Optimization of Crude Palm (*Elaeis guineensis*) Oil Bleaching using Zeolite-Fe by Response Surface Methodology

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ABSTRACT

A carcinogenic 3-monochloropropane 1, 2 diol ester (3-MCPD ester) can be formed during processing crude palm oil (CPO). Chlorine is one of the precursors for the formation of 3-MCPD esters. This study aimed to optimize the bleaching conditions using zeolite-Fe for reducing chlorine concentration by the Response Surface Methodology (RSM) and further evaluate the characteristics of the bleached CPO. Factors such as bleaching time, zeolite-Fe concentration, and bleaching temperature were evaluated and further optimized. The results showed that Fe in the modified zeolite-Fe increased about 71.89% compared to natural zeolite. Zeolite-Fe concentration, bleaching time, and bleaching temperature had a significant effect on chlorine adsorption. The optimum bleaching process was obtained at a zeolite-Fe concentration of 5% (w/w) and bleaching temperature of 80 ℃ for 30 minutes. Bleached CPO had a chlorine concentration of 25 ± 1 ppb, carotenoid of 467.70 ± 13.71 ppm, and a DOBI (Deterioration Of Bleachability Index) value of 2.17 ± 0.01 R.

Keywords: Chlorine; crude palm oil; oil bleaching; zeolite-Fe

INTRODUCTION

Crude palm oil (CPO) is one of the food-grade vegetable oils after oil refining. It is obtained from several stages of palm fruit (*Elaeis guineensis*) processes. After CPO is extracted, the oil will go through the refining process. The purpose of the oil refining process is to reduce and eliminate oil impurities such as carotene, free fatty acids, phospholipids, and metals that can affect the color and flavor of the oil. The refining process purifies CPO to improve the quality and extend its shelf life.

The stages in the refining process of CPO are degumming, bleaching, and deodorization. Degumming aims to remove phospholipids by adding phosphoric acid, citric acid, or water. Bleaching reduces the carotene in the bleached CPO that will be produced, which will affect the color of the oil produced. The bleaching stage usually uses bleaching earth. Deodorization removes free fatty acids, which will affect the odor of the oil produced. Deodorization is usually carried out at high temperatures. The temperature used is higher than 200 °C (Hrncirik and Van, 2011).

The formation of 3-monochloropropane 1,2 diol ester (3-MCPD ester) occurs during CPO refining, and it shows genotoxic effects on in vitro testing (Strijowski *et al*., 2011). 3-MCPD esters are mostly formed at the deodorization stage, which is carried out at high temperatures. One of the precursors for the formation of 3-MCPD ester is the presence of chlorine and diacylglycerol in oil (Matthaus *et al*., 2011).

Chlorine is present in oil palm fruit before being processed into oil (Nagy *et al*., 2011). According to Craft *et al*. (2011), inorganic chlorine is from the soil, water, and fertilizer used in the fertility of oil palm trees. The

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bioconversion of inorganic chlorine into organochlorine occurs in palm fruit. Organochlorine is a source of chlorine in CPO. A high chlorine concentration promotes the formation of 3-MCPD ester (Freudenstein *et al*., 2013). Thus, reducing chlorine before and during the process is an alternative to reducing the formation of 3MCPD esters.

An alternative for reducing chlorine before fruit processing is washing the fruit using water and ethanol (Arris *et al*., 2020). On the other hand, alternatives to reducing chlorine during oil processing are by washing CPO (Arris *et al*., 2020), lowering phosphoric acid dosage during degumming (Sim *et al*., 2020), and bleaching CPO using zeolite as an adsorbent (Ramli *et al*., 2011). Besides, 3-MCPD in the product could be reduced by adsorption after oil deodorization (Arris *et al*., 2020).

In this research, reducing chlorine concentration was performed by bleaching the CPO using activated zeolite-Fe. Factors such as bleaching time, zeolite-Fe concentration, and bleaching temperature were evaluated and further optimized. Furthermore, chlorine concentration, carotenoid, and a DOBI *(*Deterioration Of Bleachability Index*)* value were analyzed.

MATERIALS AND METHODS

Material

CPO was obtained from South Sumatra, Indonesia. Zeolite was obtained from Klaten, Central Java, Indonesia. $FeCl_3$, HCl, NaOH, magnesium oxide, ammonium molybdate, ammonium vanadate, nitric acid, glacial acetic acid, chloroform, ethanol, acetone, toluene, and isooctane were obtained from Merck (Darmstadt, Germany). The Hanna HI 761 reagent was obtained from Woonsocket, Rhode Island, USA.

Activation of Zeolite

The activation of zeolite was prepared according to Suhartana *et al*. (2018) and Anis (2019). The zeolite was separated by sieving it using a 100 mesh sieve. The zeolite, which passed the sieve, was washed with distilled water, filtered, and dried in an oven at 105 °C for 4 hours. The washed zeolite was soaked in 6 N HCl at a ratio of 1:10 for 24 hours and then filtered. The obtained Zeolite-H was then rewashed with distilled water; then, it was dried at 105 °C for 4 hours. The obtained Zeolite-H was further soaked in $FeCl_3$ at a ratio of 1: 5. The mixture was then stirred and heated at 85 °C for 4 hours. Then it was neutralized by adding 2 N NaOH, washed with distilled water, and filtered. Washing was performed until Cl was not detected by analyzing using $AgNO_3$. The obtained zeolite-Fe was filtered and dried at 105 °C for 4 hours. The dried zeolite-Fe was then heated at 550 °C for 2 hours. The water concentration of the activated Zeolite-Fe was analyzed using the thermogravimetry method, and its chemical elements were analyzed using Energy Dispersive X-Ray (EDX).

Degumming and Bleaching of Crude Palm Oil

Degumming and bleaching of CPO were performed according to Zulkurnain *et a*l. (2012) and Anis (2019). Degumming of CPO was carried out by heating CPO at 80 °C, then adding 1% distilled water (w/w). It was carried out for 30 minutes and stirred at a speed of 200 rpm. The degummed CPO was bleached by adding the activated zeolite-Fe (5% w/w) and incubated at 80 °C for 30 min. The mixture was stirred at a speed of 400 rpm. The zeolite-Fe was further separated by centrifugation. The total chlorine, carotene concentration, and DOBI value of the bleached CPO were analyzed.

Experimental design and optimization

Screening design

Screening design was performed to evaluate the factors that affected and determined the process. Firstly, the effect of the bleaching time on the total chlorine was evaluated. The CPO was heated at 80 °C. The zeolite-Fe (5% w/w) was then added and incubated for 20-60 minutes (Zulkurnain *et al*., 2013; Ramli *et al.,* 2011). Secondly, the effect of the zeolite-Fe concentration on the total chlorine was determined. The CPO was heated at 80 °C. The zeolite-Fe was further added (1.5 - 15% w/w) (Zulkurnain *et al*., 2013; Silva *et al.,* 2013), then incubated for 30 min. The third factor was the bleaching temperature. The CPO was heated at 60-140 °C (Ramli *et al.,* 2011). The zeolite-Fe 5% (w/w) was added and incubated for 30 min.

Optimization

The results of screening the bleaching time, zeolite-Fe concentration, and bleaching temperature were used to determine the actual levels of the Box-Behnken design. This design was then used to optimize the bleaching process using the response surface methodology. The responses were total chlorine, carotene concentration, and DOBI values with 17 experiments. The data showed significant results if $p< 0.05$.

Total Chlorine

The total chlorine on the oil was analyzed using the IP 77 method (1972) with modifications. The oil used was approximately 32 g. The solvent contained 40 mL of toluene, 25 mL of ethanol, 15 mL of acetone, and 50 mL of water. The extracted chlorine from the oil was then analyzed using a kit, namely Hanna HI 761 Total Chlorine Checker from Woonsocket, Rhode Island, USA.

Carotenoid Concentration

The carotenoid concentration was analyzed using a Genesys 10S UV-Vis spectrophotometer, Thermo Scientific (USA), based on MPOB (2005) method. A total of 0.1 ± 5x10-4 g of the oil was weighed, then dissolved in 25 mL isooctane. The Absorbance of carotenoid was measured at 446 nm and converted to carotenoid concentration in ppm, using the Equation 1.

$$\text{Carotenoid concentration (ppm)} = \frac{25\ x\ \text{Abs}\ x\ 10000}{\text{Mass}\ x\ 2610} \quad (1)$$

DOBI value

The deterioration of bleachability index (DOBI) was analyzed using a Genesys 10S UV-Vis spectrophotometer, Thermo Scientific (USA), based on MPOB (2005) method. A total of 0.1 ± 5x10-4 g of the oil was weighed, then dissolved in 25 mL isooctane. Absorbance was obtained at wavelength 446 nm and 269 nm (Equation 2).

$$\text{DOBI} = \frac{\text{Abs 446 nm}}{\text{Abs 269 nm}} \quad (2)$$

RESULT AND DISCUSSION

Characteristics of Zeolites and Zeolites-Fe

Table 1. Characteristics of Zeolites and Zeolites-Fe

Parameter	Zeolite	Zeolite-Fe
Water concentration (%)	11.08±0.12	0.43±0.20
Si (%)	55.83	55.68
Fe (%)	13.52	23.24
Al (%)	8.38	8.30
Ca (%)	12.32	5.49
K (%)	6.59	3.96
S (%)	0.85	1.27
Ti (%)	1.40	1.03

Table 1 shows the characteristics of the zeolite-Fe used in the experiment. The zeolite-Fe was modified from the natural zeolite to increase the adsorption capacity (Aidha, 2013). The results showed that the water concentration in the zeolite-Fe decreased by 96.12% compared to the natural zeolite. Such a decrease in water concentration in zeolite-Fe was due to the physical activation process of the zeolite. The zeolite was heated at 105 °C and calcined at 550 °C for 2 hours. The heating and calcination of the zeolite caused the water to evaporate, resulting in a decrease in the water concentration of the zeolite-Fe.

The concentration of the zeolite elements (Table 1) was almost similar to Wahyuni results (2003). Zeolites contain interchangeable cations. The cations in these zeolites include IA and IIA groups. The analysis results using EDX showed that Fe, Al, Ca, and K were elements that acted as cations that could be exchanged on the zeolites. Fe concentration in the zeolite-Fe increased by 71.89% compared to the natural zeolite. Thus it showed a successful modification of the zeolite using $FeCl_3$. The color of the zeolite-Fe changed to brown-orange, also showing success in the zeolite modification (Suhartana *et al*., 2018).

Effect of Zeolite-Fe Concentration, Bleaching Time, and Bleaching Temperature on Chlorine

At least three factors, namely the bleaching time, zeolite-Fe concentration, and bleaching temperature, affected the total chlorine. Based on Figure 1(a), an increase in the bleaching time from 20 to 30 minutes decreased the chlorine concentration in the oil by 37.50%. It is due to an increase in contact time. A further increase in the bleaching time to 40 minutes increased the chlorine concentration. It may be due to a leaching of Fe from the zeolite-Fe. A further increase in the bleaching time did not cause an increase in the total chlorine. Thus, the best bleaching time was at 30 minutes. The bleaching times of 20, 30, and 40 minutes were chosen for Box Behnken design and coded -1, 0, and +1, respectively.

Besides, an increase in the zeolite-Fe concentration from 1.5% to 5% (w/w) decreased the chlorine concentration in the oil by 66.66% (Figure 1(b)). The chlorine concentration in the oil decreased because the concentration of the zeolite-Fe added to the oil was higher. A further increase in the zeolite-Fe concentration to 15% (w/w) increased the chlorine concentration. It is suggested that the Fe concentration in oil increases due to leaching of Fe from zeolite-Fe when the zeolite-Fe concentration increases, causing Fe to interact with the chlorine in the oil, decreasing the interaction between the chlorine and zeolite-Fe. The concentration of zeolite-Fe (5% w/w) resulted in the lowest chlorine concentration of the oil. The zeolite-Fe concentrations of 1.5, 5, and 15% were chosen for Box Behnken design, and they were coded -1, 0, and +1, respectively.

Furthermore, the increase in the bleaching temperature from 60 to 80 °C increased the chlorine adsorption in the oil by 54.54%. However, a further increase in the temperature to 140 °C decreased the chlorine adsorption, thus increasing the chlorine in the

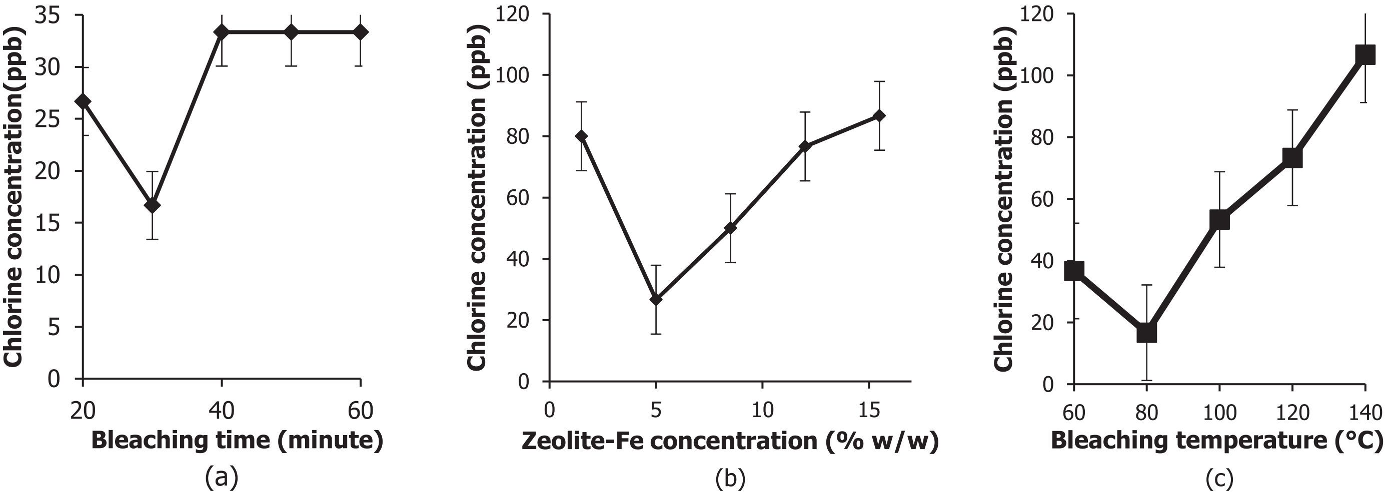


Figure 1. Effect of time (A), zeolite-Fe concentration (B), and bleaching temperature (C) on chlorine concentration

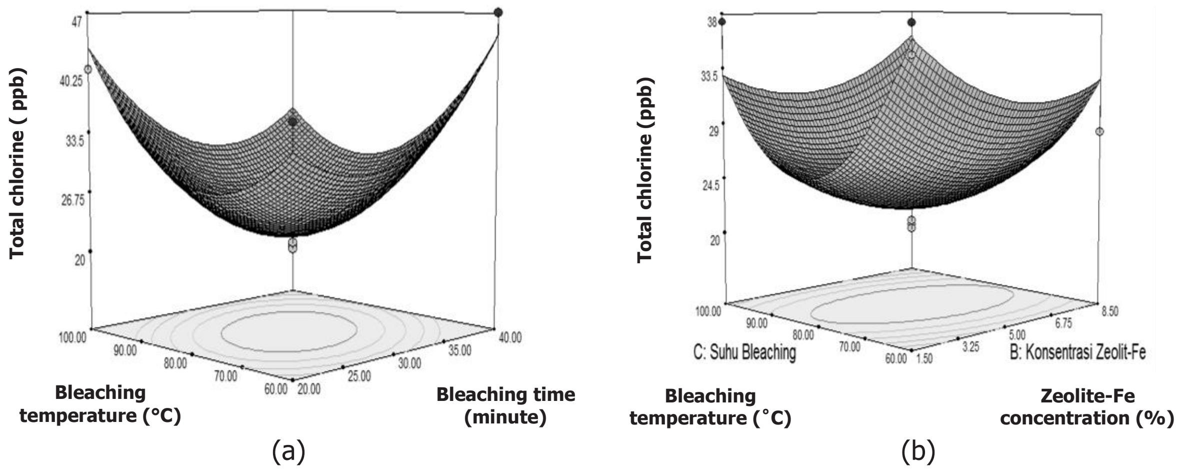


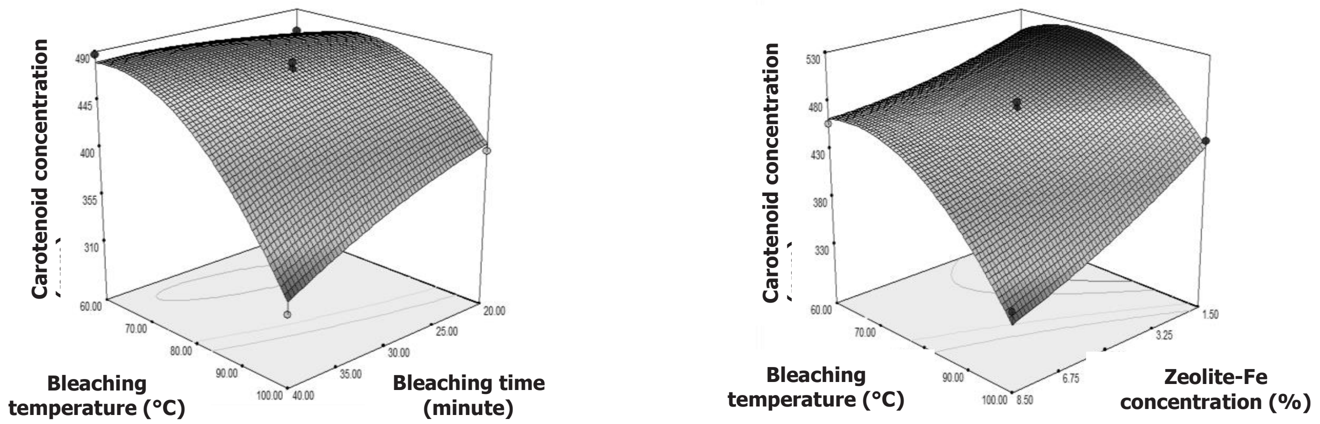
Figure 2. Effect of time and bleaching temperature on total chlorine (a) and Effect of zeolite-Fe concentration and bleaching temperature on total chlorine (b)

oil by 5.4 times. It is suggested that an increase in temperature increases the surface activity of zeolite-Fe. However, a higher temperature may defect the active surface of zeolite-Fe (Chi and Cummings, 1978), lowering chlorine adsorption. In other words, the bleaching temperature at 80 °C resulted in the lowest total chlorine in oil. The bleaching temperatures of 60, 70, and 80 minutes were chosen for Box Behnken design, and they were coded -1, 0, and +1, respectively.

Effect of Time, Zeolite-Fe Concentration, and Bleaching Temperature on Total Chlorine

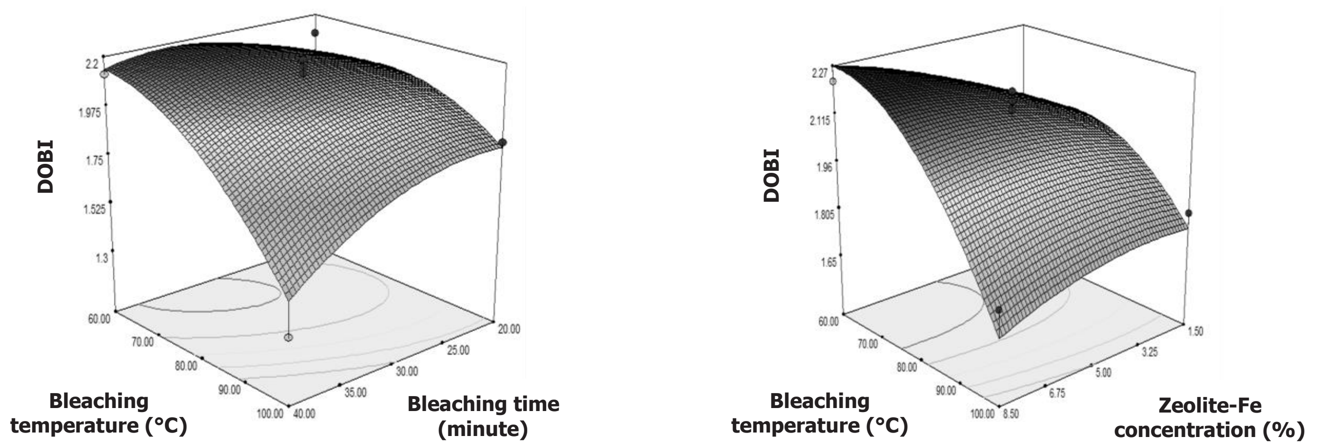
Based on Figure 2(a), when the bleaching was performed at a longer time and a higher temperature, the chlorine in the oil decreased until an optimum point was achieved. After reaching the optimum point, the chlorine increased. This is because the longer the bleaching time and the higher the bleaching temperature, the lower the adsorbed chlorine (Suhartana *et al*., 2018) due to Fe leaching and a decrease in the surface affinity of zeolite-Fe. The interaction between the bleaching time and the temperature had a significant effect ($p<0.05$).

As shown in Figure 2(b), the interaction between the zeolite-Fe concentration and bleaching temperature did not have a significant effect ($p>0.05$). This is because an increase in the zeolite-Fe concentration did not affect the chlorine adsorption compared to the bleaching temperature. Based on the theory, an increase in the concentration of zeolite-Fe as an adsorbent can increase chlorine adsorption so its adsorption efficiency increases (Javadian *et al*., 2015).



(a) (b)

Figure 3. (a) Effect of time and bleaching temperature on carotenoid concentration (b) Effect of zeolite-Fe concentration and bleaching temperature on carotenoid concentration



(a) (b)

Figure 4. (a) Effect of time and bleaching temperature on DOBI value (b) Effect of zeolite-Fe concentration and bleaching temperature on DOBI value

Effect of Time, Zeolite-Fe Concentration, and Bleaching Temperature on Carotenoid Concentration

The interaction between the bleaching time and the temperature had a significant effect ($p<0.05$). Based on Figure 3(a), the longer the bleaching time and the higher the bleaching temperature, the lower the carotenoid concentration. A higher temperature causes a higher carotenoid adsorption due to an increase in the affinity between adsorbent and adsorbate, in this case: zeolite-Fe and carotene (Silva *et al*., 2013).

Based on Figure 3(b), the carotenoid concentration decreased with an increase in the zeolite-Fe concentration and the bleaching temperature during the bleaching process because of the carotenoid adsorption on the zeolite-Fe. According to Nwabanne and Ekwu (2013), the higher the adsorbent concentration, the higher the carotene adsorption. This is because carotene is partially adsorbed during a bleaching process. The interaction between the zeolite-Fe concentration and bleaching temperature significantly affected carotenoid concentration ($p<0.05$).

Effect of Time, Zeolite-Fe Concentration, and Bleaching Temperature on DOBI Value

Figures 4(a) and (b) show that the DOBI value of the oil decreased with an increase in the bleaching temperature. The temperature had a significant effect

(p<0.05). DOBI is one of the parameters for measuring oil quality. DOBI value is obtained from the ratio between carotene concentration and secondary oxidation products in oil. DOBI decreases if carotenoid concentration decreases during bleaching at high temperatures. DOBI decreases when oil is heated at a temperature higher than 55 °C, DOBI decreases (Hasibuan, 2016).

Characteristics of Bleached Crude Palm Oil Using Zeolite-Fe

CPO bleaching using zeolite-Fe shows higher chlorine adsorption. The chlorine concentration of the bleached CPO was 44.85% lower than that of CPO, which was bleached using zeolite without modification. The bleached CPO using the zeolite-Fe had a chlorine concentration of 81.25% lower than the CPO because the addition of Fe^{3+} solution caused Fe^{3+} ions to replace exchangeable cations in the zeolites. The Fe ions bound the chlorine, thus decreasing the chlorine concentration in the CPO bleached using the zeolite-Fe (Elizalde-Gonzalez *et al*., 2001).

The carotenoid concentration in the CPO bleached using the zeolite-Fe was 7.49% lower than the CPO bleached using the zeolite without modification. The carotenoid concentration of the CPO bleached using the zeolite-Fe was about 18.92% lower than the CPO. In fact, zeolite-Fe is physically and chemically activated. Zeolite activated using $FeCl_3$ causes zeolite-Fe to have a better ability to adsorb carotenoids than zeolites without modification.

The CPO bleached using the zeolite-Fe had a lower DOBI value of 3.98% than the CPO bleached using the zeolite without modification. The DOBI value of the CPO bleached using the zeolite was 5.65% lower than the DOBI value in the CPO, because the carotenoid concentration was lower in the CPO bleached using the zeolite-Fe. DOBI value is closely related to the carotenoid concentration in oil (Hasibuan *et al*., 2017).

CONCLUSION

Modification of zeolite using $FeCl_3$ increased the Fe concentration in zeolite-Fe, about 71.89% compared to natural zeolite. Zeolite-Fe concentration, bleaching time, and bleaching temperature significantly affected chlorine adsorption. The optimum bleaching condition using zeolite-Fe for CPO was at a bleaching time of 30 minutes, at a zeolite-Fe concentration of (5% w/w), and at a bleaching temperature of 80 °C. It could reduce the total chlorine by 81.25%. The bleached CPO contained 467.70 ± 13.71 ppm carotenoid and 2.17 ± 0.01 DOBI.

CONFLICT OF INTEREST STATEMENT

The authors declare that there is no conflict of interest.

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