Optimization of Neutralization in the Integrated Refining of Red Palm Oil: Effects on Free Fatty Acids and Water Content

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ABSTRACT

Red Palm Oil (RPO) is widely recognized for its high provitamin A content, particularly carotenoids, and its abundance of natural antioxidants, including tocopherols and tocotrienols. These compounds make RPO a valuable functional food ingredient with significant nutritional and health benefits. However, refining RPO remains challenging because improvements in oil quality must be achieved without causing substantial losses of these bioactive compounds. This study evaluated an integrated refining sequence comprising degumming, bleaching, neutralization, and deodorization applied to crude palm oil at moderate temperatures to maintain nutritional quality. The effects of NaOH concentration (14–18 °Bé) and reaction time (10–20 minutes) were investigated as the main variables influencing free fatty acid (FFA) reduction and water content stability. The results showed that the neutralization step was the most decisive stage, achieving an FFA reduction of nearly 60%, substantially higher than the reductions obtained during degumming–bleaching (28%) and deodorization (17%). The optimum operating condition was observed at 15 minutes with an appropriate NaOH concentration, where FFA reached its lowest level while water content remained relatively stable. In contrast, extending the reaction time beyond the optimum increased FFA levels, likely due to reverse hydrolysis caused by excess water formed during the process. Overall, this study defines an effective processing window for refining RPO under mild conditions and identifies neutralization as the critical control step. Further work is recommended to evaluate carotenoid retention and antioxidant stability throughout the refining sequence.

1. Introduction

Red palm oil (RPO) is a product of processing crude palm oil (CPO) extracted from the mesocarp of Elaeis guineensis fruit [1], [2], [3], [4]. Through selective purification (e.g., moderate-temperature deacidification–deodorization), RPO retains bioactive components—mainly carotenoids (α- and β-carotene) and vitamin E (tocotrienol/tocopherol)—which give it its red-orange color and its functional value as a source of provitamin A [1], [2]. This potential is relevant for increasing vitamin A intake in populations at risk of deficiency, while still meeting quality requirements (low FFA, oxidative stability, and food safety) [2], [3]. The main challenge lies in the thermo-oxidative degradation of carotenoids during processing and supply chain (sterilization, drying, storage), as well as the trade-off between nutrient retention and oil quality [1], [3].

Crude palm oil (CPO) has a reddish-yellow color, indicating a high carotenoid content, particularly α-carotene and β-carotene, which act as provitamin A and natural antioxidants. In addition, CPO is also rich in vitamin E in the form of tocopherols and tocotrienols, as well as other bioactive compounds such as riboflavin, niacin, lycopene, and various minerals including calcium, magnesium, potassium, and phosphorus, which contribute to the nutritional value and functional properties of palm oil [4], [5], [6]. Recent studies show that these minor compounds play an important role in improving oxidative stability and providing health benefits in palm oil products, even after undergoing certain processing stages [4], [5], [6], [7].

In recent years, the development of Red Palm Oil (RPO) has focused on optimizing processes to preserve carotenoids, vitamin E (tocopherols, tocotrienols), and other bioactive compounds without compromising oil quality. Several recent studies emphasize that acid degumming with phosphoric or citric acid is an important method for purifying Crude Palm Oil (CPO) to produce Red Palm Oil (RPO) with high carotenoid retention. A combination of 0.1–0.3% phosphoric acid at 90°C for 20 min was reported to retain 82.7% of carotenoids while reducing free fatty acid (FFA) content to 0.46% [8]. Similar results were reported by [9] with optimization of time and pH, showing that pH below 4.5 can minimize pigment loss during the process. The use of citric acid produced RPO quality comparable to that obtained with phosphoric acid; however, phosphoric acid was more effective at reducing phospholipid levels by approximately 80–90% [10]. also found that citric acid produced RPO quality comparable to that of phosphoric acid, but phosphoric acid was more effective at reducing phospholipid levels by 80–90% [10]. In addition, Lau *et al.* studied palm-pressed fiber oil and found that acid degumming reduced phosphorus to <10 ppm while maintaining 90% of vitamin E and 70% of β-carotene [11]. Meanwhile, a previous study reported that process parameters, such as acid concentration and stirring time, significantly affected phospholipid reduction and increased the oil's oxidative stability [12]. Industrial-scale research also supports these findings, noting that combining acid degumming with neutralization and deodorization yields RPO with a more stable color and lower peroxide values [13].

Another method is to combine the neutralization process with vacuum deodorization. This technique was reported to maintain β-carotene levels above 50 ppm while effectively suppressing peroxide formation, thereby enhancing the oil's resistance to oxidation [14]. A pilot-scale study also supports these findings. They showed that using moderate temperatures (≤180°C) in deodorization under high vacuum pressure can reduce carotenoid damage by up to 30% compared to traditional high-temperature methods [15]. Meanwhile, [16] identified optimal conditions for red palm oil olein fractionation, showing that shorter processing time and lower temperature can yield oil with low free fatty acid content and high β-carotene retention. Other studies also confirm that this method not only preserves carotenoid content but also reduces peroxide formation by up to 25% during storage compared to conventional processes [17]. The key to this process is controlling the temperature, pressure, and processing time. The combination of neutralization and vacuum deodorization can produce healthier, higher-quality red palm oil.

On the other hand, the bleach-free process is an effective strategy to minimize carotenoid loss because bleaching earth can absorb 20–50% of pigments, whereas high-temperature deodorization (260–280°C) can cause significant thermal degradation [1], [2], [18]. A cold filtration approach has also been developed to reduce thermal damage to carotenoids and vitamin E, although its capacity to remove free fatty acids and volatile compounds remains limited [3], [19]. Recently, enzymatic interesterification has been explored to modify the fatty acid distribution in triglycerides without exposure to high temperatures, thereby improving the retention of bioactive compounds compared to conventional chemical processes [20], [21]. Thus, the selection of RPO production methods must consider the trade-off between nutritional quality, oxidative stability, process complexity, and industrial costs [22], [23], [24].

While studies have explored individual low-temperature steps, systematic data on the effects of alkali concentration and contact time during neutralization within an integrated low-temperature degumming, bleaching, neutralization, and deodorization sequence on core quality parameters such as FFA and water content are lacking. This gap is critical because neutralization is a major determinant of final quality and yield. This study aimed to (1) determine the effect of NaOH concentration and reaction time during neutralization on the FFA and water content of RPO produced via an integrated low-temperature degumming, bleaching, neutralization, and deodorization process, and (2) identify the optimum conditions for minimizing FFA without promoting reverse hydrolysis.

2. Research Methodology

2.1. Materials

The materials used in this study included CPO samples, phosphoric acid (H_3PO_4), distilled water (aquadest), bleaching earth, sodium hydroxide (NaOH), ethanol, and phenolphthalein indicator. Crude palm oil (CPO) samples with a free fatty acid (FFA) content of 3.5% and the bleaching earth were obtained from the marketplace. The phosphoric acid used had a concentration of 85% wt, while the sodium hydroxide had a concentration of 98% wt. The ethanol used was from Merck, with a concentration of 99.8% v/v.

2.2. Procedures

The experimental procedure consists of four main stages: degumming, bleaching, neutralization, and low-temperature deodorization. Each stage is carried out sequentially under predetermined operating conditions, as described in the following subsections. This study was designed as a two-factor experiment to evaluate the effects of NaOH concentration and reaction time during the neutralization stage. NaOH concentration was varied at three levels (14, 16, and 18 °Bé), while reaction time was set at 10, 15, and 20 minutes [25], [26]. The experimental combinations are summarized in Table 1.

Table 1. Experimental matrix of the two-factor neutralization study

Run	*NaOH Concentration (°Bé)*	*Reaction Time (min)*
1	14	10
2	14	15
3	14	20
4	16	10
5	16	15
6	16	20
7	18	10
8	18	15
9	18	20

1) Degumming

A total of 100 g of CPO sample was heated on a magnetic stirrer hot plate at 70°C and stirred with a magnetic rod until a homogeneous mixture was achieved. Next, 0.05% phosphoric acid was added to the sample. The mixing of CPO with a phosphoric acid solution was carried out for 30 minutes at a stirring speed of 600 rpm, following typical degumming procedures [27], [28].

2) Bleaching

After degumming, the oil mixture enters the bleaching stage. At this stage, bleaching earth at a concentration of 0.8% wt is added to the oil. This process is carried out at 95–100°C for 30 minutes, with continuous stirring, conditions consistent with previous studies [29], [30]. The addition of bleaching earth helps absorb pigments, impurities, and other contaminants that can affect the oil's color and quality [31]. Once the contact time is complete, the stirrer and hotplate are turned off to stop heating and mixing. Next, the oil mixture that has undergone the bleaching process is filtered. This filtration aims to separate the bleaching earth saturated with impurities from the oil, leaving the oil clearer and cleaner [29], [31].

3) Neutralisation

The experiment began by placing bleached CPO oil into a beaker. Next, sodium hydroxide (NaOH) solutions at 14 °BE, 16 °BE, and 18 °BE were added to the oil. The mixture was heated to 70°C while stirring for 10, 15, and 20 minutes, respectively, to neutralize free fatty acids and form soap (soapstock) as a by-product [32]. After the neutralization process is complete, the mixture is centrifuged for 5 minutes at 1000 rpm to separate the soap phase from the neutral oil. The resulting oil is then washed with hot water at 50°C to remove any remaining sodium hydroxide and soap. The process of separating water from oil is repeated using a centrifuge, yielding a purer, neutral oil ready for the next stage of processing [32], [33].

4) Deodorization

Next, the oil mixture to be deodorized is placed in a covered Erlenmeyer flask and connected to a vacuum pump. The heating process is carried out by setting the temperature to 110°C. Once that temperature is reached, the deodorization is conducted for 60 minutes under vacuum (-76 cmHg) to remove volatile compounds, odors, and residual free fatty acids that could impact oil quality. After the deodorization period, the resulting oil is analyzed for free fatty acid content and water content as quality parameters [33]. Free fatty acid (FFA) content was determined according to AOCS Official Method Ca 5a-40 by acid–base titration using NaOH as the titrant and phenolphthalein as the indicator. Water content was determined using the gravimetric oven-drying method. Approximately 10 g of the oil sample was dried at 100°C until a constant weight was reached, cooled in a desiccator, and reweighed. Water content was calculated from the weight loss during the drying process and expressed as a percentage.

3. Results and Discussion

The degumming and bleaching processes are essential stages in palm oil refining to reduce the levels of phospholipids, gums, and pigments that affect oil quality. The success of these two stages is greatly influenced by the type of adsorbent, operating conditions, and the chemical properties of the pigments, particularly carotenoids, which are known to be difficult to remove due to their lipophilic nature [30], [31], [33].

Fig. 1. Oil is produced from the degumming and bleaching process.

Fig. 1 shows the results of observations after the degumming and bleaching processes, in which CPO undergoes a color change to dark brown, due to phospholipid gums that are only partially adsorbed by bleaching earth [32], [34]. After filtration, the oil returns to red, indicating that carotenoid pigments remain. This condition is consistent with previous studies stating that carotenoids are difficult to eliminate even after bleaching, thus continuing to contribute significantly to the final color of palm oil [30], [34].

a b c

Fig. 2. Oil produced from the neutralisation process

Fig. 2a shows the formation of a precipitate upon the addition of NaOH during the neutralization process, consistent with studies [35] confirming that the reaction of FFA with an alkali produces soap. The dispersed soap causes the oil phase to appear cloudy before separation. Separation using a centrifuge shows efficient phase separation, with soap collecting at the bottom and oil remaining in the upper phase, as shown in Fig. 2b. These results are consistent with reports [36] and [37] on the effectiveness of centrifugation in suppressing residual soap. Fig. 2c shows the appearance of oil after washing, where CPO appears clearer.

Fig. 3 shows the deodorization stage, where the CPO, originally a clear red, turns orange-red, indicating that water from the neutralization stage remains. The vapor formed during the deodorization process acts as a stripping agent, removing odors, while vacuum conditions accelerate the diffusion of volatile compounds, making the oil clearer. These results are consistent with research [38], which shows that vacuum-based deodorization can suppress rancid odors more quickly than conventional methods.

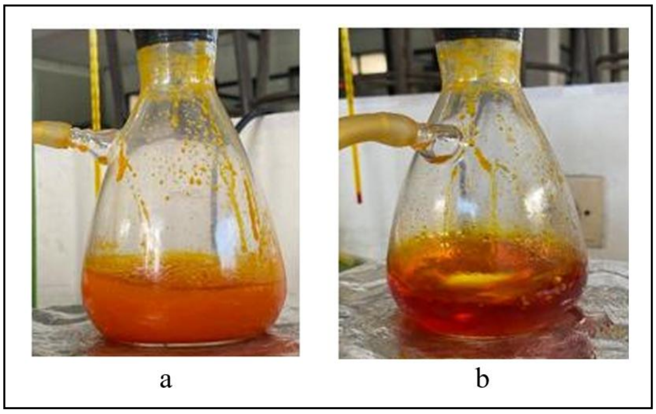

Fig. 3. Deodorization process

3.1. Effect of NaOH Concentration on FFA

Excess free fatty acids cause problems in storage, such as darker colors, unpleasant odors, and accelerated chemical and organoleptic instability. Neutralization with a NaOH solution is the most common method because it effectively reduces FFA levels via saponification. However, if the base concentration is excessive, the reaction can attack neutral triglycerides, leading to excess soap formation, oil loss, and phase separation difficulties. Therefore, optimizing the NaOH concentration is crucial to achieve a balance between FFA reduction efficiency and minimal oil loss. As an alternative, steam distillation-based physical purification has also been widely developed to maintain the final quality of vegetable oil [38].

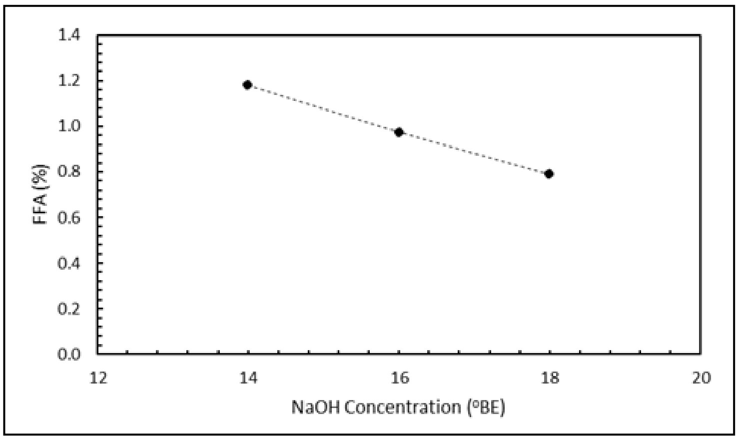

Fig. 4. Effect of NaOH concentration on FFA at reaction time 10 min

Fig. 4 shows the significant effect of NaOH concentration in reducing the Free Fatty Acid (FFA) content in oil. The initial FFA content of approximately 3.5% in CPO can be reduced to 0.8% at a concentration of 18°BE, indicating that this strong base is capable of neutralizing free fatty acids through the formation of sodium soap and water, following reaction equation (1). This trend is in line with the findings of [25], which reported that an increase in NaOH concentration accelerates the reduction in FFA levels but also increases the formation of soap and emulsions that can interfere with the oil separation process.

$$R\text{–}COOH + NaOH \rightleftharpoons R\text{–}COONa + H_2O \quad (1)$$

Fig. 4 also shows that the decrease in FFA levels is no longer proportional to the increase in base concentration, indicating diminishing returns in the neutralization process. This finding is in line with the report [26], which shows that excessive NaOH use actually triggers greater soap formation than increasing FFA removal efficiency. Therefore, it is necessary to determine the optimal base concentration to ensure efficient chemical consumption while preventing damage to oil quality.

3.2. Effect of Reaction Time on FFA

Fig. 5 shows that FFA levels decreased sharply after 10 minutes of reaction, then reached a low point at the 15th minute. This condition indicates that the neutralization process with NaOH was most effective within that time span. However, when the reaction was extended to 20 minutes, the FFA levels actually increased again. This finding is consistent with the report [26], which explains that prolonged reactions can trigger a reverse reaction, allowing some of the formed soap to break back down into free fatty acids.

This trend is even clearer when compared with the data in Fig. 6. At the 20th minute, a significant increase in water content is seen. This excess water is thought to shift the reaction equilibrium to the left, as shown in reaction equation (1), thereby hydrolyzing the formed soap back into FFA [26]. Thus, the relationship between Fig. 5 and Fig. 6 shows that the increase in FFA at longer reaction times is closely related to the formation of excess water. From these results, it can be concluded that the optimum reaction time is 15 minutes because, at that point, the FFA is at its lowest and the water content remains relatively low.

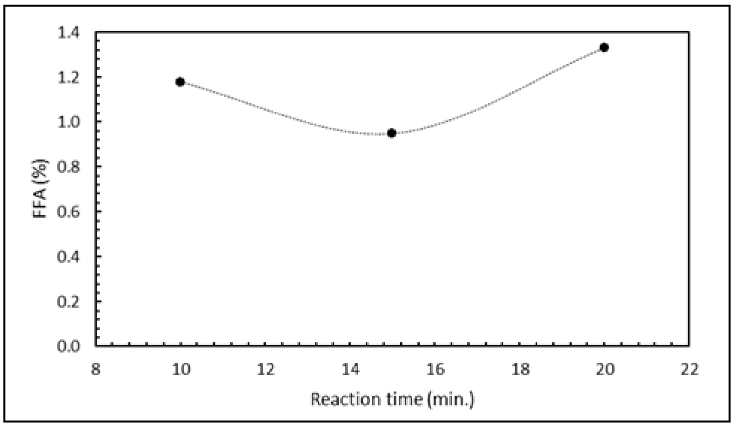

Fig. 5. Effect of reaction time on FFA at 14°BE

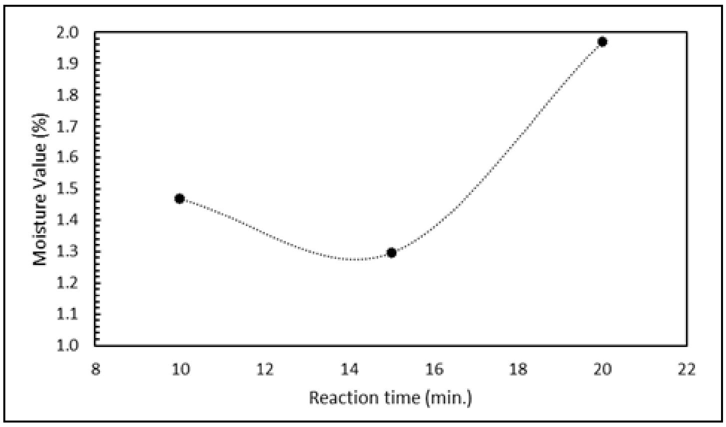

Fig. 6. Effect of reaction time on moisture value at 14°BE

3.3. Comparison Performance to Reduce FFA

Fig. 7 shows that each stage of the refining process has a different effectiveness in reducing FFA levels. In the degumming and bleaching stages, the reduction in FFA is only around 28%. This is reasonable because the main purpose of this stage is to remove phosphatides, pigments, and other minor impurities, not specifically to neutralize free fatty acids. In contrast, during the neutralization stage, FFA reduction is much greater, reaching around 60%. This mechanism occurs through a direct reaction between NaOH and FFA, producing soap that is more readily separated from the oil phase. These results are in line with the findings of [39], which confirms that neutralization is indeed the most effective method for reducing FFA. However, its use must be regulated to prevent oil loss caused by excessive soap formation.

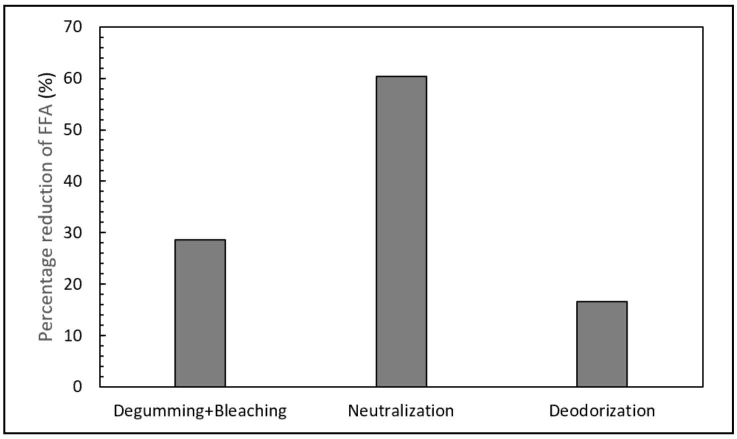

Fig. 7. Percentage reduction of FFA at each processing stage at 18ºBE and 15 min

Meanwhile, deodorization only reduces FFA by about 17%. This limitation arises because the deodorization process primarily removes volatile compounds and a small portion of short-chain fatty acids, thereby improving aroma and extending the oil's shelf life. The study [39] also showed that the main function of deodorization is to improve sensory quality, rather than to be the primary stage in FFA reduction. Thus, the combination of these results confirms that neutralization is the key to reducing FFA levels, while degumming, bleaching, and deodorization play complementary roles in maintaining the oil's physical and sensory quality.

4. Conclusion

This study shows that an integrated refining process involving degumming, bleaching, neutralization, and deodorization can effectively reduce free fatty acid (FFA) levels in red palm oil while maintaining stable moisture content. Among these stages, neutralization played the most decisive role, accounting for nearly 60% of the total FFA reduction. Based on the complete experimental matrix covering NaOH concentrations of 14, 16, and 18 °Bé and reaction times of 10, 15, and 20 minutes, the optimum condition was clearly identified at a reaction time of 15 minutes and a NaOH concentration of 16 °Bé, where the lowest FFA level was achieved without a noticeable increase in water content. A key scientific contribution of this work is the quantitative demonstration that extending the neutralization time can promote reverse hydrolysis, as evidenced by a simultaneous increase in FFA and moisture content, thereby defining a critical process window for effective FFA control. Future studies are recommended to further evaluate carotenoid (β-carotene) and tocopherol retention under the optimum conditions using HPLC, assess oxidative stability parameters such as peroxide value, induction period, and oxidative stability index, and conduct a comprehensive quality assessment in accordance with SNI standards to confirm the nutritional and practical applicability of the refined oil.

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