การแยกกรดไขมันอิสระที่สภาวะสุญญากาศจากน้ำมันปาล์มดิบกรดสูง
Vacuum separationof free fatty acids from high-free fatty acidscrude palm oil

Nantanit Tohpong[1]
Wiriya Duangsuwan[2]E-mail: wiriya.dua@gmail.com,
Poonsuk Prasertsan[3]E-mail:Email: poonsuk.p@psu.ac.th
[1]Master student in Biotechnology Program, Department of Industrial Biotechnology,
Faculty of Agro-Industry, Prince of Songkla University, Songkhla 90112 Thailand
Email: nantanit.tp@gmail.com
[2,3]Department of Industrial Biotechnology, Faculty of Agro-Industry,
Prince of Songkla University, Songkhla 90112 Thailand

Abstract

The high-free fatty acids crude palm oil (H-FFAs CPO) usedin this investigation mainly contained palmitic (42.07%), oleic (38.86%), linoleic (7.98%) and stearic (3.95%). Three 7.2-hour batches of vacuum distillation of 28-L H-FFAs CPO in a 40-L reactor were performed. For batch 1, 2 and 3, the beginning and setting temperatures were 27.4 and 230°C; 29.5 and 250°C; 30.5 and 250°C, respectively. The vacuum pressures were20, 160 and 30 mmHg, respectively. The original and final FFAs content in the CPO were 25.70 and 14.34%; 14.95 and 10.60%; and 10.29 and 5.22%, respectively. The FFAs profile of the CPO before and after the process, and of the FFAs obtained from every batch were also reported. The FFAs in the CPO could be reduced from 25% to 5% within 15 hours for 1 batch of the propose reactorat temperature of 230°C and pressure of 20 mmHg.

Keywords: crude palm oil, separation, free fatty acid

บทคัดย่อ

น้ำมันปาล์มดิบที่มีกรดไขมันอิสระสูงประกอบไปด้วยกรดไขมันหลักคือกรดปาล์มิติก 42%, โอเลอิก 38.86%, ลิโนเลอิก 7.98% และสเตียริก 3.95% ทดลองให้ความร้อนภายใต้สภาวะสุญญากาศในถังปฏิกรณ์ขนาด 40 ลิตรจำนวน 3 กะ แต่ละกะใช้ตัวอย่างน้ำมันปาล์มดิบ 28 L ระยะเวลาทดลองเฉลี่ย 7.2ชั่ วโมงต่อกะ สำหรับกะที่ 1, 2 และ 3 อุณหภูมิเริ่มต้นและอุณหภูมิระเหยของน้ำมันปาล์มดิบที่ใช้คือ 27.4 และ 230°C; 29.5 และ 250°C; 30.5 และ 250°C ตามลำดับ ค่าความดันสุญญากาศที่ใช้คือ 20, 160 และ 30 mmHg ตามลำดับ ปริมาณกรดไขมันอิสระเริ่มต้นและสุดท้ายในน้ำมันปาล์มดิบในแต่ละกะเท่ากับ 25.70 และ 14.34%; 14.95 และ 10.60% ; 10.29 และ 5.22% ตามลำดับ ชนิดและสัดส่วนของกรดไขมันในน้ำมันปาล์มดิบก่อนและหลังการทดลอง และในกรดไขมันอิสระที่แยกได้ ได้รายงานไว้ในผลการทดลองในภาพรวมหากใช้ถังปฏิกรณ์ชุดนี้ปริมาณกรดไขมันอิสระในน้ำมันปาล์มดิบสามารถลดลงจาก 25% เป็น 5% ภายใน 15 ชั่วโมง ที่อุณหภูมิ 230 °C และที่ความดัน 20 mmHg

Introduction

The free fatty acid (FFA) content is a significantrequirement that affects the quality of crude palm oil (CPO).Separation of FFA from high-free fatty acids crude palm oil (H-FFA CPO) will cause the CPO have good quality. FFA can be removed by various methods such as using vacuum distillation (Molekul, 2016), polyvinyl alcohol (PVA)-cross-linked polyvinylidene fluoride (PVDF) membrane (Azmi *et al.*, 2015),andenzyme (Saparin *et al.*, 2016). Vacuum distillation of FFA in a film evaporator is an effective method for simultaneously decreasing the FFA and water contents in high-FFA CPO (Cvengros and Cvengrosova, 2004).In addition, vacuum distillation technique has more advantages that it can keep the evaporating temperature to be minimum to avoid destruction of raw material (Shao *et al*., 2007), and the product FFA can be used for further applications(Molekul, 2016).

Objective

The objective of this research work is to separate FFAs from H-FFA CPO for using as raw material for production of glycerides. The proposed reactor will be investigated to find if it can separate FFAs at the desired operating time, temperature and vacuum pressure. Also, the separation efficiency will be obtained.

Research Scope

This workused a 40-L reactor which was designed and built by the research team. The working volume was 28 L. The reactor was heated by the electric heater and the vacuum pressure was obtained via the vacuum pump. The H-FFA CPO without any pretreatment, such as bleaching and degumming, was heated under vacuum condition and the vapor was condensed via the water-cooled condenser. The collected products were analyzed for FFA profile.

Literature Review

FFA can be removed by various methods.Molekul*et al*. **(2016)**did the experiment of molecular distillation at evaporation temperature 150-265°C and pressure 10 mmHg (10 torr) to reduce FFA in CPO from 9.4%-0.9%.Azmi *et al*. **(2015)**used polyvinylidene fluoride (PVDF) hollow fiber crosslinked with 100 ppm polyvinyl alcohol (PVA) to exhibit the highest FFA rejection of 5.93% (the remaining FFA in the permeate is about 5.93% compared with the feed) after 3 hours at feed temperature of CPO 65°C and pressure 2 bar. Saparin *et al*. **(2016)**lowered the FFA level in high FFA CPO by lipase *Candida Antartica* enzyme with the presence of glycerol under 100 mbar vacuum in the rotary evaporator at 260 rpm and found the FFA reduction was about about 71-88% at 2% glycerol and 46-75% at 1% glycerol.

The molecular distillation is efficient for reducing FFA and the product FFA is obtained for further application such as bio-lubricant and bio-plastic production(Molekul,

2016), whereas more efforts are needed to put into further improve the efficiency of FFA removal using membrane-based technology(Azmi *et al.*, 2015). In addition, reducing FFA using lipase *Candida Antartica* enzyme with glycerol have found a drawback as the increase of partial glycerides in the CPO(Saparin *et al.*, 2016).

Methodology

(1) Experimental setup

The experimental setup was shown schematically in Fig. 1. A batch reactor consisted of a 40-L stainless steel cylindrical evaporation tankequipped with an electric heater and vacuumed using a vacuum pump. There were a first condenser connected directly on the top of the evaporator and the second condenser connected separately via flexible hoses between the first condenser and the vacuum pump. The cooling tanks together with the cooling water pump were connected to both condensers via flexible hoses.

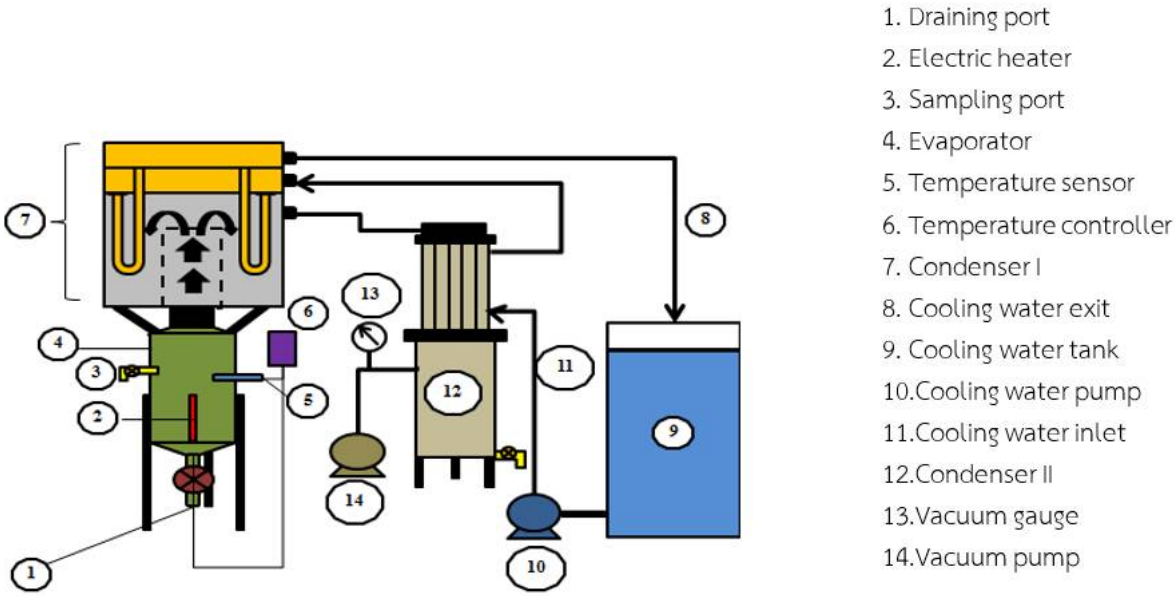

Fig.1 Schematic diagram of a reactor for separating FFA from H-FFAs CPO

The experiment of each batch began with pouring 28 liters of H-FFAs CPO into the evaporator. Then the first condenser was installed on the top of the evaporator. All hoses were connected according to Fig. 1. Ice was put into the cooling tank to reduce the temperature of cooling water to be around 13-20°C. The vacuum pump was turned on until reaching the desired pressure and then the heater was turned on to heat the oil. The oil temperature gradually climbed to reach the desired temperature. After the setting temperature was achieved, the reactor was continued running further for 4 hours before

stopping and turned off all equipment. Samples were collected, temperature and pressure were recorded at the beginning and every 20 minutes during the time from beginning to reach thedesired temperature, and every 30 minutes from reaching the desired temperature until the end of experiment. Samples of CPO collected from the evaporator and distillates collected from the condenser after the experiments were analyzed for FFA content, FA profiles and moisture content. The feed and products were also weighted for doing mass balance. Electrical power consumption was calculated and compared.

(2) Raw material

The high-FFA CPO used in this work was obtained from the local palm oil mill in Songkhla province, Thailand.

(3) Analysis

FFA content based on palmitic acid was determined according to standard methods as described in [6]. Fatty acid compositions of all samples were analyzed using an Agilent 6890 series gas chromatograph (GC) with a flame ionization detector (FID) and a capillary column (30 x 0.25 x 0.25 mm). The FID was set at 240°C with a flow rate of 0.8 ml/min. The injector temperature was also set at 240°C. Hydrogen was used as the carrier gas. The peaks were identified by measuring the retention time of the samples and comparing them with reliable standards analyzed under the same conditions. The percentage of each fatty acid was the relative peak area of a fatty acid species to the total peak area of all the fatty acids in the CPO or distillates samples.

Results and Discussion

(1) Temperature and Pressure of the Reactor During Investigation

The initial and setting temperaturesof the H-FFAs CPOand the setting vacuum pressuresinside the evaporator and condenser of the three batches were shown in Table1. The CPO showed semi-solid state at the beginning of heating due to their initial temperatures, which were 27.4, 29.5 and 30.5°C respectively for batch 1, 2 and 3, were not high enoughto make them totally become a liquid form. Asindicatedin [7] that melting point of palm oil was 34.2°C. In the same way, reported by [8] that CPO mainly contained palmitic acid (44%), oleic acid (39.1%), linoleic acid (10.1%), stearic acid (4.5%) and myristic (1.1%) which their melting points were 62.9, 16.3, -6.5, 70.1 and 54.4°C, respectively. Therefore, at room temperature, solid form of CPO mainly due to palmitic, stearic and myristic, whereas liquid form mainly due to oleic and linoleic.

In case of workingtemperature and pressure of CPO, the three batches were controlled to work at temperatures of 230, 250 and 250°C, respectively, and vacuum pressures of 20, 160 and 30 mmHg, respectively. However, the CPO had to be heated up from the beginning temperatures to those setting temperatures for each workingpressure.

Fig. 2 showed the time courses of temperatures and vacuum pressures for three batches. It can be seen from Fig. 2 that this reactor took about three hours to heat the CPO up to the desired temperatures. The boiling points of palmitic acid, oleic acid, linoleic acid, stearic acid and myristic at pressure 10 mmHg were reported in [8] to be 212, 223, 224, 227 and 193℃, respectively. In this present work, the minimum working pressure of 20 mmHg was achieved, which was limited by the ability of vacuum pump together with the leakage resistance of the gasget at the condenser. Therefore, this investigation had to use the oil temperatures to be higher than the above boiling points of fatty acid.

Table 1 The temperature of oil, vacuum pressure and free fatty acid content of each batch.

Batch	Temperature of H-FFAs CPO in evaporator (°C)		Setting vacuum pressure in evaporator and condenser (mmHg)
	initial	setting	
1	27.4	230.0	20
2	29.5	250.0	160
3	30.5	250.0	30

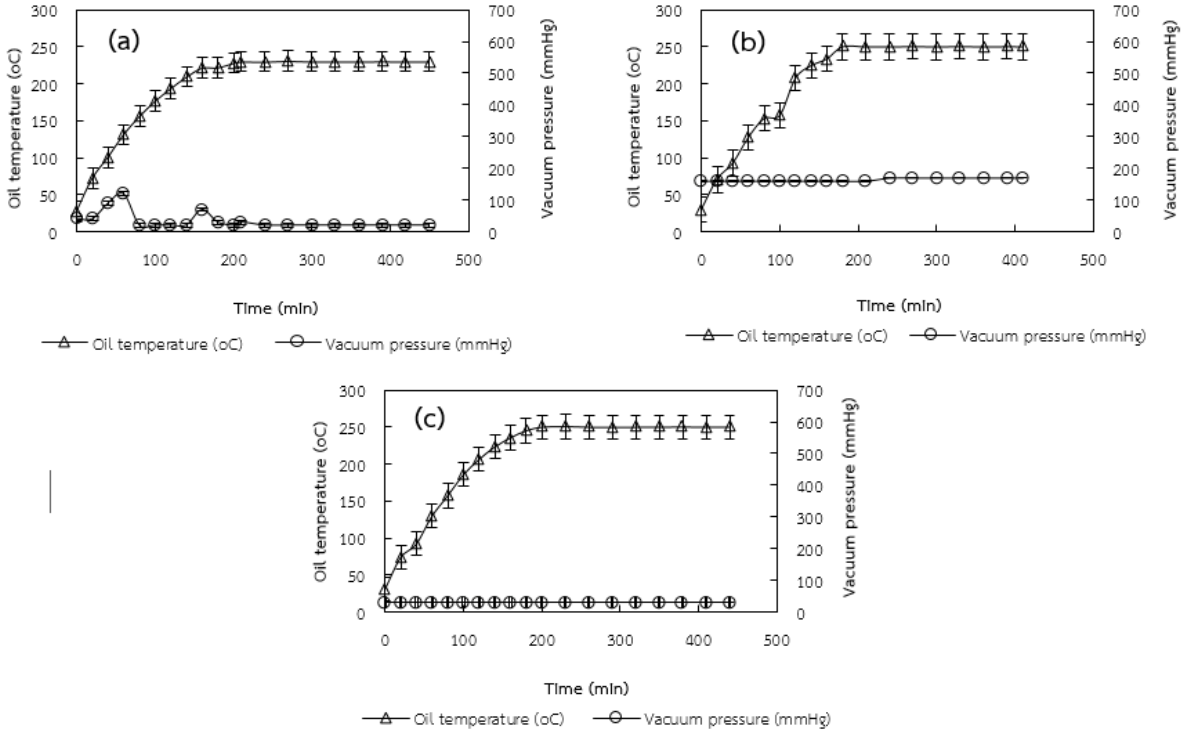

Fig. 2 Profiles of oil temperature and vacuum pressure during separation of free fatty acid from H-FFAs CPO. (a) Batch 1; (b) Batch 2; (c) Batch 3

(2) Fatty Acid Profiles of the High-FFA CPO and the Distillates

The fatty acid compositions in the high-FFA CPO both before and after experiment, and in the distillates collected from the evaporator were shown in Fig. 3For batch 1, the CPO before and after experiment mainly contained palmitic acid (42.07% and 42.21%), oleic acid(38.86% and 39.56%), linoleic acid (7.98% and 7.81%), stearic acid (3.95% and 4.02%), myristic acid (0.92% and 0.91%) and lauric acid (0.23% and 0.23%), respectively.For batch 2, the CPO before and after experiment mainly contained palmitic acid (41.86% and 41.72%), oleic acid(41.10% and 41.88%), linoleic acid (8.16% and 7.61%), stearic acid (4.20% and 4.19%), myristic acid (0.96% and 0.96%) and lauric acid (0.28% and 0.26%), respectively. And for batch 3, the CPO before and after experiment mainly contained palmitic acid (41.18% and 43.24%), oleic acid(42.27% and 40.22%), linoleic acid (8.57% and 7.62%), stearic acid (4.21% and 4.41%), myristic acid (0.96% and 0.96%) and lauric acid (0.26% and 0.25%), respectively.

The distillates obtained from batch 1, 2 and 3 contained FFA of 73.46%, 76.7% and 89.28%, respectively. They were mainly composted of palmitic acid (56.86%, 55.92%, 55.79%), oleic acid(23.60%, 28.81%, 26.19%), linoleic acid (4.67%, 4.98%, 4.87%), stearic acid (2.02%, 2.79%, 2.60%), myristic acid (3.45%, 2.67%, 3.11%) and lauric acid (2.17%, 1.35%, 1.94%), respectively**.** However, about 10 to 25% of the compositions in the distillates were not FFA, which could be the oil themselves. Thus, the results of fatty acid profiles could be the fatty acids from the oil and the FFA.

The initial and final FFA contents in the CPO were 25.70% and 14.34%, respectively for batch 1; 14.96% and 10.61%, respectively for batch 2; and 10.29.70% and 5.22%, respectively for batch 3. The FFA removal between three batches can be compared obviously as in Fig. 4. The FFA content in the CPO at the end of batch 1 and at the beginning of batch 2 were similar. Likewise, the FFA content at the end of batch 2 was similar to those at the beginning of batch 3. This can be implied that the reactor can reduce FFA from around 25% to be 5% if continuous running for 15 hours in one batch was performed

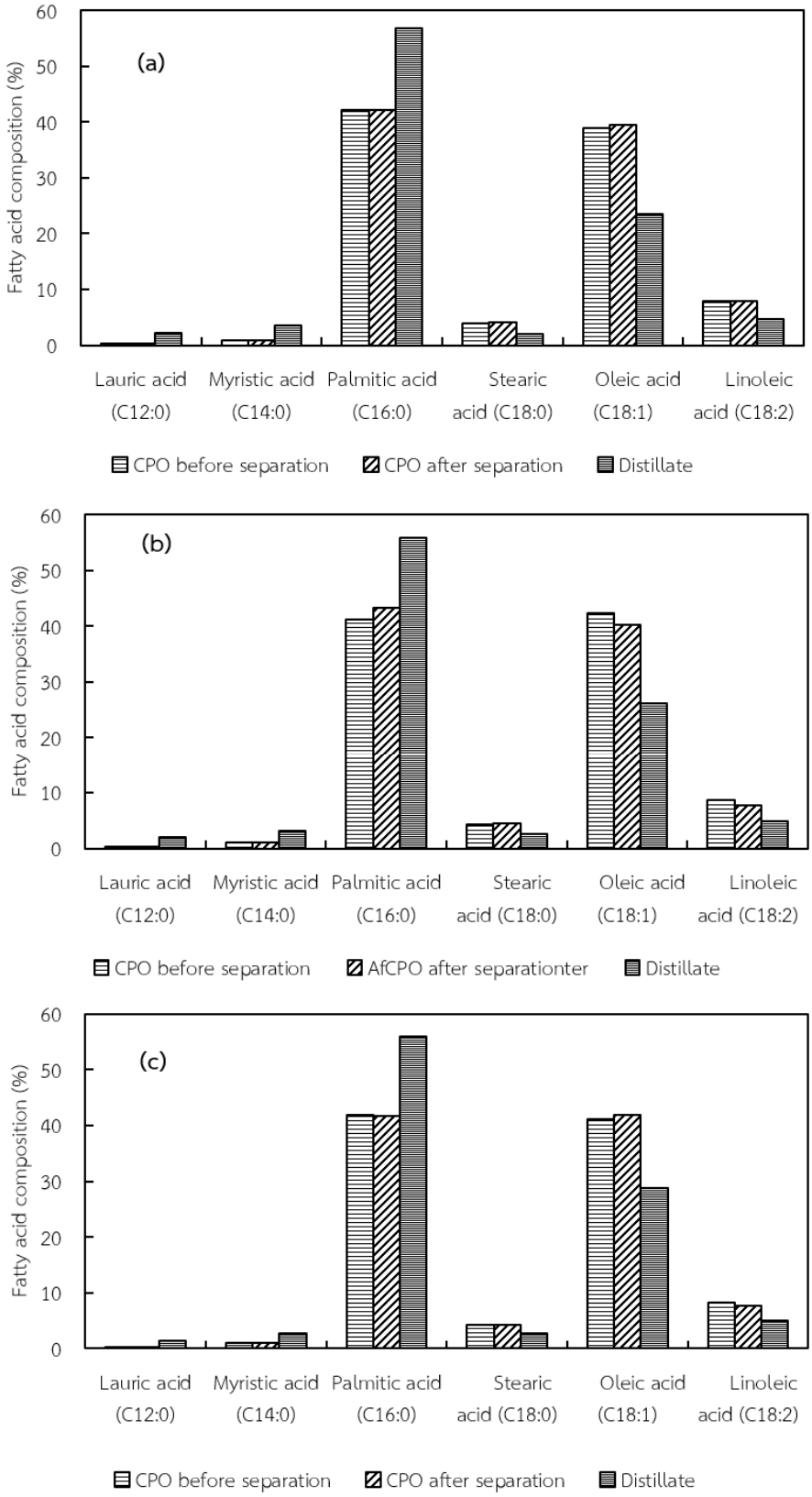

Fig. 3The fatty acid compositions in the H-FFA CPO both before and after experiment, and in the distillates collected. (a) Batch 1; (b) Batch 2; (c) Batch 3.

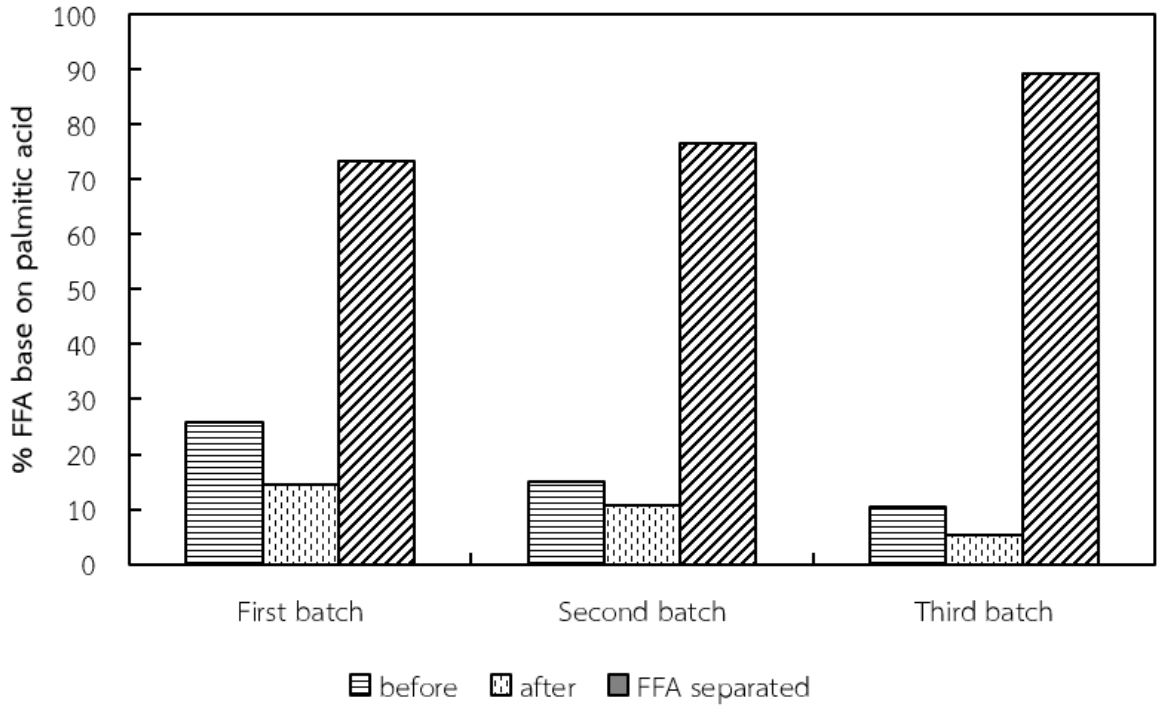

Fig. 4 Free fatty acid contents of high-FFA CPO before and after experiments, and distillate collected from experiment.

Table 2.Showed the efficiency of the condenser to trap the distillates. Although batch 1, 2 and 3 respectively lost the mass of CPO about 0.65, 0.77 and 0.54 kg at the evaporator, the condenser could collect just 8.36, 11.11 and 19.13% of the lost mass, respectively. More than 80 wt.% of vapor evaporated from the evaporator lost to the surround air. This reactor consumed about 26 kW-hr for one batch. . Further efficiency improvement for both the evaporator and condenser will be done in the future.

Table 2 Recovery efficiencyand energy consumption of 3 batches of experiment.

Batch	CPO (kg)			FFA obtained from condenser (kg)	Recovery efficiency (%)	Energy consumption (kW-hr)
	Initial	Final	Lost			
1	25.50	24.85	0.65	0.05433	8.36	27.15
2	25.45	24.68	0.77	0.08558	11.11	24.62
3	24.92	24.38	0.54	0.10330	19.13	26.45

Conclusion

The proposed work showed the feasibility of reducing FFAs in the crude palm oil by means of heating under vacuum pressure using a batch reactor. The FFAs in the H-FFAs

CPOcould be reduced from 25% to be less than 5% within 15 hours for 1 batch under the minimum temperature of 230°C and the maximum pressure of 20mmHg. However, improvement of separation efficiency of the reactor and development of continuous process will be a future work. This work is a project continued from the project of oil separation from palm oil mill effluent. The FFAs obtaining from separation process can be a raw material for various applications such as production of glycerides, biodiesel, biolubricants, bioplastics, cosmetics, animal feed, soap and detergent.

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