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Surface Area Enhancement of Modified-Candle Soot using Ethanol Treatment for Carbon Dioxide Adsorption

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ABSTRACT

Carbon dioxide (CO_2) is one of the greenhouse gasses that has various effects to the earth atmosphere. In recent years, carbon-based adsorbent used for CO_2 adsorption. To increase the adsorption of CO_2, the surface treatment should affect the adsorption sites. However, the mechanism of the ethanol surface treatment was lack of study. Hence in this study, we investigate the effects of ethanol treatment on candle soot surface and adsorption performance. Briefly, the raw soot (FS) was collected directly from the inner flame. The collected soot was treated with aqueous ethanol and labelled as washed soot (WS). Both FS and WS were studied for the significant effect on its physical, chemical properties and adsorption capacities. FS and WS showed the agglomeration of particles (mean diameter > 1). FS washed with ethanol significantly increased total pore volume (from 0.18 to 0.65 cm^3/g) with almost the same surface area (from 111.01 to 113.54 m^2/g). The CO_2 adsorption capacity was revealed that the maximum adsorption capacity obtained by FS was 68 mg/g and slightly increased after the pre-treatment (69 mg/g). Meanwhile, Freundlich isotherm model fitted to the experimental data based on basis of the regression coefficient (R^2) which are 0.979 (FS) and 0.993 (WS). Based on the isotherm, after ethanol treatment particles suggesting heterogeneous surface with large number of CO_2 adsorption sites compared to non-treated soot.

Keywords:
Soot; adsorbent; CO_2 capture; isotherm; surface modification; adsorption capacity; Van der Walls forces; cavitation bubble

1. Introduction

The excessive amount of carbon dioxide (CO_2) emission and atmospheric aerosol has led to the increment of the earth's temperature which causes global warming and climate change. The method for later has been extensively developed based on monitoring [1,2] as compared to a shortcoming of

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CO_2 issues. The major causes of CO_2 emission in the atmosphere are human activities such as the combustion of fossil fuel, agriculture activities, and industries [3]. To mitigate these issues, carbon capture and storage (CCS) technology has been developed. There have three different routes in CO_2 capture which are oxy-fuel combustion, pre-combustion, and post-combustion. Extensively, the post-combustion route has been employed by industries due to easiness in retrofitting and low capital cost [4]. There are various methods in post-combustion such as absorption, adsorption, cryogenics, and membranes. Presently, a conventional method for post-combustion is via an amine scrubbing system, but due to numerous side effects, an alternative way should be pursued. Solid adsorption has been proposed to overcome the aforementioned limitations. It is one of the most promising options due to its low energy consumption, low equipment costs, and easiness of application [5].

Solid adsorbents from carbonaceous materials received more attraction among the researchers as they 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, high CO_2 uptake, and scalable root for carbon production [6,7]. Recently, considering the excessive carbon soot production, researchers have come out with solutions by utilizing it in various applications such as activated carbon [8,9]. Soot or known as an organic pollutant is produced from incomplete combustion of any carbon-based materials such as fossil fuels, woods, plants, wax, etc. Surprisingly, soot generated from paraffin wax candles contains high purity of carbon than other carbon soot sources [10]. It is made of heavy straight-chain hydrocarbons obtained from crude petroleum oil [11,12]. They are solid and crystalline mixtures of hydrocarbons consisting of linear n-alkane and branched iso- and cyclo-alkane with carbon ranging from C_{16}-C_{30} and higher [13]. As it contains low inorganic materials, candle soot is widely used in energy storage applications because of its porous properties and high surface area. For instance, Potphode and Sharma [14] have treated the candle soot at a high temperature (450 °C) to increase its purity and improve its surface area (608 m^2/g) for charge storage in supercapacitors. Besides, candle soot has also been explored in oil-water separation [15,16], organic pollutants removal [17], and fluorescent carbon nanoparticles [18].

The porosity and specific surface area (SSA) are subjected to CO_2 adsorption also play an essential role to enhance the adsorption capacity. To date, there has been little discussion about the uses of candle soot as a gas adsorbent. The candle soot was utilized as a CO_2 adsorbent because it contains high carbon content, high availability, and low-cost material. In this present work, the soot particles were collected from the inner flame of a candle which is superhydrophobic properties. The hydrophobic adsorbent can give a better adsorption capacity as it provides much more adsorption sites for adsorbate [19]. However, it will hinder the process of surface modification (e.g. impregnation). Therefore, in this work, the candle soot collected undergo the treatment with aqueous ethanol as the interaction between volatile organic solvents (e.g. alcohol) with carbon-based nanomaterials will alter the surface wettability of carbon soot [20]. The immersion of carbon soot into the organic solvent will increase the number of oxygen functional groups (hydrophilic active sites) on the surface. To the best of our knowledge, the study of surface area by modified soot with ethanol treatment is still lacking in the literature. Thus, in this study, we aim to investigate the effect of ethanol treatment on the CO_2 adsorption performance of candle soot. To further understand the candle soot's before and after the ethanol treatment, the physicochemical characteristics also evaluated. Afterward, these samples will be subjected to a CO_2 adsorption test, from which the adsorption capacity will be analysed.

2. Methodology

2.1 Synthesis of Candle Soot

The paraffin candles (1280, Jelita, Malaysia) used in this study were used without further purification as the source materials for carbon soot. The candle soot was synthesized from the inner flame as the setup in Figure 1(a) by using a direct-deposition method as reported by previous research [21]. Briefly in Figure 1(a) showed the synthesis pf candle soot nanoparticles as adsorbent. The methodology for synthesizing encompassed two primary phases: collection for candle soot and pre-treatment phases. As shown in Figure 1(a), for the collection phase, soot was produced through the combustion of a candle with the resultant particles being gathered on ceramic-based substrate measuring 5 cm in diameter, positioned on inner flame of candle. The deposited soot on the ceramic-based substrate was scratched to collect the soot particles. The process was done repeatedly until a desired weight of the collected soot was obtained. The gathered soot designed as fresh soot (FS), subsequently underwent a pre-treatment procedure using ethanol. In this study, ethanol (C_2H_6O, 99.8%,) was purchased from Systerm, Malaysia, and deionized water was used without further purification.

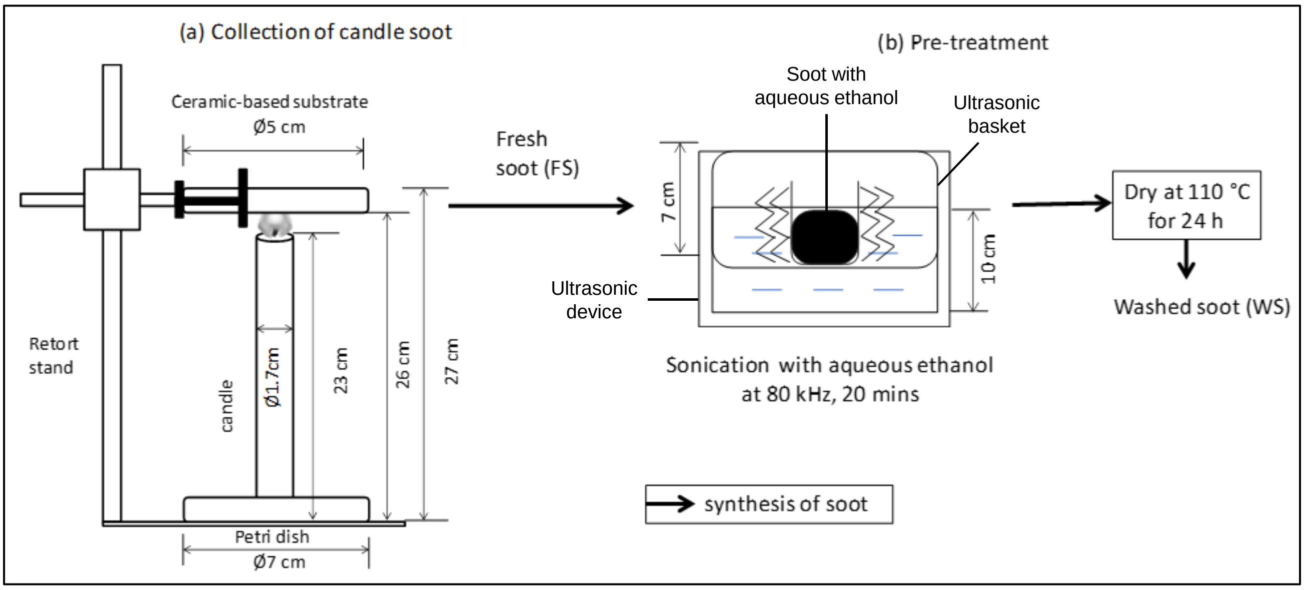

Fig. 1. Synthesis of candle soot (a) Candle soot was collected from the inner flame by direct burning method (b) The pre-treatment of soot by sonication with aqueous ethanol

2.2 Ethanol Chemical Surface Modification of Soot

In the pre-treatment phase (Figure 1(b)), the collected fresh soot (FS) was washed with ethanol and deionized water at a ratio of 1:1 under sonication for 20 minutes at 80 kHz and 100 W until well dispersed (Figure 1(b)). This step as done by previous work [22] was originally to detach the candle soot particle from the substrates. This sonication techniques facilitates the disintegration of larger soot aggregates and expected to remove the impurities and unburned hydrocarbons of the candle soot. This process also ensures the soot maintained the structural integrity of carbon-based materials. Thereafter, the candle soot in a suspension was filtered and dried via oven overnight at 110 ± 2 ℃ under vacuum condition until the weight remained constant. The dried sample was labelled as washed soot (WS) and used for CO_2 adsorption and further analysis.

2.3 Physicochemical Properties

To study the morphological changes of the fresh soot (FS) and washed soot (WS) were identified through an ultimate analysis using CHN/S analysis (TruSpec, LECO, Germany). Next, the surface morphology washed soot (WS) was characterized by Field Emission Scanning Electron Microscopy, FESEM (JSM-7800F, JEOL Ltd., Japan). Prior to analysis, the soot sample was coated with platinum (Pt) with a thickness of ~2.4 nm. The surface morphology was observed at magnification of 10 000 – 100 000x with 100 nm resolution and performed at 5 kV.

To investigate, 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 deionized water using an ultrasonic sonicator before sample analysis. The refractive index used for soot was 1.55-0.50i and the logarithmic Gaussian function was applied to determine the particle distribution.

Next, the surface area analysis was carried out using nitrogen (N_2) adsorption-desorption analysis using Brunauer-Emmet-Teller (BET) surface area equipment (Quantachrome instrument, Nov Atouch, USA). Before the measurements, about 0.15 g of sample was degassed at 250 °C for 3 hours to remove the impurities. Data obtained from the adsorption-desorption isotherm was used to find BET surface area. The surface area was calculated using the BET equation over a relative pressure range of 0.05-0.35. Total pore volume was obtained from the distribution curves of pore volume by Barrett-Joyner-Halenda (BJH) method. Finally, the Fourier Transform Infrared Spectroscopy, FTIR for spectroscopy analysis was carried out to determine the functional groups present in the soot particles. This was done using IRT Tracer-100 (FTIR, Shimadzu, Japan) at wavelength 400-4000 cm^{-1} under atmospheric conditions. The atmospheric correction has been done for each sample to remove the noise that might affect the reading.

2.4 Carbon Dioxide Adsorption Capacity Measurement

The adsorbent performance for CO_2 adsorption was measured using Sorption Measuring Instrument with Magnetic Suspension Balance system by (MSB, Rubotherm, Germany). Magnetic suspension balance was used to measure CO_2 adsorption in various materials [23], including carbon-based materials due to its reliability in the evaluation. The sample was loaded into the magnetic suspension balance at 80 °C in vacuum conditions until its weight stabilizes to remove adsorbed ambient moisture. The buoyancy measurement of the sample was carried out at 25 °C and 30 bars to determine the weight and volume of the sample. For the CO_2 adsorption performances, the test was conducted at 500 mL min^{-1} of 100% CO_2 gas flow, at 25 °C temperature until maximum pressure of 30 bar is reached with 5 bar per step. The adsorption capacity was determined by the difference in the sample's weight and measured in mg/g.

2.5 Adsorption Isotherm Models

The experimental data of adsorption was studied by applying it to the isotherm models such as Langmuir, Freundlich, and Dubinin Radushkevich (D-R) [24]. The non-linear and linearized equations of these isotherm models are summarized in Table 1. Where P_e (bar) represents equilibrium pressure, q_e (mg/g) is the total of CO_2 adsorbed, q_m (mg/g) is the maximum CO_2 adsorption capacity adsorbed and K_L is the Langmuir constant (1/bar), K_F ($\text{mg/g.bar}^{1/n}$) and n are the Freundlich constant, β (mol^2/J^2) is D-R constant, ε (J/mol) is the Polanyi potential which equivalent to $RT \ln (1/1+P_e)$ and E is adsorption energy which equal to $1/\sqrt{2B}$. The correlation coefficient (R^2) is to evaluate the validity of these models. The best fitting towards the isotherm model is determined by R^2 closest to one.

Table 1
Adsorption isotherm models and correlation coefficient equation

Isotherm	Non-linear equation	Linear equation	Equation
Langmuir	$Q_e = \frac{Q_m K_L P_e}{1 + K_L P_e}$	$\frac{P_e}{Q_e} = \frac{1}{Q_m} P_e + \frac{1}{K_L Q_m}$	(1)
Freundlich	$Q_e = K_F P^{1/n}$	$\log Q_e = \log K_F + \left(\frac{1}{n}\right) \log P_e$	(2)
D-R	$Q_e = Q_m e^{-\beta \varepsilon^2}$	$\ln Q_e = \ln Q_m + \beta \varepsilon^2$	(3)
Correlation coefficient			
$R^2 = \frac{\sum(Q_m - \overline{Q_e})}{[\sum(Q_m - \overline{Q_e}) - (Q_m - Q_e)\,]^2}$			(4)

3. Results

3.1 Elemental Analysis of Materials

The physical and chemical composition of the fresh soot (FS) and washed soot (WS) samples were summarized in Table 2. The carbon contents obtained by FS and WS were at 93.91% and 67.52% respectively. These samples were remarkably high in carbon content as compared to biomass materials which typically range between 28-47% [25,26]. High carbon content is favourable for the CO_2 adsorption process due to van der Waals interaction forces [27]. The carbon content for the FS sample was significantly high as compared to the previous study by Liang *et al.,* [28]. However, after treatment with aqueous ethanol, the carbon content was reduced by 28% (from 93.91% to 67.52%) due to the volatility effect. As soot is alkane-based, it is easily volatile after ethanol treatment results in a decrease in carbon content. Similar results were obtained by Liang *et al.,* [28], where they found that the carbon content of soot decreased after washing with alcohol. The carbon associated (H, N, and O) obtained by both samples were in adequate amounts.

Table 2
Elemental and physical properties of fresh soot (FS) and washed soot (WS)

Elemental composition (%)	FS	WS
Carbon	93.91	67.52
Hydrogen	0.81	0.88
Nitrogen	0.17	0.08
Oxygen (by difference)	5.49	31.52
Characteristics		
BET surface area (m^2/g)	111.01	113.54
Pore volume (cm^3/g)	0.18	0.65
Average pore size (nm)	1.66	8.28
Particle mean diameter (µm)	4.41	6.76

3.2 Physicochemical Properties

The physical properties of the fresh soot and wash soot was evaluated using the FESEM. The surface morphology of the soot was showed by FESEM images for WS as depicted in Figure 2. It showed an agglomeration in size diameter which was more than 1 µm (Figure 2(a)). The shape of WS particle as shown in Figure 2(b) displayed clustering in sphere shape in form of closely packed aggregates which is consistent with previous work by Raj *et al.,* [29]. It also showed the carbon structure consisted of some intraparticle pores that can facilitate the transport process of the CO_2 molecules towards the inner side of the porous carbon.

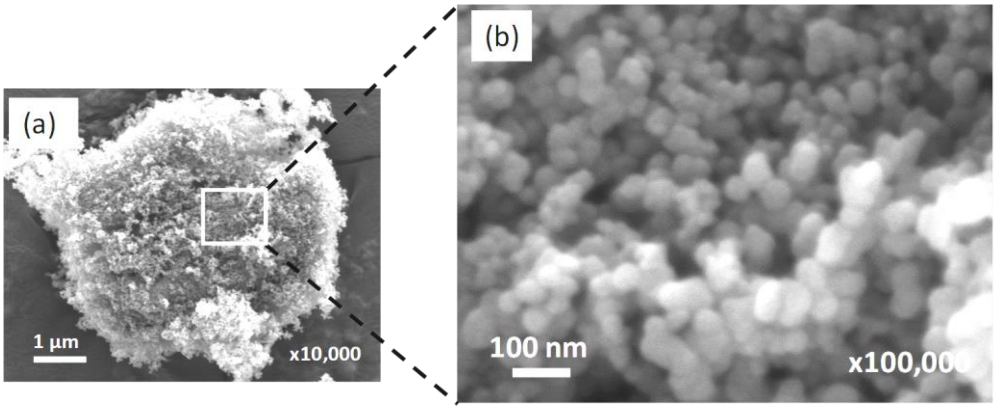

Fig. 2. FESEM of ethanol treated washed soot at (a) Magnification: X10,000 (b) Magnification: X100,000

To further investigate the effects of the ethanol on the surface agglomeration and surface defects in the candle soot before and after treatment, laser diffraction technique was done. The particle size distribution for both samples was obtained by laser diffraction technique, displayed the diameter size within range 0.1 to 1000 µm as illustrated in Figure 3. The average diameter measured for FS and WS samples were 4.41 µm and 6.76 µm respectively as presented in Table 2, which indicating the agglomeration of particles. It was correlated with the FESEM image in Figure 2(a) for the WS sample which was more than 1 µm. The diameter of FS is which is bigger than reported previously [21]. This might be caused by unstable colloidal dispersion in the water suspension due to the aggregation of strongly bonded particles and caused it to agglomerate [30].

For this study, the inner flame or known as a yellow region has been chosen for particles collection. The inner flame was characterized as superhydrophobic and large agglomerates with particle diameter size 20-55 nm as reported in [10]. For the WS sample, the size particles are bigger than FS. According to previous study [30], size reduction can promote agglomeration. Agglomeration was taking place to reduce surface free energy. This study found higher agglomeration indicated the particle has a high surface area which is supported by BET surface area analysis. The washed soot also showed increased in the surface area, which indicates the defects on the surface of washed soot exposed after the removal of the impurities on the surface.

The BET surface area (SA) and pore size distribution of the samples were retrieved from the N2 adsorption/desorption isotherm method is presented in Figure 4. Figure 4(a) displayed the isotherms obtained by fresh soot (FS) and washed soot (WS) represented type IV isotherm which is given by mesoporous adsorbents with hysteresis loop H2. Both samples exhibit initial monolayer-multilayer adsorption on the mesopore walls and are followed with pore condensation. The capillary condensation has occurred at high relative pressure where there was pore-blocking between the primary particles [31]. The absorption volumes increase when the relative pressure was higher than 0.8, indicating the presence of mesoporous particles with few micropores in both samples. The BET surface area and the corresponding pore volume of FS and WS samples were displayed in Table 2. The surface area and pore volume obtained by FS was 111.01 m2/g and 0.18 cm3/g while for WS were 113 m2/g and 0.6 cm3/g respectively. After washing with aqueous ethanol, the total pore volume increased by 2.6%, and SA was increased by 1.8 %. These increments indicated the ethanol treatment has affected the porosity of the carbon surface but did not alter the carbon structure as shown in Figure 2.

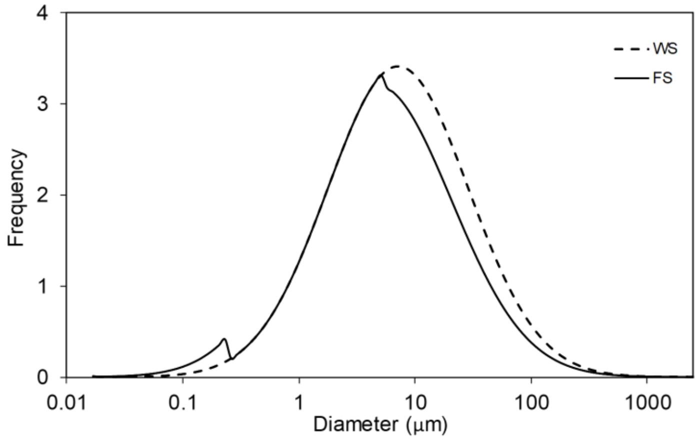

Fig. 3. Particle distribution of fresh soot (FS) and washed soot (WS) with an average diameter of more than 1 μm

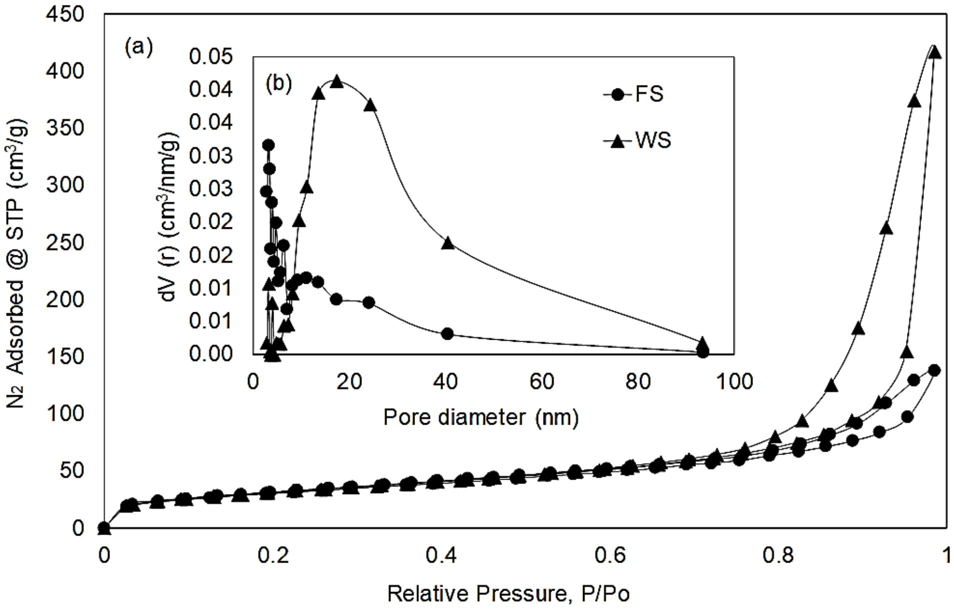

Fig. 4. Nitrogen adsorption/desorption isotherm (a) Nitrogen adsorption and desorption of fresh soot (FS) and washed soot (WS) samples attributed to type IV isotherms with hysteresis loop H2 and (b) pore distribution of FS and WS

In additional, with the sonication during the treatment of soot, the cavitation bubbles were expected to remove the insoluble matters from the soot pores as shown in Figure 5. The FS will contain both soluble organic matters and insoluble matters in the pores. The soluble organic matters will be removed by the ethanol treatment. Meanwhile, the insoluble matters will be removed by the implosion of cavitation bubbles [32], to go deeper in the pores to remove the insoluble matters, which will assist ethanol to clean the soot pores and provide spaces for CO_2 adsorption. This also supported by the previous study [22], which used soot particles as “ping-pong ball” alike mechanisms and cavitation bubbles to remove the magnesium oxide particles from the substrate.

The pore distribution was determined by BJH analysis as presented in Figure 4(b). Both samples have mesoporous size distribution of around 3 nm to 17 nm. This analysis clearly showed that the ethanol treatment did affect the porosity of the soot which can be enhanced the adsorption capacity.

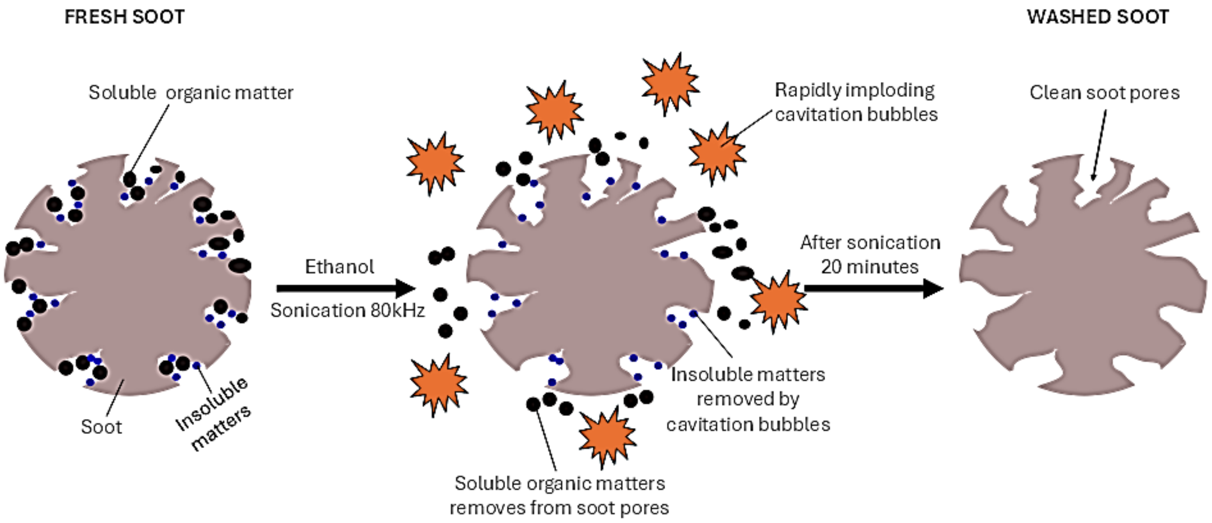

Fig. 5. Mechanism of removal of soluble organic and insoluble matters from the soot pores, using the ethanol treatment assisted with cavitation bubbles

Figure 6 presented the FTIR spectrum of fresh soot (FS) and washed soot (WS) samples in the range 400-4000 cm^{-1}. The spectrum of the FS sample shows the presence of CH_2 and CH_3 asymmetric vibrations at peak 2970 and 2880 cm^{-1} respectively indicating the combustion of diolefin in candle soot was partially completed [33]. Based on studies by Liang *et al.,* [28], the inner flame region released more organics due to the slightly oxidized wax molecules under the low fraction of oxygen. The spectrum of WS samples showed a huge decrease in intensity of the above-mentioned peaks indicating the enhanced quality of candle soot [29]. The peak at 1048 cm^{-1} corresponded to the stretching vibration of C-O bonds. It was probably due to carboxyl groups (C-O) stretching of oxidized carbon [28]. The intensity of this peak decreased and formed a broad peak in the WS sample represented a lower number of such bonds in candle soot structure after the treatment process.

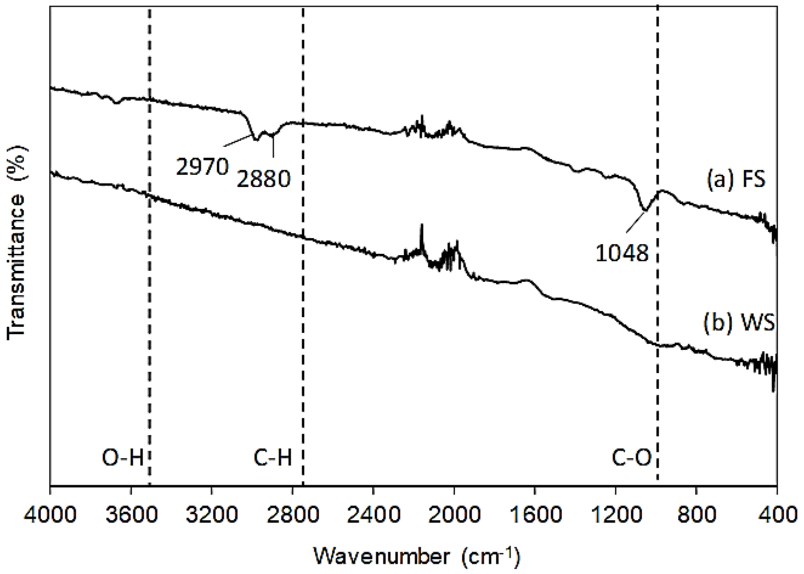

Fig. 6. FTIR spectrum of (a) Fresh soot (FS) (b) Washed soot (WS)

3.3 CO_2 Adsorption Amount and Isotherms Study

Figure 7 depict the CO_2 adsorption amount of fresh soot (FS) and washed soot (WS) tested under 30 bars and 25 °C respectively. Based on Figure 7, it was noticeable that the adsorption capacity for both samples did not show a highly significant effect. As shown by experimental data, CO_2 uptake was gradually increasing by the pressure for both samples. At 1 bar, the CO_2 uptake reached 22 mg/g and 19 mg/g for FS and WS samples respectively. As the pressure increases, the amount of CO_2 increases gradually until reached the maximum capacity at 30 bar which are 68 mg/g (1.55 mmol/g) and 69 mg/g (1.57 mmol/g) for FS and WS samples respectively. The amount of CO_2 adsorbed increases at higher pressure as more CO_2 molecules will force into to enter carbon surface and adsorbed on pores surface by van der Waals interaction force [34].

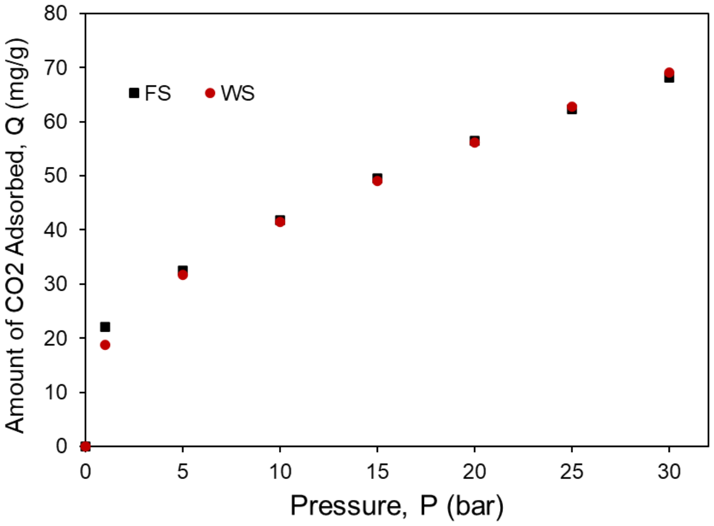

Fig. 7. Experimental data for CO_2 adsorption for fresh soot (FS) and washed soot (WS)

Based on the physical properties of the samples, the CO_2 adsorption capacity of the samples is linked to the BET surface area and pore volume. A surface area will provide more sites for CO_2 molecules to adequate the carbon surface as mentioned by Sui *et al.,* [35]. As FS and WS have a significantly larger surface area, thus this allowed both samples to have higher CO_2 adsorption capacity at 30 bar pressure. Rashidi *et al.,* [36] using peat as a precursor (Norit ® SX2) which owned 660 cm^2/g of surface area has possessed a higher CO_2 adsorption capacity (84 mg/g). The enhanced pore volume in the ethanol treated candle soot associated with the removal of the volatile matter on the pores, which exposed the clogged small pores diameter and defects. This thus related to the higher adsorption capacities in the ethanol treated soot compared to fresh soot.

This mechanism also found by Wu *et al.,* [37], which have obtained the highest CO_2 uptake by the porous carbon (160 mg/g) as it owned the highest surface area and pore volume which are 1943 m^2/g, respectively. A similar correlation was retrieved by previous study [38] in biomass-derived activated carbon and obtained the experimental CO_2 uptake at 602 mg/g with 2716 cm^2/g of surface area.

Table 3 illustrated the CO_2 adsorption equilibrium isotherm for FS and WS samples based on the adsorption amount. The Langmuir constant, k_L and Freundlich constant, k_F are related to adsorption capacity [39]. The value is correlated with the variation of suitable area and porosity of the adsorbent which implies that larger surface area and pore volume will result in higher adsorption uptake as displayed by FS and WS samples. The value of 'n' in the Freundlich model is higher than 1 which implies the favourable in CO_2 adsorption to take place on porous carbon. FS and WS showed physical and multilayer adsorption of CO_2 as the value of 'n' is higher than 1 which is similarly obtained by Sarwar *et al.,* [38]. Dubinin-Radushkevich isotherm gives the value of mean free energy of adsorption, E within the range 3-4 kJ/mol which suggesting that the CO_2 adsorption is physical, as the value of E is below 8 kJ/mol. The R^2 coefficient of the correlation shows that the Freundlich model gives the best fit towards the experimental data. The R^2 value obtained by FS and WS were 0.979 and 0.993 respectively. It implies that the surface of the carbon soot is heterogeneous and multi-layer CO_2 adsorption occurs and do not restrict to monolayer.

Table 3
Experimental and isotherm model for fresh soot (FS) and washed soot (WS)

Type	FS	WS
Experimental		
1 bar	22 mg/g	19 m/g
30 bar	68 mg/g	69 mg/g
Langmuir		
Q_m	76.92	79.37
K_L	0.162	0.145
R^2	0.955	0.955
Freundlich		
n	3.005	2.615
K_F	20.65	17.90
R^2	0.979	0.993
D-R		
Q_m	51.34	51.27
β	4×10^{-07}	4×10^{-07}
E	1.118	1.118
R^2	0.661	0.720

The novelty of the current study is founded upon the analysis of the effects of ethanol treatment on the surface characteristics and CO_2 adsorption performance of candle soot, a carbon-rich material often overlooked in carbon capture investigations. In contrast to traditional carbon sources, candle soot, which is derived from paraffin wax, demonstrates an exceptionally high level of carbon purity, with the initial carbon content quantified at 93.91%. This research distinctly evaluates the way ethanol treatment modifies the porosity and surface area of the soot, thereby enhancing its CO_2 adsorption potential, a factor of paramount importance in tackling the urgent challenge of atmospheric CO_2 emissions. The findings indicate that the treatment not only reduces the carbon content to 67.52% but also significantly improves the structural properties of the soot, thereby promoting enhanced transport of CO_2 molecules through intraparticle pores. Furthermore, this research enhances the understanding of the mechanisms that govern the impact of ethanol on soot, thereby filling a critical gap in the current body of literature pertaining to the enhancement of carbon-based adsorbents for CO_2 sequestration.

4. Conclusions

In summary, the ethanol treated candle soot was utilized for enhanced CO_2 adsorption for the first time. The physicochemical properties of the non-activated candle soot were studied to evaluate the performance in CO_2 adsorption. Both samples showed a better performance in CO_2 adsorption capacity which was confirmed through the experimentation and characterizations. The particle analysis of fresh soot (FS) and washed soot (WS) samples were showing the agglomeration size which was more than 1 μm indicated the samples were high in surface area to the volume ratio, which supported by BET increase in surface area. Both samples revealed the increment in adsorption capacity at higher equilibrium pressure which is 68 mg/g and 69 mg/g respectively. Experimental data of adsorption displayed agreement to the Freundlich isotherms by giving a closest value 1 of R^2, indicating a heterogeneous surface with varying adsorption energies. Based on isotherms, this proved the ethanol treatment enhance the adsorption of CO_2 uptake. This finding will allow us to understand the ethanol treatment not only improved the surface area and porosity of the soot but also facilitated the removal of volatile components, thereby exposing more active sites for CO_2 interaction. Further research is required to develop the potential of candle soot with different surface modification methods to maximize the CO_2 adsorption.

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