Enhanced carbon dioxide adsorption by potassium hydroxide modified peat soil as porous adsorbent

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Abstract. Carbon dioxide (CO\textsubscript{2}) capture is a strategy established to mitigate issues related to CO\textsubscript{2} emission through various methods, for instance by adsorption. Soil, a natural resource, depending on its particular type are abundant carbon precursor. However, previous studies have neglected the possibility of soil modification for CO\textsubscript{2} adsorption. In this study, a natural soil, peat soil was used to prepare dried, carbonised, and alkaline-modified soils. Through nitrogen adsorption-desorption analysis, the porosity of the alkaline modified soil has higher BET surface area and total pore volume of 72.08 m\textsuperscript{2}/g and 0.11 cm\textsuperscript{3}/g respectively. Furthermore, the functional groups of OH, C=O, and S=O presence in the adsorbents which were determined by FTIR also capable to assist the adsorption of CO\textsubscript{2}. Hence, the performance of the soil after alkaline modification increased by 85% compared to the non-modified soil where the adsorbent recorded an adsorption capacity of 11.70 mg/g. Moreover, the isotherms and kinetics fitting suggest that the adsorption of CO\textsubscript{2} onto the adsorbents was governed by physisorption. In conclusion, modified soils showed better performance for CO\textsubscript{2} adsorption. Thus, further investigations such as optimisation of the modification process or amine functionalisation are anticipated in development of soil-based adsorbents.

1. Introduction

Carbon capture, utilization, and storage (CCUS) is a method introduced to prevent massive accumulation of CO\textsubscript{2} in the atmosphere. Carbon dioxide is a slightly acidic gas which is reported to be the major contributor to greenhouse gases emission. Emission of this gas is either from natural or anthropogenic sources. Natural emission of CO\textsubscript{2} is unavoidable as it is a part of natural process. For instance, a recent catastrophic natural disaster that took place in Australia which destroyed more than half a million of its flora and fauna has emitted about 350 million tonnes of CO\textsubscript{2}. Furthermore, the aggressive and rapid development in many industries such as in food production has led to the gradual increase of CO\textsubscript{2} concentration in the atmosphere [1]. Accumulation of CO\textsubscript{2} has long associated with negative environmental effects such as global warming and climate change. It has been established that carbon capture can be done through several technologies such as absorption, cryogenic separation, and adsorption. Previously, absorption by amine-based solution is the most utilised method, however it has high potential in releasing harmful products. Meanwhile, in cryogenic separation the high cost is the major limitation. Therefore, in recent years, researchers have shown an increased interest in adsorption process by renewable sources due to its ability to achieve high adsorption capacity. Besides that, the use
of renewable precursors as adsorbent which are abundant and cheap could be a great advantage to the environment and economy.

Soils are the uppermost layer of the earth and have been used in several applications such as in construction, bioremediation, and wastewater treatment. For instance, a study has been conducted used dried clay soil in wastewater treatment to remove dye concluded that the soil was effective and serve as low-cost adsorbent [2]. To date, there are only a few studies that have been carried out to investigate the ability of non-modified and modified soils for CO₂ adsorption. A study by Quiroz-Estrada et al. investigated the ability of dried soil to adsorb CO₂. The study reported the highest adsorption capacity of 4.67 mg/g by dried soil with 25 m²/g and 0.033 cm³/g of BET surface area and total pore volume respectively at pressure and temperature approximately at 0.03 bar and 250 °C respectively [3]. The existing literatures on CO₂ adsorption by soils had ignored the possibility that soils are modifiable, hence it can be predicted that the adsorption capacity by modified soils can be enhanced.

In this study, a natural peat soil was collected in Sabak Bernam, Malaysia and used as carbon precursor in development of CO₂ adsorbents with enhanced adsorption capacity. Although peat soil is abundance in Malaysia it is regards as a problematic soil due to high acidity and low in nutrient which make it unsuitable for cultivation. Moreover, it is also has low shear strength and bearing capacity which make it less beneficial in construction [4]. Reported by previous literatures, peat soil has high carbon content of 60% and showed great adsorption capacity for water purification [5, 6]. Precursors with high carbon content is good in the synthesis of activated carbon.

Thus, by considering the availability of the soil, its potential to be modified and then used as CO₂ adsorbent were investigated and compared in this study. There are several modification methods available such as chemical activation. Chemical activation requires activating agents from variety of chemicals such as salts, acid, and alkali. In this study, an alkali solution, potassium hydroxide (KOH) which is widely used in previous literatures to activate biomass or carbon precursors was used to activate the soils. For instance, KOH-activated peanut shell and sunflower seed synthesised in a study recorded a high surface area and pore volume compared to its pristine materials respectively where only few pores can be observed [7]. Therefore, modification of these natural soils with alkaline agent is expected to produce soil-based adsorbent with higher porosity which will contribute to a higher adsorption capacity.

2. Methodology

2.1 Soils collection and samples preparation

In this study, a natural peat soil was used as precursor to investigate its enhanced performance for CO₂ adsorption after each stage of modification. The peat soil was collected at depth of 0.5 m from surface in Sabak Bernam. Three types of samples were prepared from the soil in this study; dried, carbonised, and alkaline modified soils. Initially, the raw soil was dried in an oven at 110 °C overnight to remove moisture and the oven-dried soil was obtained. Subsequently, the dried soil was carbonised at 300 °C for 3 hours in a muffle furnace and then the carbonised soil was obtained. In alkali activation, 30 ml of potassium hydroxide (KOH) with 5M concentration was used to impregnate about 20 g of carbonised soils and placed at 110 °C for 24 hours [8]. Then, the impregnated soil was crushed and washed to neutral pH. All adsorbents included in this study was sieved to particle size of 300-100 µm to ensure the uniformity of the adsorbents and kept in an airtight bottle before being used for CO₂ adsorption study.

2.2 Samples characterisation

2.2.1. Proximate and Ultimate Analysis of the Raw Soil.

Firstly, the moisture and ash content of each soil was determined by using standard ASTM 2974-97. For moisture content calculation based on equation (1), the raw soils were dried in an oven at 110 °C overnight. Then, the ash content was determined by carbonisation of the dried soil at 440 °C for 3 hours and the calculation follows equation (2).

\[
\text{Moisture content, } \% = \frac{(A-B) \times 100}{A} \quad (1)
\]

\[
\text{Ash content, } \% = \frac{C \times 100}{B} \quad (2)
\]
where; A is the initial mass of the soil (g), B is the mass of the oven-dried soil (g) and C is the mass of ash (g).

Meanwhile the ultimate analysis was carried out using the elemental analyser (CHN628 series, USA) to determine the chemical composition of the soil such as carbon, hydrogen, nitrogen, oxygen, and sulphur content.

2.2.2. Nitrogen Adsorption and Desorption Analysis.

Nitrogen adsorption and desorption isotherm was conducted to analyse the physical properties of the prepared adsorbents with the means of a volumetric adsorption instrument (NOVAtouchTM LX3 Surface area & pore size analyzer, USA) with accompanying software (Quantachrome TouchWinTM). Initially, all samples were degassed at 300 °C for 3 hours to remove all gaseous and vapours. Then, the sample was cooled down at room temperature and moved to the analysis port of the instrument for nitrogen adsorption measurement. The instrument records various pressures of gas in the sample cell due to adsorption and desorption. Series of data obtained was used to determine the BET surface area, total pore volume, and the average pore size for each adsorbent. Pore size distribution (PSD) was calculated by the Barrett-Joyner-Halenda (BJH) method, applied to the N₂ adsorption boundary isotherm.

2.2.3. Fourier Transform Infrared Spectroscopy. Surface functional groups of the adsorbents are characterised by FTIR (Perkin Elmer, Spectrum 100, USA). In this study, attenuated total reflection (ATR) measurement was employed. For the measurement, it requires IR source, ATR crystal, and a detector. In the beginning, a small amount of sample was placed on the ATR crystal which has higher refractive index and then the sample was pressed to ensure that the sample had a good contact with the crystal. Then, the IR light was passed through the crystal and internally reflected while some were absorbed by the sample located on top of the crystal. Lastly, the reflected light was collected at a detector which carries the absorption information. Different elements will absorb light at different frequencies, therefore it will have different absorbance wavelength. The resulting signal at the detector is a spectrum representing a molecular ‘fingerprint’ of the adsorbents.

2.3. Carbon dioxide adsorption

2.3.1. Volumetric adsorption system.

The performance of each sample included in this study was determined by using a differential pressure volumetric adsorption system as shown in Figure 1 which consists of two cells; reference cell and sample cell. About 1 g of sample was placed in the sample cell, while the reference cell was left empty and the adsorption was carried out at atmospheric temperature and elevated pressure of 10 bar. During the adsorption process, pressure in the sample cell will drop and the pressure difference between the two cells will be detected in voltage. The voltage is converted to pressure by calibration curve constructed. Then, the adsorption capacity of each adsorbent was determined by using equation (4).

\[
\text{Amount of CO}_2 \text{ adsorbed (n) = } \frac{PV}{RT} \tag{3}
\]

Where; P is the pressure (bar), V is the determined volume (L), R is the gas constant of carbon dioxide (8.314 X 10⁻² L.Bar.K⁻¹.mol⁻¹), and T is the temperature (K).

Later, the adsorption data obtained from the volumetric adsorption system was used to fit with two kinetic models; pseudo-first-order and pseudo-second-order which the equations are as listed in equation (4 & 5).

\[
\text{Pseudo-first-order: } q_t = q_e (1 - e^{-kt}) \tag{4}
\]

\[
\text{Pseudo-second-order: } q_t = k_1 q_e^2 t / 1 + k_2 q_e t \tag{5}
\]

Where; \( q_e \) is the amount of CO₂ adsorbed at equilibrium (mg/g), \( q_t \) is the amount of CO₂ adsorbed at time t (mg/g), and \( k_1 \) & \( k_2 \) (g/mg.min) are the sorption rate constants for pseudo-first-order and pseudo-second-order respectively.
2.3.2. Gravimetric adsorption system.

Adsorption test of carbon dioxide was carried out by using a sorption measuring instrument with magnetic suspension balance (Rubotherm, Germany). This system was applied to carbonised soil only, where it went through three configuration steps; pre-treatment, buoyancy, and adsorption. Initially, each sample underwent pre-treatment and buoyancy configuration to remove moisture from the sample and to measure its actual mass respectively. Then, adsorption test for each sample was carried out at 25 °C and pressure up to 30 bar. Isotherm fitting with three models; Langmuir, Freundlich, and Dubinin-Radushkevich (D-R) isotherms as shown in equation (6-8) was done with the data obtained from gravimetric adsorption system.

Langmuir isotherm: \( C_e / q_e = (1 / K_L q_m) + (C_e / q_m) \)  
Freundlich isotherm: \( \log q_e = (\log K_F) + (1/n \log C_e) \)  
Dubinin-Radushkevich (D-R) isotherm: \( \ln q_e = \ln q_m - \beta \varepsilon^2 \)

Sorption energy: \( E = 1/(\sqrt{2\beta}) \)

Where; \( q_e \) (mg/g) is the equilibrium adsorption capacity, \( C_e \) (mg/L) is the equilibrium concentration, \( q_m \) (mg/g) is the maximum amount of adsorbed carbon dioxide per unit mass, \( K_L \) (L/mg) is the Langmuir constant, \( K_F \) (L/g) and \( 1/n \) are the Freundlich constants, \( \beta \) (mol²/J²) is the D-R constant, \( \varepsilon \) is the Polanyi sorption potential, and \( E \) is the sorption energy (kJ/mol).

3. Results and Discussion

3.1 Proximate and Ultimate Properties of Peat Soil

Proximate and ultimate properties of the raw soil are presented in Table 1. Moisture content of peat soil was about 36%, however, the moisture was effectively removed through drying process. An adsorbent with low moisture content is good as recent studies reported that when the moisture content is high, the adsorption capacity of an adsorbent for gas molecules will be reduced [9, 10]. This negative relationship can be observed because the presence of water molecules which will compete with gas molecules or even block the adsorption sites available. In terms of ash content, peat soil has high ash content that implies a high presence of inorganic matter and minerals which is common in soil [11]. Normally,
material with low ash content and high carbon content is more preferred in development of CO$_2$ adsorbents. Important to note, the carbon content of the collected peat soil was 15.39% is the most crucial component for the synthesis of activated carbon. A study conducted by Uraki et al. which used peat soil as the carbon precursor reported that the peat soil from Kalimantan used has almost 60% carbon content [5]. Similarly, a study conducted by Mudjijati et al. also reported almost similar carbon content for peat soil [6]. The finding shows that even a similar soil from different origin might have different compositions as the materials that made up a soil varies from places to places.

| Soils       | Moisture content (%) | Ash content (%) | Fixed carbon (%) | Nitrogen content (%) | pH   |
|-------------|----------------------|-----------------|------------------|----------------------|------|
| Peat soil   | 35.9                 | 72.85           | 15.39            | 0.5677               | 5.33 |

3.2.1 Nitrogen adsorption and desorption isotherm.

The porosity of the samples included in the study are presented in Table 2. Generally, the each stage of modification of the soil successfully improved the porosity of the adsorbents. Through the analysis, the isotherm of nitrogen adsorption-desorption suggests that it is a typical Type II isotherm which indicates the heterogeneity of the surface and point B labelled in the isotherm graph in Figure 1 (a) indicates that multilayer adsorption was occurred. Furthermore, in the graph also observed a hysteresis loop type H3 for all samples that suggest the nature of slit-like pores which is common for soil particles. The carbonisation and activation process with KOH both are responsible for the improvement. After activation with KOH, the soil has higher BET surface and total pore volume of 72.0846 m$^2$/g and 0.10584 cm$^3$/g respectively. Similar observation was reported in most literatures for instance, alkaline modification with KOH successfully convert the non-porous fungi char with a very low surface area of 2 m$^2$/g to microporous adsorbent with very high surface area and pore volume of 2264 m$^2$/g and 0.92 cm$^3$/g respectively [12]. During carbonisation process, the char become carbon rich by eliminating non-carbon atoms which generates more pores [13]. Meanwhile, development of porosity with KOH is induced by the ability of potassium to intercalate into the carbon network which creates more pores [14]. Improvement in porosity observed was directly enhanced the adsorption capacity for CO$_2$ in this study. The correlation was also found in previous studies, for instance, a study conducted by Creamer, Gao, & Zhang concluded that the adsorption process may be determined by the porous structure of an adsorbent [15].

| Samples | BET surface area (m$^2$/g) | Total pore volume (cm$^3$/g) | Average pore size (nm) |
|---------|---------------------------|------------------------------|------------------------|
| DS      | 11.599                    | 0.03741                      | 6.4507                 |
| CS      | 31.7831                   | 0.0645                       | 4.0591                 |
| KOH-S   | 72.0846                   | 0.10584                      | 2.9367                 |
In contrary, the average pore size was reduced in every stage of modification. Pore development in a solid adsorbent can happened through three stages; opening of previously blocked pores, creation of new pores, and widening of the existing pores during the modifications process [16]. Reduction of the average pore size observed in this study is great as smaller pores are better in adsorption of the small size molecules CO$_2$ with the size of 0.209 nm [17]. Figure 1(b) is the illustration of pore size distribution of the adsorbents by BJH analysis. As seen in the graph, all adsorbents showed multiple modal distributions across the microporous and mesoporous range. Besides that, the intensity for each range of pore size between micropore and mesopore are higher in carbonised and alkaline modified soils subsequently.

Figure 2. a) Nitrogen adsorption-desorption isotherm of a typically Type II isotherm with hysteresis loop type H3, and b) PSD of samples after each stage of modification.

3.2.2. Fourier transform infrared spectroscopy.
The FTIR spectrum shown in Figure 3 covers the wavelength range from 4000 to 1000 cm$^{-1}$ for all adsorbents included in this study. The FTIR spectra of peat showed the OH stretching bands at 3700 to 3000 cm$^{-1}$. This functional group remains presence after modification indicates that it is thermally stable. Oxygen functionalities such as OH group was reported to enhance the adsorption of acidic molecule of CO$_2$ gas [18]. Twin peaks at 2922 and 2850 cm$^{-1}$ are attributed to an asymmetrical and symmetrical stretching of C-H aliphatic hydrocarbons. The disappearance of these bands in CS were observed due to depolymerisation of the material at high temperature. Peak at 1633 cm$^{-1}$ is attributed to C=O bonds in carboxylic acids and this peak remains presence in all adsorbents in this study. Whereas, the presence of sulfonyl group is confirmed by peak at 1396 cm$^{-1}$ and similarly this group remains presence in all adsorbents. These functionalised groups carbonyl (C=O, C=S) and sulfonyl (S=O, S=S) were reported to manifest a higher stability in interaction with CO$_2$ which become valuable factor in term of adsorption of CO$_2$ [19].
3.3 Carbon dioxide adsorption.

3.3.1 Adsorption capacity by volumetric adsorption system and kinetic fitting.

The fundamental of adsorption test with volumetric system is that it is determined by the change of pressure. During the adsorption test, the pressure decreased as more carbon dioxide is adsorbed. In this study, the change in pressure was detected by Hantek device in voltage and then converted to differential pressure using the equation given in the previous section. From the calculation, the adsorption capacity for DS, CS, and KOH-S are presented in Figure 4. The performance of adsorbents in this study gradually increased after each stage of modification. Enhancement recorded by CS in this study was about 25.04%. Meanwhile, KOH-S with higher surface area, pore volume, and smaller average pore size has greater adsorption capacity of 11.70 mg/g which is 85% higher than DS.
In the present investigation, two kinetic models namely pseudo-first order and pseudo-second order were tested for all adsorbents. For all studied adsorbents, the correlation coefficient for the pseudo-second-order are higher compared to correlation coefficient obtained from pseudo-first-order kinetic model. Moreover, there are smaller difference between theoretical and experimental equilibrium adsorption capacity observed for all adsorbents with pseudo-second-order kinetic model as shown in Figure 5. The experimental and modelling results suggest that CO₂ adsorption by soil-based adsorbents were mainly controlled by physisorption. Similar observation was found in a study conducted by Creamer et al. where the CO₂ adsorption by biochar was reported to be best described by pseudo-second-order kinetic model. They further concluded that the adsorption process was governed by physisorption where CO₂ molecules are weakly bound to the surface of biochar [15].

![Figure 5. Adsorption kinetics of CO₂ by soil-based adsorbents.](image)

### 3.3.2 Adsorption capacity by gravimetric adsorption system and isotherm fitting.

The CO₂ adsorption by gravimetric system is determined by the change of adsorbent weight during the process. The adsorption of carbon dioxide by the CS at 25 °C and pressure up until 30 bar by magnetic suspension balance, is presented in Figure 6. Adsorption was considerably fast at low pressure; however it slowed down after about 15 bar, indicating it was approaching equilibrium. The maximum adsorption capacity achieved by CS at 30 bar was 16.4 mg/g. Besides that, the adsorption capacity for the carbonised soils gradually increased as the pressure increased. This is because at higher pressure, the gas molecules are pushed to reside in the pores [20].
Figure 6. Adsorption of CO$_2$ by carbonised soil by gravimetric adsorption system.

The adsorption data obtained by gravimetric system was used to do isotherm models fitting. The models applied were Langmuir, Freundlich, and Dubinin-Radushkevich (D-R) isotherms and the results are presented in Table 3. As can be seen from the table, the Freundlich isotherm model showed excellent fit to the experimental data with high correlation coefficients which indicates the heterogeneity of the adsorbent. Furthermore, the D-R isotherm, an empirical adsorption model generally used to express adsorption mechanism with Gaussian energy distribution onto heterogeneous surfaces. The correlation coefficients for all adsorbents with D-R isotherm was found to be lower than the Langmuir and Freundlich isotherm models as shown in Table 3. However, the constant obtained from the D-R isotherm fitting is useful as it can be used to determine the type of adsorption involved either physisorption or chemisorption. The constant of $\beta$ obtained was used to calculate the sorption energy, $E$ (kJ/mol) using the given equation (8). If the magnitude of $E$ is between 8-16 kJ/mol, the sorption process is suggested to govern by chemisorption, while for values <8 kJ/mol, the sorption process is through physisorption. Particularly for CS, the magnitude of $E$ is 1.290 kJ/mol suggesting that the adsorption mechanism of carbon dioxide by soil-based adsorbents were governed by physisorption.

| Table 3. Isotherm fitting of CO$_2$ adsorption with Langmuir, Freundlich, and Dubinin-Radushkevich (D-R) models. |
|---------------------------------------------------------------|
| Langmuir Isotherm                                              |
| Samples           | Isotherm | $q_m$ (mg/g) | $K_L$ (L/mg) | $R^2$ |
| CS                | Langmuir 1 | 17.8617 | 0.0098 | 0.9929 |
|                   | Langmuir 2 | 15.6057 | 0.0059 | 0.9628 |
|                   | Langmuir 3 | 15.9622 | -0.6425 | 0.8701 |
|                   | Langmuir 4 | -16.3875 | -0.5590 | 0.8701 |
| Freundlich Isotherm                                          |
| Samples           | $K_F$ (L/g) | $n$ | $R^2$ |
| CS                | 6.6500 | 3.6318 | 0.9943 |
| Dubinin-Radushkevich (D-R) Isotherm                          |
| Samples           | $q_m$ (mg/g) | $\beta$ (mol$^2$/J$^2$) | $E$ (kJ/mol) | $R^2$ |
| CS                | 14.30 | 3 x 10$^{-7}$ | 1.290 | 0.8345 |
4. Conclusion
In conclusion, increased porosity after each stage of modification is the key determinant for the enhanced adsorption capacity achieved in this study. Modified KOH recorded the highest surface area and pore volume of 72.0846 m²/g and 0.1058 cm³/g respectively. Moreover, development of micropores and mesopores are observed through the modification process. Furthermore, the presence of hydroxyl, carbonyl, and sulfonil functional groups presence in all adsorbents are able to assist CO₂ adsorption. Thus, enhanced performance was achieved by KOH-S with adsorption capacity of 11.70 mg/g which is 85% higher than the non-modified DS. Both kinetic and isotherm models fitting suggest that the adsorption of CO₂ by soil-based adsorbents was governed by physisorption. Future research and development can be directed to the optimization of the modification process where greater physical and chemical properties of soil-based adsorbents are possible.

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