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Effect of Flowrate and Circulation Time on Fractionation of Refined Bleached and Deodorised Palm Oil using Progressive Freeze Concentration Method

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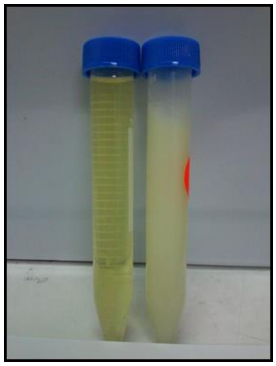

Abstract

This research concerns on the possibility of separating refined, bleached and deodorised palm oil (RBDPO) into olein and stearin using another alternative through Progressive Freeze Concentration (PFC) to replace the conventional process of dry fractionation. The process was carried out in stainless steel coil crystalliser and the quality of olein and stearin and the performance of crystalliser to purify the olein were analysed. The quality of oil was evaluated through iodine value (IV) and Slip Melting Point (SMP), while for the performance of PFC, effective partition constant, K, and yield of olein were used to determine the system efficiency. The purified olein were obtained for IV and SMP at higher flowrate (2800 mL/min) and longer period of time (60 min) are 55.87wijs and 23.04°C, 55.89wijs and 23.08°C, respectively. Meanwhile, the best K and yield of olein were obtained at higher flowrate and time, giving values of 0.2369, 67.99% and 0.238 and 67.93%, respectively.

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

Abstrak

Kajian ini tertumpu kepada kebolehan pemisahan bagi minyak kelapa sawit yang ditapis, diluntur dan dinyah bau (RBDPO) kepada olein dan stearin menggunakan alternatif lain melalui Pemekatan Pembekuan Progresif (PFC) untuk menggantikan proses pemisahan konvensional. Proses ini telah dijalankan di dalam penghablur gegelung keluli tahan karat dan kualiti olein dan stearin dan prestasi penghablur untuk ketulenan olein telah dianalisis. Kualiti minyak telah dinilai melalui Nilai Iodin (IV) danTakat Lebur Gelincir (SMP), manakala bagi pelaksanaan PFC, pemalar permisahan berkesan, K dan hasil olein telah digunakan untuk menentukan kecekapan sistem. Olein yang tulen diperolehi bagi IV dan SMP pada kadar aliran yang lebih tinggi (2800 mL/min) dan tempoh masa yang lebih lama (60 min) pada 55.87 wijs dan 23.04°C, 55.89 wijs dan 23.08°C. Sementara itu, K terbaik dan hasil olein telah diperolehi pada kadar aliran dan masa yang lebih tinggi memberikan nilai sehingga 0.2369, 67.99% dan 0.238 dan 67.93% masing-masing.

Kata kunci: Pemekatan pembekuan progresif; minyak kelapa sawit; minyak kelapa sawit yang ditapis; diluntur dan dinyah bau; nilai iodin; takat lebur gelincir; penghabluran

1.0 INTRODUCTION

Palm oil is one of the well-known edible oils worldwide besides coconut oil, palm kernel, or olive oil, which is used as raw material for food or non-food industries. Prior to consumption as edible oil, crude palm oil has to go through various processes including refining, bleaching, deodorizing and finally fractionation.[1] There are three types of fractionation, which are dry fractionation, detergent fractionation, and solvent fractionation. Among the three fractionation processes, dry fractionation is the most common process used, which involves crystallisation of olein at its freezing/melting temperature (20-24°C).[2] 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 fractionation required a greater capital investment than dry fractionation[3] and dry fractionation only requires crystalliser, filter and washing unit.

The fractionation process is more or less similar to a process known as suspension freeze concentration (SFC), which is a

process of aqueous solution concentration, where water molecules are crystallised out as a suspension in the mother liquor, whilst leaving behind a more concentrated solution. [3]SFC is a process based on melt crystallisation principle where substance with higher melting/freezing point in the mixture would be crystallised first by bringing down the temperature of the mixture/solution to the melting/freezing point while stirring. This will eventually leave behind the substance with lower melting/freezing temperature in the form of mother liquor and the substances of higher melting/freezing point in solid form.[4]

In this type of crystallization process, subsequent processes to obtain optimum separation of the crystals from the solution involve several unit operations including filters and washers, which add to the capital investment. Therefore, another alternative method called progressive freeze concentration (PFC) could provide a better alternative. PFC is also a process which uses the concept of melt crystallisation where the separation occurs based on melting point of crystal. The component with higher melting pointis firstly crystallised layer by layer from themixture and a single block of crystal will be produced at the end of the process.[5] Meanwhile, the component with lower melting point will remain in liquid form. Separation of crystals is then easier, just by draining off the resulted concentrate from the system. In fractionation of palm oil, the entity that would be crystallised is the stearin, which would leave behind a more purified olein.

Although the process is simpler, the problem faced by PFC is that it gives lower productivity. This research assess the performance of a relatively new design of crystalliser for PFC process in separating olein from refined, bleached and deodorized palm oil (RBDPO). In addition, it is a simpler and cheaper alternative for fractionation of palm oil.

■2.0 EXPERIMENTAL

The crystalliser is called a coil crystalliser (CC), that could provide higher productivity and efficiency for progressive freeze concentration process due toits higher surface area. For this study, the CC made of stainless steel as shown in Figure 1 and 2 was used to carry out the separation process of RBDPO. Before the real experiment was carried out, preliminary screening experiment was carried out to determine the suitable range of two parameters which are circulation flowrate and time. The range of these parameters was also considered based on the extensive reading from previous literature.

To start the operation, RBDPO obtained from a local palm oil refinery was pumped by a peristaltic pump into the CC. After the CC was filled with RBDPO; the CC was then immersed in a water bath. The CC was connected to a peristaltic pump via silicone tube and the RBDPO was circulated in the system for a period of time. Before circulating at the desired temperature, the CC which is full of RBDPO must firstly be heated at a temperature of 70°C to avoid the formation of unwanted crystal and to remove previous thermal history.[6]

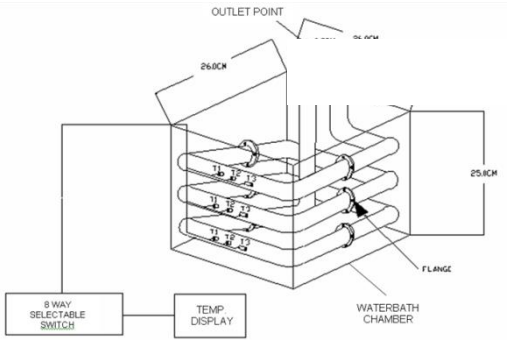

Figure 1 Coil crystallizer

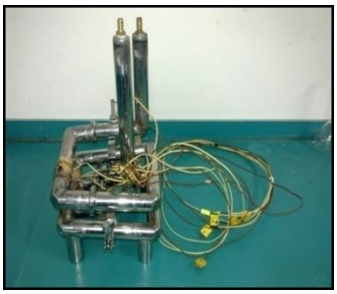

Figure 2 Stainless steel

After that, the process was proceeded to a designated analysis on the effect of circulation flowrate, in which the circulation flowrate were varied from 2000 ml/min to 2800 ml/min, while circulation time, initial IV and water temperature were fixed at 60 minutes, 52.5 wijs and 28°C, respectively. The fixed value of circulation time and water temperature were selected based on the best results obtained from the preliminary screening, meanwhile the standard initial IVof 52.5 wijs used in refinery industry of palm oil was chosen as the fixed value of initial IV for this study.[7] In addition, the melting point of olein and sterinare 48°C and 24°C, respectively.[2] The circulation flowrate of RBDPO from 2000 ml/min to2800 ml/min in CC would cause the formation of stearin layer at the inner wall surface of the CC and leaving behind a more purified olein. The process was also carried out at different circulation times ranging from 40 to 60 minutes and the other three parameters were fixed at circulation flowrate of 2800 ml/min, initial IV of 52.5 wijs and water temperature of 28°C.

Throughout the process, eight different points of the CC were equipped with thermocouples to measure the temperature of the water, the RBDPO and the CC wall. The measured data was logged to the computer via PicoLog recorder for easy process monitoring. After the desired temperature has been reached for a certain period of time, the circulation was stopped and the purified olein was drained out, which leaves behind the stearin layer as shown in Figure 3, which has to be melted or thawed and analysed for its purity. For the next experiment, the CC was flushed with hot water. The temperature of the waterbath then will be increased to 48°C to enable the stearin to melt and detach from the CC wall. The experimental set-up is shown in Figure 4.

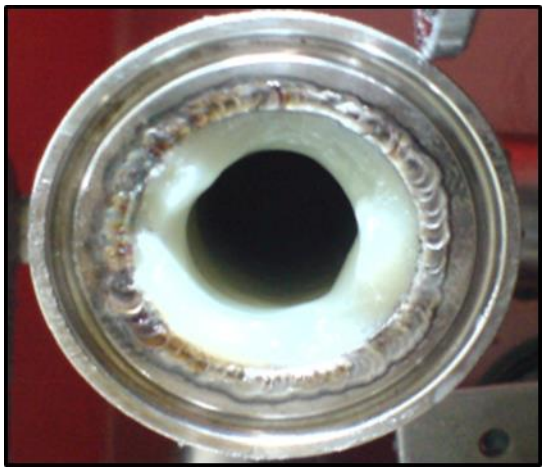

Figure 3 Close-up of stearin crystal layer formed

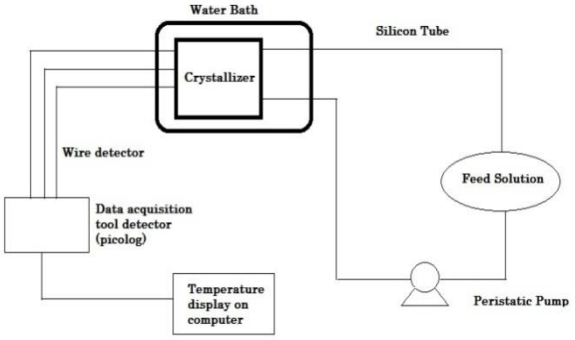

Figure 4 Experimental setup

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 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, temperature of fractionation,[6] circulation time and circulation flowrates. The quality of oil is measured based on the degree of unsaturation or double bond of oils and fats, as indicated by the IV. It also indicates the ease of oxidation of oils and fats.[8] Meanwhile, the SMP is an indicator to measure the characteristic of the melting and solidification properties of oils and fats. The chain length of fatty acids, trans fatty acid content, unsaturation rations, and the position fatty acids in glycerol backbone are changed relying on the value of SMP.[9] Other than that, the SMP of fat is an important attribute of many specification trade of quality oil and in some country it is an element of the legal definition of food product.[10]

A. Quality of Palm Oil

Quality of palm oil relies on the IV and SMP of olein and stearin. The IV olein and stearin were measured using MPOB Test Methods of p3.2-2004 and p4.2:2004,[11] respectively. The p3.2-2004 method is technically equivalent to ISO 3961:1996, while MPOB Test Method p4.2-2004 is originated from AOCS Official Method Cc3-25.[12]The IV is calculated by the following equation:

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

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; and

m= Mass(g) of the sample

The determination of IV is crucial to give a measurement of unsaturated oil, and saponification value, which will give a measure of the average molecular weight of the constituent fatty acid. The lower fatty acid in the palm oil gives better quality and high IV olein.[13] Meanwhile SMP is defined as the temperature of a column of fat in an opened capillary tube, which moves up when it is subjected to a controlled heating in waterbath. Palm oil consists of fats that have a complex mixture of glycerides, therefore does not have sharp melting points, unlike pure chemical substance.

All the graphs below show the results of IV and SMP in palm olein and palm stearin as affected by circulation flowrate and circulation time. The data collected is a little bit different from the standard reference of dry fractionation but is still acceptable according 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 is 56.0-57.0 wijs, IV stearin is 33.0-48.0 wijs, SMP olein is 24.0-19.9°C, and SMP stearin is 53.4-44°C.[7,10,14] The less satisfactory data might due to the fact that the final product was not filter pressed due to the high cost of the filter press and a multicomponent melt may fail to produce a pure crystalline product at a single stage. This phenomenon can be best described either by the incorporation of impurities into the growing crystals due to kinetic effect or an equilibrium distribution of impurities in the liquid and the crystalline phase (thermodynamic)[15] Hence, recrystallization step which could increase the purity of crystal is required in order to obtain a pure product. If the solubility of the crystal component decreases considerably with the decreasing temperature, a high yield can be expected.

In Figure 5 and Figure 6, IV and SMP for palm olein and stearin are plotted against circulation flowrate. The trend for IV data is in contrast with SMP data for both olein and stearin. It appears that, high circulation flowrate gives high olein IV and low stearinIV, which opposes the SMP result where high circulation flowrate gives low SMP of olein and high SMP of stearin. The crystallization of fat is a slow process due to the high viscosity of liquid fat and relatively a large size of molecule, so the process needs at high flowrate in order to make a pure crystalline product.

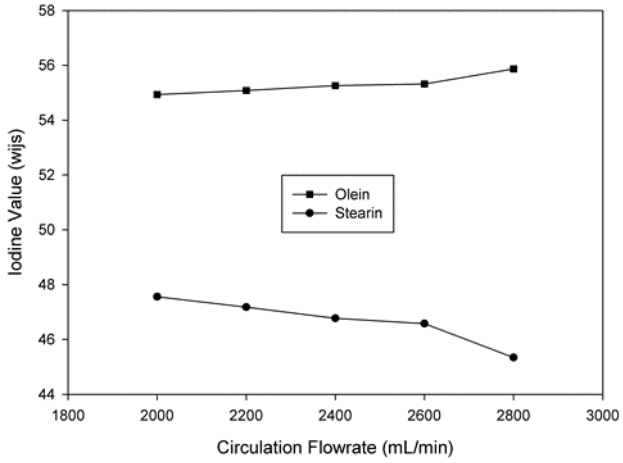

Figure 5 Iodine value for olein and stearin versus circulation flowrate

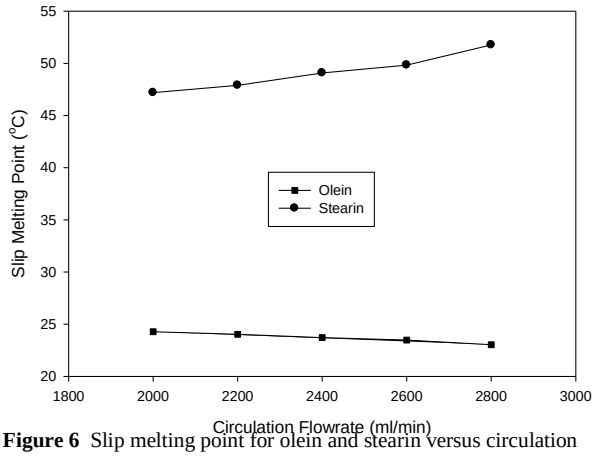

Figure 6 Slip melting point for olein and stearin versus circulation flowrate

Although the process of this fractionation needs a high flowrate, the higher flowrate of 3000ml/min and above would give insufficient result of olein and stearin due to entrapment of olein in the stearin crystal layer and this would dramatically break down the crystal of stearin aggregates according to the preliminary experiments carried out.

Fractionation of RBDPO into olein and stearin is classified as fractional crystallization from the melt. Therefore, to develop crystalline phase into the high growing crystals, a fast kinetic (nucleation and growth) effect is a vital role as 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 enhances the heat transfer between the RBDPO and the cooling surface, while giving the optimal rates of crystal growth and producing a compact layer of suitable morphology and sufficient purified of olein and thus achieving high IV.[15] However, it is necessary to handle the flowrate at only certain desired flowrate and not too high due to small diameter of the crystallizer only about 0.8 mm. The liquid olein might be trapped in the crystal layer of stearin, which could result in less pure stearin layer.

A long circulation time is needed to completely build the crystal layer of stearin, which would result in a complete separation of olein and stearin. Circulation time of 60 minutes gives high IV for olein and low for stearin of55.89 wijs and 23.1 wijs 45.32°C, respectively, as illustrated in Figure 7. Meanwhile in Figure 8, it is shown that circulation time of 60 minutes gives a low value of SMP for olein and high value of SMP for stearin of 45.32°C and 51.63°C, respectively. Besides, it appears that low circulation time results in a low IV olein, low SMP stearin, high IV stearin and high SMP olein. This is in agreement with the findings from previous researchers, where it was found that for palm olein, it is evident that the short tempering period does not always allow adequate crystallization of RBDPO. It was also mentioned that 1 hour is proven to be insufficient but 8 hours or more is adequate for complete crystallization.[11]

Nevertheless, this studied process is using a completely different equipment and the circumstantial of equipment distinguishes the operating condition for circulation or tempering time. Therefore, if the longer circulation time is used in this process, more crystalline solid will be developed on the inner wall surface of the crystalliser.

Compared 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.[16]

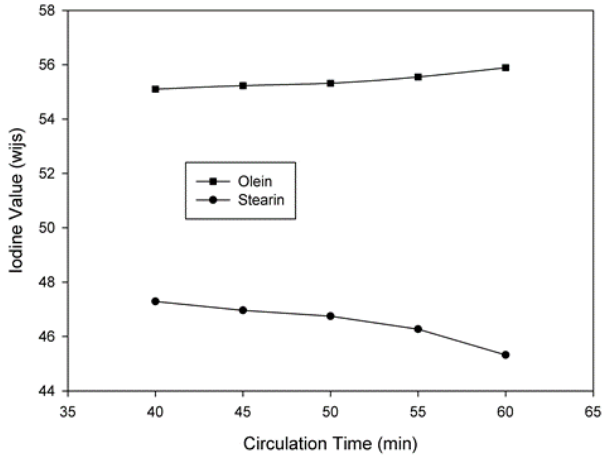

Figure 7 Iodine value for olein and stearin versus circulation time

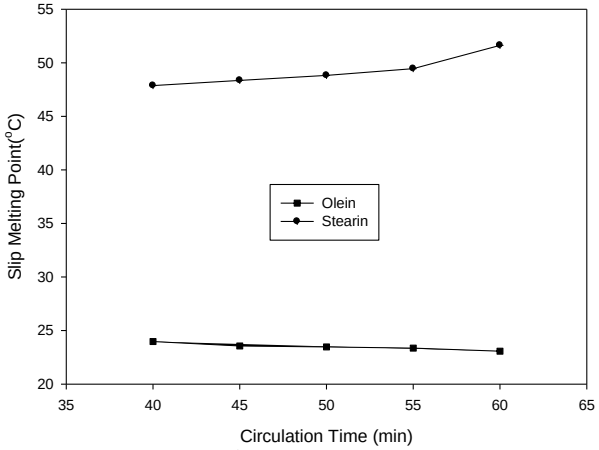

Figure 8 Slip melting point for olein and stearin versus circulation time

B. *Effect of Operating Condition on Yield and K*

The quality of palm oil and effectiveness of the process of PFC depends on the value of yield and effective partition constant, K. This correlation of yield and K relies on circulation flowrate and circulation of time. This research also focuses on the yield of olein rather than yield of stearin because olein is the premium product of RBDPO fractionation.

Palm oil is a highly efficient producer of vegetable oil compared to other vegetable oil crops.[17] Hence, it is significant to measure the yield of olein in order to know the finest and best quality palm olein based on the manipulated operating condition of circulation flowrate and time. The yield is calculated by using the following equation:

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

Results for yield and K against circulation flowrate and circulation time are plotted in the graphs below. Figures 9 and 10 show that the yield becomes higher when circulation flowrate and circulation time are increased. This obeys the theory which indicates that the yield has a correlation with flowrate and time, where a better yield needs a high range of flowrate and long operation time. The common yield of olein from dry fractionation or conventional fractionation using vacuum suction filter gives a yield of 65-68%.[18] Therefore, the results obtained from this study are completely acceptable.

The speed of RBDPO going through the CC during operation will affect the end product, if the speed is too slow, the slurry or stearin crystal layer will become hard and brittle as the fat is cooled rapidly when it touches the wall of the crystalliser. Meanwhile, if it is too fast, the crystals would attach to each other instead of orientating themselves in the right position.[19] On the other hand, too high or too slow of a feeding and circulation flowrate gives insufficient cooling, promoting post-crystallization, weak triglyceride backbone or hardening.[20]

In Figure 9, high yield is shown at flowrate of 2800 ml/min, thus is the best and the most suitable flowrate for this process. Meanwhile from Figure 10, it is evident that the higher yield (67.92%) is obtained at 60 minutes of circulation time. Similar to circulation flowrate, in order to get high yield of olein, longer contact time is required.

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[21] below:

$$K=1-[(\log(C_o/C_L))/(\log(V_L/V_o))] \quad (3)$$

where C_o is concentration of the initial solution of RBDPO, C_L is concentration of the purified olein, V_o is the initial volume of RBDPO, and V_L is the volume of purified liquid (olein). But before calculation, firstly the concentration of solute is calculated based on the percentage of C18:1 (oleic acid), which is commonly high in palm olein.

It is found that K decreases with increasing circulation flowrate and time as illustrated in Figure 9 and Figure 10. The best performance is at flowrate of 2800 ml/min and 60 minutes circulation time. Higher growth rate and nucleation of stearin crystal is caused by high flowrate and long circulation time which is undesirable to produce a low K value for this system. The higher the crystal growth rate, the more stearin would be trapped together and will strongly build the triglyceride (palmitic acid) bone according to the different melting point.

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 is found to be inversely proportional to the circulation flowrate and circulation time.

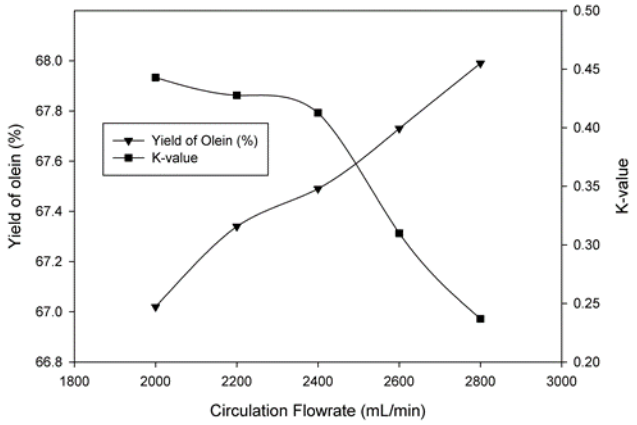

Figure 9 Yieldof olein and K-value against circulation flowrate

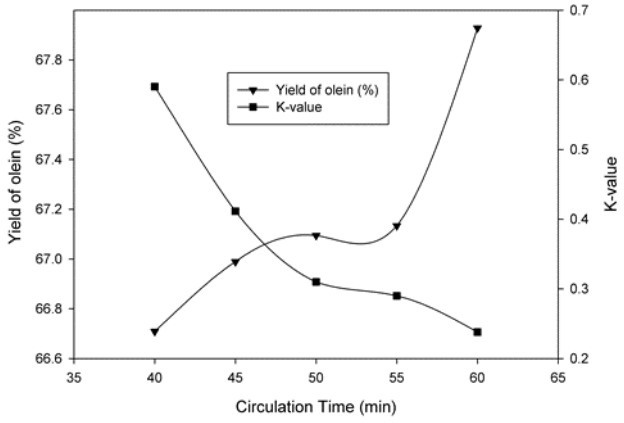

Figure 10 Yieldof olein and K-value against circulation time

■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 circulation flowrate and circulation time which possibly can produce the best quality at the flowrate of 2800ml/min and circulation time of 60 minutes. Meanwhile, higher percentage of yield at 66-68% is obtained for both operating parameter. For IV and SMP, the result obeys the quality of palm based-standard reference, thus could serve as an attractive alternative for fractionation of RBDPO.

Acknowledgement

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