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Adsorption and Desorption Properties of Beta-Carotene in Crude Palm Oil (CPO) Using Empty Fruit Bunch-Based Activated Carbon Composite with Metal-Organic Frameworks (MOFs) - Fe(TAC)

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ABSTRACT

The rapid expansion of oil palm plantations in North Sumatra, from 31,730,961 hectares in 2016 to 41,667,011 hectares in 2018, has resulted in significant solid waste generation, particularly empty fruit bunches (EFB). This study aims to utilize EFB as a precursor for activated carbon production and evaluate its effectiveness in adsorbing beta-carotene from crude palm oil (CPO). EFB was processed through carbonization and activation to create a composite adsorbent KA-Fe(TAC) synthesized with metal-organic frameworks (MOFs). Characterization using X-ray diffraction (XRD) confirmed the crystalline structure of the composite. Results indicated that the optimal adsorption efficiency for activated carbon was 8.925%, while the KA-Fe(TAC) composite achieved 21.645%. The findings suggest that the KA-Fe(TAC) composite significantly enhances beta-carotene adsorption capacity compared to conventional activated carbon. Additionally, the optimal contact time for adsorption was found to be 60 minutes for activated carbon and 150 minutes for the KA-Fe(TAC) composite, indicating improved efficiency over time.

Keywords: Crude Palm Oil, Activated Carbon, Beta-Carotene Adsorption

1. INTRODUCTION

According to data from the Indonesian Central Bureau of Statistics (2018), the area of oil palm plantations increased from 31,730,961 ha in 2016 to 41,667,011 ha in 2018. One of the solid wastes produced from oil palm is Palm Empty Bunches (TKKS). TKKS is currently not optimally utilized and is only left to accumulate. TKKS contains lignocellulose compounds consisting of cellulose, hemicellulose and lignin with percentages of 45.95%, 16.49% and 22.84% respectively. The existence of a high enough lignocellulose content makes TKKS potentially used as a basic material for making activated carbon.[1]

Activated carbon is used as an adsorbent for anions, cations, various organic and inorganic compounds, in the form of solutions and gases, which have selective absorption properties, which favor non-polar materials over polar materials. Activated carbon is a porous solid or powder containing 85-95% carbon compounds. Apart from being a fuel, carbon can be used as an absorbent that has good absorption power. In making activated carbon, the step taken is activation. Activation is to increase the size of the pores of activated carbon and form pores that bind together, with increasing micro pore volume and internal surface area, absorption is faste.[2]

Porous polymers or metal organic frameworks (MOFs) are porous microcrystalline materials, which over the past few years have shown their potential as sorbents for various applications, ranging from drug delivery to gas separation and adsorption. To enhance adsorption, we synthesized composite materials/MOFs with the aim of extending high adsorption.[3] Adsorption is the absorption of a substance (molecule or ion) on the surface of an adsorbent. In the adsorption mechanism where there is a process of molecules that were originally in the solution, attached to the surface of the adsorbent physically. A molecule can be adsorbed if the adhesion force between the adsorbate molecule and the adsorbent molecule is greater than the cohesion force on each molecule. One type of adsorbent that is used and widely developed for adsorption and heavy metals is activated carbon because it has a large adsorbing capacity and can be regenerated.[4]

The carotenoids contained in crude palm oil (CPO) will interact with the adsorbent used to bind the carotenoids without undergoing a chemical reaction, ensuring that the CPO remains chemically unchanged. This adsorption process can be conducted at room temperature and does not require high energy.[5]

Crude palm oil (CPO) is obtained from the extraction of the flesh of the oil palm fruit (*mesocarp*). CPO is the richest source of natural carotenoids in the form of retinol (pro-vitamin A).[6] Research[7] states that carotene is an orange pigment, the orange color in crude palm oil is caused by this carotene content. Carotene is a member of the carotenoid group which is included in the terpene compound group. Various methods of taking carotenoids from coconut oil have been carried out including the adsorption process. Research examining the absorption mechanism of β-carotene adsorption process using activated carbon composite MOFs Fe(TAC).

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The main materials used in this research are palm empty bunches Crude Palm Oil (CPO). The chemicals used were Phosphoric acid (H_3PO_4), Terephthalic acid (TAC), Hydrofluoric acid (HF), Nitric Acid (HNO_3), Ferric Chloride ($FeCl_3$), distilled water, and filter paper. The equipment used in this study included glassware, burette, stative, clamps, analytical balance, 200 mesh sieve, oven, furnace, hot plate, X-Ray Diffractometer (XRD), and UV-Vis Spectrometer.

2.2. Research Procedure

2.2.1. Preparation of Palm Empty Bunches

Palm Empty Bunches was taken from PT PKS Pagar Merbau, North Sumatera. The collected Empty Palm Kernel Bunches are washed using running water and dried in the sun until dry. The dried Empty Palm

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Kernel Bunches were chopped into small pieces then pulverized using a grinder and sieved with a 200 mesh sieve.[8]

2.2.2. Moisture Content of Palm Empty Bunches

The moisture content of Palm empty bunches can be determined by drying the biosorbent in an oven. Petri dishes were subjected to a 15-minute drying process at 105°C in an oven. Subsequently, 2 grams of biosorbent were placed in a petri dish of known weight and subjected to the same drying process for a period of 2 hours. Following this, the biosorbent were cooled in a desiccator for 15 minutes and weighed again until a constant weight was obtained.

2.2.3. Biosorbent Carbonization

FEB adsorbent prepared sample then insert to the furnace and setup the temperature at 500ºC for 2 minutes. After finished the carbonization the sample was cooled to a room temperature and transferred in a closed container.[9]

2.2.4. Activated Carbon Activation

Activated carbon that has been carbonized is prepared and weighed. A ratio of 1:10 is used between activated carbon and 10% H3PO4, and then it is soaked for 24 hours. The activated carbon is filtered using filter paper and a vacuum filter, then washed with distilled water until a neutral pH is achieved. The activated carbon is dried for 24 hours at a temperature of 105 °C in an oven, cooled and placed in a sealed container.[9]

2.2.5. Synthesis of the Carbon-Based Composite Fe(TAC)

The synthesis of the carbon-based composite Fe(TAC) was conducted in two stages with a molar ratio of Fe : TAC : KA (1:2:3). The first stage involved dispersing terephthalic acid into activated carbon. An ethanol solution containing terephthalic acid was prepared and then dispersed into the activated carbon. The amount of the dispersed solution was determined based on the specified ratio. The mixture of terephthalic acid and activated carbon was then soaked for 24 hours. The second stage involved mixing iron (III) chloride, hydrofluoric acid, nitric acid, and distilled water. Subsequently, both beakers were combined and refluxed for 8 hours at a temperature of 90 °C. The resulting product was characterized using X-ray diffraction (XRD) with an X-ray diffractometer.[10]

2.2.6. Determination of the Optimal Adsorption Mass Effect on CPO

Crude Palm Oil (CPO) was introduced into five beakers, each containing 100 mL of CPO. Modified Fe activated carbon was then added in varying amounts of 2, 4, 6, 8, and 10 grams to each beaker. The mixture was heated using a hot plate at a temperature of 60 °C and homogenized using a magnetic stirrer at a constant speed of 120 rpm for 120 minutes. Following this, the mixture was filtered using Whatman No. 1 filter paper, and the filtrate was stored for β-carotene concentration analysis using a UV-Vis spectrophotometer.[11]

2.2.7. Determination of the Optimal Contact Time effect on CPO adsorption

Modified Fe activated carbon and CPO were added to a beaker in the optimal mass ratio previously determined. The mixture was heated on a hot plate at 60 °C and stirred with a magnetic stirrer at a constant

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speed of 120 rpm for varying contact times of 30, 60, 90, 120, and 150 minutes. Afterward, the mixture was filtered using Whatman No. 1 filter paper, and the resulting adsorption filtrate was stored for β-carotene concentration analysis with a UV-Vis spectrophotometer.

2.2.8. Analysis of β-Carotene Concentration

The CPO sample was first melted over a water bath and then labeled accordingly. A sample weighing 0.1001 grams was placed into a 25 mL volumetric flask, followed by the addition of isooctane up to the mark on the flask. The mixture was homogenized until the CPO was completely dissolved, after which the absorbance of the sample was measured using a UV-Vis spectrophotometer at a wavelength of 446 nm.

3. RESULTS AND DISCUSSION

3.1. Analysis of Characterization Results

The XRD characterization results of the KA-Fe(TAC) composite indicate that the KA-MOFs Fe(TAC) exhibits a crystalline structure. The sharp peaks observed are likely indicative of metal contact, appearing at 2 theta values in the range of 15°-40°. The activated carbon composite MOFs Fe(TAC) shows a similar pattern; however, it is evident that the intensity has decreased, which may lead to differences in crystal orientation.

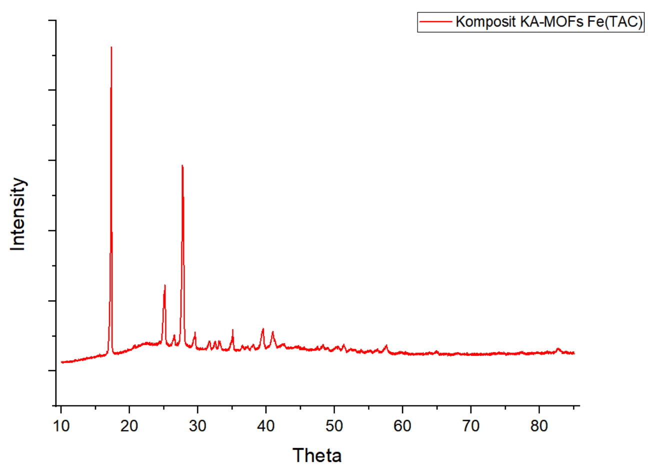

Figure 1. XRD Patterns of KA-Fe(TAC) Composites

The results presented in Table 1 indicate that the KA-Fe(TAC) composite exhibits the same crystal systems, namely monoclinic and triclinic. This suggests that the KA-Fe(TAC) composite is homogeneous and that the composite has been successfully formed, as evidenced by the differences in crystal systems, which also imply variations in crystal orientation.

The determination of the optimal mass of activated carbon was conducted to ascertain the amount of adsorbent required to maximize the absorption of β-carotene present in CPO. The β-carotene adsorption process on CPO utilized several mass variations to identify the optimal adsorbent mass for β-carotene uptake.

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The mass variations employed were 2 g, 4 g, 6 g, 8 g, and 10 g for both activated carbon and composite KA-Fe(TAC), maintaining consistent mass variations. The adsorption was carried out under identical conditions of temperature, stirring speed, and contact time for each sample mass. When a solution contains more than one type of adsorbable substance, these substances will compete for the surface or pores of the adsorbent. The high adsorption capacity of activated carbon is due to its abundant pores and large surface area, allowing it to absorb more organic molecules from solutions or gases compared to bleaching earth. The combined bleaching process, involving heat treatment with activated carbon, can generate new color-producing compounds, such as oxidation products of tocopherols. Activated carbon is not classified as a selective adsorbent; therefore, other coloring substances will also be adsorbed, leading to faster saturation of the activated carbon by these substances and a reduced ability to absorb β-carotene.

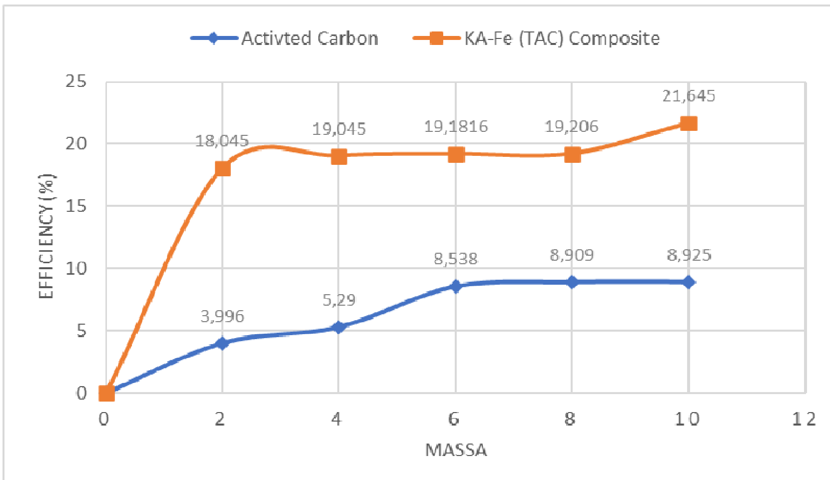

Figure 2. Beta-carotene Adsorption Efficiency at Optimal Mass

In Figure 2, it can be observed that the adsorption efficiency of activated carbon and the KA-Fe(TAC) composite increases with the increasing mass used, indicating that the optimal condition for β-carotene has not yet been found. The most optimal absorption efficiency for activated carbon is at a mass of 10 grams, achieving an absorption efficiency of 8.925%, while for the KA-Fe(TAC) composite, the optimal mass is also 10 grams, with an efficiency of 21.645%.

Contact time can significantly influence performance and absorption capacity, thereby affecting the effectiveness of a sample. The optimum adsorption time for beta-carotene was determined by varying the contact time after establishing the optimal mass. The contact time variations included 30 minutes, 60 minutes, 90 minutes, 120 minutes, and 150 minutes. Adsorption was conducted at a temperature of 60°C with a stirring speed of 120 rpm. According to [12], the adsorption process on activated carbon involves strong physical bonds within its porous structure. The bonds formed can be categorized as London or van der Waals interactions. The van der Waals forces that occur during adsorption on the silica surface arise from dipole-dipole interactions.

In Figure 3, it can be observed that the optimum adsorption efficiency of activated carbon occurs at the 60th and 150th minutes for the Fe-(TAC) composite. The amount of β-carotene adsorbed on activated carbon is 44.662%, which is slightly lower than that of the Fe-(TAC) composite at 46.30%. Based on this study, it

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can be concluded that the adsorbent has not been fully saturated with the adsorbate, indicating potential instability of the adsorbent.

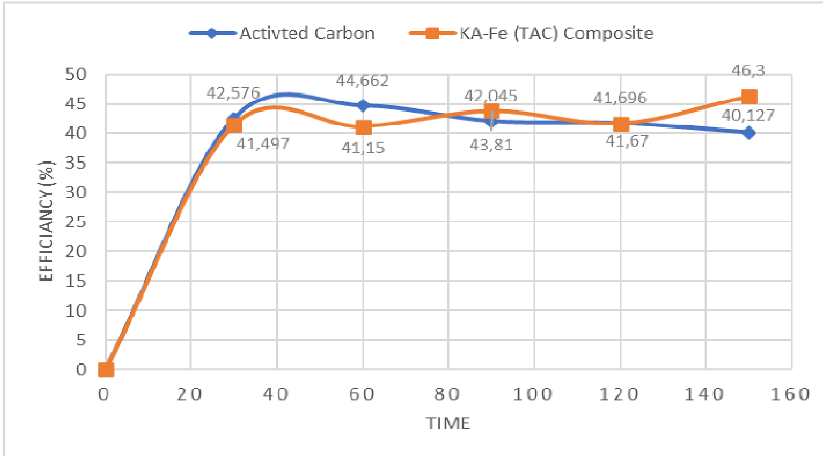

Figure 3. Adsorption Efficiency of Beta-Carotene at Various Contact Times

4. CONCLUSION

The characterization results indicate that the KA-Fe(TAC) composite forms a crystalline structure, as evidenced by the sharp peaks observed in the XRD analysis, corresponding to monoclinic and triclinic crystal systems. The optimal conditions for using activated carbon in the adsorption of β-carotene show an efficiency of 8.925%, while the efficiency for the KA-Fe(TAC) composite is significantly higher at 21.645%. Additionally, under optimal time conditions, the adsorption efficiency for activated carbon reaches 44.862%, whereas the KA-Fe(TAC) composite exhibits an efficiency of 46.30%. These findings highlight the effectiveness of the KA-Fe(TAC) composite in enhancing adsorption performance compared to standard activated carbon.

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