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# Characteristics and carbon dioxide adsorption performance of candle soot-activated by potassium hydroxide

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**Abstract.** Incomplete combustion by fossil fuel has contributed to the increase in carbon dioxide (CO<sub>2</sub>) emission as well as carbon soot production. Several methods have been done via carbon capture and storage (CCS) strategy to mitigate CO<sub>2</sub> emission such as via adsorption. However, a method to utilize the carbon soot production was still in question. Less discussion on utilizing soot as gas adsorbent up to this date. Therefore, this paper aimed to investigate the CO<sub>2</sub> adsorption performance from fresh candle soot (FS) and soot activated by potassium hydroxide (KOH) with mass ratio 2/1 KOH to soot (KOH-S1). The activation process was done by using muffle furnace at 700 °C for 1 h under vacuum condition. FTIR spectrum of the KOH-S1 appeared at 3500, 1639, 1048 and 1740 cm<sup>-1</sup> due to KOH treatment before activation. The mean diameter for KOH-S1 particles was larger (22.328 μm) than FS (4.413 μm) due to agglomeration. The adsorption capacity obtained for KOH-S1 and FS were 24.4092 and 10.3052 mg/g respectively. It has been demonstrated that KOH-S1 has excellent potential to be used for CO<sub>2</sub> adsorption and more studies need to be conducted for its development.

## 1. Introduction

In this day and age, the emission of the greenhouse gases mainly carbon dioxide (CO<sub>2</sub>) is rising at alarming rate and generate many serious environmental issues such as global warming and climate change. The major cause of CO<sub>2</sub> emission in the atmosphere is human activities such as combustion of fossil fuel, agriculture activities, and industries. To reduce the amount of CO<sub>2</sub> released into atmosphere, there are four main methods are being developed for CO<sub>2</sub> capture and separation (CCS) such as absorption, adsorption, cryogenics and membranes. Among these methods, adsorption with solid adsorbents is one of the most promising options due to its low energy consumption, low equipment costs and easiness for application [1]. Solid adsorbents from carbonaceous materials received more attraction among the researchers as it can be derived from any carbon precursors such as graphene, graphene oxide carbon nanotube and activated carbon from waste or biomass materials which are cost-effective and scalable route for carbon production.

Recently, considering the carbon soot production, researchers have come out with the solutions by utilizing it in various applications such as activated carbon. Soot or known as organic pollutant produced from incomplete combustion either from fossil fuels or biomass materials. Candle soot is generated through burning a paraffin wax, contains high purity of carbon. Candle soot widely used in energy storage application because it has porous properties and high surface area. Potphode and Sharma [2] treated candle soot at high temperature (450°C) to increase its purity and improve its surface area (608 m<sup>2</sup>/g) for charge storage in supercapacitors. Beside, candle soot also been explored in oil-water separation [3, 4], organic pollutants removal [5] and fluorescent carbon nanoparticles [6]. Subjected to



CO<sub>2</sub> adsorption, the porosity and specific surface area (SSA) also play an essential role to enhance the adsorption capacity. There has been little discussion about the uses of candle soot as gas adsorbent. Candle soot contains 92 % of carbon content [7] which is good for carbon precursor of activated carbon. By applying either physical or chemical activation, the SSA and pore volume of the candle soot can be improved. As compared to physical, chemical activation has showing the excellent performance in develop well-controlled porosity and high SSA by previous literature.

Among the chemical agents, KOH can create the largest volume of micropore less than 1 nm which is good for CO<sub>2</sub> adsorption as it depends on the volume of this micropore [8]. KOH activator showed a higher CO<sub>2</sub> uptake by the previous literature. Martín-Jimeno and his co-workers [9] reported KOH-activated carbon xerogels that adsorbed 4.9 mmol/g of CO<sub>2</sub> (0 °C, 1 bar). Besides, Yang and his team [10] performed KOH activation of the carbons prepared from polymers and achieved CO<sub>2</sub> uptakes of 6.5 mmol/g (0 °C and 1.13 bar). Hence, the aim of this paper is to investigate the CO<sub>2</sub> adsorption performance using KOH-activated candle soot. The candle soot will be impregnated with KOH and activated at high temperature, where its characteristics after activation will be compared with a fresh soot. Afterwards, these samples will be subjected to CO<sub>2</sub> adsorption test, from which the adsorption capacity will be evaluated.

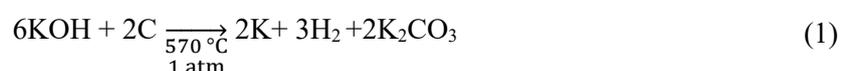
## 2. Methodology

### 2.1. Materials

The candle soot was synthesized from the inner flame of paraffin wax candle by using direct-deposition method as reported by Faizal and his co-workers [11]. The deposited soot on the substrate was scratched to collect the soot particles. The process was done repeatedly until a desired weight of the collected soot was obtained. Other reagents: Potassium hydroxide (KOH) pellets MW=56.11 (Sigma Aldrich, US), ethanol (99.5%, System, Malaysia) and hydrochloric acid (HCl) (37%, System, Malaysia) were used without further purification.

### 2.2. Preparation of Candle Soot by KOH activation

The collected fresh soot (FS) was washed with ethanol and deionized water at ratio 1:1 under sonication for 20 minutes at 80 kHz and 100 W until well dispersed. This step as done by Raj *et al.* [12] was to remove the impurities and unburned hydrocarbons of the candle soot. The candle soot in a suspension was filtered and dried overnight at 110 °C to remove the moisture completely from the carbon soot. The dried soot was soaked in KOH solution of 10% concentration with mass ratios of KOH/soot 2/1 (KOH-S1) and then, the resulted slurries was heated at 110 °C for 24 h [13]. The activation process was done using muffle furnace at activation temperature 700 °C for 1 hour under vacuum condition. The activation of soot possibly started with the reaction at 570 °C as in equation (1), then further proceeds through reaction of metallic K with KOH to K<sub>2</sub>O [14].



According to Wang and Kaskel [15], at about 600 °C, KOH was completely consumed. Formation of K<sub>2</sub>CO<sub>3</sub> decompose into CO<sub>2</sub> and K<sub>2</sub>O at temperature higher than 700 °C and completely disappears at approximately at 800 °C. The resulting CO<sub>2</sub> can be further reduced by carbon to form CO at high temperature. At over 700 °C, K<sub>2</sub>O and K<sub>2</sub>CO<sub>3</sub> could be reduced to produce metallic K which efficiently intercalating into carbon lattices of the carbon matrix during activation to form the micropore. The intercalated metallic K and other K compounds were removed by washing with 1 M HCl solution and deionized water until neutral pH was achieved. Lastly, the sample was dried at 110 °C for 24 h to remove the moisture.

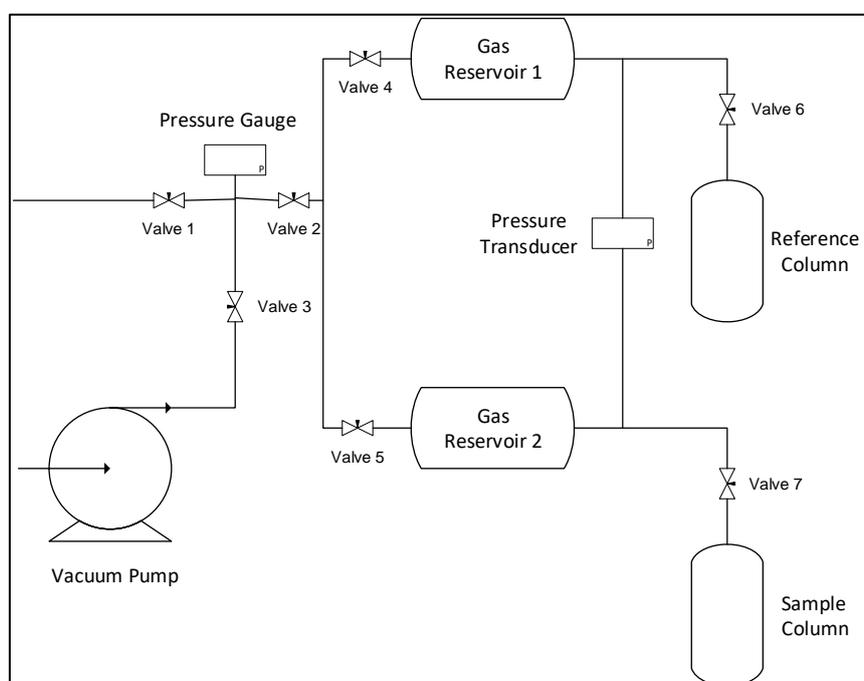
### 2.3. Characteristics Analysis

**2.3.1. Fourier-Transform Infrared Spectroscopy (FTIR) Analysis.** FTIR spectroscopy analysis was carried out to determine the functional groups present in the soot particles before and after KOH activation. This was done using IRT Tracer-100 (Shimadzu, Japan) at wavelength  $400\text{--}4000\text{ cm}^{-1}$  under atmospheric condition. The atmospheric correction has been done for each sample to remove the noise that might affect the reading.

**2.3.2. Particle Size Analysis.** The particle size distribution of the soot was determined by using Laser Diffraction Particle Size Analyzer (SALD 2300, Shimadzu, Japan). The soot suspension with 0.01% was prepared by mixing 0.1 g of soot with 100 ml of water using ultrasonic sonicator prior to sample analysis. The refractive index used for soot was 1.55-0.50i and logarithmic gaussian function was applied to determine the particle distribution.

#### 2.4. Carbon Dioxide Adsorption Capacity Test

The carbon dioxide adsorption capacity was analyzed using differential pressure (volumetric) adsorption system (Figure 1) for determining equilibrium gas-solid adsorption isotherms. As adsorption occurs at the gas-solid interface, the weight of solid increases and the pressure of the gas decreases. The pressure difference two columns are considered as  $\text{CO}_2$  adsorbed. For  $\text{CO}_2$  adsorption process, the sample was placed inside the sample column. Prior to that, the pressure inside the column was equalized using vacuum pump. The reservoirs were filled with  $\text{CO}_2$  at 10 bar for 15 minutes before letting the gas in contact with the sorbent for 30 minutes. For baseline correction, after equalizing the pressure in the columns and filling the reservoirs with nitrogen gas ( $\text{N}_2$ ) at 10 bar, the gas was let in contact with the sorbent for 15 minutes. Both process condition was in the room temperature ( $25\text{ }^\circ\text{C}$ ). The calculation of the adsorption capacity ( $Q$ ) in unit  $\text{mg/g}$  will be done in excel spreadsheet and the graph of  $\text{mg/g}$  versus time will be constructed.

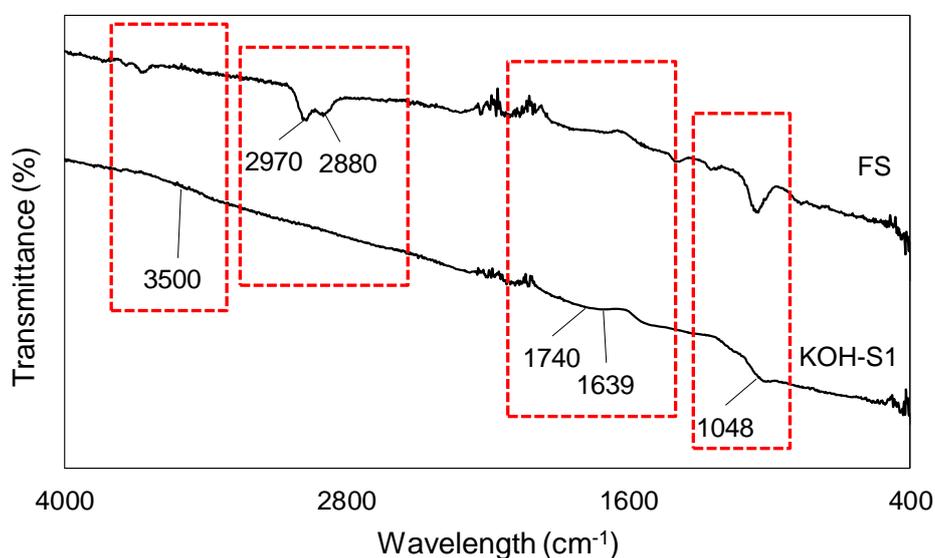


**Figure 1.** The schematic diagram of the differential pressure (volumetric) adsorption system.

### 3. Results and Discussion

### 3.1. Formation of functional group after KOH activation

FTIR spectra of FS and KOH-S1 samples were shown in Figure 2. The FTIR spectrum of FS only shows the presence of CH<sub>2</sub> and CH<sub>3</sub> asymmetric vibrations at peak 2970 and 2880 cm<sup>-1</sup> respectively indicating the combustion of diolefin in candle soot was partially completed [16]. KOH treatment resulted in the formation of different functional groups such as hydroxyl group, carbonyl group and carboxyl group on the carbon surface [17]. FTIR spectrum of the KOH-S1 slightly appeared at 3500, 1639, 1048 and 1740 cm<sup>-1</sup>. The absorption peak at 3500 cm<sup>-1</sup> corresponds to O-H bond stretching vibration of water molecules which absorbed onto the carbon matrices. C=C bond stretching vibration in the aromatic ring resulted in an absorption peak at 1639 cm<sup>-1</sup> and the absorption peak at 1048 cm<sup>-1</sup> was attributed to the stretching of C-O bond. The peak at 1740 cm<sup>-1</sup> correspond to C=O stretching vibration associated with the presence of carboxyl group. All the peaks observed in KOH-S1 indicated that KOH treatment has preserved these O-containing groups from fresh soot which will enhance the adsorption capacity [17].



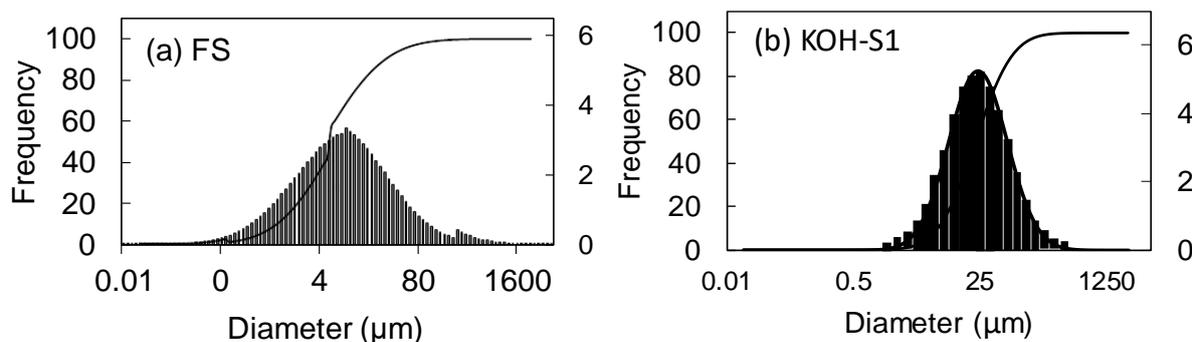
**Figure 2.** FTIR analysis for fresh soot (FS) and KOH-S1.

### 3.2 Size Distribution of Candle Soot Particles

The laser diffraction technique has been performed to measure the particle size distribution of the fresh soot and KOH-S1. Based on the result illustrated in Figure 3, size distributions of KOH-S1 (Figure 3 (b)) has narrower distribution compared to fresh soot (Figure 3 (a)). The mean diameter for both samples obtained were 22.328  $\mu\text{m}$  and 4.413  $\mu\text{m}$  respectively as showed in Table 1. The particle size for FS sample was bigger than reported previously [11]. This might be caused by the aggregation due to strongly bonded particles and caused it to agglomerate. For this study, inner flame or known as yellow region has been chose for particles collection. Inner flame was characterized as superhydrophobic and large agglomerates with particle diameter size 20-55 nm as reported by Mulay and his team [7]. When hydrophobic particles are suspended in water, it can cause expansion and breaking of the hydrogen bonds between water particles. In order to sustain the hydrophobic soot particle dispersion in the water, longer time is needed to sonicate the soot suspension. Thus, the result obtained for fresh soot can be considered as large agglomerates as it might be the short of sonication time which caused the particles were not well-dispersed in water suspension.

In case for KOH-S1 sample, the size of particle should be smaller than FS due to reduction size by intercalated metallic K and K compound in the carbon lattice [15]. According to Kocjan and his co-workers [18], the size reduction can promotes agglomeration. Agglomeration was taking place in order to reduce surface free energy. Higher agglomeration indicated the particle has high surface area and

small pore size. The narrow particle distribution obtained by KOH-S1 sample can be indicated that hydroxide activation can promote the agglomeration which caused the diameter size become bigger. The authors believe that the activation process has create some new pores on the soot surface which increase its surface area. As CO<sub>2</sub> adsorption can be enhanced by high surface area and pore volume of the adsorbent, it is important to consider KOH-S1 as one of the potential adsorbents for CO<sub>2</sub> adsorption.



**Figure 3.** Particle size distribution of (a) fresh soot (FS) (b) KOH-S1 suspension in water.

**Table 1.** The data of the particle size of each sample.

Sample	Mean (μm)	Median (μm)	Std dev
Fresh soot (FS)	4.413	4.413	0.644
KOH-S1	22.328	22.329	0.394

### 3.3 Adsorption capacity of candle soot

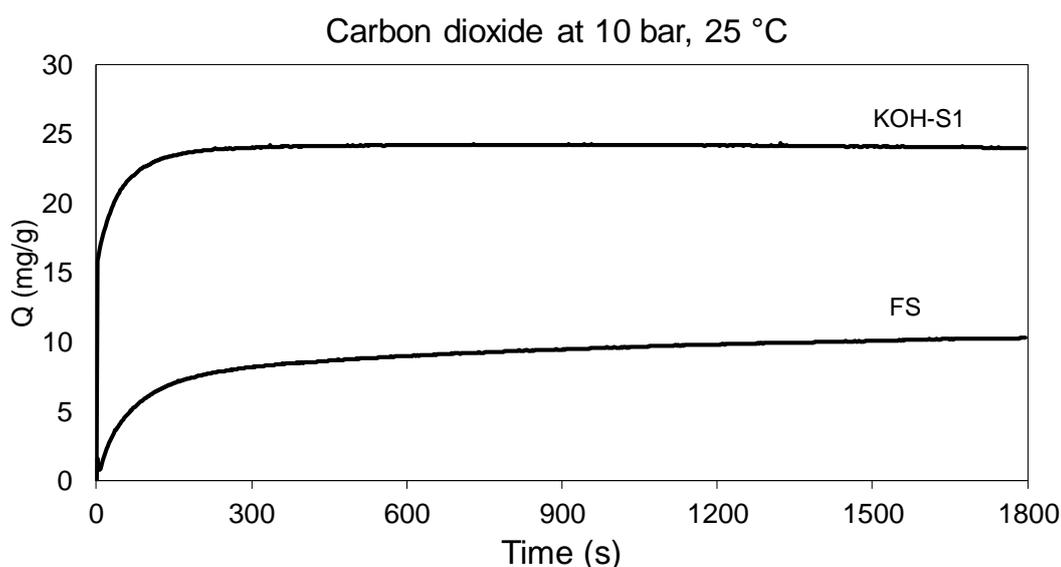
Figure 4 shows the CO<sub>2</sub> adsorption isotherm of fresh soot (FS) and candle soot-activated of KOH (KOH-S1) at 10 bar and 25°C. This adsorption test was carried out to investigate the effect of the adsorbent from candle soot before and after KOH activation on the CO<sub>2</sub> adsorption performance. From Figure 4, KOH-S1 sample recorded a higher adsorption capacity with 24.4092 mg/g as compared to FS sample which is 10.3052 mg/g (Table 2). This result indicated that the candle soot-activation of KOH give a significance effect to the adsorption capacity.

High adsorption capacity shown by KOH-S1 was possibly due to the effective micropore developed from intercalating metallic K and K compound at 700 °C as stated by Wang and Kaskel [15]. Based on literature, activation by KOH can create the largest volume of micropore less than 1 nm which can enhance the CO<sub>2</sub> adsorption [8]. The higher agglomeration showed in KOH-S1 particles can indicate the particle has high in surface area to the volume ratio that caused by high surface energy to be minimized. In addition, this physical property of the carbon soot should have to look insight its specific surface area and the pore volume which also plays essential factors in giving value to the adsorption capacity.

The ratio of KOH to the soot also need to be considered as it can promote the production of metallic K and K compounds to soot particles. If the ratio is too high, it might cause the excessive potassium ions (K<sup>+</sup>) presence which lead to deformation of the soot surface as reported by Musa *et al.* [19]. The excessive of K<sup>+</sup> will open up more pore which can distort the carbon surface by interconnecting through pores which can hinder the CO<sub>2</sub> adsorption. Thus, the ratio of KOH/soot should be one of the factors that need to be investigated for our future work in order to control the pore formation which lead to higher adsorption capacity. In addition, the adsorption kinetic should be studied to provide the crucial study about mechanism of sorption, optimum conditions and possible rate controlling step.

**Table 2.** The CO<sub>2</sub> adsorption capacity of the fresh soot (FS) and KOH-S1 samples at 10 bar, 25 °C.

Sample	Adsorption capacity (mg/g)
FS	10.3052
KOH-S1	24.4092



**Figure 4.** CO<sub>2</sub> adsorption capacity against time of fresh soot and KOH-S1 at 10 bar, 25 °C.

#### 4. Conclusion

In summary, the candle soot was activated by KOH at 700 °C for the first time. The physical and chemical properties of the candle soot-activation of KOH was studied to evaluate the performance in CO<sub>2</sub> adsorption. The KOH activation was showed the formation of peaks at 3500, 1639, 1048 and 1740 cm<sup>-1</sup> due to KOH treatment on the candle soot. The fresh soot particle was also increase in size after KOH activation due to large agglomeration which indicated high in surface area to the volume ratio. The CO<sub>2</sub> adsorption test was proved that KOH-S1 can obtain higher adsorption capacity (24.4092 mg/g) than fresh soot (10.3052 mg/g) by 47 % which have proven the improved in characteristic of fresh soot by KOH activation. This result gives an opportunity to the authors in utilizing the KOH-activated soot as CO<sub>2</sub> adsorbent for the future work.

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