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Effect of Activation Duration of Water Hyacinth-Based Activated Carbon Using Bilimbi (Averrhoa bilimbi) as an Activator on Free Fatty Acid Content in Crude Palm Oil (CPO)

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Abstract
Crude palm oil (CPO) quality is significantly affected by its free fatty acid (FFA) content, which must be minimized to meet industrial standards and preserve oil stability. This study aimed to evaluate the effect of activation duration of water hyacinth-based activated carbon, using bilimbi (*Averrhoa bilimbi*) extract as a natural activator, on the reduction of FFA levels in CPO. A true experimental design was employed using activated carbon with activation times of 4, 8, 16, and 24 hours. A total of 30 CPO samples were tested, including untreated controls. The FFA content was determined using the alkalimetric titration method, and statistical analysis was conducted using ANOVA and linear regression. The results showed a consistent decrease in FFA content with increasing activation time: 9.38% (4 h), 6.32% (8 h), 6.29% (16 h), and 5.54% (24 h), compared to the control (12.54%). A significant negative correlation was found between activation duration and FFA levels ($p = 0.000$, $\beta = -0.852$), indicating improved adsorption performance over time. Although the 24-hour treatment yielded the greatest FFA reduction, levels remained slightly above the Indonesian National Standard threshold of 5.0%. These findings highlight the potential of water hyacinth-based activated carbon activated with bilimbi extract as a sustainable bioadsorbent, while also suggesting that further process optimization is needed to achieve industrial compliance.

Keywords: Water hyacinth, starfruit, crude palm oil

Introduction

Crude palm oil (CPO) is an oil product obtained from the processing of oil palm fruit flesh (mesocarp). To produce CPO, fresh fruit bunches (FFB) undergo several processing stages, including sterilization, threshing, digestion, pressing, and oil purification[1]. CPO is one of the main products derived from oil palm processing. Approximately 95% of CPO production in Indonesia comes from Sumatra and Kalimantan. CPO and its derivatives are widely used in various industries, such as the oleo-food complex (e.g., palm cooking oil, margarine, and shortening), the oleochemical complex (e.g., detergents, soaps, and shampoos), and the biofuel complex (e.g., biodiesel, biogas, bio-premium, and bio-aviation fuel)[2] .

When evaluating the quality of CPO, two main factors are considered. The first factor involves the content of impurities, water, and free fatty acids (FFA). The second factor includes sensory properties such as taste, aroma, clarity, and purity [3]. The sustainability of the palm oil industry largely depends on maintaining product quality. One of the key issues affecting CPO quality is the high concentration of free fatty acids, which leads to a decrease in market value[4].

Based on preliminary tests conducted on CPO samples obtained from a palm oil processing plant in Selutung, Mandor District, Landak Regency, on December 7, 2023, the average FFA content was found to be 6.05%, exceeding the maximum standard quality limit of 5.0% (SNI 2901:2021).

The increase in FFA levels in CPO can result from hydrolysis or oxidation reactions. These reactions convert oils into more saturated structures by breaking the carbon–carbon double bonds of unsaturated lipids in the oil (Ilyas & Husin, 2023). Hydrolysis is accelerated by factors such as heat, moisture, acidity, and catalysts (enzymes). The formation of FFAs increases with the duration of hydrolysis, which can negatively impact CPO quality, causing rancidity, discoloration, off-flavors, and reduced production yields [5].

One effective method to reduce FFA levels in CPO is by using adsorbents. Commonly used adsorbents include bleaching earth, zeolite, and activated carbon [6]. In the bleaching stage of CPO purification, bleaching earth is typically employed[2]. However, the diverse availability of natural materials presents the potential for developing bio-adsorbents such as activated carbon, offering an alternative to conventional bleaching earth[7].

Crude palm oil (CPO) quality is critically affected by its free fatty acid (FFA) content, which must be minimized to meet industry standards and maintain product stability. While conventional adsorbents like bleaching earth are widely used to reduce FFA, there is limited research on the use of sustainable, natural bio-adsorbents such as activated carbon derived from water hyacinth activated with bilimbi fruit extract. Moreover, the influence of activation duration on the efficiency of such bio-adsorbents in reducing FFA levels remains unclear. This study addresses this gap by systematically evaluating the effect of varying activation times of water hyacinth-based activated carbon, using bilimbi extract as a natural activator, on FFA reduction in CPO. The findings are expected to contribute valuable insights into optimizing environmentally friendly adsorbent production to improve CPO quality while minimizing chemical use.[8]

One natural material that can be utilized to produce activated carbon is water hyacinth. Water hyacinth is notable for its high cellulose content, its ability to absorb heavy metals and sulfide compounds, and the fact that cellulose accounts for approximately 64.51% of its composition. Considering its high cellulose content, water hyacinth can be used as a bio-adsorbent in CPO purification. The hydroxyl groups in the cellulose molecular structure enable the adsorption of polar molecules. Activated carbon can adsorb polar compounds more effectively than non-polar ones due to its polar nature, derived from hydroxyl functional groups [9].

According to Pasaribu et al 2022 [10], increasing the amount and surface area of adsorbent particles enhances the number of ion-binding sites for FFAs, thereby improving adsorption efficiency. FFA adsorption increases with longer contact times. Their study utilized activated rice husk charcoal treated with H_2SO_4, where the optimal conditions were achieved using 40 grams of adsorbent over 5 hours, resulting in an FFA reduction from 6.3018% to 3.7673%.

Activated carbon can be produced using physical or chemical activation methods. Physical activation involves heating to evaporate moisture within the crystal pores, increasing the surface area[11]. Chemical activation uses salt, base, or acid solutions, commonly employing chemicals such as KOH, NaOH, H_3PO_4, H_2SO_4, HCl, and Na_2CO_3. However, these substances pose environmental risks [12].

In addition to environmental hazards, Dita (2020) [13] noted that using strong bases like NaOH can be dangerous without proper safety precautions. The corrosive nature of NaOH can cause burns upon skin contact. If chemical residues remain in the activated carbon due to inadequate washing, they can pose health risks when the carbon is used in food or beverage applications.

To minimize chemical use, natural materials can serve as safer alternatives. One such natural activator is Averrhoa bilimbi (bilimbi), a tropical fruit commonly grown in home gardens and plantations in Indonesia[14]. Bilimbi contains reactive compounds such as hexadecanoic acid, sulfuric acid, ferric acid, and aliphatic acids, which are essential for activating carbon due to their oxygen reactivity[12].

Research by Hatibie et al. 2022[15] utilized acid activators derived from bilimbi fruit for producing activated carbon from corn cobs through pyrolysis. Various bilimbi solution concentrations (100%, 70%, 50%, 40%, and 20%) were tested, with the 100% concentration yielding the best results: 4.4% moisture content, 2.06% ash content, and an iodine adsorption capacity of 1002.51 mg/g. These values met the requirements of SNI 06-3703-1995, which specifies a maximum moisture content of 15%, a maximum ash content of 10%, and a minimum iodine number of 750 mg/g.

A study by Kurniasih et al. 2021[12] compared different concentrations of bilimbi solution (100%, 75%, 50%, and 25%) and activation times (4, 8, 16, and 24 hours). Based on the above discussion, this study aims to investigate **"The Effect of Activation Time of Water Hyacinth-Based Activated Carbon Using Bilimbi Fruit Activator on Free Fatty Acid Content in Crude Palm Oil (CPO)"**.

Materials and methods

This study employs a Quasi-Experimental Design to investigate the effect of water hyacinth-based activated carbon, activated using starfruit (Averrhoa bilimbi) extract, on the free fatty acid (FFA) content in crude palm oil (CPO).

Population and Sample

The population in this study consists of crude palm oil (CPO). The sample comprises CPO treated with activated charcoal derived from water hyacinth, activated using starfruit extract with immersion times of 4, 8, 16, and 24 hours. A total of 24 samples were prepared, with 6 additional control samples containing untreated CPO.

The sampling technique employed is purposive sampling, where samples are selected based on specific criteria determined by the researcher.

Examination Method

The FFA content in the CPO samples was determined using the alkalimetric titration method. The percentage of FFA by weight (w/w) was calculated based on the molecular weight of palmitic acid (256) in accordance with SNI 01-2901-2006.

Sample Preparation

Preparation of Starfruit Activator[15]

Fresh starfruit was harvested, thoroughly washed, cut into small pieces, and processed using a juicer to obtain a smooth pulp, which was subsequently filtered through gauze to yield a clear liquid extract that was stored in a sealed container until use.

Preparation of Activated Charcoal from Water Hyacinth.[16]

Water hyacinth plants were collected, cleaned, and sun-dried under a paranet net for 24 hours, followed by oven drying at 105°C for 8 hours; the dried biomass was then chopped into small pieces, carbonized in a furnace at 500°C for 30 minutes, ground using a mortar, and sieved through a 100-mesh sieve to obtain fine charcoal, which was subsequently soaked in starfruit extract for 4, 8, 16, and 24 hours for activation, filtered and washed with distilled water until a neutral pH (~7) was achieved, and finally oven-dried at 120°C for 1 hour.

CPO (Crude Palm Oil) Preparation.[17]

Fresh oil palm fruits were washed and boiled for 2 hours to facilitate seed separation, then crushed to separate the mesocarp from seeds and fibers; the resulting pulp was allowed to sediment to enable oil-water separation, after which the upper oil layer was collected and reheated to reduce moisture content, yielding crude palm oil (CPO) that was subsequently stored for further processing

CPO Refining Process

Degumming Process[18].

A total of 500 mL of crude palm oil (CPO) was placed in a beaker and heated on a hot plate, after which 0.05% (v/v) or 0.25 mL of 85% phosphoric acid (H_3PO_4) was added at 80°C with continuous stirring at 500 rpm for 15 minutes; the resulting mixture was then centrifuged at 4000 rpm for 15 minutes to remove phospholipids (gum), and the degummed oil was used for the subsequent bleaching process.

Bleaching Process [9].

Four labelled beakers, each containing 100 mL of degummed crude palm oil (CPO), were prepared and supplemented with 10 grams of activated water hyacinth charcoal—previously activated with starfruit extract for 4, 8, 16, and 24 hours, respectively—then stirred using a flocculator at 500 rpm for 1 hour, allowed to stand for 24 hours, and subsequently filtered to separate the charcoal from the CPO.

Free Fatty Acid (FFA) Content Analysis

The filtered CPO samples were analyzed for FFA content using the alkalimetric titration method, determining the FFA level as a percentage (w/w) of palmitic acid equivalent in accordance with SNI 01-2901-2006 standards.

Examination Procedure

Standardization of Oxalic Acid ($H_2C_2O_4$) Solution with Sodium Hydroxide (NaOH) [19]

Prior to determining the free fatty acid content, the NaOH solution was standardized to ensure accurate titration results by pipetting 10 mL of oxalic acid ($H_2C_2O_4$) solution into a 250 mL Erlenmeyer flask, adding 2–3 drops of 1% phenolphthalein indicator, and titrating with 0.05 N NaOH while stirring until a stable pink endpoint was reached, with the volume of NaOH used recorded and the normality calculated using the equation ($V_1 \times N_1$) = ($V_2 \times N_2$), where V_1 and N_1 represent the volume and normality of NaOH, and V_2 and N_2 represent the volume and normality of oxalic acid, respectively.

Determination of Free Fatty Acid Content (SNI 01-2901-2006)

The free fatty acid (FFA) content in crude palm oil (CPO) was determined using an alkalimetric titration method, in which 5 grams of CPO—preheated to 60°C–70°C with stirring for homogenization—was weighed into a 250 mL Erlenmeyer flask, mixed with 50 mL of a neutralized solvent (ethanol or alcohol), and heated until fully dissolved; 1–2 drops of phenolphthalein indicator were then added, and the mixture was titrated with standardized NaOH solution while continuously shaking until a stable pink endpoint was reached, with the volume of NaOH recorded for FFA calculation.

$$\frac{(VxN)NaOHxMW\ Fatty\ Acid\ (25{,}6)}{Gram\ Sampel} x100\% = palmitate\ \%$$

Where:

V NaOH: volume produced during titration (ml)

N NaOH: Normality of NaOH

Data Analysis

The collected data were processed using the SPSS software, employing linear regression analysis to examine and interpret the relationship between activation time and the reduction of free fatty acid (FFA) levels in crude palm oil (CPO).

Results

Table 1: Results of the Effect of Activation Time of Water Hyacinth-Based Activated Charcoal with Starfruit Activator on Free Fatty Acid Levels in Crude Palm Oil (CPO)

Code	Free fatty acid (%)							
	1	2	3	4	5	6	avg	decline
K	12,41	12,38	12,67	12,60	12,67	12,52	12,54	0,00
A(4h)	8,88	9,60	9,30	8,72	10,22	9,55	9,38	25,22
B(8h)	6,46	6,23	6,38	6,37	6,17	6,30	6,32	49,62
C(16h)	6,40	6,40	6,67	5,99	5,88	6,38	6,29	49,87
D(24h)	5,27	5,12	5,31	6,33	5,71	5,50	5,54	55,83

Table 1 shows that the initial free fatty acid (FFA) level in the unactivated sample was 12.54%, indicating a relatively high FFA concentration. Following a 4-hour activation process, the FFA content decreased to 9.38%. Extending the activation time to 8 hours resulted in a further reduction to 6.32%. This decline continued with a 16-hour activation period, reaching 6.29%. Finally, the 24-hour activation yielded the lowest FFA level of 5.54%. These results demonstrate a consistent decrease in FFA content with increasing activation time. Although the 24-hour activation produced the most favorable outcome, the FFA level remained slightly above the commonly accepted industry threshold of ≤5%.

Table 2 : Descriptive Analysis of the Effect of Activation Time of Water Hyacinth-Based Activated Charcoal with Starfruit Activator on Free Fatty Acid Levels in Crude Palm Oil (CPO) in (%)

Descriptive Statistics

Variabel	N	Minimum	Maximum	Mean	St Dev
K	6	12.38	12.67	12.5417	0.12671
A(4h)	6	8.72	10.22	9.3783	0.54312
B(8h)	6	6.17	6.46	6.3183	0.10647
C(16h)	6	5.88	6.67	6.2867	0.29487
D(24h)	6	5.12	6.33	5.5400	0.43745

Table 2 shows the descriptive statistical analysis of FFA data. The mean FFA level in the control group (without activation) was 12.54%. With 4 hours of activation, the mean decreased to 9.38%, followed by 6.32% after 8 hours, 6.29% after 16 hours, and 5.54% after 24 hours of activation. These values indicate a consistent downward trend in FFA content as activation time increases. The highest FFA level was observed in the control group (12.54%), while the lowest was recorded after 24 hours of activation (5.54%). These average values suggest that extending the activation time enhances the reduction of FFA, with 24 hours being the most effective duration. However, the resulting FFA level remains slightly above the standard industry limit, which is typically set at ≤5%.

Table 3: Shapiro–Wilk Test for Normality

Group	W-statistic	pValue	Interpretation
Control	0.854	0.169	Normally distributed
4 H	0.839	0.128	Normally distributed
8 H	0.906	0.413	Normally distributed
16 H	0.899	0.366	Normally distributed
24 H	0.976	0.929	Normally distributed

Table 3 shows the normality test results indicate that the data from all treatment groups followed a normal distribution, as evidenced by p-values greater than 0.05. This confirms that the data meet the basic assumptions for parametric statistical analysis. Table 4 presents the results of Levene's test for homogeneity of variances, which yielded a p-value of 0.470, indicating that the variances across groups are homogeneous and therefore suitable for ANOVA.

Table 4: Levene's Test for Homogeneity of Variances

Test	p-Value	Interpretation
Levene's Test	0.470	Variances are homogeneous

Table 5: ANOVA Test on the Effect of Activation Time of Water Hyacinth-Based Activated Charcoal with Starfruit Activator on Free Fatty Acid Levels in Crude Palm Oil (CPO)

ANOVA[a]

Model	Sum of Squares	df	Mean Square	F	Sig.
Regression	39.998	1	39.998	58.067	0.000[b]
Residual	15.154	22	0.689		
Total	55.152	23			

Table 5 shows the ANOVA results reveal a significant effect of activation time on the FFA levels in crude palm oil (CPO), with an F-value of 58.067 and a p-value of less than 0.001. This confirms that variations in activation time significantly influence FFA reduction and that the differences among treatment groups are statistically significant.

Table 6: Coefficients a Effect of Activation Time of Water Hyacinth Activated Charcoal with Starfruit Activator on Free Fatty Acid Levels in CPO (Crude Palm Oil)

Coefficients[a]

Model	Unstandardized Coefficients		Standardized Coefficients	t	Sig.
	B	Std. Error	Beta		
(Constant)	9.768	.415		23.537	.000
Charcoal Activation Time	-1.155	.152	-.852	-7.620	.000

Table 6 shows the regression analysis, in which the β coefficient was -0.852, indicating a very strong negative correlation between activation time and FFA content. This finding confirms that extending activation time significantly reduces FFA levels, thereby demonstrating the effectiveness of the activation process in improving CPO quality.

Initial FFA content in the untreated CPO was recorded at an average of 12.54%. Following treatment with activated carbon for 4, 8, 16, and 24 hours, the FFA content decreased progressively. The CPO treated with 4-hour activated carbon exhibited an average FFA content of 9.38%, while 8-hour and 16-hour treatments reduced the FFA levels to 6.32% and 6.29%, respectively. The 24-hour activation achieved the most significant reduction, resulting in an average FFA content of 5.54%.

Discussion

This study evaluated the influence of activation duration on the efficacy of water hyacinth-derived activated carbon, employing *Averrhoa bilimbi* (starfruit) extract as an activating agent, in reducing the free fatty acid (FFA) content in crude palm oil (CPO). Despite the application of activated carbon at varying activation times, the FFA concentrations in all treated CPO samples remained above the maximum permissible limit of 5% stipulated by the Indonesian National Standard (SNI 2901:2021). Although extended activation time demonstrated an improved adsorption capacity, it was insufficient to achieve full compliance with industrial-grade oil specifications. These findings suggest that activation time, while critical, may require further optimization or integration with additional refining techniques to effectively meet quality standards.

The persistently high initial FFA levels observed in the untreated CPO (mean: 12.54%) are likely attributable to several extrinsic factors, particularly environmental and post-harvest handling conditions. Elevated rainfall during harvesting periods increases the moisture content in oil palm fruits, thereby promoting the hydrolysis of triglycerides into FFAs. Furthermore, substandard hygienic practices can introduce microbial contaminants that catalyze lipid degradation. Fruit maturity also exerts a considerable influence, with both underripe and overripe fruits known to exhibit elevated FFA levels due to disrupted enzymatic activity and membrane integrity.

Anomalies identified within the 16-hour activation group, where certain replicates demonstrated unexpectedly increased FFA levels, may reflect inconsistencies in sample weighing or uncontrolled fluctuations in thermal conditions during processing. These variables can alter the kinetics of adsorption and degradation reactions. Supporting this observation, Nurfiqih et al. 2021[20] reported that insufficient thermal energy may hinder the progression of chemical reactions, resulting in incomplete FFA reduction.

Moisture content analysis of the activated carbon revealed an inverse relationship between activation time and water content, with values declining from 0.63% at 8 hours to 0.28% at 24 hours. All measurements fell within the acceptable threshold (<15%) as prescribed by SNI 06-3730-1995, confirming the suitability of the material for adsorption applications. These findings are consistent with the hygroscopic nature of activated carbon and suggest that lower concentrations of activating agents or higher dilutions may increase moisture retention[12].

The degumming process, integral to the physical refining of CPO, aims to remove phospholipids that adversely affect oil quality attributes such as flavor and color. Process parameters including agitation speed and temperature were found to significantly influence degumming efficiency. The addition of phosphoric acid further enhanced this process by facilitating the formation of precipitable complexes with phospholipids, as previously demonstrated by Ristianingsih et al. 2011 .[21]

The activation mechanism is driven by the mildly acidic nature of starfruit extract, which facilitates the removal of volatile impurities and enhances porosity through chemical etching. Aisyah et al. (2019) reported that extended activation time correlates with increased surface area of activated carbon, attributable to greater pore development. This structural modification is believed to underpin the improved FFA adsorption observed. Moreover, starfruit extract contains multiple bioactive constituents—such as oxalic acid, essential oils, phenolics, flavonoids, and pectin—that may contribute synergistically to its activating potential.[14]

The synergistic effect between Averrhoa bilimbi extract and the porous structure of activated carbon derived from water hyacinth may enhance the reduction of free fatty acids (FFA) in crude palm oil (CPO). Bilimbi extract contains bioactive compounds such as oxalic acid, flavonoids, and phenolics, which can facilitate the activation process by increasing the surface area and porosity of the carbon material. This enhanced porosity improves the adsorption capacity of the activated carbon, leading to more effective FFA removal. Studies have shown that the acidic nature of bilimbi extract aids in the development of micropores during activation, which are crucial for adsorbing small molecules like FFAs. Therefore, the combination of bilimbi extract and water hyacinth-based activated carbon presents a promising natural approach for improving CPO quality by reducing FFA content.[22–24]

Inferential statistical analysis using ANOVA revealed that activation time had a statistically significant effect on FFA reduction ($p < 0.001$). The linear regression model exhibited a strong negative correlation ($\beta = -0.852$) between activation duration and FFA content, further supporting the hypothesis that increased activation time enhances the adsorptive efficiency of the carbon material.

In summary, water hyacinth-based activated carbon, when activated using *Averrhoa bilimbi* extract, presents a viable natural alternative for the reduction of FFA in CPO. However, the results suggest that further process optimization—such as increased activation temperatures, extended activation durations, or integration with complementary refining stages—is necessary to meet regulatory quality standards. Given the abundant cellulose (17%) and hemicellulose (43%) content in water hyacinth, the material represents a sustainable and cost-effective precursor for developing high-performance bioadsorbents in the edible oil industry.[25]

Conclusion

The study demonstrated that activated carbon derived from water hyacinth and activated with bilimbi (*Averrhoa bilimbi*) extract effectively reduced free fatty acid (FFA) levels in crude palm oil (CPO). The 24-hour activation duration produced the lowest FFA content at 5.54%, showing the best adsorption performance among all treatments. While the 24-hour activation showed the greatest reduction in FFA, additional refinement steps are necessary to meet the industrial quality standard of ≤5% FFA.

Conflict of interest

Authors state no conflict of interest.

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