Jurnal Teknologi

Full paper

Effect of Coolant Temperature on Progressive Freeze Concentration of Refined, Bleached and Deodorised Palm Oil

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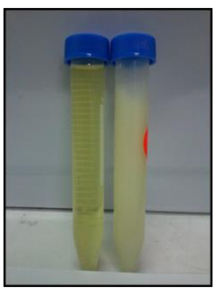
Article history

Received :4 October 2013
Received in revised form :
17 March 2014
Accepted :8 May 2014

Graphical abstract



Abstract

This study focused on the possibility of separating refined, bleached and deodorized palm oil (RBDPO) into olein and stearin by using progressive freeze concentration (PFC) as an alternative method to replace the conventional fractionation process. PFC has the potential to be a more effective technique for olein-stearin separation, with minimal changes in the product's quality for producing high quality edible oil. Apart from that, it requires fewer unit operations compared to conventional methods. In this research, the parameter of coolant temperature was selected to investigate the performance of PFC using stainless steel crystallizer. In order to determine the system efficiency, effective partition constant (K) was investigated, while the quality of the oil was evaluated by iodine value (IV), slip melting point (SMP) and the percentage of olein yield. From the results, all the determinant parameters were found to be optimum at the coolant temperature of 28°C. At this optimum point, K value, IV, SMP and olein yield were found to be 0.2715, 55.84 wijs, 23.10°C and 67.8537%, respectively.

Keywords: Progressive freeze concentration; palm oil; refined bleached and deodorised palm oil; iodine value; slip melting point

■1.0 INTRODUCTION

Palm oil is richly edible and an important member of vegetable oil group besides coconut oil, palm kernel, or olive oil which are used as raw material in food or non-food industries. The oil palms (*Elaeis or macaw-fat*) are very well-known worldwide as a raw material for various products such as soap, frying oil, biodiesel, edible fats in the confectionery, powder detergent, ice cream, mayonnaise, pomades, candle and so on. Different types of oil can be extracted from the oil palm fruit such as crude palm oil, refined, bleached palm oil (RBDPO), palm stearin, palm olein, fractionated palm olein and palm mid-fraction. Out of these, Malaysia's most exported oil is palm oil and palm olein.

Globally, Malaysia has the capacity to produce and consistently export best palm oil in a large quantity. Oil palms are now grown in tropical countries as an agricultural crop due to its ability to act as a high-yielding source of edible and technical oil. In fact, most of them are having a minimum 1600 mm/yr of rainfall, geographically within 10° of the equator.

Prior to consumption as edible oil, crude palm oil has to go through various processes including refining, bleaching, deodorising and finally fractionation[1]. There are three types of fractionation, which are dry, detergent, and solvent fractionations. Among all of these processes, dry fractionation is the most common process used, which involves crystallisation of olein at its melting point (22-24°C)[2] temperature. Dry fractionation is mostly used in Malaysia's refinery industries because of its own advantages in the process compared to detergent and solvent fractionation. In particular, solvent and detergent fractionations need greater capital investment than dry fractionation[3].

The dry fractionation process is similar to a process known as suspension freeze concentration (SFC). SFC is a conventional way of freeze concentration (FC). FC has emerged as an interesting alternative in concentration field in terms of both the construction and operation of the equipment[4]. The principle of FC is based on the solidification phenomena of water. When a crystallized ice appears and grows from solution, the ice crystal expels impurities to build up pure crystal during freezing process. Thus the impurities are piled on the liquid phase to increase concentration of the mother liquor[5].

A typical SFC process is composed of three processing units which are ice nucleator, a recrystalliser and ice crystal separator. Ice nucleator normally used in SFC is scraped surface heat exchanger (SSHE) to generate ice nuclei and to maintain high heat transfer by scraping the ice layer formed. The limited size of ice crystals formed from ice crystal scraping need an additional step in order to increase the size of ice crystals thus increase the process complexity. Furthermore, due to the large surface area of small ice crystals, the product obtained is not highly pure and increase the difficulty to separate it from mother liquor[6]. Apart from that, SSHE that is normally used in SFC process is the most expensive type of heat exchanger, which is leading to high capital cost[7].

On the contrary, improved method of FC which is PFC is discovered by Matthews and Coggeshall in 1959, in which a single ice crystal is formed on the cooled surface[8]. PFC is applying the same concept with SFC but the major difference between these two methods is the size of ice crystal formed. The solution that needs to be concentrated flows over a cooled surface, which causes crystallization process, occurs on the surface. Further growth of ice crystal is producing ice crystal in layer form. The large size of ice crystal resulting in lower surface area and less impurities is trapped at the ice-liquid interface. The separation of ice occurs when concentrated solution is collected and flushed while the ice crystal adheres to the surface making the separation process easier [9]. Since the process is involving less unit operations, hence it is expected that the process to be much simpler and lowering initial investment compared with previous method.

The present paper aims to study and evaluate the performance of PFC process to separate olein from RBDPO. In particular, this technique has never been applied in RBDPO application. By applying FC concept where water components usually crystallise, in this research stearin at melting point 44-58°C will be crystallise instead and concentrated olein at melting point 22-24°C is obtained[2]. The potential of this process is investigated by determination of the following: K, IV, SMP and the percentage of olein yield. Moreover, this study also provides a new alternative which is simpler and cheaper for fractionation of palm oil.

2.0 EXPERIMENTAL

2.1 Material

RBDPO was used as a sample solution throughout the experimental work. Meanwhile, pure water used in water bath, functioning as a medium for cooling and heating process.

2.2 Equipment

The crystalliser is known as coil crystalliser (CC), which could provide higher productivity and efficiency for progressive freeze concentration process through its higher surface area. For this study, the CC was made of stainless steel, as illustrated in Figure 1, and it was used to carry out the separation process of RBDPO. The crystallizer has three layers or stages and also fitted with stainless steel flanges, thus the chamber could easily be split into two to collect the products from the process. In addition, nine temperature detectors, thermocouple type K, are engaged in each stage of the crystallizer in order to determine the temperature profile which later are displayed by Pico Log recorder software through connected computer.

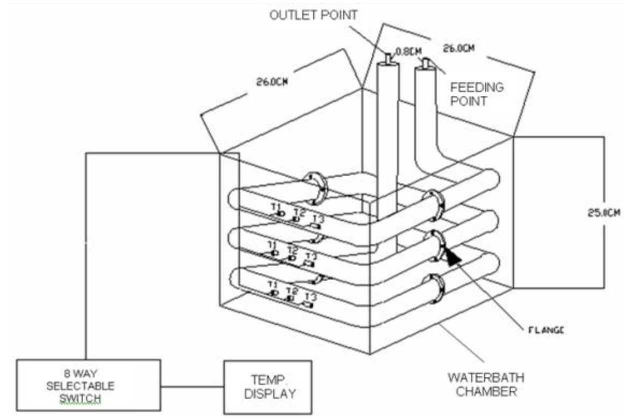


Figure 1 Coil crystalliser

2.3 Experimental Procedure

To start the operation, RBDPO obtained from a local palm oil refinery was pumped by a peristaltic pump into the CC until the crystalliser is completely filled. Then, it was immersed in a refrigerated water bath. The CC was connected to a peristaltic pump by a silicone tube and the RBDPO was circulated in the system for a period of an hour. Before starting the circulation at the desired temperature, the CC containing RBDPO must first be heated at a temperature of 70°C to avoid the presence of unwanted crystal and also to remove previous thermal history[10].

After that, the process proceeds to the designated reduced temperature, usually from 28°C until 24°C, which corresponds to the melting point of stearin and olein, which are about 48 °C and 20°C, respectively. The circulation flowrate of RBDPO of approximately 2800 mL/min in the CC would cause the stearin layer to be formed on the inner wall surface of the CC and leaving behind a more purified olein. The process was also carried out with constant initial iodine values of palm oil at 52.5 wijs as usually used in the refinery of palm oil.

Throughout the process, nine different points of the CC were fixed with thermocouples to measure the temperature of the coolant, RBDPO, and the wall of the crystallizer. The measured data were displayed on the computer after being detected by PicoLog recorder for easier monitoring process. After the desired temperature and period of time, the circulation was stopped and the purified olein was drained out, leaving behind stearin layer as shown in Figure 2, which has to be melted or thawed, and its purity was then analysed. Before starting the successive experiment, the CC was flushed with hot water. The temperature of the water bath was then increased to 48°C to melt stearin completely and detached it from the wall of the crystallizer. The experimental setup is shown in Figure 3.

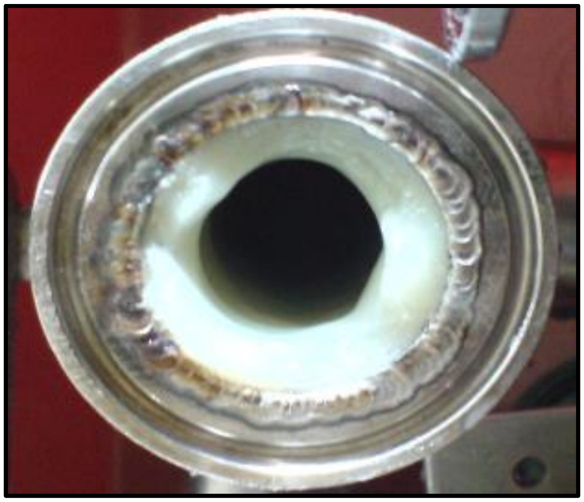


Figure 3 Close-up of stearin crystal layer formed

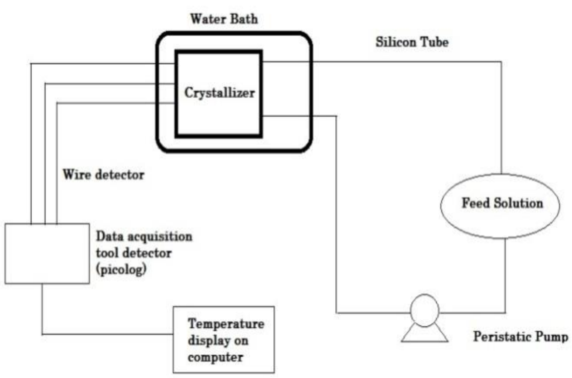


Figure 4 Experimental setup

3.0 RESULTS AND DISCUSSION

In this study, the quality of palm oil was determined by iodine value (IV) and slip melting point (SMP), while the efficiency of the PFC system was determined by yield and effective partition constant, *K*. The yield and *K* of the product could be influenced by the type of fractionation process, cooling rates and temperature of fractionation[11]. The quality of oil was measured based on the degree of unsaturation or double bonds presence in oils and fats, as indicated by the IV. It also reflects the ease of oxidation of oils and fats[12]. Meanwhile, SMP is an indicator used to measure the characteristic of melting and solidification properties of oils and fats. The chain length of fatty acids, trans fatty acid content, unsaturation ratios, and the position of fatty acids in glycerol backbone are changed depending on the value of SMP[13]. Other than that, the SMP of fat is an important attribute of many specifications trades of quality oil, and in some country, it is an element of the legal definition of food product[14].

3.1 Quality of Palm Oil

The quality of palm oil depends on the IV and SMP values of olein and stearin. The IV of olein and stearin were measured using MPOB Test Method p3.2-2004 and p4.2:2004[15], respectively. The p3.2-2004 method is technically equivalent to ISO 3961:1996, while MPOB Test Method p4.2-2004 originated from AOCS Official Method Cc3-25[16]. IV is calculated by the following Equation (1):

$$\text{IV}\left(\frac{\text{g}}{100\text{g}}\right) = \frac{12.69C\,(V_1 - V_2)}{m} \qquad (1)$$

where,
C= Concentration of sodium thiosulfate solution (mole L^{-1});
V_1= Volume (mL) of sodium thiosulfate solution for the blank test;
V_2= Volume (mL) of sodium thiosulfate solution for the sample;
m = Mass (g) of the sample.

The determination of IV is crucial as it gives the measurement of unsaturated oil and saponification value, which is used to obtain the average molecular weight of the constituent fatty acid. Lower fatty acid in the palm oil gives better quality and high IV olein[17]. Meanwhile, SMP is defined as the temperature of a column of fat in an opened capillary tube, which moves up when subjected to a controlled heating in water bath. Palm oil consists of fats with complex mixture of glycerides; therefore it does not have sharp melting points, unlike pure chemical substance.

Table 1 summarizes the statistical evaluation of IV and SMP in RBD palm olein and RBD palm stearin under the influence of coolant temperature. The data collected was a little bit different from the standard reference of dry fractionation, but it is still acceptable by referring to the trend when the data is plotted. Usually the standard reference data collected from Palm Oil Research Institute of Malaysia (PORIM) for IV olein are 56.0-57.0 wijs, IV stearin (33.0-48.0wijs), SMP olein (24.0-19.9°C), and SMP stearin (53.4-44°C)[14,18,19]. The less satisfactory data might be related to the final product that was not filter pressed. No filter press was employed because PFC is supposed to give the final product (olein) without engaging a filtration process.

Figures 4 and 5 show that IV and SMP for palm olein against coolant temperature. The trend for IV's data is contra with SMP's data for both olein and stearin. It is appears that, high coolant temperature gives high IV olein while low for IV stearin and antagonistic with SMP's result.

Although higher temperature contributed to lower IV for stearin, but AbLatip *et al.*[20] mentioned that the temperature range for their study was from 30 to 50°C, which means that it was out of range compared to this study and the lower temperature for this study was not anywhere near to the lower temperature used by AbLatip *et al* [2].

To make this reason more reliable and acceptable, according to Kawamura[21-22] and Berger[23], α crystals formed when the crystallization was conducted below 24°C and appeared as very unstable dotted spherulites under the microscope and its lifetime depends on the temperature, while β crystals, which appeared as very stable dendritic spherulites, were formed at temperature above 26°C.

Fractionation of RBDPO into olein and stearin is classified as fractional crystallization from the melt. Therefore, in order to develop crystalline phase into high growing crystals, a fast kinetic (nucleation and growth) effect is an important parameter in crystallization from the melt. The flow of slurry or RBDPO creates an optimal supersaturation and rates of nucleation and growth. It also enhanced the heat transfer between RBDPO and the cooling surface, and at the same time gives the optimal rates of crystal growth to produce a compact layer of suitable morphology and sufficient purity of stearin, thus achieving high IV[24].

Table 1 Operating conditions to investigate the effect of coolant temperature

Flow rate (mL/min)	Temperature (°C)	Time (min)	Initial iodine value (wijs)	IV (wijs)		SMP (°C)	
				Olein	Stearin	Olein	Stearin
2800	28	60	52.5	55.84	45.45	23.10	51.71
2800	27	60	52.5	55.63	45.95	23.40	50.78
2800	26	60	52.5	55.37	46.53	23.51	49.57
2800	25	60	52.5	55.00	47.32	24.00	48.45
2800	24	60	52.5	54.89	47.58	24.15	46.49

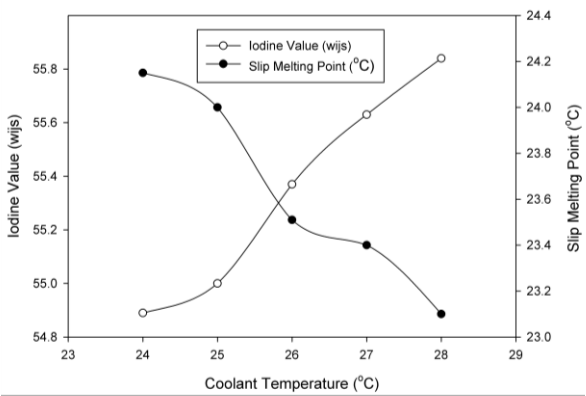


Figure 4 SMP and IV for olein at different coolant temperature

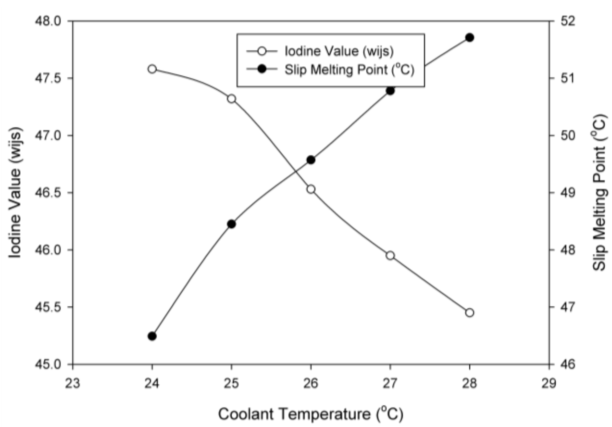


Figure 5 SMP and IV for stearin at different coolant temperature

Nevertheless, this process study used differs of equipment and the circumstantial of equipment distinguish the operating condition for coolant temperature. Therefore, if the temperature is too low used in this process, a lot of crystalline solid develop in the wall of crystallizer until the tube silicon cannot bear the pressure from inside of the crystallizer and buzz out. The problem face due to the small diameter of CC crystallizer in only 8mm and its too small to manage at too low temperature which is effect build of olein into solid phase. In comparison to the previous research which used dry fractionation, the results from PFC in this research are still highly acceptable in terms of IV and SMP[25].

3.2 Effect of Operating Condition on Yield and K

The quality palm oil and effective of the process of PFC is depending on the value of yield and effective partition constant, K. This correlation of yield and K are relying on coolant temperature. This research also more focus to the yield of olein rather than yield of stearin cause olein is the premium product for RBDPO fractionation.

The yield of palm oil is a highly efficient producer of vegetable oil and higher than any other vegetable oil crops[26]. Hence, it is significant to measure yield of olein in order to know the finest of effective and best quality for palm olein based on the manipulated operating condition of coolant temperature. The yields are calculated by using following Equation (2):

$$Yield = \frac{IV\ (palm\ oil) - IV\ (stearin)\ x\ 100}{IV\ (olein) - IV\ (stearin)} \quad (2)$$

Statistical evaluations for yield and K are tabulated in Table 2. Yield becomes higher when coolant temperature increased. This confirms the theory that the yield has a correlation with coolant temperature, where a better yield needs high range of temperature. The common yield of olein from dry fractionation or conventional fractionation using vacuum suction filter gives a yield of 65-68%[19]. Therefore, the results obtained from this study are completely acceptable.

In Figure 6, highest yield was observed at temperature of 28°C, thus is the best and the most suitable temperature for this process. In PFC system, the effective partition constant, K is a crucial indication of successful separation. From the K value, the performance of the PFC system can be evaluated. The values of K are calculated using the Equation (3) below:

$$K = \frac{C_S}{C_L} \quad (3)$$

It was found that K decreased with increasing coolant temperature, as illustrated in Figure 6. Higher growth rate and nucleation of stearin crystal is caused by low temperature but in this study range the coolant temperature is at high temperature (28°C) due to Malaysian palm olein has higher melting point at the range of 22-24°C[2], which means olein tends to crystallize at the temperature below that range and trapped together with palm stearin on the inner wall of CC. This situation makes the inner wall of CC covered with crystal solids and gives products of low quality and impurities, and led to loss of palm olein (i.e. low yield of olein)[27]. When the crystal growth rate is higher, more stearin would be trapped together and will strongly build the triglyceride (palmitic acid) bone in accordance to the different melting point. In this point, the temperature is importance in order to build strongly stearin but depend on the adjustment of temperature range with not to low and not too high.

Furthermore, olein in the mother liquor of RBDPO is prone to entrapment in the stearin crystal layer, thus increasing the concentration of stearin and produces higher K value. K value was found to be inversely proportional to the coolant temperature.

Table 2 Effect of circulation flowrate and coolant temperature on olein yield and K

Manipulated Operating Condition	Range	Yield	K
Coolant Temperature (°C)	28.0000	67.8537	0.2715
	27.0000	67.6653	0.2999
	26.0000	67.5339	0.3690
	25.0000	67.4479	0.5693
	24.0000	67.3051	0.6221

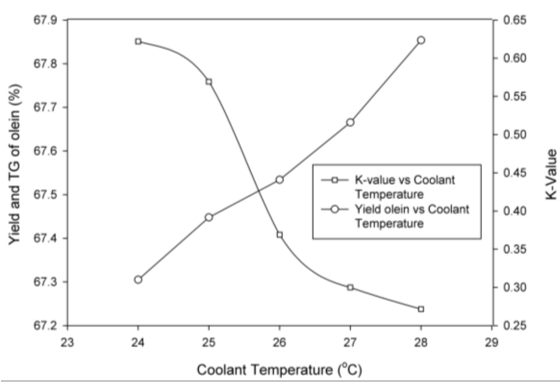


Figure 6 K-value and yield of olein at different coolant temperature

■4.0 CONCLUSION

Fractionation of RBDPO through PFC system is completely different from the conventional system, but the process still relies on the influence of coolant temperature. High quality olein was produced at the temperature of 28˚C while, higher percentage of olein yields at 67.85% and for K-value was obtained at 0.2715. For IV and SMP, the result conformed to the quality of palm based-standard reference, thus this process could serve as an attractive alternative for fractionation of RBDPO. All analyses support the objective of this study and give enough evidence for good performance, quality and productivity of olein in the PFC system.

Acknowledgement

The authors would like to thank UniversitiTeknologi Malaysia and Government of Malaysia for the financial and technical support in carrying out this research.

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