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Processing of red palm oil by modified acid degumming method

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Abstract - Red palm oil (RPO) is a high carotenoids product that resulted from crude palm oil (CPO). The conventional process of RPO includes degumming, neutralization, deodorization, and fractionation processes. This research aimed to develop the simple and economical acid degumming method for processing of red palm oil. 85% phosphoric and 99.5% citric acid were used and the factors used in the study were acid (phosphoric:citric acid 0.06:0.04,0.08:0.02,0.10:0 % w/w), temperature (90, 100, 110 °C) and time (20, 25, 30 minutes). The sample was stirred at 200 rpm. In addition, the neutralization process was further studied. The results indicated that the optimal condition for degumming was 90 °C for 20 minutes and the optimum ratio of acid to separate the gum is 0.06% phosphoric acid (w/w) and 0.04% citric acid (w/w) citric acid. The sedimented gum was separated from the oil by washing the oil with water at 60 °C. After degumming the oil contained 82.70±6.49% yield carotenoids, 3.50±0.55% free fatty acid (FFA), 3.01±0.73 umol Trolox eq/g DPPH-free radical scavenging activities and 4.43±0.05 umol Trolox eq/g FRAP-free radical scavenging activities. The free fatty acids and phosphorus content were separated by NaOH at 80 °C for 30 min with constant stirring. Soap from the saponification reaction and excess NaOH were washed out with water at 60 °C. After neutralization, the oil contained 0.46±0.05% fatty acids, 637.60±8.64 mg/kg carotenoids, 74.48±9.39% oil yield, < 0.1 mg/kg iron, the copper and phosphorus contents were not detected.

Keywords: Crude palm oil, acid degumming, red palm oil

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1. Introduction

Red palm oil (RPO) is a high carotenoids product that resulted from the refining and fractionation process of CPO. The CPO contained 3.10±0.14% FFA, 639.90±5.30 mg/kg carotenoids, 111 mg/kg phosphorus, 20.27 mg/kg iron, and 55.56 mg/kg copper. RPO has been used in food, health, and skin care productions (Nagendran *et al.*, 2000; Chaijan & Panpipat, 2021). The conventional process of RPO includes degumming, neutralization, deodorization, and fractionation processes. CPO contains gums, which comprise of several compounds such as phospholipid, glycolipid, sugar, protein, and water-soluble compounds (Rinćon *et al.*, 2020) Gums develop off flavor and darkening in oil (Jiang *et al.,* 2015) and cause dirty to the equipment (Sulihatimarsyila *et al.*, 2019). The conventional degumming using a specified quantity of acids, and the sediments were removed by centrifugation.

Yields of carotenoids decrease in response to temperature and heating time because heat enhance the decomposition of carotenoids molecule, as well as increase conversion *trans* to *cis* isomers (Bonnie & Choo, 1999). Azis *et al.*(2016) reported that deodorization at 140 ^{o}C for 1 hour under the vacuum distillation system is recommended to produce RPO with high carotene retention. Mayamol *et al.* (2007) reported that at 150 ^{o}C there is a faster rate of destruction of carotenes in red palm olein. Heat treatment also leads to lipid oxidation which yields free fatty acids (Lee *et al.*, 2007).

The acids solution was used to remove the non-hydratable phosphatides by changing it to the hydratable form (Dijkstra, 2017). The acid treatment with phosphoric acid is able to convert the non-hydratable phosphatides to hydratable ones better than using citric acid (Szydlowska-Czerniak, 2007). However, phosphoric acid increases the phosphoric content of the oil. Thus, mixing phosphoric acid and citric acid is usually preferable (Shahidi, 2005). Thus, the mixing ratios of acids should be paid attention. For the acid to react with the non-hydratable phosphatides, vigorous stirring was applied to generate a very fine dispersion especially when the reaction was almost completed and a very low residual phosphatide content was reached. Also, the water/oil dispersion was not stable as the acid droplets tended to coalesce and the interface was decrease, leading to more diffusion distances and slow reaction. The principle of water washing process has been reported (Pratik & Surekha, 2018). The warm water was sprayed into the oil to provide such an oil/water interface that the hydratable phosphatides were able to fully hydrate and move into the water phase (Szydlowska-Czerniak, 2007). In the separation stage, as the degumming solution used acids that dissolved in water (Pratik & Surekha, 2018), water washing was applied continuously until pH of the washing water got back to normal or before adding acids and the gum residue could not notice in the washing water. The excess washings leading to increase oil loss and waste water, as well as degumming costs. Our preliminary study found that rinsing with water for 6 times was able to bring pH of the water back to normal level. In many instances, gum might remain unnoticed, but the remaining gum can be further eliminated in the neutralization step (Shahidi, 2005).

In this research, the modified acid degumming treatment that could keep

carotenoids loss to the minimum and optimize the use of chemical reagents, temperatures and times was studied. The simple and economical sediment removing technique obtained from this study would be suitable for SMEs, which are important sector and currently have a high number in Thailand.

2. Materials and methods

2.1 Material

The CPO prepared from the steam sterilization process by Srisuk Palm Co., Ltd., Prachuap Khiri Khan, Thailand on the year 2019. The chemicals used were either analytical or HPLC grade.

2.2 Degumming

The CPO samples were analyzed for FFA (AOAC, 2000), phosphorus, iron, copper (AOAC, 2016) and melting point (DSC, Perkin Elmer, USA). The 200 g CPO were degummed at 90 °C, 100 °C, or 110 °C with vigorous stirring at 200 rpm (Agitator Model MR 3001, Heidolph, Germany) for 20, 25 or 30 min (modified from the method of Chompoo *et al.* (2019)). The acids used were the mixture of 85% phosphoric acid and 99.5% citric acid at the ratios of 0.06:0.04 (A), 0.08:0.02 (B) or 0.10:0.0 (C)% by weight of the oil. The acid solution was diluted 9 times before mixing with the CPO. Accordingly, the concentrations of phosphoric acid to citric acid were 5.1%:3.98% (A), 6.8%:1.99%(B) and 8.5%:0%(C). The sedimented gum was separated from the oil by washing the oil with water at 60 °C. Next, the oil was evaporated to remove water at 80 °C under vacuum condition for 30 min by using the rotary evaporator (RV 10 auto V: IKA brand, Germany). The degummed oil was analyzed for oil yield, carotenoids content (Ribeiro *et al.*, 2008), FFA, phosphorus, copper, and antioxidant activities by DPPH method (Mansouri *et al.*, 2005) and FRAP method (Mah *et al.*, 2017). The obtaining data were used to determine the optimal degumming condition.

2.3 Caustic neutralization

After the oil was degummed, the free fatty acids in the oil were removed through neutralization process. The proper quantity of NaOH that was calculated according to molecular weight of oleic acid (Shahidi, 2005) was taken into the oil. The neutralization was controlled at 80 °C for 30 min with constant stirring. Separation of the resulting soap was carried out by using the separating vessel. The oil samples were vacuum dried at 50 °C, 150 mmHg, until the moisture content values were less than 0.1%. The CPO yield, carotenoids content (Ribeiro *et al.*, 2008), free fatty acids, phosphorus content, iron, copper and antioxidant activities by DPPH method (Mansouri *et al.*, 2005) and FRAP method (Mah *et al.*, 2017) were determined

After received optimal conditions for degumming and neutralization, do the degumming according to Chompoo *et al.* (2019) method to compare with the method that has been done. Optimal condition of Chompoo *et al.* (2019) was acid degumming with citric and phosphoric acids at 90°C for 25 min with continuous agitation. Water degumming followed the acid degumming, and it was carried out by adding 5% (w/w) water and cooling the oil down to 35°C

before centrifuging to remove the gums. The excess of free fatty acids was removed by using 7% NaOH.

2.4 Statistical analysis

Analysis of variance (ANOVA) was carried out and the determination of significant differences among means was done by the Duncan's multiple range tests ($p \leq 0.05$). All data were the means of triplicate determinations with standard deviations.

3. Results and discussion

3.1 The optimal degumming condition

The yields of CPOs obtained from various ratios of acids (Figure 1) did not indicate the significant difference ($p > 0.05$), but after the degumming process it showed the decrease trend. Yields of the degummed CPO ranged from 80 - 92%. The studied times did not statistically affect the CPO yield ($p > 0.05$). The decreasing of CPO yield was mainly due to the water that was used in degumming process. Doing many washings might increase chance of losing CPO.

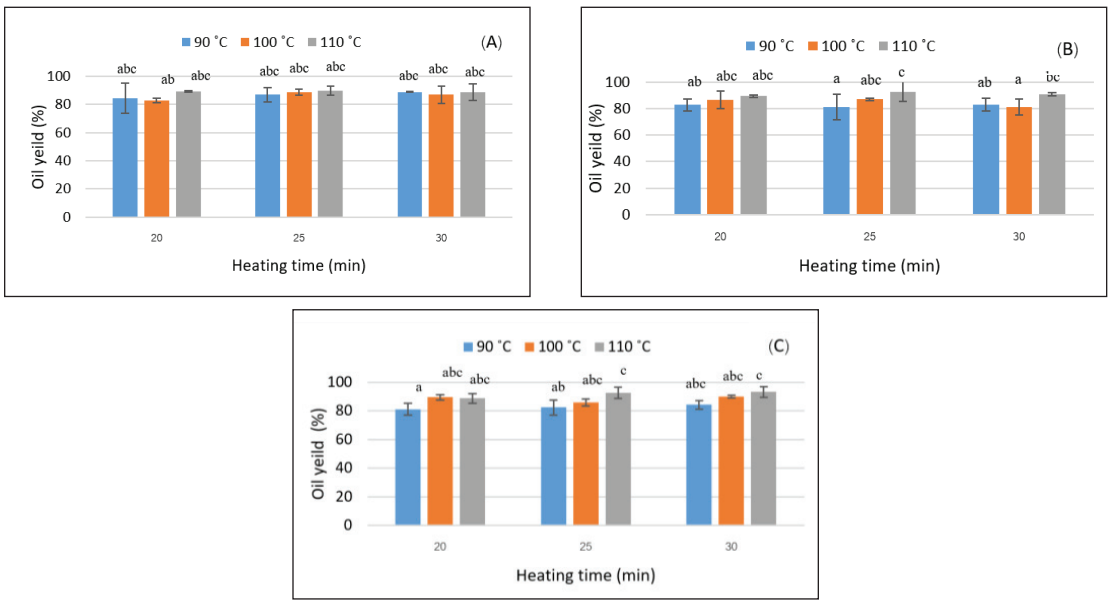

Figure 1. Effect of degumming conditions toward CPO yields (%). A: 0.06% phosphoric acid and 0.04% citric acid, B: 0.08% phosphoric acid and 0.02% citric acid, C: 0.10% phosphoric acid and 0% citric acid. The results are presented as means (n=3) and standard deviation (shown in error bars). The means with the different letters ($^{a,b,c,\cdots}$) indicated significant difference ($p \leq 0.05$).

Figure 2 showed effect of degumming conditions toward carotenoids yields. There was about 73-85% in carotenoids content after degumming, which was in accordance with the previous studies (Bonnie & Choo, 1999; Paisan *et al.*, 2017; Sulihatimarsyila *et al.*, 2020). Although temperature, heating time, and ratio of the degummed acids on the reduction of carotenoids yields were not significant difference ($p > 0.05$),

but the decrease tended to be noticeable when using higher temperatures and longer heating times, except at 0.1% phosphoric acid and 0% citric acid. The decrease was highest when using 0.06% phosphoric acid and 0.04% citric acid per weight of oil (Figure 2). Overall results suggested that yields of carotenoids were not affected by temperature, heating time, and ratio of the degummed acids. However, using the ratio of 0.08% phosphoric acid and 0.02% citric acid per weight of the oil at 90 °C for 20 min provided the highest yield of carotenoids 84.52±4.95%.

Figure 3 showed that free fatty acids slightly increased with increase in temperature and heating times. Using 0.06% phosphoric acid and 0.04% citric acid per weight of oil at 20 min yielded the lowest FFA content ($p \leq 0.05$). Overall results (Figure 1-3) suggested that using 0.06% phosphoric acid and 0.04% citric acid per weight of oil would be the best mixture for the acid degumming.

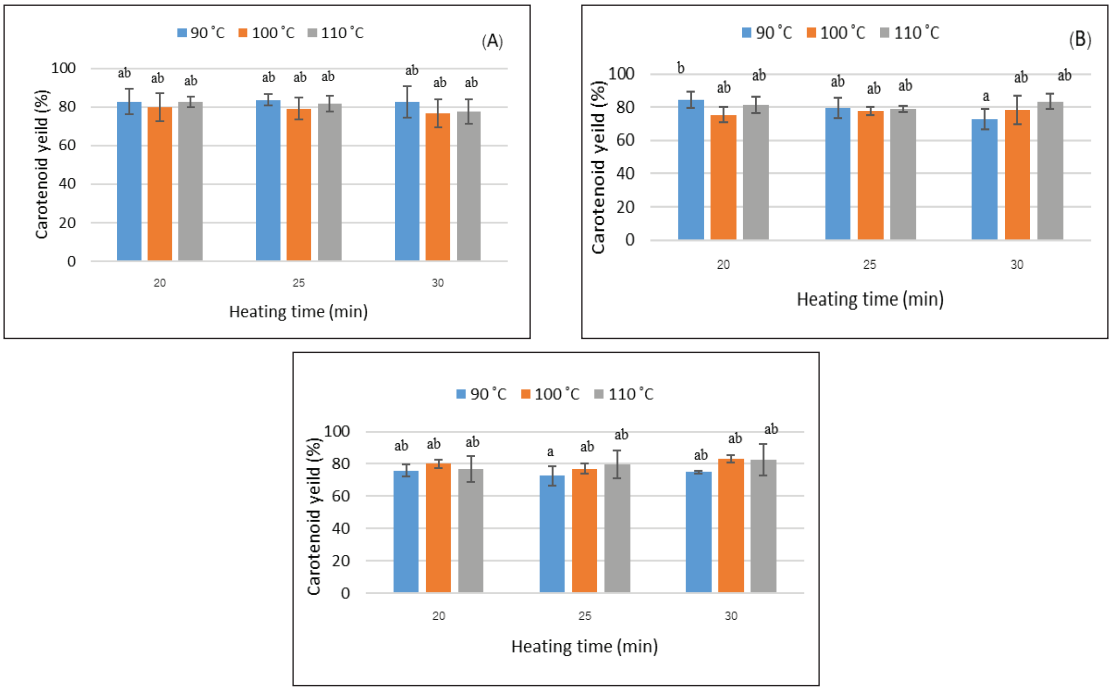

Figure 2. Effect of degumming conditions toward carotenoids yields (%). A: 0.06% phosphoric acid and 0.04% citric acid, B: 0.08% phosphoric acid and 0.02% citric acid, C: 0.10% phosphoric acid and 0% citric acid. The results are presented as means (n=3) and standard deviation (shown in error bars). The means with the different letters ($^{a,b,c,\ldots}$) indicated significant difference ($p \leq 0.05$).

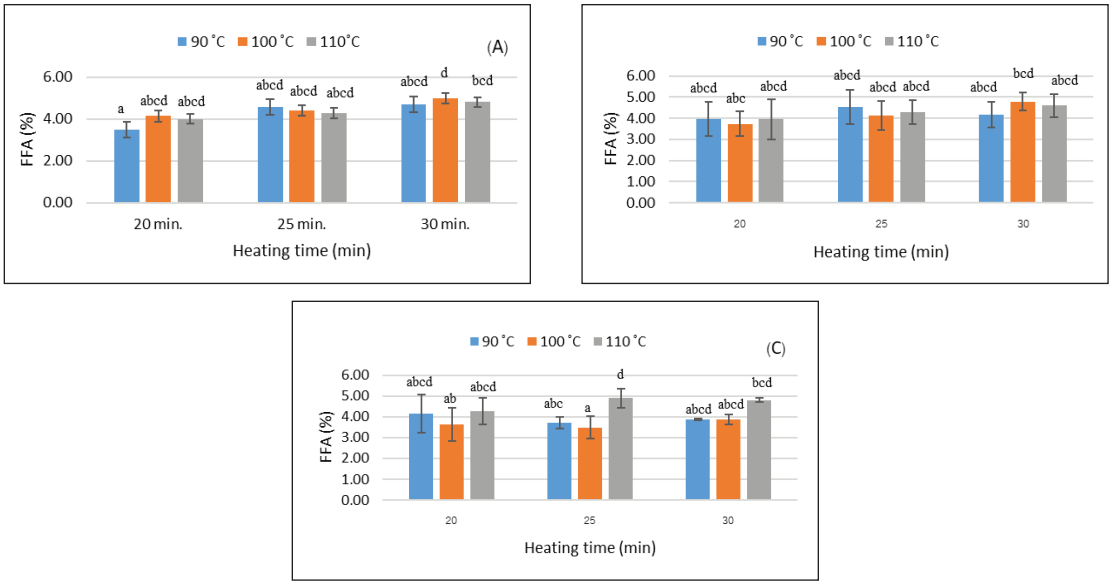

Figure 3. Effect of degumming conditions toward FFA (%). A: 0.06% phosphoric acid and 0.04% citric acid, B: 0.08% phosphoric acid and 0.02% citric acid, C: 0.10% phosphoric acid and 0% citric acid. The results are presented as means (n=3) and standard deviation (shown in error bars). The means with the different letters ($^{a,b,c,\cdots}$) indicated significant difference ($p \leq 0.05$).

With consideration on potential antioxidant value of RPO, the 0.06% phosphoric acid and 0.04% citric acid treatment was further investigated for the proper temperature and heating time on antioxidant activities. As for the free radical scavenging capacity measured by DPPH method, it was found that using temperature of 90 °C for 20 min provided the highest scavenging capacity($p \leq 0.05$) of 3.01 µmol Trolox eq/g, which indicated that the oil contained high amounts of antioxidants (Rossi *et al.*, 2007). Whereas the FRAP results revealed that temperature had less effect on electron donor ability, except when using longer heating time (Figure 4). This may be because palm oil contains antioxidants that were potent in capturing free radicals such as vitamin E and coenzyme Q10 (Unnithan *et al.*, 2011).

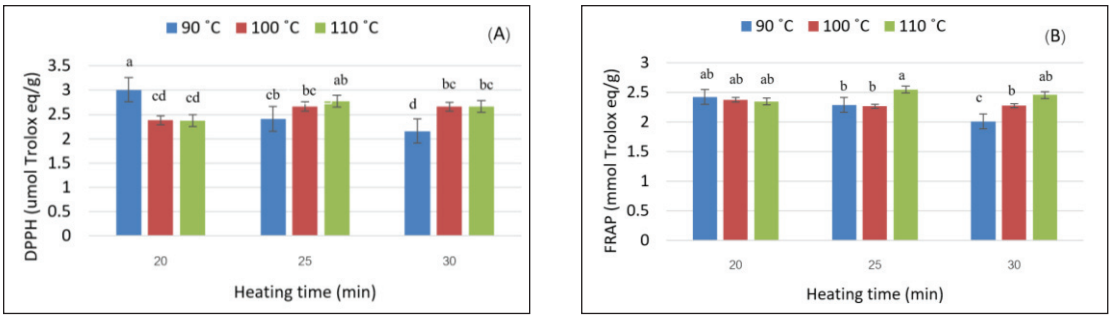

Figure 4. Effect of degumming conditions toward antioxidant activities analyzed. A: DPPH, B: FRAP. The results are presented as means (n=3) and standard deviation (shown in error bars). The means with the different letters ($^{a,b,c,\cdots}$) indicated significant difference ($p \leq 0.05$).

Overall results suggested that the optimal degumming conditions were the use of 0.06% phosphoric acid and 0.04% citric acid per weight of the crude oil and degum at 90 °C for 20 min. The heating times of 15-30 min was also reported in refine palm oil in industry by Shahidi (2005) and used degumming times 20 min for red palm oil by Chompoo *et al*, (2019). The oil degummed at the optimal condition contained 3.5% FFA, 625.92±6.59 mg/kg carotenoids or 82.7±6.49% carotenoids yield, 84.35±10.69% CPO yield, 68.49 mg/kg phosphorus, 19.25 mg/kg iron, and 0.84 mg/kg copper. Compared to those amounts in the CPO, the vast decreased occurred in phosphorus and copper contents. However, when compared with the standard of edible oil, free fatty acids and phosphorus were more than the standard of CPO. The excess free fatty acids and phosphorus content could be reduced again in the following neutralization step.

3.2 Neutralization process

The neutralization process carried out at 80 °C for 30 min with constant stirring. Separation of the resulting soap was done by using the separating vessel. After neutralization, the average of FFA was decreased from 3.5±0.05% to 0.46±0.05%, which were within the standard of not more than 0.6%. Thus, saponification could significant decrease free fatty acids in palm oil as expected. Soap from the saponification reaction was washed out with water at 60 °C. Phosphorus was trapped in the soap (Shahidi, 2005; De Greyt, 2013), thus the soap washing resulted in a reduction of the phosphorus content. In this study, the phosphorus content reduced from 68.49 mg/kg to an undetectable level. The oil was washed by water until NaOH and soap were removed completely, which was noticed by pH that was neutral. The volume of washing water and oil were 1.5:1, which was higher than that reported by Mayamol *et al.* (2007) where the volume of water and oil were 1:1. The reason for using more amount of washing water was because lower concentration of NaOH was used in this study, which resulting in more emulsion between oil and soap. Thus, it required extra washing water to discharge the soap. It was advantageous that neutralization process could reduce iron to less than 0.10 mg/kg and the copper and phosphorus contents were not detected, which were within the product standard (Codex Alimentarius International Food Standards, 1999). The neutralized oil contained 637.60±8.64 mg/kg carotenoids or 74.21±9.35% carotenoids yield, and 74.487±9.39% CPO yield. Carotenoid concentrations of oil was higher after neutralization, because in neutralization process NaOH reacts with fatty acids or oils to form soap (saponification), which dissociated. This resulted in a higher concentration of carotenoids which considered to be one way to extract carotenoids (Varzakas & Kiokias, 2016).

Table 1 concluded the properties of the oil prepared from the optimal degumming procedure of this study and those prepared from the method of the previous work (Chompoo *et al.*, 2019) (Figure 5). The differences were that their study employed the acid degumming without diluting the acids solution, and the gum was separated from the oil by filtration method. This study modified their acid degumming procedure by diluting the acid solution to promote oil and acid to react. The acid solution was also mixed with the oil

under vigorous agitation to generate a very fine dispersion that facilitated the contact between oil and acids. In addition, gum separation was operated by washing with warm water at 60 °C to keep the oil in a liquid state, which reacted better. Accordingly, it was shown that losing CPO and carotenoids in the modified acid degumming method was less than those obtained in the conventional degumming method. Thus, the simplicity and efficacy of this modified procedures (Figure 5) would be beneficial for the SMEs who are involved in the CPO processing.

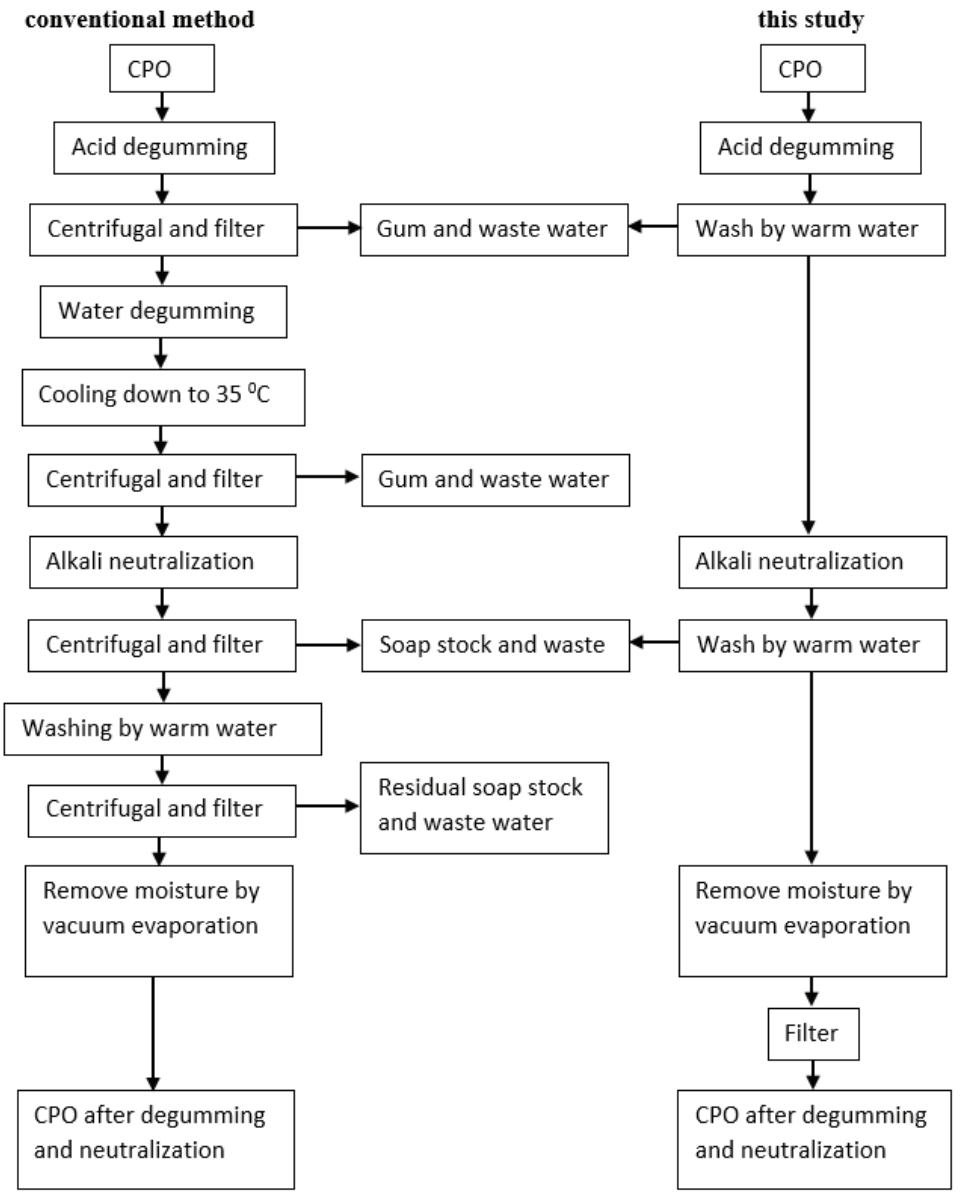

Figure 5. Flow chart degumming and neutralization between conventional method (Chompoo *et al.*, 2019) and this study method.

Table 1. Properties of the oils after degumming and neutralization by conventional and modified procedures.

		Acid degumming by conventional method	Modified acid degumming of this study
Acid solution		**0.08% of 75% phosphoric acid and 0.02% of 20% citric acid per volume of the oil**	**0.06% of 85% phosphoric acid and 0.04% of 99.5% citric acid per weight of the oil, and dilute 9 times**
Degumming conditions		90 °C, 25 min.	90 °C, 20 min.
CPO	Carotenoids (mg/kg)	639.90±5.30	639.90±5.30
	FFA (%)	3.10±0.14	3.10±0.14
	P (mg/kg)	111	111
	Fe (mg/kg)	20.27	20.27
	Cu (mg/kg)	55.56	55.56
CPO after degumming	Carotenoids (mg/kg)	606.79±22.68 ^{ns}	625.92±6.59 ^{ns}
	FFA (%)	3.67±0.22 ^{ns}	3.50±0.55 ^{ns}
	Carotenoids Yield (%)	76.80±6.13 ^{ns}	82.7±6.49 ^{ns}
	CPO Yield (%)	77.73±2.99 ^{a}	84.35±10.69 ^{b}
	P (mg/kg)	ND	68.49
	Fe (mg/kg)	0.28	19.25
	Cu (mg/kg)	1.02	0.84
CPO after degumming and neutralization	Carotenoids (mg/kg)	646.60±19.79 ^{ns}	637.60±8.64 ^{n}s
	FFA (%)	0.45±0.06 ^{ns}	0.46±0.05 ^{ns}
	Carotenoids Yield (%)	66.19±0.97 ^{a}	74.21±9.3^{5} b
	CPO Yield (%)	65.54±2.12 ^{a}	74.48±9.3^{9} b
	P (mg/kg)	ND	ND
	Fe (mg/kg)	ND	<0.1
	Cu (mg/kg)	<0.1	ND

Note: The values presented were the means ± standard deviations of triplicate analysis. The English letters that label the data values in the same horizontal that differ from each other indicated that they are statistically significant different at the 95% confidence level ($p \leq 0.05$). ns indicated not significant different ($p > 0.05$). ND indicated not detected.

4. Conclusion

The optimal acid degumming of the CPO was using the ninefold diluted of 0.06% (w/w) phosphoric acid and 0.04% (w/w) citric acid at 90 °C for 20 min with vigorous agitation. This condition provided the highest yield of carotenoids (84.52±4.95%) and the highest free radical scavenging capacity measured by DPPH method (3.01±0.73 umol Trolox eq/g) and FRAP method (4.43±0.05 umol Trolox eq/g). The sedimented gum was

separated from the oil by washing the acid oil with water at 60 °C. The excess of FFA and phosphorus content were removed by neutralization reaction. The soap and excess NaOH were washed out with water at 60 °C. After neutralization, the oil contained 0.46±0.05% FFA, 637.60± 8.64 mg/kg carotenoids, 74.48±9.39% CPO yield, < 0.1 mg/kg iron, while the copper and phosphorus contents were not detected. The simplicity and efficacy of this studied method would be beneficial for production of RPO by the SMEs.

5. Acknowledgement

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