solution to remove traces of residual soap particle. The washed water was tested with phenolphthalein indicator to ensure that it was free from soap particles. The washed oil was dried under vacuum to remove traces of water and moisture.

Bleaching

Filtered, degummed, and neutralized palm oil was bleached under closed system using vacuum pump to remove all pigments and oxidation products. Oil was heated in (1) liter round-bottom flask (pyrex) to 110˚C and 3% of bleaching earth by weight of oil was added to it. Vacuum was set at 25 in.Hg and the temperature was raised to 180˚C. The oil mixture was agitated by a magnetic stirrer spinning at the rate of (200) rpm for 60 minutes. The spent bleaching earth was separated by filtration using special filter paper. The same experiment was carried out using varying amount of bleaching earth, such as 4% and 5% by weight.

Figure 1. – Laboratory Apparatus for Bleaching under Closed System

Fractional Crystallization

Filtered, degummed, neturalized and bleached palm oil (5% by weight bleaching earth being the most favorable) was heated to 72˚C. The melted palm oil was chilled to 28˚C first with cold water and then with ice .After the oil temperature had reached to 28˚C, the oil was stabilized at that temperature. During (6) hours of crystallization, the oil was agitated by a magnetic stirrer spinning at (50) rpm. Then, the crystallized palm oil was centrifuged to separate liquid olein and solid stearin by means of a mechanical centrifuge and the olein was again filtered with special filter paper.

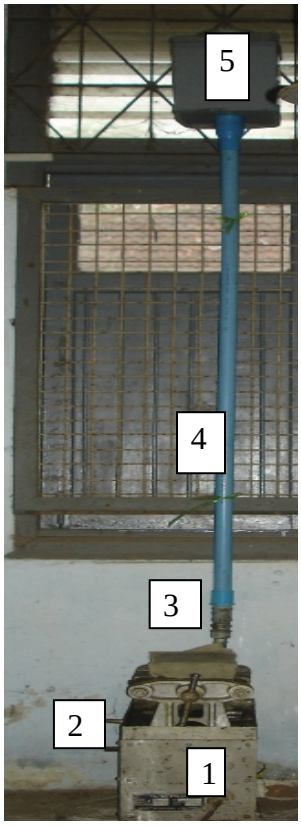
Then the olein obtained was fractionated again at that temperature to remove residual solid stearin from the olein.

Similar experiment was conducted at different temperature, 25 ˚C.

Calibration of Yashino–system Plate-and-frame Filtration

The refined, bleached and deodorized palm oil (RBDPO) was first degummed with water and phosphoric acid to remove phosphatides. Then, the degummed, refined, bleached and deodorized palm oil (DRBDPO) was fractionated based on the Yashino system dewaxing of rice bran oil. The DRBDPO was preheated to a slip melting temperature of 72˚C and cooled to a crystallization temperature 25˚C by adding first with cold water and then with ice. During (3) hour of crystallization, the oil was agitated by a magnetic stirrer spinning at (50) rpm and the crystallized palm oil was left over night at

that temperature. Then, the oil was fed into the small tank of the plate-and-frame filter press at a head pressure of 1.59 psi and the olein was obtained from the outlet of the filter press. (3) sets of plates and frames were used for filtration and the filter area is 16 in^2, as in Figure (2).



1. Plate-and-frame filter press
   - No. of plates - 3
   - No. of frames - 3
2. Outlet
3. Gate valve
4. PVC pipe (1″diameter and 5′ height)
5. Small tank with chilling jacket

Figure 2. – Laboratory Apparatus for Calibration of Yashino system Plate-and-frame Filtration

Analysis of Palm Oil

Acid Value (AV)

The acid value is a measure of the quality of oil. It is defined as the number of milligrams of potassium hydroxide necessary to neutralize the free acids in 1g of sample. For edible purposes, AV of oil must be less than 1.

Peroxide Value (PV)

The peroxide value is a measure of the amount of oxygen chemically bound to an oil or fat as peroxide, particularly hydroperoxide. It is defined as the weight in milli-equivalents of peroxide per 1000g of sample, which

oxidize potassium iodide under the conditions of the fat. Apart from the acid value, the analysis of rancidity should include the determination of peroxide value.

Iodine Value (IV)

The iodine value of a fat is a measure of its degree of unsaturation. It is determined by the amount of halogen absorbed and is conventionally expressed as the weight of iodine absorbed by 100 parts by weight of the fat. It should be noted that the higher the iodine value, the greater the degree of unsaturation of the fat.

Refractive Index (RI)

The refractive index of a substance is the ratio of the speed of light in a vacuum to the speed of light in the substance. It is related to the degree of saturation but it is affected by other factors such as free fatty acid content, oxidation, and heat treatment. The refractive index of oil was determined by using Abbe Refractometer.

Color

Color is an important criterion in the determination of quality of finished oils and the processing efficiency on an edible oil refinery. Red, yellow and blue colors are used for edible oils. Color was determined by using Lovibond Tintometer (1 inch cell).

Results and Discussion

Crude palm oil (CPO) sample and refined, bleached and deodorized palm oil (RBDPO) sample were analyzed for physical and chemical properties by the official and tentative methods of A.O.C.S (American Oil Chemists' Society) and the data are shown in Table (1). The physical and chemical properties of palm oil after every stage of purification from CPO are shown in Table (2).

In the process of degumming, varying quantity of hot water such as 2% and 3% (by volume) respectively was used to remove hydratable phosphatides and 3% was found to be the optimum condition in that it yields the maximum amount of gum (1.2 g). The water-degummed oil (3% by volume hot water being the most suitable) was further refined with different percentages of concentrated phosphoric acid such as 0.1%, 0.2%, 0.3% (by

volume) to remove non-hydratable phosphatides. In this process, 0.2% by volume of phosphoric acid gave the favorable condition in which the maximum amount of gum (3 g) is obtained. The optimum data of degummed palm oil (with 0.2% phosphoric acid) are shown in Table (2).

Palm oil contains high free fatty acid content. The degummed oil (3% by volume hot water and 0.2% by volume of phosphoric acid being the most suitable) was neutralized with 22˚Be´ caustic soda solution to reduce the free fatty acid. The results are shown in Table (2). In neutralization step, the acid value was reduced from 24.166 to 0.4488 and the refining loss was 41% (by weight). Bailey, 1945 reported that the higher the acid value, the greater the refining loss.

In the experiment of bleaching the neutralized oil, varying amount of bleaching earth such as 3%, 4% and 5% (by weight) was used and it was found that 5% was considered as the optimum. The optimum data of bleached oil (with 5% bleaching earth) are shown in Table (2). Although there was a slight increase in acid value of the bleached oil, there also occurred a distinct color reduction from (7R, 41Y) to (3R, 9Y).

Palm oil was fractionated under different crystallization temperatures such as 28˚C and 25˚C. The olein separated from the crystallized oil slurry was checked by the iodine value and refractive index of the oil before and after fractionation. The experimental data are tabulated in Table (3) and Table (4).

The fractionation of oil at 25˚C results in a higher iodine value and refractive index of olein, a higher stearin yield. Fractionation was improved by using a two-stage process. An olein obtained by the first stage fractionation was separated into a fraction called as super olein. Second fractionation at 25˚C was found to be the most favorable conditions in which the iodine value of super olein was raised to 65.35 and the refractive index of those was also increased to 1.4655. Walker, R.C., (1983) reported that the higher the iodine value, the greater the degree of unsaturation of the oil.

Yashino-system low pressure plate-and-frame filtration was calibrated to remove stearin from the refined, bleached and deodorized palm oil (RBDPO). In this type of filtration, the oil flew as a result of gravitational force. The filter cloth retained the solid (stearin) in the slurry which added successive layers to the cake as the filtrate (olein) passed through the cake and filter cloth. The results obtained are tabulated in Table (6). By this

method, the iodine value and the refractive index of olein were increased to 58 and 1.464, respectively although the olein yield per cent was very low. Hamm, W., (2005) reported that the feed oil quality, composition, pre-treatment and crystallizer design affect the crystallization and separation. The cooling surface must be large enough in relation to the volume of oil to produce a slurry suitable for separation.

The final products filtered, degummed, neutralized and bleached palm olein at 25°C (FDNB palm olein) and degummed, refined, bleached and deodorized palm olein (DRBD palm olein) were analyzed for their physical and chemical properties and then compared with the crude palm oil (CPO) sample and refined, bleached and deodorized palm oil (RBDPO) sample respectively. The data are shown in Table (5) and Table (7).

Table (1) Physical and Chemical Properties of Crude Palm Oil (CPO) and Refined, Bleached and Deodorized Palm Oil (RBDPO)

Parameter	**CPO**	**RBDPO**
Colour : Red	46	1.0
Yellow	Nil	8
Blue	1.1	Nil
Refractive Index	1.461	1.4625
Iodine Value (Wijs)	52.452	53.298
Acid Value	20	0.3366
Peroxide Value (meq/kg)	11.584	8.4

Table (2) Physical and Chemical Properties of Palm Oil after each step of Purification from Crude Palm Oil (CPO)

Parameter	**CPO**	**After Filtration**	**After Degumming**	**After Neutralization**	**After Bleaching**
Colour : Red	46	10.8	8.5	7	3
Yellow	Nil	43	41	41	9
Blue	1.1	Nil	Nil	Nil	Nil
Refractive Index	1.461	1.4615	1.4615	1.4620	1.4630
Acid Value	20	21	24.166	0.4488	1.122

Table (3) First Fractionation of Filtered, Degummed, Neutralized and Bleached Palm Oil at Different Temperatures

	Temperature (˚C)	
	28˚C	**25˚C**
Olein Yield (%)	52	45
Stearin Yield (%)	48	55
Iodine Value		
Before Fractionation	52.452	52.452
After Fractionation	57.74	59
Refractive Index		
Before Fractionation	1461	1.461
After Fractionation	1.464	1.4645

Table (4) Second Fractionation of Filtered, Degummed, Neutralized and Bleached Palm Olein at Different Temperatures

	Temperature (˚C)	
	28˚C	**25˚C**
Superolein Yield (%)	35	28.5
Stearin Yield (%)	65	71.5
Iodine Value		
Before Fractionation	57.74	59
After Fractionation	63.45	65.35
Refractive Index		
Before Fractionation	1.464	1.4645
After Fractionation	1.465	1.4655

Table (5) Physical and Chemical Properties of Crude Palm Oil (CPO) and Filtered, Degummed, Neutralized and Bleached Palm Olein (FDNP palm olein)

Parameter	**CPO**	**FDNP palm olein**
Colour : Red	46	2.5
Yellow	Nil	10
Blue	1.1	Nil
Refractive Index	1.461	1.4655
Iodine Value (Wijs)	52.452	65.35
Acid Value	20	0.5049
Peroxide Value (meq/kg)	11.584	6.8

Table (6) Fractionation of Degummed, Refined, Bleached and Deodorized Palm Oil at 25˚C using Yashino-system Plate-and-frame Filter Press

Olein Yield (%)	9
Stearin Yield (%)	81
Iodine Value	
Before Fractionation	53.298
After Fractionation	58
Refractive Index	
Before Fractionation	1.4625
After Fractionation	1.4640

Table (7) Physical and Chemical Properties of Refined, Bleached and Deodorized Palm Oil (RBDPO) and Degummed, Refined, Bleached and Deodorized Palm Olein (DRBD palm olein)

Parameter	RBDPO	DRBD palm olein
Colour : Red	1.0	1.5
Yellow	8	14
Blue	Nil	Nil
Refractive Index	1.4625	1.4640
Iodine Value (Wijs)	53.298	58
Acid Value	0.3366	0.2992
Peroxide Value (meq/kg)	8.4	6.8

Conclusion

Acid value and peroxide value of the crude palm oil exceeded the international standard values as acid value and peroxide value were found to be 20 and 11 respectively. According to PORIM on crude palm oil, the standards set for crude palm oil are 5 maximum acid value and 5 maximum peroxide value, respectively. Owing to the deterioration of oil, there were more refining loss and less percentage of yields.

After fractionation, it was observed that fractionation temperature and residence time have a fundamental importance on the formation and character of the crystal. Higher fractionation temperature results in a lower stearin yield and a lower iodine value and refractive index of olein. Lower fractionation temperature results in a higher stearin yield and higher iodine value and refractive index of olein. Longer residence times induced lower growth rates and made longer filtration times. In order to obtain higher iodine value and to improve the flow properties of olein, the oil should be fractionated to a low temperature in a two-stage process.

Acknowledgements

The author is grateful to Professor Daw Aye Nyunt Kyi, Head of Department of Industrial Chemistry, University of Yangon, for her kind permission to carry out this research at the Industrial Chemistry Department of Yangon University. The author wishes to express sincere gratitude and heartfelt thanks to supervisor Dr. Pansy Kyaw Hla, Professor**,** Industrial Chemistry Department, University of Yangon and co-supervisor U Myint Pe, Part-time Professor, Industrial Chemistry Department, University of Yangon for their detailed specific instructions and valuable suggestions.

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