

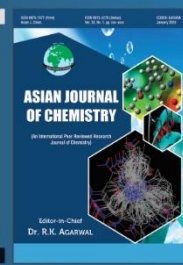


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Composite of Oil Palm Empty Fruit Bunches-derived Carbon with Metal Organic Frameworks for Recovery of β -Carotene in Crude Palm Oil

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This study focuses on the synthesis and characterization of activated carbon composites from oil palm empty fruit bunches (OPEFB) modified with a metal organic frameworks (MOFs) based on Cu(II) and benzene dicarboxylic acid (BDC) for adsorption and isolation of β -carotene in crude palm oil (CPO). Two types of composites were synthesized with variations in the ratio of activated carbon: Cu(II):BDC ligand, namely PC-CB-1 (3:1:2) and PC-CB-2 (2:1:3) using the reflux method. XRD characterization showed PC-CB-2 had a higher crystallinity, while PC-CB-1 was dominated by the amorphous phase of activated carbon. SEM analysis confirmed that PC-CB-1 has porous structure compared to PC-CB-2. Adsorption tests indicated that PC-CB-1 had the highest β -carotene adsorption capacity ~ 10.6 mg/g for 90 min and PC-CB-2 showed a lower adsorption capacity (~ 6.3 mg/g) but excelled in the desorption (isolation) process with efficiency of $\sim 88\%$ in the first 30 min. This indicates a trade-off which carbon dominance (PC-CB-1) enhances capacity through surface area, while MOFs dominance (PC-CB-2) improves selectivity and ease of release through π - π and coordination interactions. This study concludes that controlling the composite ratio is a key parameter in optimizing adsorbent performance for purification and isolation of bioactive compounds from CPO.

Keywords: Activated carbon, Metal organic frameworks, Carbon composite, β -Carotene, Adsorption, Crude palm oil.

INTRODUCTION

Crude palm oil (CPO) is one of Indonesia's strategic commodities, with substantial economic value and widespread applications in the food, cosmetics and bioenergy industries. It contains carotenoid pigments, particularly β -carotene, which impart its characteristic red-orange colour and serve as valuable bioactive compounds [1,2]. Although β -carotene is desirable for nutraceutical applications, its concentration may require control or separation during oil refining to meet specific quality standards. Selective adsorption and isolation methods are consequently important for improving CPO processing and enabling carotenoid recovery.

Conventional pigment-removal methods such as bleaching with bleaching earth or commercial activated carbon, can suffer from limited selectivity, nutrient losses and inadequate adsorption efficiency [3,4]. Dependence on imported

commercial adsorbents may further increase production costs and develop sustainability concerns. These limitations have encouraged the development of alternative materials from biomass waste, particularly oil palm empty fruit bunches (OPEFB), which can be converted into activated carbon with a developed pore structure and high surface area [5,6]. The OPEFB-derived activated carbon offers abundant surface functional groups and a well-developed porous structure. However, conventional activated carbon materials often have limited molecular selectivity, which can restrict their ability to selectively adsorb non-polar compounds such as β -carotene.

Combining activated carbon with porous coordination polymers (PCPs) offers a potential route to improve adsorption selectivity. PCPs constructed from transition-metal ions, such as Cu(II) and aromatic ligands such as benzene dicarboxylic acid (BDC), possess ordered crystalline structures, porous networks and interaction sites that may support mole-

cular recognition through π - π stacking and metal-associated interactions [7-10]. Integrating OPEFB-based activated carbon with Cu(II)-BDC PCPs (CB) is expected to combine the adsorption capacity and pore structure of carbon with the specific interaction capabilities of the PCP phase [11-15].

Previous investigations have examined activated carbon, silica, clay and mesoporous carbon for β -carotene adsorption. Reported capacities include approximately 86.21 mg g⁻¹ for florisol and 25.32 mg g⁻¹ for silica gel. Mesoporous carbon-coated monoliths have achieved capacities of 22.37 mg g⁻¹ in methyl ester and 62.12 mg g⁻¹ in isopropanol at 50 °C [16-18]. These values depend strongly on the pore structure, surface chemistry, solvent composition and operating conditions. Recent studies [19,20] have examined β -carotene adsorption from vegetable oils, including CPO, using acid-activated clays and iron-modified adsorbents. The effects of carbon-PCP composition on adsorption capacity, selectivity and β -carotene recovery remain insufficiently explored.

Studies have reported improved β -carotene removal from CPO using OPEFB-based activated carbon and metal-modified composites, with Cu-containing materials exhibiting enhanced adsorption and rapid desorption [21,22]. The relative proportions of activated carbon, Cu(II) and benzene dicarboxylic acid (BDC) may influence pore accessibility, crystallinity, active-site distribution and adsorbate-adsorbent interactions, yet their effects on β -carotene adsorption and recovery remain insufficiently explored. Highly porous adsorbents may offer high adsorption capacity but hinder desorption, while stronger specific interactions may improve affinity at the expense of pore accessibility. Systematic control of the carbon/MOF ratio is therefore important for understanding the balance between physical adsorption and specific physico-chemical interactions and for optimizing adsorption capacity, selectivity and β -carotene recovery.

The novelty of this study lies in systematically varying the composition of OPEFB-derived activated carbon and Cu-BDC to establish a structure-performance relationship for β -carotene separation directly from CPO. Two carbon-PCP composites, with activated carbon(II) ratios of 3:1:2 (PC-CB-1) and 2:1:3 (PC-CB-2), were synthesized and characterized. Their adsorption and desorption behaviour were evaluated in terms of adsorption capacity, separation efficiency, β -carotene recovery, and possible interaction mechanisms. Comparing carbon-dominant and PCP-dominant compositions is intended to clarify how the material balance affects adsorption and recovery functions. The study seeks to establish a composition-based design strategy for biomass-derived carbon/PCP adsorbents and contribute to the development of selective, efficient and sustainable materials for β -carotene recovery in the palm oil industry.

EXPERIMENTAL

The materials used in this study included oil palm empty fruit bunches (OPEFB) obtained from PTPN II, Medan, crude palm oil (CPO), Cu(NH₃)₂·3H₂O, distilled water, H₃PO₄, KI, isooctane, isopropanol, neutral alcohol, phenolphthalein indicator, Whatman No. 1 filter paper, Cu metal, benzene-1,4-

dicarboxylic acid (BDC) and ethanol. The equipment used included laboratory glassware, an analytical balance, thermometer, grinder, 200-mesh sieve, oven, furnace, hot plate, desiccator, X-ray diffractometer (XRD), scanning electron microscope (SEM) and UV-Vis spectrophotometer.

Preparation of OPEFB: The OPEFB samples obtained from PTPN II, Medan, were initially washed thoroughly with water to remove adhering impurities and subsequently dried under sunlight to reduce their moisture content. The dried OPEFB was then cut into smaller pieces and ground using a grinder. The ground material was sieved using a 200-mesh sieve to obtain a relatively uniform particle size. The resulting OPEFB powder was collected and used for subsequent preparation of the activated carbon.

Activation of carbon: The dried OPEFB was initially carbonized in a furnace at 500 °C for 2 h. After carbonization, the material was allowed to cool to room temperature and subsequently transferred to a closed container to prevent contamination and moisture uptake. The resulting carbonized material was weighed and 10 g of carbonized OPEFB was immersed in 100 mL of 10% H₃PO₄ solution and allowed to stand for 24 h for chemical activation. Following activation, the material was separated from the activating solution by filtration using Whatman No. 1 filter paper under vacuum. The activated carbon was then thoroughly washed with distilled water until the washing solution reached neutral pH. The washed activated carbon was dried in an oven at 105 °C for 24 h. After drying, the activated carbon was cooled to room temperature and stored in a closed container until further use.

Synthesis of PC-CB activated carbon composite: The synthesis was carried out in two stages. The first stage involved dispersing benzene dicarboxylic acid into activated carbon. An ethanol solution of benzene dicarboxylic acid (0.6 g benzene dicarboxylic acid per 10 g ethanol) was prepared and dispersed into activated carbon (about 1 g solution per 0.9 g activated carbon) with a ratio of 1:10:0.6. The second stage was the addition of reagents needed to produce PC-CB-1 crystals, with a solution composition of 0.315 g Cu(NO₃)₂·3H₂O; 0.19 mL HNO₃; 0.2 mL HF; 20 mL H₂O. Both mixtures were combined and heated at 90 °C for 9 h using the reflux method. The solution was then cooled to room temperature, filtered with filter paper and washed three times with distilled water, then washed three times with absolute ethanol and air-dried. The PC-CB-2 carbon composite was prepared in the same way using 0.315 g Cu(II) solution, 0.9 g benzene dicarboxylic acid and 0.6 g palm activated carbon.

Crude palm oil (CPO) quality analysis: The initial CPO was analyzed for β -carotene content, free fatty acid (FFA) levels and peroxide value (PV). These parameters were measured again after adsorption and desorption to assess changes in the chemical properties of oils during the separation process.

β -Carotene adsorption isotherm

Effect of optimum adsorbent mass: CPO was placed in five beakers, with 100 mL added to each, followed by activated carbon at doses of 2, 4, 6, 8 and 10 g. The mixtures were heated at 60 °C and stirred at 120 rpm for 120 min using a magnetic stirrer. After treatment, each mixture was filtered through Whatman No. 1 filter paper to separate the adsorbent.

The same procedure was repeated using PC-CB-1 and PC-CB-2 carbon composites.

Effect of optimum contact time: Activated carbon and CPO were placed into a beaker glass using the optimal mass ratio previously determined. The mixture was then heated on a hot plate at 60 °C and homogenized with a magnetic stirrer at a constant speed of 120 rpm for 30, 60, 90, 120 and 150 min. Then, the mixture was filtered using Whatman No. 1 filter paper. The β -carotene content was analyzed using a UV-Vis spectrophotometer.

β -Carotene desorption: Desorption was evaluated using the adsorbent recovered from CPO after the optimum adsorption contact time. Isopropanol (350 mL) was added to the adsorbent and the mixture was heated at 50 °C and stirred at 180 rpm for 45 min. It was then filtered through Whatman No. 1 filter paper and the β -carotene content in the filtrate was determined using a UV-Vis spectrophotometer. Equilibrium isotherm and thermodynamic analyses were not performed in this study. Since CPO was used directly as the adsorption medium, its initial β -carotene concentration was not analytically varied to establish the conditions required for Langmuir and Freundlich isotherm modelling. Adsorption experiments were not conducted across a range of temperatures, as temperature-dependent changes in CPO viscosity and physical properties could affect mass transfer.

RESULTS AND DISCUSSION

X-ray diffraction: The X-ray diffraction patterns (Fig. 1) of PC-CB-1 and PC-CB-2 reveal crystalline and amorphous contributions associated with the Cu(II)-benzene dicarboxylic acid (Cu-BDC) framework and biomass-derived activated carbon, respectively. The main diffraction peaks at $2\theta \approx 16-18^\circ$, $25-26^\circ$ and $28-29^\circ$ are attributed to the crystalline Cu-BDC phase. PC-CB-2 exhibits sharper and more intense peaks than PC-CB-1, suggesting higher crystallinity and structural order at the higher PCP composition (carbon(II) = 2:1:3). In comparison,

the broader, less intense peaks and raised baseline of PC-CB-1 (3:1:2), particularly across $2\theta \approx 20-30^\circ$, which indicate a higher contribution from the amorphous carbon phase. This is consistent with the disordered structure and limited graphitic domains commonly associated with biomass-derived activated carbon. Small reflections in the $30-45^\circ$ range may arise from additional crystalline phases or semi-crystalline carbon, although their assignments require further analysis. No prominent impurity peaks were observed [23,24].

The differences between the patterns indicate that composite composition affects the relative contributions of the crystalline PCP and amorphous carbon phases. The more crystalline structure of PC-CB-2 may provide a better contribution from Cu(II) sites and aromatic surfaces to specific adsorbate interactions, while the carbon-rich PC-CB-1 may favour adsorption through accessible pores and surface sites. These structural features may influence β -carotene adsorption and recovery. The absence of substantial peak shifts suggests that the principal diffraction features remain broadly similar in both composites. Slight peak broadening in PC-CB-1 may reflect smaller crystallites or lattice disorder, although additional analysis would be needed to establish its origin. Thus, the XRD patterns reveal differences in crystallinity and phase contributions between the composites, providing a structural basis for examining their β -carotene adsorption behaviour [25,26].

SEM studies: The SEM images (Fig. 2) illustrate the surface morphology of the OPEFB-derived activated carbon-MOF composites, PC-CB-1 and PC-CB-2, prepared at different composition ratios. Both materials exhibit heterogeneous structures comprising the carbon matrix and crystalline porous coordination polymer (PCP) phases. PC-CB-2 is characterized by micrometre-sized particles arranged in compact aggregates. Its rough surfaces contain layered features and crystalline fragments attached to the matrix, consistent with the growth and distribution of the Cu-BDC phase across the carbon surface. The dense morphology agrees with the higher crystallinity observed by XRD. Partial coverage of the carbon surface by the crystalline phase may restrict access to some pores and hinder the diffusion of large molecules such as β -carotene.

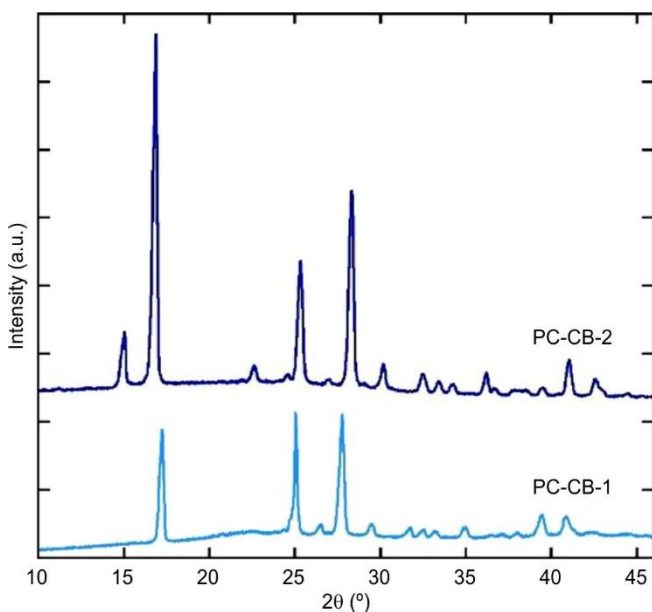


Fig. 1. XRD patterns of PC-CB-1 and PC-CB-2 carbon composites

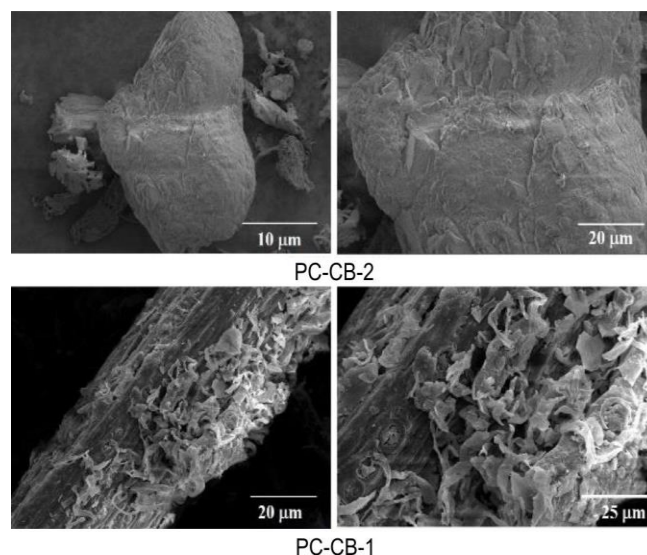


Fig. 2. SEM images of PC-CB-1 and PC-CB-2 carbon composites

PC-CB-1 displays a more open and irregular morphology, with carbon fibre structures clearly visible as the main framework. Numerous cavities and gaps occur across the surface, while small particles are dispersed throughout the carbon matrix. These features are consistent with a better contribution from the activated carbon phase and a less aggregated distribution of the MOF component. The visible cavities and gaps may provide accessible pathways for mass transfer and adsorption, although surface area and pore volume require confirmation through complementary measurements. The morphological differences between the composites are associated with their composition ratios. In PC-CB-2, the denser, MOF-rich structure may provide more Cu(II) sites and aromatic ligands for specific interactions with β -carotene, such as π - π interactions. In PC-CB-1, the more open carbon-rich structure may facilitate molecular diffusion and adsorption within accessible pores. These possible contributions to adsorption behaviour require further investigation and cannot be established from SEM images alone [6,27,28].

Distinct crystalline particles, which are completely separated from the carbon matrix are not readily apparent in the images. This may be consistent with integration of the MOF phase into the composite, potentially through anchoring or in situ growth on the carbon surface. SEM alone, however, cannot confirm interfacial bonding or establish the mechanism of composite formation. The images reveal composition-dependent differences in morphology: PC-CB-2 has a denser structure, while PC-CB-1 retains a more open surface with visible cavities and gaps. These features provide a basis for examining the relationship between composite structure and β -carotene adsorption, with pore accessibility potentially contributing to uptake and MOF-associated chemical sites potentially influencing adsorbate interactions [27-29].

Adsorption and desorption properties of β -carotene in CPO β -carotene

Effect of adsorbent mass: Fig. 3 presents the effect of adsorbent mass on β -carotene adsorption by PC, PC-CB-1 and PC-CB-2. All three materials exhibit an initial increase in adsorption capacity as the adsorbent mass increases, followed by a decrease beyond the optimum dosage. For PC-CB-1, increasing the adsorbent mass from 0 to approximately 2 g raises the adsorption capacity to a maximum of about 3.0 mg/g. This initial increase may be attributed to the higher availability of adsorption sites and surface area, which improves the opportunity for β -carotene molecules to interact with the adsorbent. Beyond the optimum dosage, the capacity per unit mass decreases. Particle aggregation may reduce the effective surface area and limit the utilization of available sites [27]. The lower adsorbate-to-adsorbent ratio at higher dosages may further contribute to this decline, commonly described as the adsorption-capacity dilution effect [30].

PC-CB-2 follows a similar trend but exhibits a lower adsorption capacity than PC-CB-1. Its maximum capacity is approximately 1.0 mg/g at an adsorbent mass of around 4 g, followed by a gradual decrease. The denser morphology observed by SEM may restrict access to some pores and limit β -carotene diffusion, potentially offsetting the contribution of the Cu-BDC phase to adsorbate interactions. These results

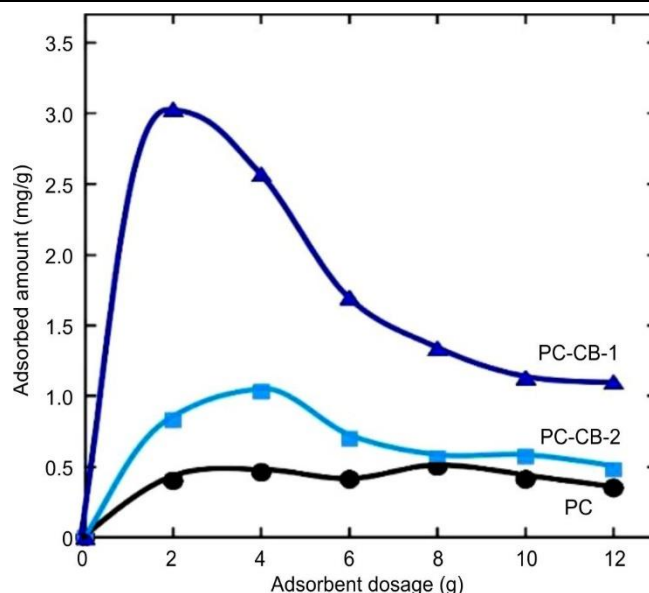


Fig. 3. Effect of adsorbent dosage on β -carotene adsorption in CPO using PC, PC-CB-1 and PC-CB-2

suggest that the adsorption capacity depends not only on the presence of potential active sites but also on their accessibility and the transport of molecules to those sites.

The adsorption behaviour of the composites may arise from the combined contributions of the activated carbon and Cu-BDC phases. The more open morphology of PC-CB-1 may facilitate β -carotene diffusion and pore filling. Interactions between the conjugated structure of β -carotene and aromatic domains in the carbon matrix or BDC ligands may contribute to adsorption. Cu-containing sites may provide further interaction sites. These mechanisms are proposed on the basis of the structural, morphological and adsorption-desorption results; they have not been directly confirmed by spectroscopic evidence.

Unmodified activated carbon (PC) exhibits the lowest adsorption capacity, reaching a maximum of approximately 0.45 mg/g. Its capacity initially increases with adsorbent mass but remains relatively stable thereafter. This lower uptake may be associated with the absence of the Cu-BDC component and its potential contribution to adsorbate interactions. The available results do not, on their own, establish that PC adsorption is governed exclusively by physisorption or that the composites exhibit stronger specific interactions. Further chemical characterization would be needed to distinguish the contributions of pore filling, π - π interactions and Cu-associated sites.

Among the three materials, PC-CB-1 achieves the highest adsorption capacity. Its more open structure may provide better access to adsorption sites and support pore filling, whereas the denser morphology of PC-CB-2 may impose limitations on pore accessibility and molecular diffusion. The comparison points to a possible balance between accessible adsorption capacity and the chemical interactions associated with the MOF component. Selectivity, however, cannot be established from adsorption capacity alone and would require measurements of competing compounds or adsorption selectivity [31,32].

The decrease in capacity per unit mass at higher dosages may be related to the lower amount of β -carotene available per gram of adsorbent, particle aggregation and reduced utilization of adsorption sites. Site overlap and internal diffusion limitations may also contribute [33]. These results emphasize the importance of optimizing adsorbent dosage for β -carotene recovery. PC-CB-1 reaches its maximum capacity at a lower dosage than PC-CB-2, which may reduce the quantity of adsorbent required under the tested conditions. The adsorption trends are consistent with the XRD and SEM observations, which identify differences in the crystallinity and surface morphology between the composites.

Effect of contact time: Fig. 4 shows the effect of contact time on the amount of β -carotene adsorbed by the three types of adsorbents: PC, PC-CB-1 and PC-CB-2. In general, all three systems demonstrate a rapid increase in adsorption capacity at the initial stage, followed by reaching an optimum condition and then tend to decrease or approach equilibrium. For PC-CB-1, β -carotene adsorption increases rapidly during the first 30 min, reaching approximately 8.5 mg/g. This initial uptake may be associated with the availability of unoccupied adsorption sites and efficient transport of β -carotene from the bulk solution to the adsorbent surface. The capacity continues to rise, reaching a maximum of about 10.6 mg/g at 90 min, before decline slightly to approximately 9.0 mg/g at 150 min. This decrease may arise from partial desorption or redistribution of adsorbed molecules. Moreover, changes in the balance between adsorption and desorption, along with variations in site accessibility, may affect the measured capacity at extended contact times [33].

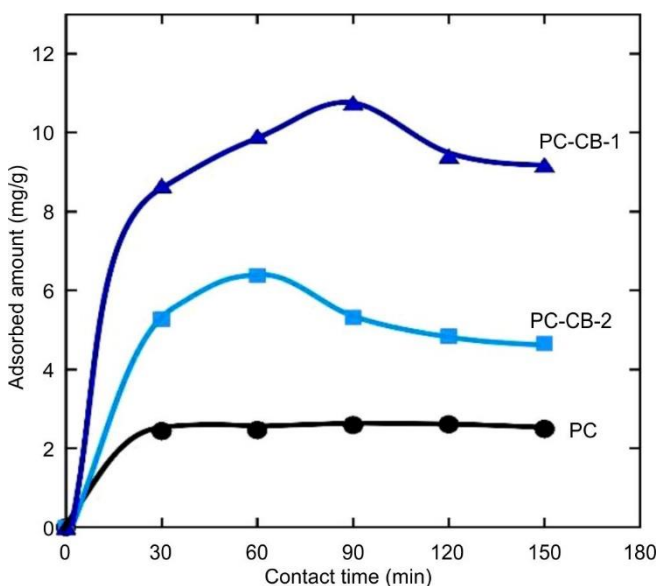


Fig. 4. Effect of contact time on the β -carotene adsorption capacity of PC, PC-CB-1 and PC-CB-2.

PC-CB-2 exhibits a similar trend but a lower adsorption capacity. Its maximum value of approximately 6.3 mg/g occurs at around 60 min, followed by a decrease to 4.5 mg/g at 150 min. The earlier maximum may reflect differences in the accessibility and distribution of adsorption sites. The denser morphology observed by SEM may restrict β -carotene trans-

port into the internal structure, potentially limiting uptake. The role of π - π interactions and interactions with Cu(II) containing sites remains a possible explanation for adsorption behaviour.

Unmodified activated carbon (PC) shows the lowest capacity, reaching approximately 2.5 mg/g within 30–90 min. The capacity then remains relatively stable. This behaviour may reflect the availability of accessible adsorption sites and the characteristics of the carbon surface. The contribution of physisorption is plausible, but the adsorption mechanism cannot be assigned exclusively without further surface and spectroscopic characterization [34,35].

Comparison of the three materials shows that PC-CB-1 achieves the highest measured adsorption capacity, with its maximum occurring at 90 min. PC-CB-2 reaches its maximum earlier, at around 60 min, but has a lower capacity. The more open morphology of PC-CB-1 may facilitate β -carotene transport and access to adsorption sites, while the denser structure of PC-CB-2 may impose greater diffusion limitations. These observations are consistent with a possible contribution from pore accessibility in determining adsorption performance. The decline in capacity beyond the observed maxima for both composites may be associated with partial desorption or changes in the distribution of β -carotene between the liquid and solid phases. Under the conditions examined, the contact times corresponding to the maximum measured capacities are approximately 90 min for PC-CB-1 and 60 min for PC-CB-2.

Adsorption mechanism: The adsorption of β -carotene by OPEFB-derived activated carbon–MOF composites containing a Cu(II)–benzene dicarboxylic acid framework may involve several concurrent processes, such as pore filling, π - π interactions and interactions with Cu(II)-associated sites. The relative contribution of these processes may depend on the composite composition, which influences the distribution of the carbon and MOF phases and the resulting physicochemical properties.

β -Carotene is a non-polar molecule with an extended π -conjugated structure. Its adsorption is expected to depend on pore accessibility and interactions with suitable surface domains. During the initial stage, β -carotene molecules are transported from the liquid phase to the adsorbent surface and may enter accessible pores. Pore filling is proposed to contribute substantially to uptake, particularly in composites with a higher activated carbon content. In PC-CB-1, the more open morphology observed by SEM may facilitate molecular transport and access to adsorption sites. Mesopores, if present in sufficient proportion, could support the diffusion of these relatively large molecules more effectively than narrow micropores.

The π - π interactions between the conjugated system of β -carotene and aromatic domains in the BDC ligands or carbon matrix may contribute to adsorption affinity. The higher MOF content in PC-CB-2 provides a higher proportion of BDC containing material, which could increase the availability of aromatic interaction sites. Cu(II) centres may provide further interaction sites within the composites. Direct coordination between β -carotene and Cu(II) should be treated cautiously, as β -carotene lacks conventional polar donor groups and the present results do not provide direct evidence of Cu- β -carotene

bonding. Interactions involving the conjugated system may be possible, but their nature and strength require confirmation through suitable spectroscopic or other mechanistic studies. The higher Cu-BDC content of PC-CB-2 may increase the proportion of Cu-associated sites, but it does not by itself establish stronger adsorption or improved stability.

The observed differences between PC-CB-1 and PC-CB-2 are consistent with composition-dependent adsorption behaviour. PC-CB-1 exhibits a higher measured adsorption capacity, which may be associated with its more open morphology and higher accessibility of adsorption sites. PC-CB-2 has a denser structure and higher MOF contribution, but its potential aromatic and Cu-associated interaction sites do not translate into a higher capacity under the tested conditions. The available data are insufficient to conclude that PC-CB-2 has higher selectivity or adsorption stability. Controlling the ratio of activated carbon, Cu(II) and BDC is consequently an important consideration in developing these composite adsorbents. The appropriate composition will depend on the desired balance of adsorption capacity, selectivity and recovery performance.

β -Carotene desorption: Desorption experiments were conducted over 150 min to evaluate the release of β -carotene from PC, PC-CB-1 and PC-CB-2. The kinetic profiles (Fig. 5) show that the carbon-MOF composites released higher amounts of β -carotene than unmodified PC. Among the tested materials, PC-CB-2 exhibited the highest desorption capacity. During the first 30 min, it showed a rapid release, reaching approximately 4.7 mg/g. The amount desorbed then declined gradually to about 3.7 mg/g at 150 min. This decrease may be associated with β -carotene re-adsorption onto available sites or changes in analyte stability in the desorption medium. Further experiments would be required to distinguish between these possibilities [3,16].

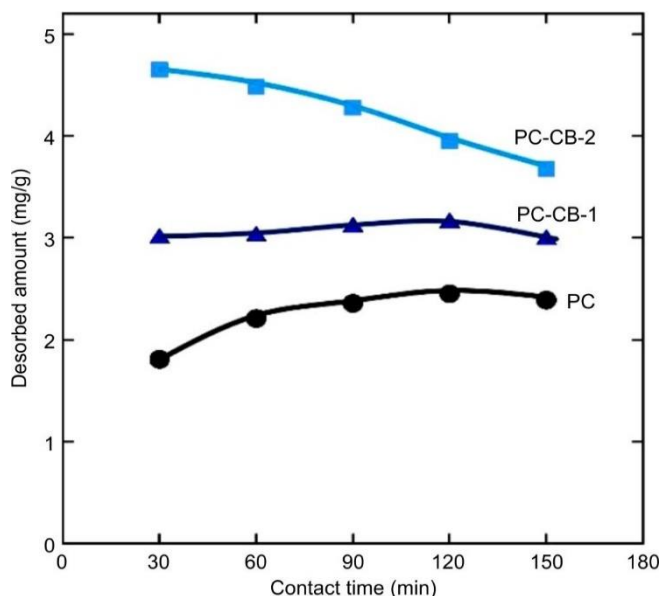


Fig. 5. Comparative results of desorption of β -carotene from PC, PC-CB-1 and PC-CB-2

PC-CB-1 and PC displayed comparatively gradual desorption profiles. PC-CB-1 maintained desorption values around

3.0 mg/g, while unmodified PC showed the lowest release, increasing from approximately 1.8 mg/g initially to 2.4 mg/g at 120 min, after which the profile approached a plateau. These differences suggest that incorporating the Cu-BDC component alters the desorption behaviour of the carbon matrix. The higher desorption capacity of PC-CB-2 indicates that it released more β -carotene under the conditions tested. The difference may be related to changes in the interactions between β -carotene and the composite surface, although the desorption data alone cannot establish changes in binding energy or identify the underlying mechanism [4]. The rapid initial release from PC-CB-2, followed by a decrease, also emphasizes the importance of optimizing the desorption contact time to limit possible readsorption or analyte loss. PC-CB-2 may be suitable for applications requiring rapid β -carotene release, but its regeneration efficiency and practical performance should be assessed through repeated adsorption-desorption cycles and a complete mass balance.

β -carotene desorption efficiency: In addition to desorption capacity (mg/g), β -carotene release was assessed in terms of desorption percentage over time (Fig. 6). This parameter provides a comparative measure of the fraction of adsorbed β -carotene released from each material. Although unmodified PC exhibited a relatively low desorption capacity, its desorption percentage increased steadily, reaching approximately 96% at 150 min. This result indicates that a large fraction of the β -carotene adsorbed on PC was released under the conditions tested. The gradual increase may be consistent with progressive diffusion and release from accessible adsorption sites [4,36].

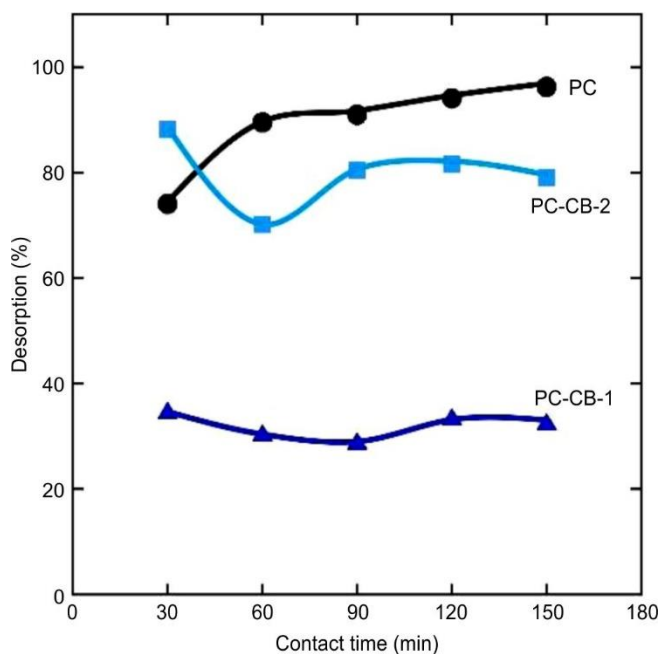


Fig. 6. β -Carotene desorption efficiency of PC, PC-CB-1 and PC-CB-2 as a function of contact time.

PC-CB-2 exhibited a fluctuating desorption profile. Its desorption efficiency reached approximately 88% at 30 min, decreased to around 70% at 60 min and then recovered and stabilized at approximately 80%. The initial high value indi-

cates rapid β -carotene release, while the subsequent decrease may reflect readsorption or changes in the distribution of β -carotene between the adsorbent and desorption medium. By comparison, PC-CB-1 exhibited a consistently lower desorption percentage, remaining within the range of approximately 30–35%. This indicates that a smaller fraction of the adsorbed β -carotene was released during the experiment. The result may reflect differences in pore accessibility or adsorbate–surface interactions within the composite.

Comparison of desorption capacity and desorption percentage provides a more complete assessment of the three materials. PC sample released a high proportion of its adsorbed β -carotene but had a relatively low capacity. PC-CB-1 exhibited a higher adsorption capacity in the preceding experiments but a low release percentage. PC-CB-2 combined a comparatively high desorption capacity with a substantial release percentage, particularly during the initial contact period [23,36]. These findings make PC-CB-2 a candidate for further evaluation in β -carotene recovery applications.

Effect of CPO quality after adsorption: The quality of crude palm oil (CPO) after treatment with the different adsorbents was evaluated by measuring free fatty acid (FFA) content and peroxide value (PV). These parameters are commonly used to assess hydrolytic deterioration and primary lipid oxidation, respectively. All adsorbents reduced the FFA content from the initial value of 4.15% in raw CPO. The largest decrease was observed with PC-CB-1, which lowered the FFA content to approximately 3.3% (Fig. 7).

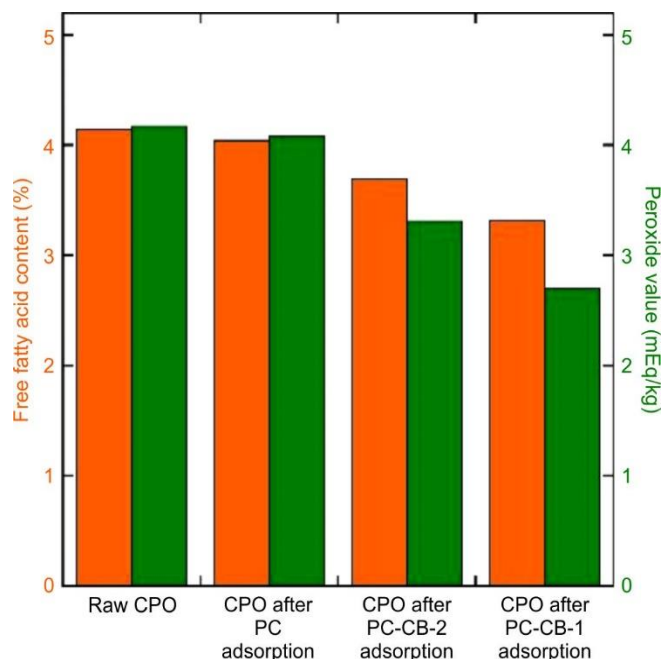


Fig. 7. Effect of β -carotene adsorption using PC, PC-CB-1 and PC-CB-2 on the FFA and peroxide value of CPO

PC-CB-1 showed a higher reduction in FFA content than PC-CB-2 and unmodified PC. This difference may be associated with variations in surface chemistry and pore accessibility that affect interactions with fatty acid molecules. The limited reduction obtained with PC suggests that its surface properties may be less favourable for FFA removal under the

conditions tested. The PV of raw CPO was 4.2 mEq/kg. Following adsorption, PC-CB-1 reduced the PV to approximately 2.7 mEq/kg, while PC-CB-2 lowered it to around 3.3 mEq/kg. Unmodified PC caused little change relative to the raw oil. The lower PV values obtained with the composites suggest that they removed some primary oxidation products from CPO.

The FFA and PV results identify PC-CB-1 as the most effective of the tested materials for reducing both parameters under the experimental conditions. Its performance may be related to differences in accessible surface sites and pore structure. The desorption experiments further support the possibility of recovering β -carotene from the composites. PC-CB-2 achieved a desorption efficiency of ~88% within 30 min.

Conclusion

Oil palm empty fruit bunches (OPEFB)-derived activated carbon-MOF composites based on Cu-BDC were successfully synthesized and investigated for β -carotene adsorption and recovery from crude palm oil (CPO). PC-CB-1 exhibited the highest adsorption capacity of 10.6 mg g⁻¹, which was attributed to its more porous structure and the predominance of pore-filling mechanisms. PC-CB-2 achieved a desorption efficiency of 88%, potentially associated with π - π and coordination interactions involving the MOF phase. PC-CB-1 was particularly effective in improving CPO quality, as confirmed from the results in the reductions of free fatty acid content and peroxide value. The results establish the activated carbon-to-MOF ratio as a key parameter influencing adsorption capacity, selectivity and recovery efficiency. Based on these results, it is concluded that OPEFB-derived activated carbon-MOF composites may offer a sustainable approach to β -carotene recovery and CPO purification, with the relative proportions of the two components providing a means of tailoring their performance.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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