SPIN 7 (2) (2025) 187-196

SPIN
JURNAL KIMIA & PENDIDIKAN KIMIA
https://journal.uinmataram.ac.id/index.php/spin

COMPARATIVE EFFECTIVENESS OF ACTIVATOR HCl AND NaOH ON PALM SHELL ACTIVATED CARBON IN CRUDE PALM OIL (CPO) REFINING

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DOI: 10.20414/spin.v7i2.14518

History Article
Accepted:
Sept 19, 2025
Reviewed:
Nov 20, 2025
Published:
Dec 20, 2025

Keywords:
Activated Carbon Adsorption, CPO Purification, Palm Shell Biomass

ABSTRACT

This study aims to utilize palm kernel shell (CKS) waste as a raw material for chemically activated carbon using HCl and NaOH. The resulting activated carbon was applied in the purification of Crude Palm Oil (CPO). Characterization was conducted using XRD, FTIR, and SEM instruments to observe crystal structure, functional groups, and surface morphology. Results showed that activated carbon had exhibited smoother and more uniform surfaces compared to the unactivated sample. CPO purification effectiveness was analyzed based on the reduction of free fatty acid (FFA), peroxide value, moisture content, and color changes. Optimal results were achieved using NaOH 3M with 72,62% *FFA reduction,* 64,44% *peroxide value reduction, 0.11% moisture content, and color improvement to a scale of 10 on the Lovibond Tintometer. Therefore, NaOH 3M activated carbon exhibited the highest adsorptive performance in CPO purification.*

ABSTRAK

Penelitian ini bertujuan dalam memanfaatkan limbah cangkang kelapa sawit (CKS) sebagai bahan baku karbon aktif yang diaktivasi secara kimia menggunakan HCl dan NaOH. Karbon aktif yang dihasilkan digunakan dalam proses pemurnian *Crude Palm Oil* (CPO). Karakterisasi dilakukan menggunakan instrumen XRD, FTIR, dan SEM untuk mengetahui struktur kristal, gugus fungsi, dan morfologi permukaan karbon aktif. Hasil penelitian menunjukkan bahwa karbon aktif teraktivasi memiliki permukaan lebih halus dan rata dibandingkan sebelum aktivasi. Efektivitas pemurnian CPO dianalisis berdasarkan penurunan *kadar free fatty acid* (FFA), bilangan peroksida, kadar air, dan perubahan warna. Hasil optimal diperoleh pada aktivator NaOH 3M dengan penurunan FFA sebesar 72,62%, bilangan peroksida 64,44%, kadar air 0,11%, dan perubahan warna hingga skala 10 pada Lovibond Tintometer. Dengan demikian, karbon aktif NaOH 3M menunjukkan efektivitas tertinggi dalam proses pemurnian CPO.

How to Cite

Leofayuda, M.S., Aisya'adah, U., Nuraini, S., ..., & Lestari, I. (2025). Comparative Effectiveness of Activator HCL and NaOH on Palm Shell Activated Carbon in Crude Palm Oil (CPO) Refining. *SPIN-Jurnal Kimia & Pendidikan Kimia*. 7(2). 187 - 196.

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p-ISSN: 2580-2623
e-ISSN: 2745-6854

INTRODUCTION

Crude palm oil (CPO) is the main raw material in the manufacture of vegetable oil, consisting of triglycerides (glycerin and fatty acids), non-triglycerides (phosphatides, raffinose, pentosan, carotene, gossypol). CPO also contains hydrocarbons (sterols, ketones, butyric acid, tocopherol) and impurities (gum), protein phosphatides, carbohydrates, water, and heavy metals, so that a purification process is required to obtain pure CPO (Yustinah et al., 2023). Oil quality is assessed based on Free Fatty Acid (FFA) content, peroxide value, water content, and color, using a purification process to meet the quality requirements for crude CPO according to SNI 01-2901-2006. FFA levels are produced by the hydrolysis and oxidation process of palm oil in the form of glycerol, but high FFA levels (>5%) cause rancidity, changes in taste, and color of CPO (Aldi et al., 2024). The peroxide value, or acid value, is determined by oxidation and hydrolysis to quantify the extent of damage to oil or fat. Unsaturated fatty acids can bind oxygen at their double bonds, forming peroxides, which cause rancidity and reduce the oil's nutritional value. Peroxide determination can be performed using standard iodimetry methods (Pandia et al., 2018).

According to Setiapraja et al. (2024), a comparison of several types of biomass waste, such as coconut shells, corn cobs, empty oil palm fruit bunches (EFBs), sawdust, and palm kernel shells (PKS), found that PKS was the most optimal for producing activated carbon due to its highest lignin content, reaching 53.85%. Lignin is the primary contributor to bound carbon formation during carbonization, thereby increasing the yield of the resulting activated carbon. The utilization of biomass waste such as palm kernel shells (PKS), coconut fiber, and corn cobs as activated carbon for crude palm oil (CPO) refining, through the results of free fatty acid (FFA) content, obtained by comparing the PKS waste with 0.322%, coconut fiber with 0.34%, and corn cobs with 0.19%. Furthermore, the peroxide value of PKS was 0.4478 mgKOH/g, and corn cobs had a peroxide value of 0.42 mgKOH/g (Kurniawan et al., 2023). PKS is a solid waste from the palm oil processing industry that has not been optimally utilized. This is because the waste is produced in large quantities; a capacity of 30 tons will produce 28-35 tons of PKS waste, which is difficult to degrade, making it effective for use as an adsorbent material for making activated carbon (Hikmawan & Naufa, 2022).

Palm kernel shells are a solid waste stream from the palm oil processing industry that is abundant but not optimally utilized. Each 30-ton CPO processing plant can produce 28–35 tons of Palm Kernel Shell (PKS) waste, which is relatively difficult to degrade and thus a potential raw material for activated carbon (Hikmawan & Naufa, 2022). Utilizing this waste not only helps reduce environmental burdens but also provides added economic value through the production of high-value adsorbents.

Palm kernel shells (PKS) are the hard part that protects the palm fruit and are a waste product from crude palm oil (CPO) processing (Raja & Giyanto, 2020). They are used as adsorbents after activation with activators such as HCl and NaOH. The results of CKS activation using an HCl: NaOH activator ratio showed a water content of (0.5644:0.6393), ash content of (0.6194:0.7862), and iodine absorption capacity of (710.0217:672.547). This indicates that the use of the HCl activator reduces water and ash content, whereas the NaOH activator increases iodine absorption capacity. The purpose of adding the activator is to increase the surface area and porosity to accelerate the clarification process of CPO (Sirajuddin et al., 2022).

Based on this description, it can be concluded that the use of activated carbon from palm kernel shells is very promising in improving CPO quality. However, the effectiveness of chemical activation with HCl and NaOH on the performance of activated carbon in reducing oil quality parameters (FFA

and peroxide value) remains to be investigated. Therefore, this study aims to compare the effectiveness of HCl and NaOH activators in the manufacture of activated carbon from palm shells and evaluate their performance in the crude palm oil (CPO) refining process.

METHODS

Tools and Materials

The tools needed for this research include beakers and Erlenmeyer flasks, filter paper, a separating funnel, a mortar and pestle, a simple furnace, a pH indicator, a burette, clamps and a stand, a forced convection oven, a desiccator, a test sieve, a magnetic stirrer, a hot plate, vials, and instruments such as a scanning electron microscope (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and X-ray diffraction (XRD). The materials needed for this research are palm kernel shell waste, sodium hydroxide (NaOH), hydrogen chloride (HCl), distilled water, phenolphthalein (PP), saturated potassium iodide (KI), sodium thiosulfate, chloroform, acetic acid, 10% phosphoric acid, 95% ethanol, and starch solution.

Research Procedures

Palm Shell Waste Adsorbent Carbonization

Palm shell waste was obtained, washed to remove impurities, dried in the open air for more than 3 days, and the fibers were removed from the Palm Kernel Shell to optimize carbon formation. The PKS was fired in a closed-container furnace at 500°C for 3 hours, ground using a pestle, and sieved through a 200-mesh sieve to obtain palm shell charcoal (Susanto et al., 2022).

Palm Shell Adsorbent Activation

Two different activators were prepared: HCl and NaOH in equal concentrations of 1M and 3M. 10 g of the Palm Kernel Shell carbon formed after the carbonization process was added, immersed in 100 mL of each activator in a 250 mL beaker. The mixture was allowed to stand for 24 hours, then washed with distilled water until it reached a neutral pH (±7) using a pH indicator. Next, the mixture was filtered through a vacuum filter to yield the Palm Kernel Shell carbon. The obtained Palm Kernel Shell carbon was then dried in an oven at 150°C for more than 2 hours to form palm shell activated carbon (CKSAC-HCl and CKSAC-NaOH 1M and 3M). It was then stored in a desiccator until further characterization (Ifa et al., 2022).

Characterization of Palm Shell Activated Carbon

The characterization of activated carbon from palm shells using chemical activation. FTIR characterization was recorded in the wavenumber range 500-4000 cm-1 at a resolution of 4 cm-1. This was used to determine the surface organic functional groups in the activated palm shells. These include the C=O functional group at a peak of 1650 cm-1, the C=C functional group between 1650-1500 cm-1, and the C-O functional group at a peak of 1250-1000 cm-1. The morphology of crude palm kernel shells (PKS) and activated carbon (CKSAC) was determined using a scanning electron microscope (SEM) (Hidayu et al., 2019). X-ray diffraction (XRD) was used to demonstrate the composition and crystallinity of activated carbon from palm kernel shells (CKS), as indicated by the peak at 2θ in the XRD characterization pattern (Hisbullah et al., 2022).

Crude Palm Oil (CPO) Purification

The degumming method was used on crude palm oil (CPO). 100 mL of CPO was prepared, heated on a hot plate to 80°C, and 10 mL of 10% phosphoric acid was added. Next, 20 mL each of CPO and palm kernel shell activated carbon (5% CPO by weight) were prepared in Erlenmeyer flasks. They were mixed using a magnetic stirrer at 400 rpm for 2 hours and allowed to stand for 30 minutes at 80°C. The CPO was then filtered 2-3 times through a funnel and filter paper, and the volume of each filtrate was measured. This process resulted in the CPO becoming clear and free of sap. Next, the CPO was tested for free fatty acid (FFA), peroxide value, water content, and color (Susanto et al., 2022).

Determination of Free Fatty Acid (FFA)

According to Marfitania et al. (2024), free fatty acid (FFA) analysis was performed by adding palm kernel shell-activated carbon to CPO via the adsorption method. This was followed by a titration method in accordance with the American Oil Chemists' Society (AOCS) 1990 standards. To a total of 2.5 g of CPO heated to 60°C, 10 mL of 95% ethanol was added and homogenized. Three drops of phenolphthalein (PP) indicator were then added. The titration was then carried out with 0.1 N NaOH. The endpoint of the CPO sample titration turned pink, and the volume of NaOH used was calculated.

$$\text{FFA (\%)} = \frac{25.6 \text{ x N x V}}{w} \text{ x } 100\% \qquad \ldots 1)$$

The FFA percentage was calculated based on the equation above, where N is the normality of the NaOH, V is the volume of NaOH, and w is the mass of the CPO.

Determination of Peroxide Value

According to Nurdjannah et al., (2022), to determine the peroxide value (PV) using the method (AOCS 2003), weigh 0.05 g of sample into a closed Erlenmeyer flask, then add a solution of acetic acid and chloroform (3:2) and stir until all the sample is dissolved and homogeneous. Next, add 0.5 mL of saturated potassium iodide solution, homogenize the solution for 1 minute, then add 30 mL of distilled water. Next, add the starch solution and titrate with 0.01 N sodium thiosulfate solution until the color disappears.

$$\text{Peroxide Value } \frac{Meg}{Kg} = \frac{(\text{S}-\text{B}) \text{ x N x } 1000}{sample\ mass\ (g)} \qquad \ldots 2)$$

The peroxide value (PV) is based on the equation above, where S is the titrant volume of sodium thiosulfate solution for the sample (mL), B is the titrant volume of sodium thiosulfate solution for the blank (mL), and N is the normality of the sodium thiosulfate solution.

Water Content

The water content was determined using the gravimetric method by evaporating the water contained in the refined CPO oil by drying it in an oven for 4 hours at 105°C to obtain a constant weight, the dry weight of the CPO. The calculated CPO sample weight was calculated by comparing the constant weight with the SNI water content standard for CPO, which is a maximum of 0.5% (Kurniawan et al., 2023).

Color Change

Color can determine the quality of CPO, as measured using a visual method. For comparison, color concentration in oil refers to the combination of red and yellow using a color scale with a Lovibond Tintometer. The working principle is color matching using the color panel on the Lovibond Tintometer (Kurniawan et al., 2023).

RESULT AND DISCUSSION

Activated Carbon Characterization

X-Ray Diffraction (XRD) Characterization

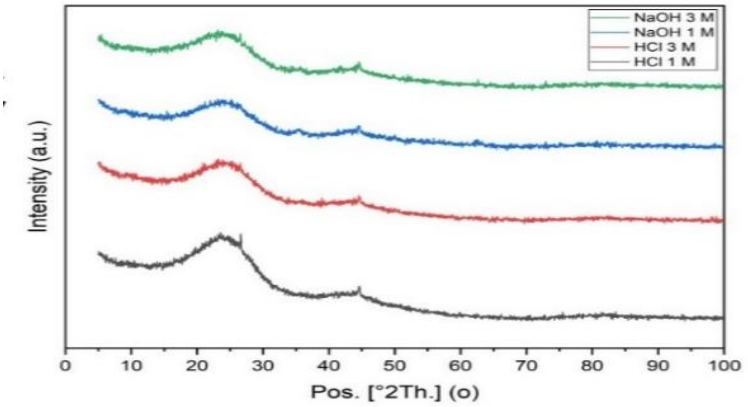

Figure 1. XRD characterization test results

Based on the results of the X-ray diffraction (XRD) test, it can be seen that the activated carbon activated by HCI and NaOH 1M and 3M successfully formed activated carbon, indicated by the formation of peaks at 2θ=20-50˚, namely 26.6135˚ and 44.6351˚, 44.672˚, 35.3689˚ and 44.5621˚, 44.5871˚, respectively. This refers to the ICSD interpretation card peak standard No.00-025-0284 with the 2"θ" peak being 26.603˚ and 44.670˚, 44.670˚, 33.153˚ and 45.547˚, 44.572˚ with each having the same miler index (HKL) value of 101, this indicates the crystal characteristics forming the same hexagonal structure. However, at the 1M NaOH peak of 35.3689˚ there is a discrepancy due to variations in the sample or the presence of unwanted additional phases..

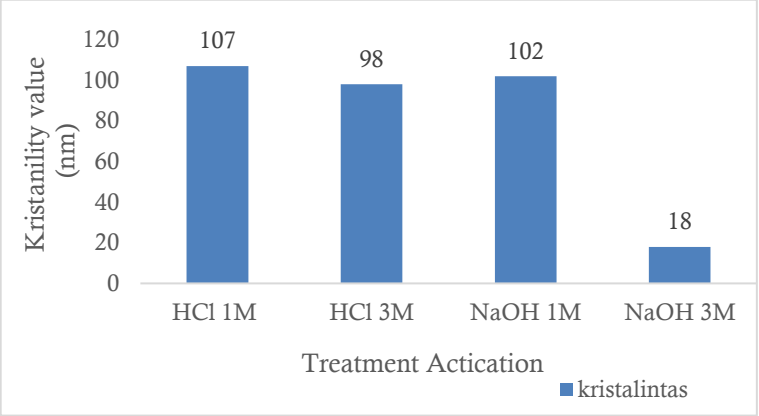

Figure 2. Crystallinity of Palm Kernel Shell activated carbon with various activations

XRD analysis can also be used to determine the average size of activated carbon crystallinity using the Scherrer equation. Based on Figure 2, the highest to lowest crystallinity of activated carbon samples activated with 1M HCl, 1M NaOH, 1M HCl and 3M NaOH are 107 nm, 102 nm, 98 nm, and 18.02 nm, respectively. Based on the crystallinity value, the 3M NaOH activated activated carbon sample shows the smallest crystallinity indicating the most amorphous structure and the diffraction pattern shows no significant sharp peaks other than at an angle of 2"θ" around 44.5871° due to chemical activation treatment which indicates a reduction in the crystalline phase. Amorphous characteristics have the potential to produce active sites in absorbing adsorbate molecules.

Fourier Transform Infrared Spectroscopy (FTIR) Characterization

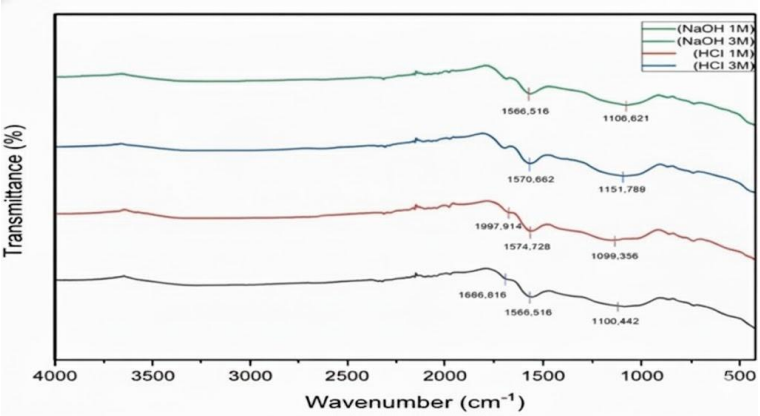

Figure 3. FTIR characterization test results

Based on the results of the FTIR characterization test analysis, the absorption band of HCl activated palm shell activated carbon obtained C=O stretching functional groups at peaks of 1697.914 cm^{-1} and 1693.808 cm^{-1}, C=C stretching functional groups at peaks of 1574.728 cm^{-1} and 1566.516 cm^{-1}. + C-O functional groups peaks of 1096.356 cm^{-1} and 1100.462 cm^{-1} while NaOH activation, C=C functional groups at peaks of 1566.516 cm^{-1} and 1570.622 cm^{-1} and C-O functional groups at peaks of 1106.621 cm^{-1} and 1151.789 cm^{-1}. The results of the FTIR analysis are in accordance with previous research conducted by (Hidayu et

al., (2019), obtained palm shell activated carbon has a C=C stretching functional group at the 1650-1500 cm^{-1} peak and C-O at the 1200-1000 cm^{-1} peak, during the activation process the activated carbon produces fewer functional groups because most of the functional groups are degraded due to the activation treatment.

Scanning Electron Microscope (SEM) Characterization

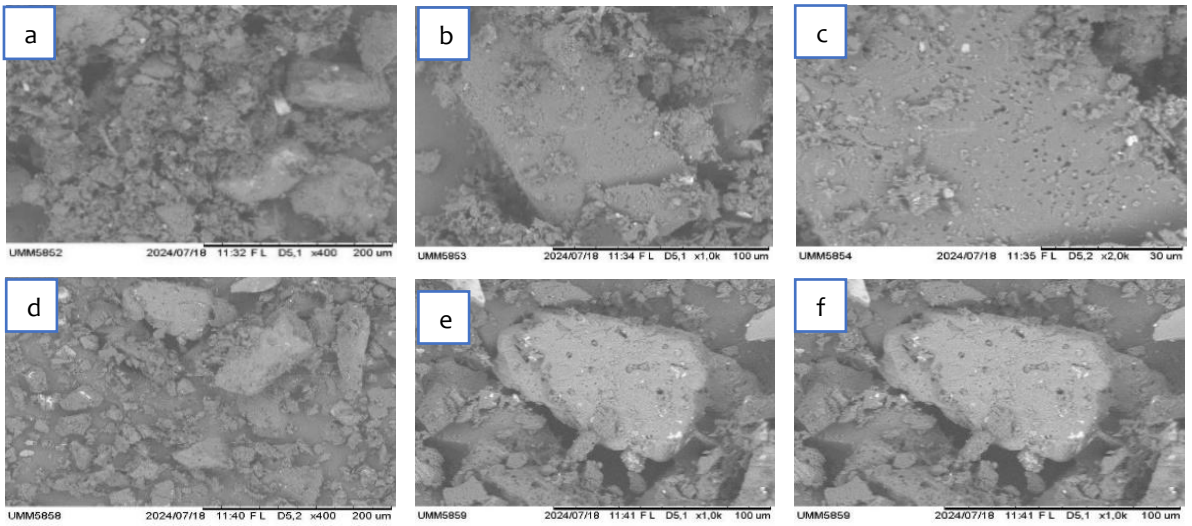

Figure 4. Activated Carbon Morphology Results of SEM Analysis: (a, b and c) before activation and (d, e and f) after activation (3M NaOH) at magnifications of 200 µm, 100 µm, and 30 µm.

This analysis shows the morphology of the palm kernel shells before and after activation with 3M NaOH. The comparison of particle diameters in µm (micrometers) at 200 µm, 100 µm, and 30 µm is used to indicate the physical dimensions of the PKS sample. Figures 4 (a), (b), and (c) show that the SEM analysis of unactivated PKS has an irregular structure with large average pores; the surface looks rough and wavy. Meanwhile, Figures 4(d), 4 (e), and 4 (f) show that the structure of 3M NaOH-activated carbon appears more regular, with more pores, and the surface is smoother and flatter. According to Susanto et al. (2023), previous research showed that unactivated palm kernel shells have a rough surface, whereas activated carbon has a regular, slightly rough surface.

Determination of Free Fatty Acid (FFA)

The free fatty acid (FFA) content in CPO is determined based on the CPO purification process during degumming and bleaching. According to Susanto et al. (2022), a 5% Palm Kernel Shell-activated carbon concentration and a CPO heating temperature of 80°C significantly affected the FFA quality parameter, resulting in the lowest FFA reduction of 3.86 wt%. Therefore, this parameter was used as a reference for comparing the FFA reduction results between activated carbon activated with HCl and NaOH (1 M and 3 M). Furthermore, the FFA reduction results also indicate that the effectiveness of activated carbon is significantly influenced by the type of activator used. Acidic activators, such as HCl, tend to produce more regular pores and increase the surface area of the activated carbon, thus increasing its adsorption capacity for free fatty acids. Meanwhile, basic activators, such as NaOH, can convert the microporous structure into a mesoporous structure, which is beneficial for the adsorption of larger molecules but can sometimes reduce the stability of the carbon surface (Aldi et al., 2024).

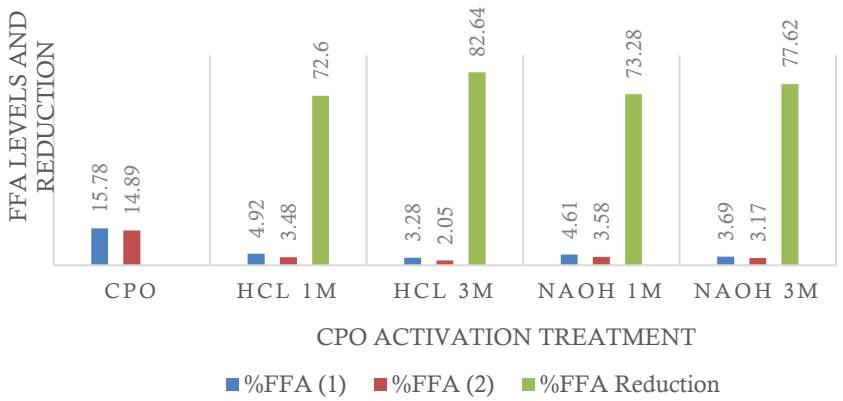

Figure 4. Activated Carbon Morphology Results of SEM Analysis: (a, b and c) before activation and (d, e and f) after activation (3M NaOH) at magnifications of 200 µm, 100 µm, and 30 µm

Based on Figure 5, the results of the crude CPO adsorption process using CKS activated carbon with HCl and NaOH activation at varying concentrations of 1 M and 3 M show a reduction in free fatty acids (FFA), with the highest percentage in the 3 M HCl CPO sample at 82.64%, while the lowest FFA percentage was in the 1 M HCl CPO sample at 72.6%. This indicates that the 3 M HCl activated CKS activated carbon produces a more uniform pore size distribution and active functional groups (carbonyl and hydroxyl) that interact more strongly with the polar free fatty acid molecules, thereby enhancing the adsorption interaction between the carbon surface and the FFA molecules. Furthermore, all refined samples showed a %FFA value lower than the SNI 01-2901-2006 threshold of 5% (Aldi et al., 2024). Thus, the 3 M HCl activated activated carbon produced the most optimal performance with a final FFA value of 2.05%, thereby improving the quality of the CPO.

Peroxide Value Determination

Table 1. Value of reduction in peroxide value on CPO purification

CPO	Peroxide Value (meg/kg)	Decreased peroxide levels(%)
CPO mentah	18.0 meg/kg	-
HCl 1M	5.2 meg/kg	71.11 %
HCl 3M	11.6 meg/kg	36.56%
NaOH 1M	13.2 meg/kg	26.67%
NaOH 3 M	64 meg/kg	64.44%

The peroxide value is used to quantify the extent of CPO damage by measuring the total peroxide content. It is well known that the higher the unsaturated fatty acid content in oil, the more likely it is to react with oxygen at its double bonds to form peroxides, thereby increasing susceptibility to oxidation, also known as auto-oxidation. Oxidation increases the peroxide value and degrades the quality of CPO. According to Fadhilah et al. (2024), SNI No. 7709-2019 states that the maximum peroxide value (PV) is 10 meq/kg. The PV values of the 3M HCl and 1M NaOH samples were relatively high, indicating low thermal oxidation stability due to the suboptimal absorption capacity of unsaturated fatty acids in the oil. In comparison, the 1M HCl and 3M NaOH samples met the standard with the highest reduction in peroxide value, reaching 71.11% and 64.44%, respectively.

Water Content

Table 2. Results of water content analysis of CPO

	SNI	Raw	HCl 1M	HCl 3M	NaOH 1M	NaOH 3M
CPO	0.5%	1.55%	0.13%	0.40%	0.90%	0.11%

Based on Table 2, the moisture content of 1.55% in crude palm oil (CPO) exceeds the limit set by SNI No. 01-2901-2006, which is 0.45%. After purification and activation, the greatest

reduction in moisture content was achieved with 3 M NaOH-activated carbon (0.11%), followed by 1 M HCl (0.13%) and 3 M HCl (0.40%). All samples met the SNI threshold for moisture reduction, except the 1 M NaOH sample, which still had a moisture content of 0.90%. Based on these results, higher activator concentrations increase the adsorbent's ability to bind water molecules, resulting in greater moisture reduction during CPO refining.

Determining moisture content is important because excessive moisture can induce hydrolysis, converting triglycerides into free fatty acids and glycerol, thereby increasing free fatty acid levels and affecting the color quality of crude palm oil (CPO). The thermogravimetric method, which relies on the evaporation of water at a specified temperature, is the standard method in the oil and fat industry for measuring the water content of CPO samples (Kurniawan et al., 2023).

Color Change

Figure 6. Comparison of (a) Lovibond tintometer color scale, (b) CPO HCl 1M, (c) CPO HCl 3M, (d) CPO NaOH 1M, (e) CPO NaOH 3M, and (f) Raw CPO

Based on the results of the visual test comparison of CPO color concentration refers to a combination of red, orange and yellow and a scale of 2 (bright) to 22 (dense) on the lovibond tintometer with degumming CPO, it was obtained that crude palm oil (CPO) results (b) were dark reddish orange on a scale of 17, (c) and (d) were reddish orange on a scale of 13 while (e) was orange on a scale of 10 while the results of raw CPO obtained a brownish orange color on a scale of 22 lovibond tintometer. Based on this statement, it is known that CPO has gone through the degumming and bleaching process and has a brighter color than raw CPO, producing a more concentrated color, which indicates that CPO has gone through the degumming and bleaching process successfully, removing impurities in the form of gum, namely triglycerides and non-glycerides.

CONCLUSION

Based on the research results, the following conclusions were drawn: XRD characterization analysis showed that CKS-AC HCl and NaOH at 1M and 3M concentrations successfully formed activated carbon, characterized by the formation of 2θ=20-50˚, and the smallest crystallinity level was found in 3M NaOH at 18.02 nm and 3M HCl at 98 nm compared to other concentrations. FTIR analysis revealed the main functional groups C=C stretching and C-O. SEM analysis showed that the structure of activated carbon had a smooth and flat surface, compared to the rough and wavy surface of unactivated activated carbon.

Based on the results of CPO purification between CKS-AC HCl and NaOH with concentration variations of 1M and 3M, the best comparison results for each activator were obtained in reducing FFA levels, namely HCl 3M 82.64%: NaOH 3M 77.62%, reducing peroxide numbers, namely HCl 1M 71.11%: NaOH 3M 64.44%, water content HCl 1M 0.13%: NaOH 3M 0.11% and color changes in the Lavibon tintometer scale HCl 3M 13: NaOH 3M 10. Based on these data, 3M NaOH gave the best results across the quality parameters FFA, peroxide value, water content, and color. However, HCl 3M showed a slightly greater FFA reduction (82.64%) than NaOH 3M (77.62%); the difference was only 5.02%, which was not statistically significant. Thus, 3M NaOH activation is determined to be the optimal condition for producing CKS-AC activated carbon with the highest adsorption effectiveness for refining crude palm oil (CPO).

ACKNOWLEDGEMENT

The author would like to express his gratitude to the Ministry of Education, Culture, Research, and Technology of the Republic of

Indonesia (Kemendikbudristek) for the funding support through the Exacta Research Student Creativity Program (PKM-RE) so that this research can be carried out well and the staff of the instrument and final project laboratory of the Faculty of Science and Technology, University of Jambi for the opportunity to use the excellent facilities.

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