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Evaluation of physically modified kenaf core adsorbent for carbon dioxide adsorptive study

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Abstract. This paper evaluates on the capability of kenaf core (Hibiscus Cannabinus L.) material as the proposed solid adsorbent for CO₂ adsorption study via physical treatment modification. The proposed physical treatment was successfully conducted by undergoing two stages of carbonization and activation treatment to improve the pore development structure of the material. The carbonized kenaf core (CKC) was prepared by carbonizing the raw kenaf core (RKC) at 800°C with constant nitrogen flow for 5 hours. Activation process took placed by impregnating KOH in the furnace with alternately switching to CO₂ up to 800°C for 2 hours. The morphological and structural characterization studies were conducted using Field Emission Scanning Electron Microscope (FESEM) and Fourier Transform Infrared Spectroscopy (FTIR). The CO₂ adsorption process was carried in the gravimetric analyzer by varying the effect of bed pressure and gas flowrate. This study revealed that the AKC has the highest CO₂ adsorption capacity of 1.70 mmol/g (1 bar) and 2.85 mmol/g (30 bar). Based on the morphology, AKC demonstrated better small micropore development that helps in providing a larger adsorption site; hence attributes to the higher CO₂ adsorption capacity. This study contributes to the development of knowledge on enhancing the CO2 adsorption on the physically-modified kenaf core material that would be used as an alternative material for CO₂ adsorbent.

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1. Introduction

Global warming is one of the serious consequences results from the high levels of greenhouse gases emission especially carbon dioxide (CO_2) towards atmosphere [1]. Energy and electricity sectors from petroleum refining, coal and natural gas have contributed to the greater percentage of CO₂ emission. Coal combustion in power plants is one of the main sources of CO_2 emissions in Malaysia, and coal remains the preferred fuel due to its abundance and lower cost, with 1.724 billion tonnes of coal reserves reported, largely in Malaysia's west [2]. Physical and chemical absorption, solid adsorption, membrane separation, and cryogenic separation are the prominent strategies used for carbon dioxide capture [1, 3, 4]. Adsorption is considered as a promising CO_2 capture technology that is benefited in terms of process simplicity, low energy regeneration and it is suitable for separating CO₂ from the diluted gas streams compared to the separation by absorption, cryogenic distillation, and membrane separation [5, 6]. Various porous solids have been considered as potential adsorbents for post-combustion CO₂ adsorption or separation for such as activated carbons (AC), carbon molecular sieves, zeolites, and metal organic frameworks (MOFs) [4]. Activated carbon, among the other adsorbents, has been regarded as a promising solid adsorbent due to its high CO_2 adsorption capacity at pressure, despite the fact that only one ambient pressure was recorded [2, 4, 7]. In comparison to the most current MOFs, activated carbon demonstrates moisture insensitivity as well as thermal and chemical resistance [2, 4, 7, 8, 9].

Despite the fact that activated carbon has excellent gas adsorption properties, its current application is limited to high pressure gases [9]. Aside from nitrogen functionalities, other heteroatomic functional groups may influence CO_2 polar interactions or hydrogen bonding within the confined pores of carbonaceous adsorbents. Furthermore, the high carbon content of the adsorbent could strengthen the London dispersion force, which is known as the inter-particle interaction between CO_2 molecules and activated carbon. [2, 10]. Carbonization and activation are the two main methods for synthesizing activated carbon from lignocellulosic materials. Carbonization, also known as pyrolysis, is a process that takes place in an inert atmosphere [1, 7, 8]. Physical activation begins with the reduction of oxygen (O_2) and hydrogen (H_2) elements in carbonaceous material by pyrolysis at 500-1000°C [7, 11, 12], followed by thermal activation at the pyrolysis temperature or higher in the presence of activating reagents like water steam, CO_2 , air, or other gases [11]. Potassium hydroxide (KOH) is a well-known activator for development of excellent activated carbon and has been identified as the most prominent alkali compound in the preparation of activated carbon. [1, 10, 13].

The first redox reaction involving carbonaceous material and KOH is proposed to set up at low temperatures, resulting in carbon oxidation and the synthesis of potassium carbonate (K_2CO_3), while the hydroxide is converted into metallic potassium (K) and H₂. This is exemplified according to the following generalized equation (1):

$$6\text{KOH} + 2\text{C} \rightarrow 2\text{K}_2\text{CO}_3 + 2\text{K} + 3\text{H}_2\uparrow\tag{1}$$

While the biomass heats up, the functional groups exist within the material degrade into volatile molecules like carbon monoxide (CO), CO₂, and water (H₂O), whereas the mass residue aromatizes forming char [1, 13]. Porosity is formed as a result of these volatile molecules escaping through the carbon framework. These molecules can also combine with KOH to generate K_2CO_3 and volatiles, resulting in a high-temperature reactions as referred to equation (2) [1]. The carbon in the material reacts with the KOH until it is totally consumed, where at point it transforms into species such as metallic K and volatiles, according to the equation (3):

$$2\text{KOH} + \text{CO}_2 \rightarrow \text{K}_2\text{CO}_3 + \text{H}_2\text{O}\uparrow \tag{2}$$

$$2\text{KOH} + 2\text{C} \rightarrow 2\text{CO}\uparrow + 2\text{K}\uparrow + \text{H}_2\uparrow \tag{3}$$

$$K_2 CO_3 + C \rightarrow K_2 O + 2 CO \uparrow \tag{4}$$

All types of KOH are consumed when the temperature rises to a minimum of 600°C [1]. Activation causes minor etching on the carbon surface at a low temperature of 600°C. Furthermore, at temperatures

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of 700°C and 800°C, it can significantly etch the carbon frameworks, allowing redox reactions to produce potassium oxide (K_2O) and K_2CO_3 [11]. Redox reactions produce K_2CO_3 , which may also react with biomass carbon to yield K_2O , metallic K, and volatiles based on equation (4-5) [1, 11, 13, 14]. The carbonization and activation reactions of depolymerization, dehydration, and gas evolution described above are attributed to the production of high-porosity activated carbons [1, 14]. Finally, the formation of metallic K at temperatures around 800°C is an important factor in creating the pore framework by integration and diffusing into the inner structure of the carbon particles and predominantly graphitic layers, resulting in the integration of new porosities as well as stretching existing pores with high specific surface area [1, 11, 13].

In Malaysia, the National Economic Action Council (NEAC) initiated the kenaf plantation effort in 1999 as a replacement for tobacco and to prevent wood-logging for the paper industry[15]. Tobaccobased industry in Malaysia was affected by the ASEAN Free Trade Area (AFTA) regulation that prevent the import and export process of tobacco [16]. As a result of the closure of all international cigarette manufacturing companies in 2018, Malaysia is no longer one of the countries producing international branded cigarettes, and instead imports branded cigarettes for domestic consumption [17]. Alternatively, kenaf became a potential crop for replacing tobacco plant [16]. Nowadays, kenaf have been widely explored for several applications due to its bast and core adsorption capacity for hydrogen adsorbent [18], oil adsorbent [19], CO₂ adsorbent [20], heavy metal adsorbent such as nickel ion [21], Cu (II) ion [22] the waxes adsorbent from batik textile in wastewater effluent [23] and for animal bedding materials [24]. This study focuses on the evaluation of physical-based modified kenaf core material for CO₂ adsorption process. The aim of physical modification is to improve the structural and physical properties of kenaf core for CO₂ adsorption study.

2. Research Methodology

2.1. Preparation of Solid Kenaf Adsorbent

Raw kenaf core (*Hibiscus Cannabinus L.*) (RKC) was selected as a precursor in this study. The kenaf core sample was purchased from National Kenaf and Tobacco Board (NKTB), Kelantan, Malaysia. This experimental work focuses on the kenaf core sample within a range of 300 to 499µm by grinding and sieving to the desired particle size.

2.1.1. Carbonization Treatment. Firstly, kenaf core was placed in the column reactor for about $\frac{3}{4}$ of the column size. The furnace was connected with N₂ tank for carbonization process and the temperature was set to 800°C [25]. The carbonization process is begun by heating the sample with heating rate of 10°C/min. When the temperature reached the desired temperature of 800°C, it was continuously burned for another two hours. Then, the sample was cooled down before it was taken out from the reactor column.

2.1.2. Activation Treatment. The char kenaf core (CKC) was treated with KOH pellets in the ratio of 1:2 which that soluble in 200 ml of distilled water by using the magnetic stirrer for two hours [7, 13]. The treated CKC was dried up overnight. The activation process was conducted by placing the CKC in the furnace by flowing the nitrogen gas until the temperature reached at 800°C. At about 800°C, the N₂ gas flowing was switched to CO₂ and continuously burned for the next two hours [7, 26]. After two hours, the gas was switched back to nitrogen gas prior being cooled at room temperature. The prepared sample was denoted as activated carbon kenaf core (AKC) modified sample.

2.2. Characterization Analysis

Morphological analysis is evaluated by using the field emission scanning electron microscope (FESEM). To execute the analysis, the sample was coated with a thin layer of carbon to prepare a suitable surface condition and prevent electrostatic charging during the scanning phase. The structural analysis is conduit through the Fourier Transform Infrared Spectroscopy (FTIR). Each of the light absorption represents

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different types of bonds depending on the structure of the attached molecule [27, 28]. In this study, the frequency range of FTIR spectroscopy are ranging from 4000 to 400 cm⁻¹ which usually measured as wave number. This wavelength range is represented as middle Infrared (Mid-IR) region, which preferred for analysis of organic sample with few micronmeter (μ m) in size [27].

2.3. CO₂ Adsorption Study

The IsoSORP Gravimetry Analyzer was piloted to evaluate the capability of physically-modified kenaf core adsorbent for CO_2 adsorptive studies. Before the adsorption process, the pre-treatment mode was applied to remove the presence of moisture content at about 80°C for 2 hours under vacuum condition. The CO_2 adsorption process was conducted by varying the pressure of bed at 1 bar and 30 bar and the gas flowing rate was varied at 100 ml/min, 300 ml/min and 500 ml/min. The temperature was kept in constant throughout the experimental procedure. Figure 1 illustrates the schematic diagram of adsorption process by using the gravimetry analyzer.



Figure 1. Schematic diagram of the IsoSORP Gravimetry Rubotherm Series for CO₂ adsorption and desorption processes.

3. Results and Discussion

3.1. Morphological Characterization Analysis

Morphological analysis was evaluated through FESEM characterization method to observe the morphology of the physically-modified kenaf core material [8]. The sample was analyzed using the Field Emission Scanning Electron Microscope (FESEM) in the magnification range of 310-330X.



Figure 2. FESEM morphology (a) CKC (b) AKC modified-adsorbent.

Figure 2 (a) presented that the porosity of the CKC is partially developed, while the other portion is destructed. The well-developed porosity is produced as a results of high pressure combustion process during the physical modification step. As shown in figure 2 (b), even though there were few pores form collapsed can be seen, in contrast, the pore structure of AKC is better than CKC. Pore synthesis during the pyrolysis is crucial for increasing the surface area and pore volume of the activated carbon. The activating agent, KOH is the main contributor towards the formation of microporous structure of AKC. Fiuza-jr et al. (2016) reported that the development of these pores opening was attributed during the carbon oxidation step to the synthesis of K₂CO₃, which then degrades into elemental K and CO₂ [14]. KOH is the most commonly used chemical for creating small micropores in the carbon skeleton, whereas K+ ions can be released during carbon processes and inserted into carbon lattices during high temperature treatments, accompanied by carbon oxidation and activation with in-situ produced CO₂ or CO molecules [13]. The carbon matrix lattices expanded due to potassium intercalation between the lattices at high temperatures of KOH activation, resulting in a combination of carbon oxidation and activation with in-situ produced CO₂ as well as formation of a large number of micropores [1, 10]. The morphological analysis assisted in the review of image differences in pore structure of kenaf core after undergoing the physical modification treatment, which resulting in better micropore structure by the activation process with KOH as the activating agent.

3.2. Structural Characterization Analysis

Fourier Transform Infrared Spectroscopy (FTIR) is conducted to evaluate the structural characterization study including the chemical functional group bonds that present, absent, and/or shrinkage for the RKC, CKC and AKC samples. Based on figure 3, the changes of the FTIR absorption band for CKC and AKC over RKC are addressed. It found that RKC presents several absorption peaks compared to CKC and AKC samples. The obvious changes are found at the absorption bands of 1029 cm⁻¹, 2914 cm⁻¹ and 3362 cm⁻¹ where RKC shows several sharp peaks, while disappear for both CKC and AKC. These trends are in line with the previous study that are contributed by the lignocellulosic composition of date seeds and the presence of functional groups of alkene, ester, aromatic, alkanone, alcohol, hydroxyl, ether and carboxyl [8]. The IR band of 3457 cm⁻¹ indicates the presence of hydroxyl group (O-H stretching 3560

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to 3419 cm⁻¹) and it is prevalent in the raw date seeds [8]. While, as pyrolysis occurred the certain absorption bands at 3457 cm⁻¹, 2924 cm⁻¹, and 2854 cm⁻¹ are disappeared that caused by the reduction in moisture content and aliphatic compounds in the date seeds or had less intense infrared peaks due to breakdown of functional groups [8]. It is reported that the peaks show at 2854 and 2932 cm⁻¹ are corresponds to the C–H bond stretching of carboxylic acids while, the new band located at 1026 cm⁻¹ is ascribed to C–O–C stretching that present in ether functional group of KOH-AC [29]. The weak C=O stretching mode is observed at 1566 cm⁻¹ in spectra of all carbon materials. At that particular wavelength (1520 cm⁻¹), it is addressed the C=O aromatic structure stretching vibration in case of activated carbon bamboo adsorbent [11]. Based on this FTIR analysis, it is proven that the physically-modified kenaf core material was successfully prepared before it will further being used in the CO₂ adsorption study.



Figure 3. FTIR spectra analysis of the RKC, CKC and AKC.

3.3. CO₂ Adsorption Capacity Study

The evaluation study for CO₂ adsorption study by RKC, CKC and AKC was investigated by using the gravimetric analyzer with the bed pressure ranging from 1, 5, 10, 15, 20 and 25 bar. For RKC, CKC and AKC, it presents an increasing trend of CO₂ adsorption capacity over increasing trend of operating bed pressure. This result is in line with the previous studies reported on the CO₂ adsorption capacity on the activated carbon adsorbent for CO₂ capture process [1, 4]. At 1 bar, the AKC exhibits the highest amount of CO2 adsorbed with 1.70 mmol/g. However, the CKC achieves about 1.43 mmol/g followed by the least CO₂ adsorption capacity at 1 bar which is the RKC with only 0.28 mmol/g. By comparing the increment percentage of CO₂ adsorption capacity from 1 bar to 25 bar of pressure, it results in the order of RKC (3.07%) > CKC (0.90%) > AKC (0.65%). Despite the fact that RKC adsorbent has a higher CO2 adsorption increment percentage, it is due to lowest amount of CO2 adsorbed at 1 bar as compared to physically-modified samples, CKC and AKC. Unfortunately, the difference in CO₂ adsorption capacity (mmol/g) between AKC and CKC with RKC is widen. The percentage increment of CO₂ adsorption is inversely proportional towards the capability of CO₂ adsorption performance. Based on this study, it is revealed that the AKC showed a better CO₂ adsorption capacity followed by CKC and RKC. Therefore, the physical treatment done on kenaf core support is apparently proven in increasing the CO₂ adsorption capacity.

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Figure 4. The CO₂ adsorption capacity of RKC, CKC and AKC over the bed pressure (bar).

Besides, as compared to the carbonization process, the activation combustion procedure took placed by alternating N₂ with CO₂ for two hours also plays an important role in achieving the excellent result in the modified kenaf adsorbent for CO₂ adsorption study. Ogunbenro *et al.* (2018) had reported that activation with CO₂ causes selective oxidation of particular areas of the date seed sample surface area by facilitating the passage of activating molecules into the pores, hence enhancing the CO₂-carbon reactions and resulting in more pores in the activated carbon [8]. On the surface of the physically activated carbon samples, well-developed and many porosity structures were clearly evident [8]. Furthermore, the trapped N₂ being kept in the micropores and unable to be released due to its strong affinity for N₂ could be related to the reduced pore volume and collapsed some areas of the pore structure.In addition, it could possibly happen due to the present of excessive potassium element that did not involve in the reaction and the removal of inorganic materials that had tendency to cause the pore to clog [1, 2, 9, 30]. The highest CO₂ adsorption capacity by AKC is 2.82 mmol/g at 25 bar. Therefore, AKC had been selected to be further studied on the effect of the gas flowrate (ml/min) for CO₂ adsorption capacity. The gas flow rate was varied at 100 ml/min, 300 ml/min and 500 ml/min and the result of CO₂ adsorption capacity is presented in figure 5.



Figure 5. CO₂ adsorption capacity at different gas flowing rates of AKC (a) 15 bar and (b) 30 bar.

As shown in figure 5 it elucidates that the higher flow rate supplied, the higher CO_2 adsorption capacity achieved at all pressure conditions. Additionally, the highest CO_2 adsorption capacity by AKC is 2.85 mmol/g at 30 bar and 500 ml/min. By increasing the feed flowing rate, the velocity of gas to the

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column increases [20]. At higher gas flowing rate, rapid adsorption occurs; hence denotes to the fast preponderance of adsorbate molecules attracted to the vacant sites of the adsorbent; thus improve the CO_2 adsorption. However, at low gas flowrate, the energy of connections between the CO_2 adsorbates and adsorbent is relatively low indicating a limited CO_2 capture capacity [20]. However, the CO_2 adsorbed by AKC at 100 ml/min gas flowrate condition is similar and slightly better than 300 ml/min, with a mmol/g difference of 0.01-0.04. This could be attributed to a collapsed pore or the fact that the CO_2 molecules were not entirely detached within the confined pore structure of AKC after the desorption treatment was repeated. Based on this study, it is suggested that the higher CO_2 adsorption capacity on AKC could be achieved at the higher gas flowing rate rather than lower flowing rate.

4. Conclusion

This paper revealed that the physically-modified kenaf core adsorbent comprised of two different techniques; carbonization and activation treatment by impregnating KOH, has significant influence on the CO₂ adsorption capacity. Increasing the bed pressure and gas flowing rate would be able to increase the CO₂ adsorption capacity. When compared to CKC and RKC, AKC performs better in terms of physical and structural characterization, as well as CO₂ adsorption studies under various bed pressure and gas flowrate conditions. The greatest amount of CO₂ adsorbed by AKC is 2.85 mmol/g at bed pressure of 30 bar and gas flowrate of 500 ml/min. To summarize, these findings are important in the context of our future plans to set up low-cost activated carbon derived from the abundant Malaysian agricultural waste or biomass.

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