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Performance Evaluation of Bentonite/Nano-SiO_2 Composite as Bleaching Earth in Crude Palm Oil Processing

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Abstract

In crude palm oil (CPO) processing industries, bleaching is important to remove unwanted impurities. Most industries use various bleaching earth with 0.8-2% w/v concentration during the bleaching process. As bleaching earth is single-used for the bleaching process, materials with higher adsorption ability are preferable to reduce the use of bleaching earth. This study aimed to evaluate the performance of bentonite/nano SiO_2 composite (BE-SiO_2) as bleaching earth in the bleaching process of CPO. The prepared BE-SiO_2 was used in the bleaching process with various BE-SiO_2 concentrations (0.1-1.3% w/v), process duration (0-2.5 hours), and process temperature (70-150°C). From the results, the rise in BE-SiO_2 concentration during the CPO bleaching process initially led to a decrease in the β-carotene concentration, acid content, free fatty acids (FFA), and DOBI number up to 1% w/v of BE-SiO_2. Subsequently, these parameters exhibited an increase with the addition of BE-SiO_2. The bleaching process effectively reduced the β-carotene concentration, acid content, FFA, and DOBI number within the first 1.5 hours of operation. While a higher amount of β-carotene was adsorbed during the adsorption process at higher temperatures, the bleached palm oil (BPO) quality was compromised due to the heat. The results showed that the most effective condition of the bleaching process was performed with 1% w/v BE-SiO_2 concentration for 1.5 hours at 90°C. From those conditions, the BPO had a β-carotene concentration of 312.311 ppm, water content of 0.112%, acid number of 0.0076, FFA of 4.864%, DOBI number of 1.311, and red/yellow of 1.5/15.

1. Introduction

The crude palm oil (CPO) production in Indonesia has shown inclining growth over the years, reaching a production number of around 47 million tons in 2020-2021. To lighten the CPO color by adsorbing carotenoids, the bleaching process is performed using 0.8-2% bleaching earth of CPO mass and performed at 100-130°C for 30 minutes [1]. As bentonite (BE) is a single-used material, the annual need for BE caused a problem in the CPO refinery. The required bleaching earth properties, such as color-adsorption effectiveness, shorter bleaching time, and waste minimization, are preferable for CPO refinery industries [2].

Different types of clay minerals, including sepiolite, attapulgite, and montmorillonite (bentonite), have been employed as bleaching agents [3]. Bentonite is the most common bleaching earth used in the CPO refinery process as it is more available in nature [4]. Yusnimar *et al.* [5] found that decreasing the size of bentonite could lower the FFA content of CPO up to 1.65% using 200 mesh-sized bentonites. Besides physical modification to reduce their size, bentonites must be chemically modified to increase their surface area and hydrophilic properties, thus improving their adsorption performance [6, 7]. However, using the acid/alkaline activation method to improve bentonite adsorption capacity is avoided as it promotes equipment corrosion and makes the oil unstable [8].

Metal oxide addition to clays gained more interest as they form microporous nanocomposites. Thus, more surface area is available for the adsorption process [9]. Ghorbanpour [10] produced bentonite nanocomposites with iron oxide, which was applied in soybean oil bleaching. The nanocomposite reduced β-carotene content by up to 85% after a 150-minute bleaching process, which is better than native bentonite. Besides iron oxide, SiO_2 also has excellent performance as a part of the adsorbent nanocomposite, such as for dye contaminant and heavy metals removal [11].

This study aimed to evaluate the performance of bentonite/nano SiO_2 nanocomposite (BE-SiO_2) for the bleaching process of CPO. The concentration of BE-SiO_2, temperature, and duration of the bleaching process was varied to obtain optimum process conditions. The evaluated parameters in this study were the change of carotenoid concentration, deterioration of bleachability index (DOBI) value, free fatty acid (FFA) value, acid value, and moisture content of bleached CPO.

2. Experimental

This research was conducted in the Chemical Industrial Process Laboratory, Politeknik ATI Padang, using materials and equipment to carry out the bleaching process. The analyses were performed in Politeknik ATI Padang and other testing laboratories. The initially prepared BE-SiO_2 was used as an adsorbent in the bleaching process with various adsorbent concentrations (0.1, 0.3, 0.8, 1.0, 1.3% w/v), bleaching process duration (0.5, 1.0, 1.5, 2.0, and 2.5 hours), and temperature process (70, 90, 110, 130, and 150°C).

2.1. Materials

The materials used in this research were bentonite-type bleaching earth obtained from Indonesia, nano SiO_2 (Merck), crude palm oil (CPO) from the CPO factory in Padang, standard BPO from the factory in Padang, n- hexane 95% (Merck) for analysis, and 96% ethanol (Merck) for modification purposes.

2.2. Preparation of BE-SiO_2

To obtain a BE-SiO_2 composite, BE (50 g) was mixed with nano-SiO_2 (15% w/w BE) in ethanol (95%, 50 mL). This modification was carried out with continuous stirring for 3 hours at 70°C. The resulting BE-SiO_2 was filtered and oven-dried at 100°C for 5 hours.

2.3. Bleaching Process

Before bleaching, CPO was degummed by adding 0.5% v/v H_3PO_4 into 100 mL of CPO with continuous stirring at 70°C [12]. After 30 minutes of the degumming process, the bleaching process was conducted by adding BE-SiO_2 in various concentrations (0.1, 0.3, 0.8, 1.0, and 1.3% w/v) and stirring for 30 minutes at 70°C. The BE-SiO_2 sample concentration with the best performance in β- carotene degradation and BPO characteristics were used for BE-SiO_2 preparation with bleaching duration variation (0.5, 1.0, 1.5, 2.0, and 2.5 hours). After the optimum bleaching process duration was achieved, the temperature was varied (70, 90, 110, 130, and 150°C).

2.4. Analyses

2.4.1. Free Fatty Acids

CPO sample (5 g) was mixed with ethanol (96%, 50 mL) at boiling temperature until they formed a homogeneous solution. A phenolphthalein indicator was added in hot conditions, and the solution was titrated using NaOH solution. The percentage of FFA was calculated using Equation 1, with $V_{titration}$ as the volume of NaOH used for titration, N_{NaOH} as the normality of the NaOH solution, and w as the weight of the CPO sample [13].

$$\%FFA = \frac{V_{titration} \times N_{NaOH} \times 25.6}{w} \quad (1)$$

2.4.2. Deterioration of Bleachability Index (DOBI) Number and β-carotene

The CPO sample (0.1 g) was diluted to 25 mL with 95% n-hexane. The mixture was mixed until it was completely homogenized. The solution was subsequently measured for its absorbance using a UV-Vis spectrophotometer (Shimadzu 1800) at 446 and 269 nm wavelengths. The DOBI number and β-carotene concentration were calculated using Equations 2 and 3, respectively, with A_{446} as the absorbance at 446 nm, A_{269} as the absorbance at 269 nm, and w as the weight of the CPO sample [14].

$$\text{DOBI number} = \frac{A_{446}}{A_{269}} \quad (2)$$

$$\beta - \text{carotene concentration} = \frac{A_{446} \times 283 \times 25}{w \times 100} \quad (3)$$

2.4.3. Morphology and Crystallinity

The BE and BE-SiO_2 morphologies were determined using scanning electron microscopy (JSM-6510 LV JEOL Ltd., Tokyo, Japan) at 20 kV under a magnification of 10,000×. Meanwhile, the crystallinity of the samples was determined based on a diffractogram produced by the MAXima_X XRD-7000 diffractometer (Shimadzu, Japan).

2.4.4. Functional Group Analysis

The change of functional groups of BE and BE-SiO_2 was analyzed through the analysis of infrared spectra produced by Fourier Transform Infrared (FTIR) spectroscopy (Perkin Elmer Spotlight 200, PerkinElmer Inc., Waltham, MA, USA) from 4000–400 cm^{-1} wavenumber.

2.4.5. CPO Color Calculation

CPO color was calculated based on the AOCS Cc 13b-45 method using the Lovibond Tintometer Color scale at 70°C. Analysis was done using glass cells <1" (25.4 mm) to obtain maximum results. The color of CPO was seen using Red (R) and Yellow (Y) readings [13].

3. Results and Discussion

In this research, modified BE-SiO_2 was used to improve the performance of bleaching earth. The evaluation of BE-SiO_2 performance was conducted by examining variations in concentration, duration, and temperature during the bleaching process.

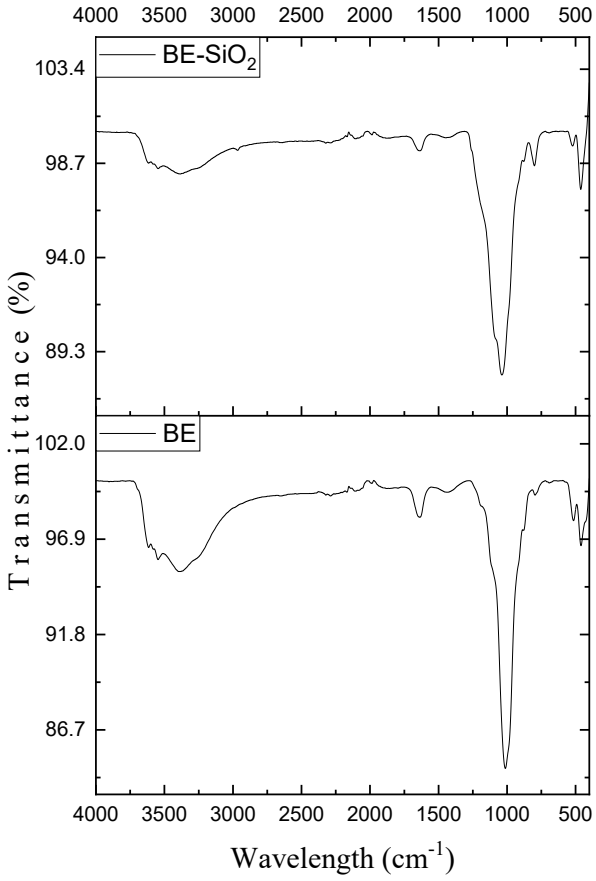

Figure 1. FTIR spectra of BE and BE-SiO_2

3.1. Characterization of BE-SiO_2

The addition of SiO_2 caused a visible change in the color of the bentonite. As the modification was conducted physically, the initial color of white SiO_2 made the color of bentonite lighter and whiter. The attachment of SiO_2 was also proved by the change of functional groups (Figure 1). Based on the IR spectra, the addition of nano SiO_2 caused shifts in the wavelength range of 3700-3500 cm^{-1}, indicating alterations in structural OH stretching vibrations. Notably, at the broad peak around 3434 cm^{-1}, a decrease in transmittance was observed, rendering the peak less discernible. This indicated that the amount of - OH bentonite decreased more intensively due to the number of octahedral replacements of SiO_2 nano atoms. These results are supported by changes in peak broadening at 1111 cm^{-1}, the longitudinal strain mode of Si–O [15].

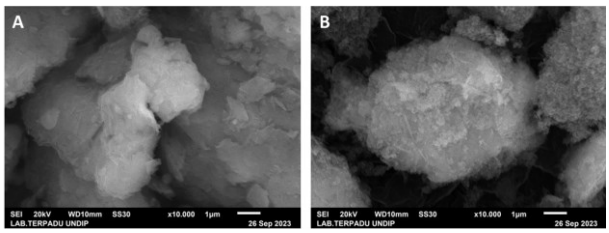

Figure 2. Morphology images of (A) BE and (B) BE-SiO_2 at 10,000× magnification

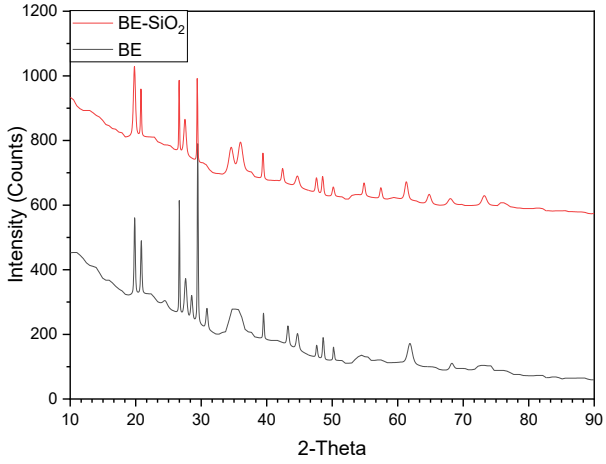

Figure 3. X-ray diffractogram of BE and BE-SiO_2

Figure 2 illustrates the morphologies of both BE and BE-SiO_2. BE-SiO_2 exhibits less agglomeration compared to native BE. The introduction of SiO_2 into BE filled the lattice, resulting in its enlargement and inhibiting agglomeration. This observation aligns with the findings reported in reference [16].

The effect of modification on the crystalline phase of bleaching earth is explained by an X-ray diffractogram (Figure 3). After modification, the intensity at 2θ=28.5296° decreases from 3.03304 Å to 2.28686 Å. This reduction caused by SiO_2 addition described a lower crystallinity of BE-SiO_2 than native BE, which increased the brittleness of the BE-SiO_2 composite. Besides intensity reduction, clearer peaks are shown at 2θ=34.5420° and 2θ=35.9680°, which highlighted the presence of montmorillonite [17] after lattice distortion caused by SiO_2. Another peak is shown at 2θ=26.61°, which is described as the characteristic of quartz [18].

Table 1. BPO quality with variations in BE-SiO_2 concentration in the bleaching process

Parameter	CPO	BPO standard	BE-SiO_2 concentration (% w/v)					
			0.1	0.3	0.5	0.8	1	1.3
Water content (%)	0.1002	0.1072	0.1002	0.1008	0.1004	0.1002	0.100	0.1002
Acid number	0.0060	0.0064	0.0070	0.0068	0.0066	0.0066	0.0064	0.0066
FFA (%)	3.84	4.096	4.505	4.352	4.224	4.224	4.096	4.101
DOBI number	2.554	1.740	2.382	2.253	2.222	1.971	2.257	2.475
Color red/yellow	2.1/21	1.5/15	1.8/18	1.6/16	1.7/17	1.7/17	1.5/15	1.6/16

* BPO standards obtained from the CPO processing industry in Padang

Table 2. BPO quality with variations in bleaching duration

Parameter	CPO	BPO standard	Bleaching process duration (hour)				
			0.5	1	1.5	2	2.5
Water content (%)	0.1002	0.1002	0.100	0.1020	0.100	0.1026	0.1006
Acid number	0.0060	0.0064	0.0064	0.0068	0.0062	0.0070	0.0066
FFA (%)	3.84	4.096	4.096	4.352	3.968	4.4480	4.224
DOBI number	2.554	1.74	2.257	1.907	1.779	1.966	1.991
Color red/yellow	2.1/21	1.5/15	1.5/15	1.7/17	1.6/16	1.6/16	1.7/17

* BPO standards obtained from the CPO processing industry in Padang

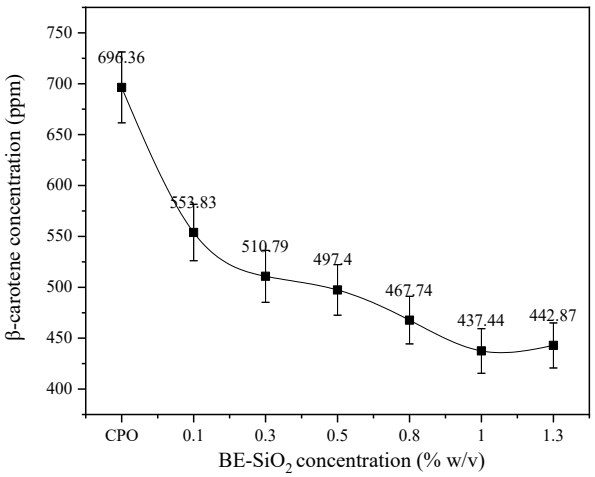

Figure 4. β-carotene concentration in BPO with varying concentrations of BE-SiO_2

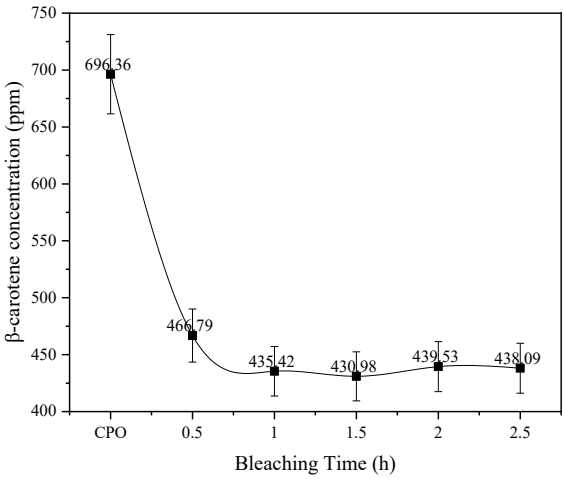

Figure 5. β-carotene concentration after bleaching process in varied durations

3.2. CPO Bleaching Process with BE- SiO_2

BE-SiO_2 performance in the bleaching process was evaluated by varying the BE-SiO_2 concentration, bleaching process duration, and temperature of the bleaching process to obtain the optimum parameters of the CPO bleaching process.

3.2.1. Effect of BE-SiO_2 Concentration

To determine the effect of BE-SiO_2 addition on bleaching process effectiveness, the concentration of BE-SiO_2 was varied from 0% to 1.3% (w/v). The bleaching process aims to reduce the β-carotene in CPO, which affects the color of palm oil [19]. As depicted in Figure 4, the addition of more BE-SiO_2 during the bleaching process enhances the adsorption of β-carotene. Consequently, a lower concentration of β-carotene in BPO is achieved after a 30-minute bleaching. Specifically, when 1% BE-SiO_2 is utilized, the β-carotene concentration decreases to 437.44 ppm. This outcome markedly outperforms the use of 1% unmodified bentonite, which yields BPO with a β-carotene concentration of 630.362 ppm. However, increasing the BE-SiO_2 concentration from 1% to 1.3% results in a higher β-carotene concentration in the BPO.

Besides the β-carotene concentration, the success of the bleaching process was indicated by water content, acid number, FFA percentage, DOBI number, and color (Table 1). The initial water content of both CPO and BPO fell below the standard threshold, and no significant alteration was observed following the bleaching process. Similar to the change in β-carotene concentration, the acid number and FFA content decreased with the increased utilization of BE-SiO_2 during bleaching. However, when the BE-SiO_2 concentration exceeded 1% w/v, there was an increase in both acid number and FFA. At the lowest point, when BE-SiO_2 concentration was 1% w/v, the acid number and FFA were the same value as the BPO standard.

Additionally, the DOBI number, which describes both the oxidation rate and the ease of palm oil processing, decreased after bleaching. However, the DOBI numbers of resulting BPOs are higher than the BPO standard. The ratio of red/yellow color in CPO changed post-adsorption and the optimal color meeting BPO standards were achieved using 1% w/v BE-SiO_2. These outcomes suggest that increasing the adsorbent concentration increases the number of active sites, thus expediting the adsorption process [18]. However, further addition of adsorbent may lower the bleaching effectiveness. Thus, a 1% (w/v) concentration of BE-SiO_2 was subsequently used for the determination of the effective duration of the bleaching process.

Table 3. BPO quality with variations in bleaching process temperature

Parameter	CPO	BPO standard	Temperature (°C)				
			70	90	110	130	150
Water content (%)	0.1002	0.1002	0.100	0.1120	0.0958	0.1026	0.0886
Acid number	0.0060	0.0064	0.0062	0.0076	0.0080	0.0080	0.0082
FFA (%)	3.84	4.096	3.968	4.864	5.120	5.120	5.248
DOBI number	2.554	1.74	1.779	1.311	1.117	1.005	0.840
Color red/yellow	2.1/21	1.5/15	1.6/16	1.5/15	1.6/16	1.7/17	1.7/117

* BPO standards obtained from the CPO processing industry in Padang

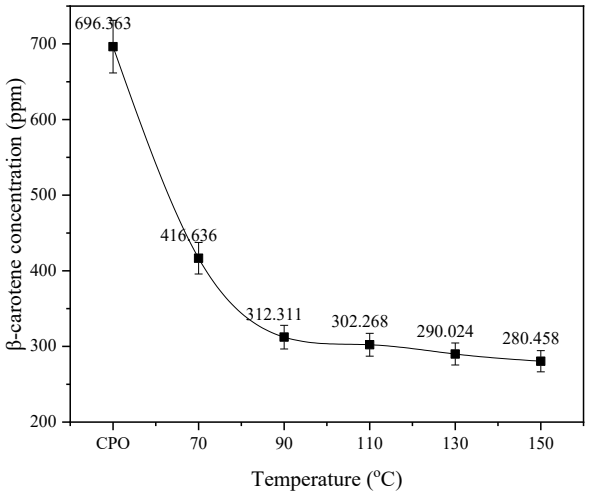

Figure 6. Effect of bleaching process temperature on β- carotene concentration

3.2.2. Effect of Bleaching Process Duration

The optimum bleaching process duration was determined at range of 0-2.5 hours with 1% w/v BE-SiO_2 at 70°C. The remaining β-carotene concentrations left after the bleaching process in different durations are shown in Figure 5. The β-carotene concentration was dropped after the 0.5-hour bleaching process. The sloping decrease further occurred up to 1.5 hours, which obtained a β-carotene concentration of 430.98 ppm, followed by an insignificant change of β-carotene concentration over time. Prolonging the duration of the bleaching process made the adsorbent saturated, which led to the possibility of β-carotene desorption. Suarya and Simpen [20] also found the desorption phenomenon after forming a saturated adsorbent.

The values of other BPO parameters after the bleaching process are shown in Table 2. The water contents of BPO are different as the bleaching duration was varied. However, only BPO produced after 0.5 and 1.5 hours of the bleaching process has a lower water content than the BPO standard. Similar to the previous discussion, the reduction in acid number and FFA were in line with the change of β-carotene concentration, which has the value of 0.0062 and 3.968, respectively, after 1.5 hours of the bleaching process. Another unobserved impurity compound in the form of colloidal suspensions in oil, phospholipids, carbohydrates, and other complex compounds may also be adsorbed in BE-SiO_2 [21]. A longer duration of bleaching proved detrimental as it led to an increase in both acid number and FFA. Moreover, FFA can act as an autocatalyst, resulting in a rapid increase in FFA levels when CPO is heated [22].

The lowest DOBI number (1.777) was also obtained after 1.5 hours of bleaching duration. DOBI is one of the CPO quality parameters to measure the level of oil damage caused by oxidation. Moreover, prolonging the bleaching duration caused the DOBI value to decrease due to the degradation of the β-carotene compound, resulting in a lower DOBI value [23]. However, at 2 hours and 2.5 hours of bleaching process, the DOBI numbers are increased, which aligns with the β-carotene concentration trend. Although the color parameter is higher than the BPO standard, other parameters indicate superior results after a 1.5-hour bleaching process. Thus, a duration of 1.5 hours was selected to establish the optimal temperature for the bleaching process.

3.2.3. The Effect of Temperature on the Bleaching Process

A study on the effect of temperature on the bleaching process using BE-SiO_2 adsorbent was conducted by varying the temperatures of 70, 90, 110, 130, and 150°C. Another condition was obtained from previous determinations, which is 1%w/v BE-SiO_2 concentration and 1.5 hours bleaching process duration. The effect of temperature of the bleaching process on β-carotene concentration is shown in Figure 6. A significant decrease of β-carotene occurred over the increasing temperature up to 90°C with a β-carotene concentration of 312.311 ppm. Increasing the temperature of the bleaching process enhanced the maximum adsorption capacity of the adsorbent [24].

Moreover, higher temperatures accelerated the β-carotene movement, which was easily caught by temperature-activated adsorbent [25], resulting in greater adsorption of β-carotene. At temperatures above 90°C, there are insignificant differences in the β-carotene concentration of BPO. After reaching a certain temperature, the maximum capacity of the adsorbent was obtained. More heat applied to BE-SiO_2 may change the physical characteristics, which decreases the number of active sites and lowers the adsorption ability [26].

The BPO characteristics were determined and compared with the BPO standard (Table 3). The results showed no correlation between the temperature of the bleaching process and the water content that resulted in BPO. However, increasing the temperature of the bleaching process accelerates the oxidation of CPO, hence increasing the acid number and FFA. According to Tan *et al.* [27], increasing temperature may also reduce oil quality due to excessive oxidation of unsaturated fats. In addition, a lower value of the DOBI number was obtained after the bleaching process at higher temperatures. As there were drawbacks to increasing the bleaching process temperature, a suitable temperature should be determined to maintain oil quality during the bleaching process. Based on the effectiveness of β-carotene adsorption and suitable color compared to the BPO standard, the appropriate temperature of the bleaching process using BE-SiO_2 was concluded at 90°C, with 1% w/v BE-SiO_2 for 1.5 hours.

4. Conclusion

The evaluation performance of the bleaching process using BE-SiO_2 was conducted in various process conditions. Increasing BE-SiO_2 concentration in CPO during the bleaching process decreased the β-carotene concentration, acid content, FFA, and DOBI number up to 1% w/v BE-SiO_2, followed by increasing the value of those parameters along with BE-SiO_2 addition. The bleaching process was performed effectively from 0 to 1.5 hours, significantly decreasing the β-carotene concentration, acid content, FFA, and DOBI number. Although more β- carotene was adsorbed during the adsorption process at high temperatures, the heat lowered the BPO quality. From the results, the bleaching process using BE-SiO_2 is effectively carried out with 1% w/v BE-SiO_2 concentration for 1.5 hours at 90°C.

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