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Analysis of FFA Quality Parameters and Peroxide Value in Crude Palm Oil Industry

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Abstract. Crude Palm Oil (CPO) is crude palm oil extracted from the palm oil industry that has not undergone a refining process. CPO quality is considered good if it meets applicable quality standards. This study aims to determine a mathematical equation model that describes the relationship between free fatty acid value (FFA) quality parameters and peroxide value (PV) in CPO samples. FFA content analysis was carried out using the alkalimetric titration method, while the peroxide value was tested using the iodometric titration method. The storage time variations used were 0 - 3 weeks. The results showed that : (1). The sample consist Free Fatty Acid value (FFAV) were 2,97%; 3,22%; 3,41%; and 3,66%, respectively, with an average of 3,31%. Meanwhile, Peroxyde Value (PV) were obtained at 1,85; 2,25; 2,65; and 3,04 meq O_2/kg. (2). According to these results, the FFAV content still meets the Indonesian National Standard (SNI) standard (<5.0%) and the PV value is also below the SNI limit (<5 meq O_2/kg). (3). Each increase in FFA will be followed by an increase in the peroxide value, which is influenced by variations in storage time. Oxidation in oil will affect oil damage, resulting in increased free fatty acid values. Therefore, the longer the storage time, the higher the peroxide value.

Keywords : Crude Palm Oil (CPO), Free Fatty Acid Value, Peroxyde Value, Storage Time

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Introduction

Crude palm oil (CPO) is a processed product produced by palm oil processing through the stages of sorting, threshing, boiling, and pressing the fruit. CPO is one of the main products with the largest production volume from oil palm plantations[1]. According to the Central Statistics Agency (BPS), in 2022, North Sumatra Province was the main national palm oil producing province after Riau and Central Kalimantan. Indonesian CPO production reached 45.58 million tons, with North Sumatra producing 6 million tons, or 13% of domestic production.

The quality of palm oil is significantly influenced by the level of ripeness at harvest and post-harvest handling until it reaches the mill. Harvesting should be done when the Fresh Fruit Bunches (FFB) reach optimal ripeness[2]. Quality is a key factor influencing consumer decisions when choosing a product. Through quality control, companies can monitor the quality of their products and make improvements if the quality is inadequate or does not meet standards[3].

PT. Perkebunan Nusantara IV Kebun Adolina is a CPO producer with its own quality standards that must be met at every stage of production. In the CPO production process, various factors can influence and cause variations in product quality. Therefore, the company needs to implement quality control to ensure CPO quality is maintained. Palm oil is significantly affected by several factors related to the quality of the oil produced. Changes in components such as free fatty acid values, peroxide value, water content, color, and impurities in the oil can occur due to conditions such as suboptimal storage, inappropriate temperature, prolonged storage time, changes in melting point, and oil clarity[4]. Previous research conducted by Hikmawan et al. (2019) showed that storage results in the hydrolysis of oil by lipase enzymes, air, and impurities, resulting in high values of free fatty acids. The longer the storage time, the more hydroperoxides are formed and accumulated, increasing the peroxide content.

Previous research conducted by Silaban et al. (2013) found that these quality parameters can be analyzed through mathematical equations with high accuracy test values for each parameter. These mathematical equations can be used to describe the patterns of data obtained and can be used to estimate certain parameter values by considering the coefficient of determination (R2). These mathematical equations can be expressed in the form of models that describe the patterns of observed data for prediction (Chapra and Canale, 1990; Walpole, 1982). The form of the equation used is a straight line equation or a linear model, by considering the value of the coefficient of determination (R2). The mathematical equation that will be used to determine the correlation of CPO quality parameters through a simple linear regression equation is Y = a + bx. In general, Y is the dependent variable, X is the independent variable, while a indicates a constant and b is the regression coefficient of each independent variable.

Based on the description above, the author is interested in raising the problem with the title "Analysis of FFA Quality Parameters and Peroxide Value in Crude Palm Oil Industry".

Experimental

Materials. The materials used in this study were palm oil samples, 95% alcohol, phenolphthalein indicator, 0.1 N KOH solution, chloroform, acetic acid, saturated KI, 0.01 N $Na_2S_2O_3$ (Sodium thiosulfate), starch indicator, oxalic acid solution. The equipment used were analytical balance, Erlernmeyer, burette, stand and clamp, beaker glass, measuring flask, measuring cup, dropper pipette, spatula, stirring rod, and aluminum foil.

Determination of Free Fatty Acid Values. As much of ± 5 grams of palm oil is weighed into a 250 mL Erlenmeyer flask and topped with 50 mL of neutral alcohol (95% ethyl alcohol). Dropped with the phenolphthaline indicator as much as 2-3 drops. Titraìt with another 0.1 N KOH solution with alcohol until it reaches a youthful color that is stable for ± seconds and the volume of KOH used is obtained.

$$\text{FFA}\,(\%) = \frac{\text{V x N x 25,6}}{\text{W}} \qquad (1)$$

Determination of Peroxide Value. As much of ± 5 grams of palm oil sample was weighed into a dark Erlenmeyer flask, dissolved in 30 mL of chloroform solvent : acetone (2:3). Added 0.5 mL of KI solvent that had been saturated with the cloth, then shaken thoroughly. Let it stand in the dark for 1 minute until it reacts, then add 30-50 mL of CO_2-free water. Titrate with 0.01 N $Na_2S_2O_3$ (Sodium thiosulfate) solution until the yellowish color almost disap-

pears, then add a few drops of the milk indicator while titrating until the blue color disappears completely. Do the same thing on the blank (sample) to get constant results.

$$Bilangan\ Peroksida\ (meq\ O2/kg) = \frac{(V - V0)\ x\ N\ x\ 1000}{W} \quad (2)$$

Data Analysis. The data obtained from the analysis are presented in tables and graphs. The relationship between the quality parameters of free fatty acid content and peroxide value was determined using a simple linear regression equation with mathematical modeling based on a straight-line equation. A coefficient of determination (R2) value approaching 1 indicates a stronger relationship between the quality parameters and a more appropriate modeling.

Result and Discussion

Determination of Free Fatty Acid Values. Determination of free fatty acid values was carried out with variations in storage time for 3 weeks, namely, control (0 weeks), week 1, week 2, and week 3. The results obtained from weighing the sample weight of ± 5 grams, the volume of 0.1 N KOH titrant used, and the results of calculating the FFA values for each variation of storage time with the average value of the FFA values produced through testing with 3 repetitions, can be seen in **Table 1**.

Based on **Table 1**, it can be seen that the value of the free fatty acid content increased with each variation in storage time. These results indicate a consistent increase every week. The average increase per week ranged from 0,19 – 0,25%, with the highest average increase occurring in the 3rd week, which indicates that the longer the storage time of the CPO sample, the higher the FFA content obtained. However, based on the average FFA content obtained (**Figure 1**), it states that the oil sample can still be used because it is still far from the maximum limit. This indicates that the FFA content value meets the quality standards set by the company, which is a maximum of 5%.

FFA exists as free acids that are not bound to triglycerides and are formed through hydrolysis and oxidation processes, usually combined with neutral fats. One result of the oil hydrolysis reaction is the formation of FFA. The FFA content in vegetable oil is one of the parameters determining the quality of the oil, where the FFA content is expressed through the acid value [8]. FFA content is one of the important parameters in determining the quality of CPO as stated in the Indonesian National Standard, namely SNI 01-2901-2006, which sets a maximum FFA limit of 5% (BSN, 2006). Although the requirement for a maximum FFA content of 5%, in the process of processing fresh fruit bunches (FFB) into CPO, efforts are made to keep the FFA content

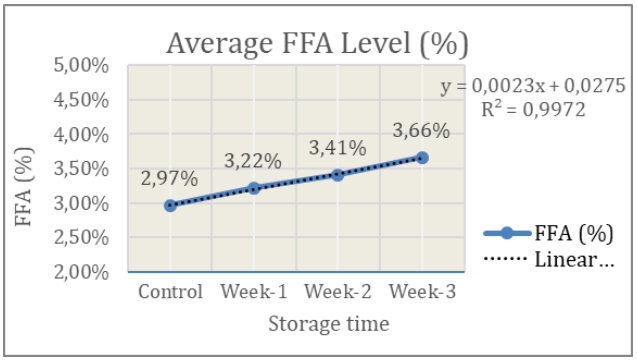

Figure 1. Graph relation between Free Fatty Acid Value and Storage Time

Table 1. Results data of Free Fatty Acid value at the storage variation (week)

No	Storage Time (Week)	Sample Weight (g)	V_{KOH} (mL)	FFAV (%)	FFAV average (%)
		5,180	7,6	3,00	
1.	Control	5,202	7,5	2,95	2,97
		5,195	7,5	2,95	
		5,001	7,8	3,19	
2.	Week-1	5,012	7,9	3,22	3,22
		5,008	8	3,27	
		5,011	8,3	3,39	
3.	Week-2	5,001	8,4	3,43	3,41
		5,018	8,4	3,42	
		5,000	8,9	3,64	
4.	Week-3	5,018	9	3,67	3,66
		5,007	9	3,68	

as low as possible. High FFA values indicate that the CPO is of low quality [9].

High values of FFA can be influenced by several factors, including water content and enzyme activity. These two factors trigger hydrolysis reaction in the fatty acid bonds, thus forming free fatty acid. An increase in water content and lipase enzyme activity can occur during the process of transportation fruit from the plantation to the factory, because the fruit metabolic activity process is still onging.[10]. In addition, less than optimal harvesting and fresh fruit bunches (FFB) techniques during the transportation process can also cause damage to the fruit such as softened fruit, falling off fruit, injured fruit or crushed fruit, which ultimately encourages the formation of the lipase enzyme. The lipase enzyme acts as a biocatalyst that accelerates the hydrolysis reaction of fat, breaking down triglycerides into glycerol and free fatty acids [11].

One way to improve the quality and quantity of CPO is by paying attention to the quality of the fresh fruit bunches (FFB). Ripe fruit is harvested when the majority of the bunch (>98%) has reached optimal ripeness, resulting in high CPO yields. However, harvesting overripe fruit will increase the FFA content and reduce oil quality. Harvesting underripe fruit will result in low FFA content but lower palm oil yield, thus reducing CPO production [12].

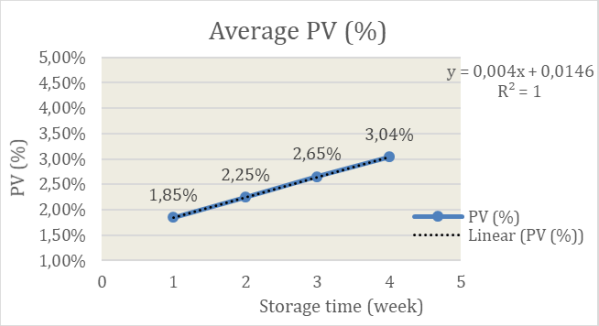

Figure 2. Graph relation between Peroxide Value and Storage Time

Determination of Peroxide Value. The determination of peroxide value was carried out with variations in storage time for 3 weeks, namely, control (0 weeks), week 1, week 2, and week 3. The results obtained from weighing the sample of ± 5 grams, the volume of $Na_2S_2O_3$ titrant used 0.01 N, as well as the results of calculating the PV coefficient for each storage time variant with the PV coefficient values produced through testing with 3 repetitions, can be seen in **Table 2**.

Based on **Table 2**, it can be seen that the peroxide value increased with each variation in storage time. These results indicate a consistent increase every week. The average increase per week ranged from 0.4 – 0.39 kg, with the highest average increase occurring in the 3rd week. The overall average value was 3.26 meq O_2/kg, indicating that the peroxide value of the CPO sample still met the established quality standard of a maximum of 5 meq O_2/kg. These results were presented in a graph as in **Figure 2** to see how the increase in peroxide value relates to variations in CPO storage time.

Peroxide content is a parameter that indicates the amount of oil that has been oxidized, namely the content of saturated fatty acids in CPO oil that are oxidized and produce compounds called peroxide compounds [13].The peroxide value indicates the presence of oil oxidation, so this value is

Table 2. Results data of Peroxide value at the storage variation (week)

No.	Storage Time (Week)	Sample weight (g)	$V_{Na\ 2S2O3}$ (mL)	PV (g)	PV average (meq O_2/kg)
		5,011	1,0	1,79	
1.	Control	5,006	1,0	1,79	1,85 meq O_2/kg
		5,015	1,1	1,99	
		5,036	1,2	2,18	
2.	Week-1	5,016	1,2	2,19	2,25 meq O_2/kg
		5,022	1,3	2,38	
		5,006	1,4	2,59	
3.	Week-2	5,007	1,5	2,79	2,65 meq O_2/kg
		5,007	1,4	2,59	
		5,015	1,6	2,99	
4.	Week-3	5,039	1,6	2,97	3,04 meq O_2/kg
		5,018	1,7	3,18	

Table 3. Table of storage time against quality parameters

No.	Storage Time	Average FFA Level (%)	Average PV (meq O_2/kg)
1.	Control	2,97 %	1,85 meq O_2/kg
2.	Week -1	3,22 %	2,25 meq O_2/kg
3.	Week -2	3,41 %	2,65 meq O_2/kg
4.	Week -3	3,66 %	3,04 meq O_2/kg
	Total	**3,31%**	**2,44 meq O_2/kg**

useful for determining the quality of oil after processing and storage [14]. The damage to the main fabric of oil is indicated by the emergence of rancid aroma which is called the rancidity process, rancidity occurs when the fatty acids undergo changes in the form of hydrolysis reactions producing hydrocarbon compounds. This peroxide formation process is accelerated by the presence of light, acidic conditions, air humidity and catalysts.

High values of peroxide indicate that oxidation is ongoing, but low values of peroxide do not necessarily indicate that the oil is free from oxidation. Based on SNI-01-3741-2013 the maximum limit for the highest peroxide consumption compared to oil is up to 5 meq O_2/kg, if the peroxide consumption exceeds this limit, Oil can be categorized as dangerous for consumption or use.

The peroxide value obtained each week tends to increase, as seen in **Figure 2**. This indicates that storage time affects the oil's oxidation rate, with the peroxide value increasing each week. Nevertheless, the test results indicate that this CPO sample is still usable because the peroxide value is still well below the established maximum limit.

The Effect of Variations in Storage Time on Free Fatty Acids and Peroxide Values. When oil is exposed to water and steam, it undergoes hydrolysis, which is initiated by water and steam, producing compounds such as monoglycerides and diglycerides in a sequential manner. The release of fatty acids from glycerol is a determinant in analyzing the level of damage to hydrolytic acids, namely through the oxidation of oil. This process is one of the causes of rancidity in oils. The primary damage to fats and oils is rancidity, caused by oxidation and hydrolysis. During hydrolysis, oil is converted into free fatty acids and glycerol. The hydrolysis reaction can cause oil damage due to the presence of water, resulting in a rancid odor. Oxidized oil forms unstable peroxides. Rancidity can also occur due to enzyme and microbial activity [15].

The FFA indicator is usually used as an indicator of oil damage. FFA is formed as a result of excessive hydrolysis which triggers oxidation reactions, with the presence of hydrolysis reactions supporting the occurrence of oxidation reactions in oil. In addition to thermal oxidation, vegetable oil produces hydroperoxide compounds as primary oxidation products which then degrade into hydrocarbon compounds, aldehydes, and ketones which then produce secondary oxidation products. Secondary oxidation products usually tend to evaporate easily and produce rancid odors in the oxidized oil [16].

Based on **Table 3**, the average results of all weekly tests show that varying storage time affects the increase in FFA levels, as does the peroxide value; the longer the storage time, the higher the peroxide value. However, in this case, the two parameters are not directly related. Increased free fatty acid levels can increase the oil's susceptibility to oxidation, thus indirectly contributing to an increase in the peroxide value. Furthermore, processes and environmental factors can trigger simultaneous increases in both parameters.

Conclusion

Based on the results of the research conducted, it can be concluded that: The sample consist Free Fatty Acid value (FFAV) were 2,97%; 3,22%; 3,41%; and 3,66%, respectively, with an average of 3,31%. Meanwhile, Peroxyde Value (PV) were obtained at 1,85; 2,25; 2,65; and 3,04 meq O_2/kg. According to these results, the FFAV content still meets the Indonesian National Standard (SNI) standard (<5.0%) and the PV value is also below the SNI limit (<5 meq O_2/kg). Variations in the length of storage time affect the increase in FFA values, as

well as the peroxide number, the longer the storage time the higher the peroxide value.

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