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Purification of Crude Palm Oil (CPO) Using Cocoa Fruit Skins and Rice Straw Waste as Bioadsorbents

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ABSTRACT: Crude palm oil is a crude oil that contains triglyceride compounds, free fatty acids, pigments, phosphatides, partial glycerides, coloring agents, and carotene k. The aims of this research are to examine the effect of contact time and mass ratio of bioadsorbents derived from cocoa pod peels and rice straw waste on the reduction of free fatty acid levels, peroxide value, and carotene content. Waste rice straw and cacao fruit peels are carbonized before the bioadsorbents are activated. Furthermore, 100 mL of heated CPO is mixed with bioadsorbent in the following mass ratios (1:1, 1:3, 1:5, and 3:1) for 60 and 120 minutes, respectively, with a stirring speed of 500 rpm and a temperature of 110°C maintained. Then it was filtered, and the filtrate was taken. The filtrate obtained was analyzed for free tatty acid content, peroxide value and carotene content. The results of this research show the best conditions for reducing free fatty acid levels, peroxide values, and carotene content, notably 1:5 in 120 minutes, yielded 93.70%, 81.58%, and 28.94% reductions, respectively.

KEYWORDS: biomass, carotene, free tatty acid, peroxide, pyrolysis

INTRODUCTION

Crude palm oil (CPO) is the primary ingredient used in the production of cooking oil (Garcia-Nunez et al., 2016). CPO contains triglyceride compounds, free fatty acids (FFA), pigments, phosphatides, partial glycerides, coloring matter, and carotene (Constant et al., 2017; Guliyev et al., 2018; Ifa et al., 2013; Onwuliri et al., 2011). Carotene compounds in oil occur as pigments (carotenoids), which could potentially cause cooking oil to turn yellow or red (F. S. Ali et al., 2014). In general, consumers prefer crude palm oil, which is yellow and clear. The pigment content of CPO can reduce clarity and accelerate damage during storage due to oxidation and hydrolysis, which can be dangerous to human health (Tian et al., 2015). The content of free fatty acids is one indicator of CPO quality (Azeman et al., 2015; De Almeida et al., 2013; Fatin et al., 2014). CPO must be purified before use to achieve the desired characteristics (Guliyev et al., 2018),(Ndé et al., 2019; Sampaio et al., 2011).

The methods commonly used for refining palm oil are physical and chemical purification (Azeman et al., 2015; Riyadi et al., 2016). Physical refining is generally preferred to avoid excessive neutral oil loss (Rossia et al., 2011). In the vegetable oil industry, the adsorption process can be used to reduce free fatty acid levels (Cren & Meirelles, 2012). Biomass-based adsorbents are commonly used (Anastopoulos et al., 2019). The majority of adsorbents are highly porous materials. Adsorbent is a substance that undergoes surface adsorption, whereas adsorbate is a substance whose molecules are adsorbed on the adsorbent's surface (Zhang, 2016). Several biomass wastes, such as coconut coir, have been reported as being used in CPO refining (Abel et al., 2020; Ifa et al., 2022), bleaching earth (Silva et al., 2014; Soetaredjo et al., 2021).

Rice straw is an agricultural byproduct made from the dried stalks and stems of cereal plants after the grains have been separated. Rice straw waste is also an agricultural waste that has not been utilized optimally, and there is quite a lot of it in the community. The majority of straw waste is only burned by farmers to prevent it from accumulating and making it difficult to cultivate the land. The use of rice straw as an adsorption material is a low-cost technology because the raw material is readily available, considering that rice production in Indonesia is increasing every year. Rice straw can also be used as an adsorbent because of its cellulose, hemicellulose, and lignin content (Bian et al., 2019; Madivoli et al., 2022; Nascimento et al., 2016).

Cacao is a plantation commodity with a significant impact on the national economy, particularly as a source of employment, income, and foreign exchange. The seed of the cacao fruit has economic value, whereas the skin, which is a byproduct of cocoa bean processing, is discarded. Cacao peels can cause environmental pollution by being left to rot and sometimes thrown away, so the economic value is still quite low. Cacao fruit peel is also a lignocellulosic waste, with 17.27% cellulose, 52.02% lignin, and 19.56% hemicellulose (Wijaya. M et al., 2017).

Converting biomass into adsorbents has two advantages: first, agricultural waste can have economic value, and second, the use of agricultural waste will help to solve some of the CPO quality problems (Abel et al., 2020).

Biomass can be converted into bioadsorbents in two stages. The first stage is the carbonization process, which involves decomposing organic cellulose into carbon elements and removing non-carbon compounds. The second stage is the carbon activation process, which removes the hydrocarbons that coat the surface of the charcoal, increasing its porosity (Na et al., 2019; Pallarés & González-Cencerrado, Ana Arauzo, 2018).

There are two methods for activating biomass: physical activation and chemical activation. In chemical activation, the biomass feedstock is first mixed with chemical activation reagents. The type of chemical reagent influences the chemical activation method (Ayinla et al., 2019). Common activation reagen are H_3PO_4, NaOH dan $ZnCl_2$, HCl, H_2SO_4 and others. Acid activation can significantly improve their surface properties, allowing them to be used as bioadsorbents in the refining of vegetable oils. Hydrochloric acid is the most commonly used acid in the activation process (Ndé et al., 2019; Usman et al., 2012) and H_2SO_4 (Joy et al., 2007; Steudel et al., 2009; Taha et al., 2011).

The aims of this research are to examine the effect of contact time and mass ratio of bioadsorbents derived from cocoa pod peels and rice straw waste on the reduction of free fatty acid levels, peroxide value, and carotene content.

RESEARCH METHODS

Research Material

CPO, the main ingredient used, was obtained from PT. Astra Agro Indonesia with an FFA content of 5.67% expressed in palmitic acid (C16:0, MW = 256 g/mol). Rice straw waste was obtained from rice fields in Gowa Regency, South Sulawesi, Indonesia, and cacao fruit skin waste was obtained from Palopo, South Sulawesi, Indonesia. Some of chemicals from Intraco, Makassae are sodium hydroxide (NaOH) p.a, hydrochloric acid (HCl) p.a, ethyl alcohol (C_2H_5OH) p.a, ishopropyl alcohol (C_3H_8O), phosphoric acid (H_3PO_4) p.a. starch p.a, acetic acid (CH_3COOH) p.a, chloroform ($CHCl_3$) p.a, phenol phthalein p.a, potassium iodide (KI) p.a, isooctane p.a, sodium thriosulfate.p.a. The main equipment used is pyrolysis and adsorption process equipment.

Bioadsorbent Production

The first stages involve cleaning rice straw waste and cacao fruit skins, which are then dried in direct sunlight for about a week. Dry rice straw and dried cacao fruit peels were both pyrolyzed for 2 hours at 500°C. After reaching the desired temperature, allow the material to cool in the reactor for several hours. The liquid smoke product resulting from the pyrolysis process is accommodated in the liquid smoke tank. The formed biochar is cooled to room temperature and then stored in a desiccator.

Activation Process

After the preparation treatment, the charcoal from the pyrolysis results was ground and passed through a 200 mesh (Lee et al., 2018). Following that, a chemical activation process was carried out using an HCl solution. In a 1000-mL beaker, 120 g of sifted rice straw waste biochar and cacao rind were added to an aquadest and concentrated HCl solution and allowed to stand for 18 hours. Then filtered and washed with distilled water until the pH was neutral (pH=7). Furthermore, the biochar was dried in a 105°C oven for 2 hours. The activated bioadsorbent is kept in a desiccator until the adsorption process requires it.

Bioadsorbent performance test

The CPO adsorption process was carried out by mixing the bioadsorbent with 100 mL of CPO in a 1000 mL beaker for 60 and 120 minutes with different mass ratios (1:1, 1:3, 1:5, 5:1, and 3:1). The mixture was heated on a hot plate to 70°C and homogenized at 270 rpm. Then the results of the adsorption process were filtered using a Buchner filter (11 mm pore size, Whatman). The filtrate is known as RPO (Refined Palm Oil) or purified CPO. CPO and RPO were characterized by levels of FFA, PV, and carotene content.

A. Determination of FFA Levels

FFA levels were determined by the titration method as written in the American Oil Chemists Society, AOCS (1990), with some modifications. In a beaker, weigh 2.5 g of preheated CPO or RPO oil (around 50°C) and mix with 50 mL of ethyl alcohol. The solution is neutralized by titration with 0.1026N sodium hydroxide. The percentage of FFA is calculated as palmitic acid where the molecular weight of the FFA is assumed to be 256 (as palmitic acid) based on Equation 1 (Henry, 2011)

$$\text{FFA as palmitic acid,\%}=\frac{\text{V x m x M}}{\text{10w}}x\ 100\% \qquad 1$$

Where: V = volume (ml) of sodium hydroxide used, m = molarity of sodium hydroxide solution used, w = weight (in grams of sample) used and M = Molecular weight of FFA. Equation 1 is equivalent to equation 2 (Fatin et al., 2014; Hashim et al., 2019; La Ifa et al., 2022).

$$\text{FFA as palmitic acid,\%}=\frac{\text{V x N x 25.6}}{\text{w}}x\ 100\% \qquad 2$$

Where: V = volume of sodium hydroxide used, N = Normality of sodium hydroxide used (0.1026), w = weight (in grams of sample) used.

B. Peroxide Value (PV) Analysis

The peroxide value of the oil samples was determined using the AOCS method, Cd 8-53 (AOCS, 2003). Weigh 5±0.05 grams of sample into a closed Erlenmeyer flask, then add an acetic acid and chloroform (3:2) solution and stir until all samples are dissolved. Add 0.5 mL of saturated potassium iodide solution, then homogenize the solution for 1 minute, and then add 30 mL of distilled water and add 1% starch. Titrate with a 0.01 N sodium thiosulfate solution until the color disappears (AOCS, 2003).

$$\text{Peroxide value (meq/kg oil)} = \frac{\text{(S-B) x N x 1000}}{massa\ sample,g} \qquad 3$$

Where: B = volume of titrant, mL of blank S = volume of titrant, mL of sample N = normality of sodium thiosulfate solution

C. Caratone β Analysis

Carotene content, expressed as β-carotene, was determined by measuring absorbance at 446 nm after homogenization and dilution in 25 mL iso-octane (Silva et al., 2014). RPO and CPO adsorption samples were weighed as much as 0.1 g into a 25 mL volumetric flask and dissolved in 25 mL of isooctane up to the miniscus line. The cuvette was filled with sample solution (adsorption-processed RPO or CPO), and the absorbance was measured at 446 nm with isooctane as a blank using a Shimadzu 1240 UV-Vis Spectrophotometer. Calculation of carotene value according to equation 4. Equation 4 is obtained from previous research conducted by (Hashim et al., 2019; Ifa et al., 2021)

$$\text{Carotene (mg/kg)}=\frac{\text{25 x 383 x Abs 446}}{\text{w x 100}} \qquad 4$$

Where: w = sample weight in g, and value 383 is the carotene calibration factor at a wavelength of 446 nm.

RESULT AND DISCUSSION

The adsorbents used in this research were cacao rind and straw waste, with mass ratios of 1:1, 1:3, 1:5, 5:1, and 3:1 and stirring times of 60 and 120 minutes. The mineral composition of straw and cacao rind waste was analyzed using XRF equipment, and the results are shown in Table 1. The most abundant component in straw and cacao fruit peel waste is silica, which is the primary component of activated bleaching earth or bentonite (Abdi et al., 2021). CPO and RPO were characterized based on FFA, peroxide and carotene values, shown in (Figures 1 to 3).

Table 1. Mineral Composition of Straw Waste, Cacao Rind, and Bentonite

Okside (%)	(Baksh & Yang, 1992)	(Ifa et al., 2021)	(Inglezakis et al., 2005)	This Research	
	Bentonite			Straw Waste	Cacao Rind
SiO_2	54,72	55-80	53,72	13,76	88,79
Al_2O_3	15,98	5-20	19,12	3,47	0,36
MgO	1,94	0-8	3,29	17.27	1.47
Fe_2O_3	2,93	2-10	4,93	1,82	0,59
TiO_2	0.12	-	0,16	0,014	0,03
Na_2O	2,04	0-2	3,64	-0.07	0,67
CaO	0.82	0-5	5,28	22.62	3.07
K_2O	0,34	0-2	0,44	18.59	4,02
P2O5	-	-	-	2.97	0.71

BaO	-	-	-	0.08	0,.05
SO3	-	-	-	3.33	0.55
MnO	1,94	0-8	3,29	0.85	0.07
Water (%)	-	10,68			

1. Effect of Contact Time and Bioadsorbent Mass Ratio on FFA Level Reduction

FFA is a product of triglyceride hydrolysis reaction and hydroperoxide decomposition reaction. This reaction causes rancidity, which produces a rancid flavor and odor in the oil. FFA levels in oil are frequently used as an indicator of oil damage. Figure 1 shows the effect of contact time on decreasing FFA levels.

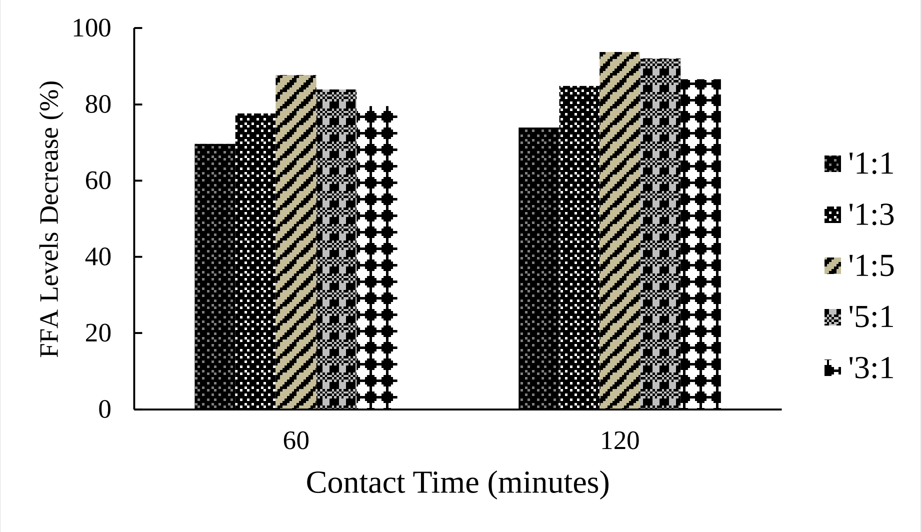

Figure 1. The Effect of Contact Time and Mass Ratio of Cocoa Peel : Straw Waste on Bioadsorbent FFA levels.

Figure 1 shows that the best time for reducing FFA levels is 120 minutes. The decrease in FFA levels started with an additional 60 minutes of 1.57% (from 5.67 to 1.57) with a 72.31% decrease in FFA levels. Figure 1 shows that the longer the contact time between CPO and the bioadsorbent, the greater the opportunity for FFA to be adsorbed in the pores of the bioadsorbent. The percentage of decreased FAA levels tends to rise over time. According to Salman et al. (2011), the longer the interaction between the solution and the adsorbent, the greater the amount of adsorbate adsorbed on the surface of the adsorbent.(Salman et al., 2011). At 120 minutes of contact time, the FFA level decreased to 0.35% (from the initial FFA of 5.67% to 0.35% with a decrease in FFA content of 93.82%). This research agrees with what was reported by (Abel et al., 2020; Amuda et al., 2013; Anang et al., 2019; Ates & Tezcan Un, 2013; Chaudhuri & Saminal, 2011; Cowan et al., 2012; Ifa et al., 2022; Oktavian et al., 2020; Purwasasmita et al., 2015; Riyadi et al., 2016) that the decrease in FFA levels is linear with contact time. The percentage decrase in FFA levels in this research was greater than research by Riyadi et al. (2016) specifically 0.13% (Riyadi et al., 2016), research by Anang et al. (2020) using zeolite, the contact time of zeolite with core oil is 4 hours which is 89.36% (Anang et al., 2020), research by Larasati et al. (2017) FFA levels are decreased by 62.5% with NaOH activated zeolite (Putranti et al., 2017). The same result was also reported by Oktavian et al. (2020) The synthesis and performance evaluation of CC bioadsorbents for refining used cooking oil reduced FFA levels by up to 93% for 45 minutes (Oktavian et al., 2020).

The addition of rice straw waste bioadsorbent mass reduces the FFA content of CPO, as shown in Figure 1. Prior to the refinement of CPO, the free fatty acid content was 5.67%. The FFA content of CPO oil decreases after it is refined. The greatest decrease in FFA levels occurred in the cacao rind bioadsorbent: straw waste ratio of 1:5 in 120 minutes (from 5.67% to 0.35% with a reduction percentage of 93.82%).

2. The Effect of Contact Time and Bioadsorbent Mass Ratio on Peroxide Value Decrease

The peroxide value is the most important value in determining the degree of damage to the oil or fat. Peroxide can be used to determine the degree of oxidation of lipids, fats, and oils as well as the amount of total peroxide in a substance. The more unsaturated a fatty acid, the more vulnerable it is to oxidation (Kouba & Mourot, 2011).

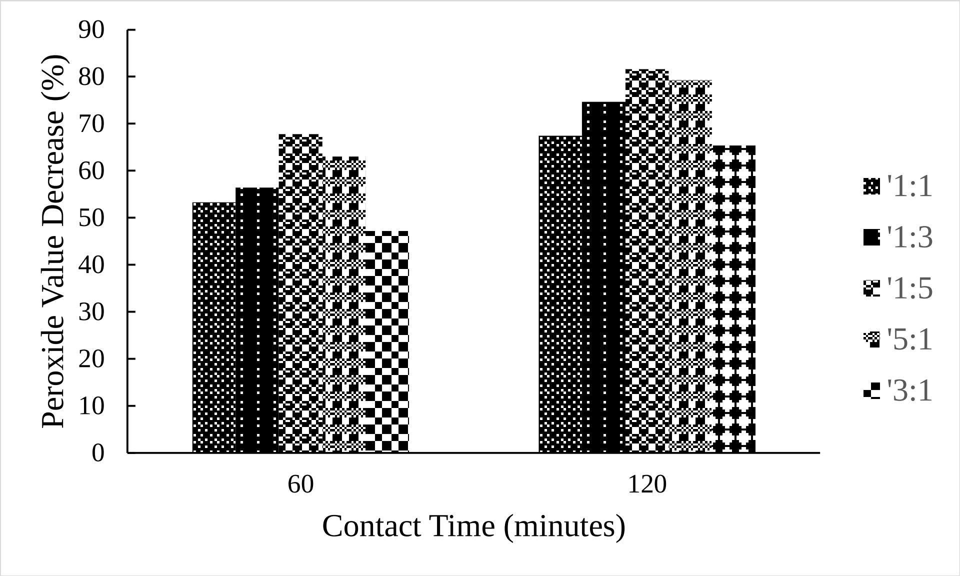

Figure 2. The Effect of Contact Time and Mass Ratio of Cocoa Peel : Straw Waste on Bioadsorbent Peroxide Value

The longer the contact period, the more adsorbate (peroxide compounds) that can be adsorbed, as shown in Figure 2. This corresponds to what has been reported by (Anang et al., 2019). Figure 2 shows that a 1:5 cacao rind: straw waste ratio at 120 minutes reduced the peroxide value. The initial value of CPO peroxide in oil was 8.63 meq/kg. The peroxide value in the oil decreased after the adsorption process, as shown by the addition of the bio-adsorbent cacao rind : rice straw waste (1:5) with a contact time of 120 minutes and 1.59 meq/kg of oil, or a percentage reduction in peroxide value of 81.58%. The application of a 5:1 bioadsorbent cacao rind:rice straw waste ratio with a contact duration of 120 minutes resulted in a 79.26% reduction in peroxide value. This difference may be due to the increased calcium content of rice straw waste compared to cocoa husk (Table 1). Peroxide value decreased with increasing ratio of cacao rind bioadsorbent: straw waste. This corresponds to what has been reported by (Abdi et al., 2021; Silva et al., 2014).

3. The Effect of Contact Time and Bioadsorbent Mass Ratio on β Carotene

The longer the contact time, the total carotene decreases (Figure 3). This is due to the longer the contact period between the bioadsorbent and CPO, the more probably carotene will be adsorbed in the pores of the adsorbent, as demonstrated by the falling levels. This corresponds to what has been reported by (Ifa et al., 2021) showed that the greatest decrease in carotene levels occurred after 60 minutes, specifically 61.32%. Riyadi et al. (2016) found that total carotene reduced significantly as contact time increased after deodorizing the oil with red palm oil for 2 hours. The percentage of decreased carotene value in this research was greater than in previous research with 51.5% decrease (Riyadi et al., 2016) used red palm oil.

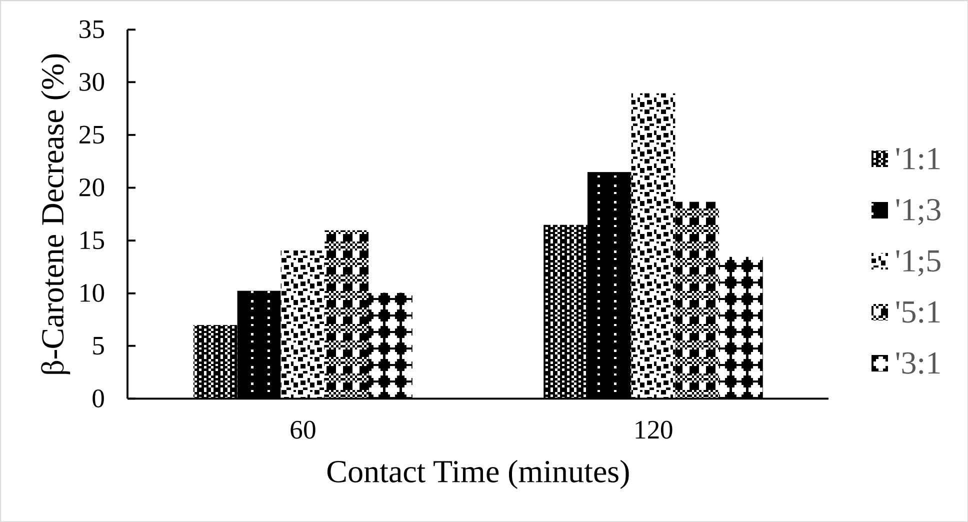

Figure 2. The Effect of Contact Time and Mass Ratio of Cocoa Peel : Straw Waste on Bioadsorbent *β-Carotene*

The amount of carotenoids in CPO was 776 ppm, which was significantly reduced after the adsorption process using various adsorbent mass ratios, as shown in Figure 3. Reduced carotenoid content is associated with a greater adsorbent mass ratio, which could be attributed to providing more surface area for adsorption and removal of β-carotene (Abdi et al., 2021). The higher the mass ratio of the adsorbent, the greater the β-carotene adsorption from palm oil. Figure 3 shows that the greatest percentage decrease in carotene value occurs at a ratio of 1:5 for 120 minutes, which is 28.94%. More carotene is absorbed when straw waste matter is added. The research results are consistent with the research by Silva et al., 2014), Total carotene decreased with the addition of BE concentrations of 0.5, 2 and 3 (%). The percentage of decreased carotene value in this research was lower than in the prior study by (Silva et al., 2014) specifically, 97.85% (from 467 to 10 mg/kg) (Silva et al., 2014), using 3% bentonite adsorbent decreased 56.37% (Ifa et al., 2021). Abdi et al. (2021) observed that utilizing 2% eggshell ash resulted in the lowest percentage of carotene content reduction (79.05%) and the highest (83.53) using 1% acid-activated eggshell powder.

CONCLUSION

Contact time and various mass ratios of bioadsorbent from cacao rind : rice straw waste were utilized in this research to determine the quality parameters of CPO, notably Free Fatty Acid, Peroxide Value, and β-carotene concentration. Increasing the adsorbent mass ratio decreased the levels of FFA, PV, and carotene concentration. The optimum conditions were 120 minutes and a mass ratio of 1:5, with decreased levels of FFA 93.70%,, PV 81.58%, and carotene 28.94%. The decrease in FFA, PV, and carotene content was affected by the bioadsorbent mass ratio and contact time. This research obtained results that were able to reduce levels of FFA, PV and carotene content of CPO by bioadsorbent from cacao rind : rice straw waste.

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