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Carbon Dioxide Adsorption on Sawdust Biochar

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Abstract

Biochar has been acknowledged for its unique property which makes it potential candidates as adsorbent for carbon dioxide (CO_2) in the flue gas system. In this study, the properties of raw sawdust biochar (SB) and amine treated sawdust biochar (NSB) are being compared. Ultimate analysis was performed using elemental analyzer to determine the carbon, hydrogen, nitrogen and sulfur contents in the adsorbent. Physiochemical characterization has been performed to characterize the biochar properties. Fourier Transform Infrared Spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET) were used to evaluate the functional groups and surface area of the biochar. Thermogravimetric analyzer (TGA) was used to discover the thermal properties, reactivity during adsorption. During the adsorption study, it was observed that raw sawdust biochar gasified at 850 °C gave the highest adsorption of 0.47 kg CO_2/kg biochar at temperature of 30 °C. The incorporation of nitrogen functionalities onto the carbon surface may cause decrement of surface area of carbon.

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Keywords: *Biochar; carbon dioxide; amine; thermogravimetric; flue gas*

1.Introduction

Extensive studies are currently being performed to reduce the discharge of CO_2 to the atmosphere. Countless organizations are finding alternatives to reduce the CO_2 concentration and considering methods to control the emissions of this greenhouse gases. The energy generated from the combustion of fossil fuels is one of the major sources of the greenhouse gasses [1]. In Malaysia CO_2 emission has increase rapidly since 1980 and this number is expected to increase massively in years to come. The amount of CO_2 emission in 1980 shows an emission of 25 million metric tons of CO_2 and increased to 160 million metric tons of CO_2 by 2006 which contributes to 540% increase from the initial amount in 1980 [2]. Monoethanolamine (MEA) solvent is widely used in the flue gas which involves introducing the gas stream to an aqueous amine solution which reacts with the CO_2 in the gas by an acid-base neutralization reaction. A problem that the industry is facing with the usage of amine in the flue gas system is the degradation of the amine as it is being recycled continuously. Other than the byproducts created by degradation of amine which reduces the adsorption capacity of CO_2, amine also accelerates the corrosion process inmachines involved [3]. One promising method to handle the carbon dioxide uptake is by using activated carbon which hasadsorptive properties such as microporous structure and high surface area [4]. Other than these criterions, the surface chemistryalso plays a role in the effectiveness of the adsorption process [5]. In this paper, the treatment of biochar is focused on MEA treatment to incorporate nitrogen component to the biochar structure.

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2. Materials and Methodology

2.1 Preparation of sawdust biochar

Sawdust biochar adsorbent used was derived from a lab scale air blown gasifier reactor operated at 850 °C to generate energy. The samples are grounded to diameter of 500µm and dried overnight at 90 °C to remove any moisture present. The biochar is separated into two parts of raw and chemically treated sample for the CO_2 adsorption capacity study. The samples were treated with monoethanolamine (MEA) and the sample was stirred for 20 minutes. The treated sample was then dried at 100 °C for 24 hours [6]. The raw biochar sample is identified as SB while the amine treated sample is regarded as NSB.

2.2 Adsorbent Characterization

Ultimate analysis was performed using CHNS elemental analyzer to determine the carbon, hydrogen, nitrogen and sulfur contents in the adsorbent. Brunauer-Emmett-Teller (BET) surface area of the biochar was analyzed using an automatic Quantacome AS1WinTM – Automated Gas Sorption Data Analyzer. Mettler Toledo TGA/SDTA851 (USA) Thermal Gravimetric Analyzer (TGA) was used to check the stability of the sample. Fourier Transform Infrared Spectroscopy (FTIR) is used to identify the chemical composition of char by recording the infrared spectrum of respective samples. The compositions of the sample can be recognized by analyzing the spectra with the relative intensities of a functional group.

2.3 Isothermal Adsorption Tests

The isothermal carbon dioxide (CO_2) adsorption analyses were carried out at temperature 30 °C to evaluate the adsorption capacity of the char sample using EXSTAR 6000 Thermal Gravimetric Analyzer (TGA). It is used to measure reactivity of carbonaceous material with CO_2. Around 10 mg of sample is placed in crucible before being placed in the conveyer. The sample is then heated up to desired temperature of 30 and 70 °C in 100 ml/min nitrogen flow. The sample is held at this temperature until the weight of sample is stabled (10 to 20 minutes). Gas is then switched to CO_2 at 100 mL/min to measure CO_2 adsorption and change back to nitrogen flow of 100 mL/min for desorption test.

3. Results and Discussion

3.1 Characterization of sawdust biochar.

The difference characteristics of the sawdust samples can be seen from the results of the ultimate analysis in Table 1. SD750 gives the highest amount of carbon content of 97.3 %, followed by SD850 of 93.4 % and SD450 of 82. 3%. SD450 and NSD850 have the highest moisture content of 10.27 % and 9.18 wt% respectively. The high volatile matter content of the biochar is due to the decomposition of cellulose, hemi cellulose and lignin. The high moisture, volatile and ash content may be due to the plant origin of the biochar samples [8]. It is foreseen that the treatment with monoethanolamine (MEA) will increase the nitrogen contents of the biochar sample and hence further improve the CO_2 adsorption. The observation of the nitrogen percentage is essential to verify the impregnation of amine onto the surface of biochar at which the nitrogen content will increase after amine treatment. All raw biochar has low value of nitrogen contents of <0.4 %. For treated biochar sample, NSD450 shows highest nitrogen content of 1.637 %. This is followed by NSD750 and NSD850 with nitrogen content of 1.385 % and 1.204 % respectively. Although high nitrogen content is preferable, the adsorption of CO_2 onto the surface of biochar is closely related to both high amounts of carbon and nitrogen contents. The biochar produced is a suitable precursor for activated carbon production as it has carbon contents of 90 % and low ash content [7]. Nevertheless the high nitrogen content does not reflect the adsorption capacity of the sample to capture CO_2.

As shown in Table 1, raw sawdust (SD) samples showed acidic characteristic with the pH range of 5.09-5.57. After amine treatment, the sawdust sample shows an increase of the pH in the range of 6.32-6.93. The value of the pH changed from acidic to basic after the immobilization of strong basic amine. Therefore the impregnation of amine onto the biochar surface is considered successful. The basic nature of the treated samples is expected to be advantageous for its application in the adsorption of CO_2 which is an acidic gas [9]. Lower CO_2 capture compared to raw biochar samples can be explained by the pore blockage of the amine film that inhibits adsorption to be performed onto the adsorbent. SD850 has the highest surface area of 182.04 m^2/g followed by SD750 and SD450. NSD750 has the lowest surface area of 0.15 m^2/g. The higher the surface area of biochar sample, the greater is the adsorption capacity of the char. The relation of the surface area of the sample coupled with the adsorption capacity for CO_2 adsorption is essential to have a clearer view on these two relations. Although the BET surface area shows low value, the effectiveness of the biochar adsorption may depend on other factors such as the pore structure, functional groups and surface chemistry [10].

SD750 and SD450 show the highest pore volume of 0.016 cm^3/g and 0.015 cm^3/g respectively. Other samples show pore volume <0.009 cm^3/g. The treated biochar sample shows a decrement of the pore volume due to pore filling effects of the amine treatment [11]. The adsorption capacity will be discovered with CO_2 adsorption test by using TGA. The pore sizes of treated sawdust sample NSD850, NSD750 AND NSD450 show the pore sizes of 9.2859, 8.8604, and 4.8265 nm respectively. Raw sawdust samples have smaller pore sizes of 1.5342-1.7141 nm. The treated biochar sample shows an increment of their pore sizes. The increase of the pore sizes are in agreement with other amine treatment onto carbon at which the nitrogen compound is attached onto the surface of the carbon [7].

According to the International Union of Pure and Applied Chemistry (IUPAC), pores are classified as micropores (<2nm diameter), mesopores (2-50 nm diameter) and macropores (>50nm diameter) [12]. The studied biochar have pore sizes between the range of <2 nm and 2-50 nm which indicates it is in the micropores and mesopores region.

Table 1.The ultimate analysis, surface area, CHNO contents and pH of raw and amine treated sawdust biochar.

		Samples					
		SD450	NSD450	SD750	NSD750	SD850	NSD850
Moisture, wt.%		10.2700	8.0100	6.4200	8.9200	7.5300	9.1800
Volatile, wt.%		28.4600	9.8800	11.1100	5.6600	4.1200	4.3700
Fixed Carbon, wt.%		64.1300	47.5800	75.9100	45.0900	65.0700	55.2100
Ash, wt.%		18.1700	6.2000	21.3900	10.9800	28.3300	9.8100
Surface Area (m^2/g)		8.7600	0.6100	11.3600	0.1500	182.0400	3.1700
Pore volume (cm^3/g)		0.0150	0.0090	0.0160	0.0050	0.0036	0.0070
Pore size(nm)		1.7141	4.8265	1.5351	8.8604	1.5342	9.2859
pH		5.1600	6.3200	5.0900	6.9300	5.5700	6.8300
Ultimate Analysis (wt%), db	C	82.3000	53.7800	97.3000	56.0700	93.4000	65.0200
	H	3.2000	3.7550	1.1000	2.0600	1.3000	1.7620
	N	0.4000	1.6370	0.0700	1.3850	0.1000	1.2040
	O	14.1000	40.8280	1.5300	40.4850	5.2000	32.0140

db: dry basis

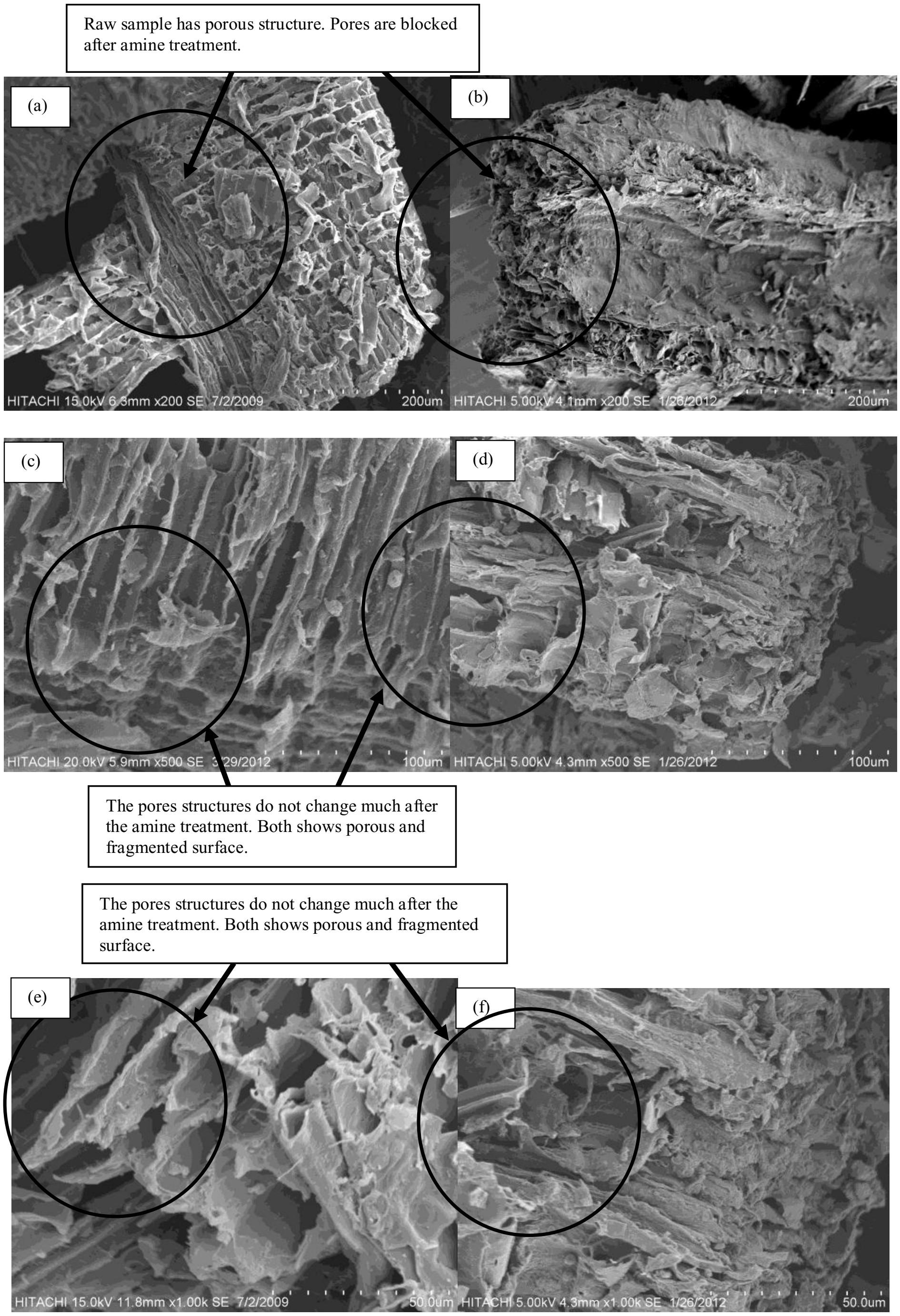
[a] Calculated by difference

Table 2 shows the amine weight percentage incorporated in biochar samples after amine treatment. SD450 shows the highest percentage of amine incorporated in the biochar sample by 7.13 %. This is followed by SD750 and SD850 of 6.03 % and 5.2 5% respectively. Therefore it can be said that the amine treatment have successfully been performed onto the biochar samples at which the nitrogen compound may increase the basicity of the biochar sample and have the potential to improve CO_2 adsorption since CO_2 is an acidic gas. However the adsorption capacity may be affected whereby the pore filling effects on the surface of sample during the impregnation of amine will inhibit the adsorption process.

Table 2. The amine weight percentage incorporated in biochar samples after amine treatment.

No	Sample	Amine weight %
4	SD 450	7.13
5	SD 750	6.03
6	SD 850	5.25

Carbon materials have porous surface, the size of which can easily be altered depending on activation methods [13]. The surface morphology of the sawdust produced at 450, 750 and 850 °C as shown in Fig. 1a, b, c, d, e and f show irregular and porous surfaces. There is increased porosity from volatiles escaping during thermal treatment [14]. This porous structure of the biochar facilitates the effectiveness of the char for CO_2 adsorption as activated carbon. The porous surface does not change much for the treated sawdust samples except for the sawdust produced at 450 °C as shown in Figure 1b which shows blocked pores after the amine loading.



Raw sample has porous structure. Pores are blocked after amine treatment.

The pores structures do not change much after the amine treatment. Both shows porous and fragmented surface.

The pores structures do not change much after the amine treatment. Both shows porous and fragmented surface.

Fig. 1 (a) SD450 biochar; (b) NSD450 biochar; (c) SD750 biochar; (d) NSD750 biochar; (e) SD850 biochar; (f) NSD850 biochar

TGA was used to study the adsorption behaviour of different types of raw and treated biochar by flowing first flowing nitrogen and then carbon dioxide into the analyzer. The amount of CO_2 being adsorbed is calculated by the weight differences of the initial and final adsorption percentages. As shown in Table 3, for adsorption at temperature of 30 °C, it is observed that for raw samples, sawdust 850 gives adsorption of 47.5 mgCO_2/g sorbent followed by sawdust 750 and sawdust 450. For amine treated samples at 30 °C, sawdust 850 gives higher adsorption of 44.8mg CO_2/g sorbent. The CO_2 capture at 30 °C is maybe due to the physisorption which takes place within the pore structure. The physisorption adsorption behaviour is described as directly proportional to the surface area by means of the raw sample has a higher surface area compared to the treated sample which its pores has been blocked by the amine film [15]. The results is seen to follow the study by Plaza et al [16] at which the adsorption of raw Norit carbon at 30 °C gives higher adsorption of 7.3 mgCO_2/g sorbent as compared to amine treated sample which gives 4.0 mgCO_2/g sorbent. The adsorption of raw zeolites 13X also gives higher adsorption of 28 mgCO_2/g sorbent as compared to amine treated sample which gives 17.53 mgCO_2/g sorbent [17]. All biochar samples give lower CO_2 adsorption at higher temperature of 70 °C as compared to lower temperature of 30 °C. For raw biochar samples, sawdust 850 gives the highest value of adsorption of 28.8 mgCO_2/g sorbent, followed by sawdust 750. For treated biochar samples, sawdust 850 also gives the highest value of adsorption of 25. 2mgCO_2/g sorbent, followed by sawdust 750 and sawdust 450 with the value of 22.6 and 12.13 mg CO_2/g sorbent respectively. The low adsorption of CO_2 compared to adsorption at lower temperature is due to the subtle contact of the carbon support with CO_2 at higher temperature [15]. When temperature increases, higher surface adsorption energy is needed and the molecule diffusion rate increases which results to the adsorbed gas becomes unsteady and desorption happens on the surface of the carbon [18]. Amine treated biochar give lower values of CO_2 adsorption compared to raw samples which due to pore blockage that takes place during the amine treatment .The loading of amine may cover the pore thus reduces the adsorption of CO_2.

Table 3. Adsorption of carbon dioxide at temperature of 30°C and 70°C

No	Sample	Temperature (°C)	Raw mg CO_2/g sorbent	Treated mg CO_2/g sorbent
1	Sawdust 450°C	30	19.7	19.1
		70	13.5	12.1
2	Sawdust 750°C	30	45.2	39.7
		70	25.4	22.6
3	Sawdust 850°C	30	47.5	44.8
		70	28.8	25.2

Fourier Transform Infrared Spectroscopy (FTIR) is used to identify the chemical composition of char by recording the infrared spectrum of respective samples. The compositions of the sample can be recognized by analyzing the spectra with relative intensities which indicates different functional group. In Fig. 2, both raw and amine treated sawdust 450 °C biochar shows high intensity in the range of 1000 – 1500 cm^{-1} which indicates carboxylic and carbonyl (basic oxides) groups and sharp peak at 1570cm^{-1} which indicates presence of ketone onto the sample [19]. The treated sample shows a decrease of intensity at 3000-3600 cm^{-1} indicating reduction of hydroxyl group of alcoholic or phenolic. The most characteristics bands of lignin can be seen at 1510 and 1598 cm^{-1} which indicates aromatic ring vibrations and between 1470-1460 cm^{-1} which signify the CH deformation and aromatic ring vibrations [20]. The spectrum of both biochar samples shows presence and IR bands linked to phenolic groups at 1000-1200 cm^{-1}. A band near 1600 cm^{-1} can be associated to quinine groups [7]. These phenolic and quinine groups which appear at higher intensity at the treated sample are acidic functional groups which may decrease the CO_2 adsorption capacity.

a)

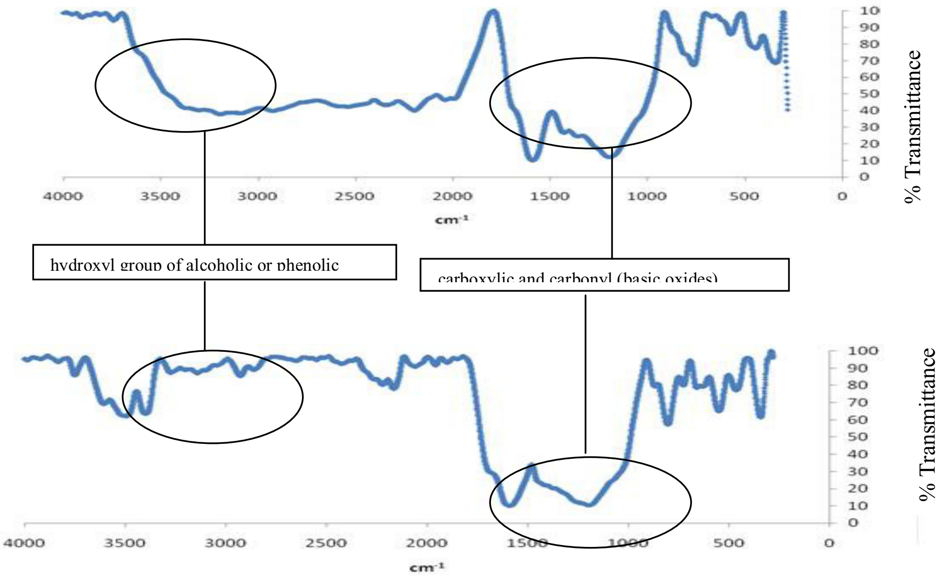


Fig. 2 FTIR spectra of (a) raw; (b) treated sawdust 450°C biochar.

Table 4 shows the summary of the group functionality of the rawand treated sawdust biochar. Treatment of biochar and activated carbon with amine particularly MEA (aminemonoethanol) help to incoporate nitrogen functionalities to the carbon sample. This is shown by the appearance of nitrogen based peaks at 1677 cm^{-1} and at 1400 cm^{-1} due to the amino groups. Bands formation of 3000-3600 cm^{-1} indicates hydroxyl group of alcoholic or phenolic and intensity at 1000-1200 cm^{-1}is largely related to phenolic groups. A band near 1480–1610 cm^{-1} can be associated to pyridine like groups. Oxygen functionalities such as chromene, ketone, and pyrone contribute to the basicity of the carbon. The sharp peak at 1570 cm^{-1} indicates presence of ketone. Although treatment of amine increases the basic functional groups, not all samples show increment of CO_2 adsorption after treatment. This may due to the presence of acidic group that may reduce the ability to capture CO_2 since it is an acidic gas. Since almost all raw samples gave higher adsorption, it can be said that the basic functional groups that is already present in the parent samples are sufficient to obtain high adsorption of CO_2.

Table 4. Summary of the group functionality of the biochar samples

Group or functionality	Wavenumber (cm^{-1})	SD450	NSD450	SD750	NSD750	SD850	NSD850
Carboxylic acids							
C=O (stretching)	1600–1800, 1720–1750,	√	√	√	√	√	√
O–H (stretching)	3530, 3500	√	√	√			
Quinones	1550–1680,1650, 1580–1620	√	√	√	√	√	√
C–H (stretching)	2600–3000, 2924	√		√		√	
Phenolic groups							
C–OH (stretching)	1000–1400, 1200–1300	√	√	√	√	√	√
O–H (stretching)	2500–3620, 3393		√	√	√	√	√
Lactones (C=O stretching)	1710, 1750, 1720, 1760	√	√			√	
Ketones (C=O stretching)	1570, 1560	√	√	√	√	√	√
Ethers (C–O stretching)	1000–1400,1100–1400	√	√	√	√	√	√
O–H (hydroxyl)	3200–3600, 3100–3600	√	√	√	√		√
C=N (Cyanide group)	1570, 1600	√	√	√	√	√	√
Pyridine-like groups	1480–1610	√	√	√	√	√	√
C–N	1190, 1250	√	√	√	√	√	√
Cyclic amides	1461–1685, 1670	√	√	√	√	√	√
N–H	1480, 1560	√	√	√	√	√	√
Nitro groups	1330–1530, 1574	√	√	√	√	√	√

Table 5 describes the amount of CO_2 captured by using 1 kg of biochar adsorbent. The CO_2 adsorption decreases as the temperature increases. For temperature 30°C, SD850 gives the higher value of 0.47 kg CO_2/kg biochar followed by SD750 and SD450. For treated sawdust at 30 °C, the values of CO_2 captured are in the range of 0.22-0.43 kg CO_2/kg. For temperature 70 °C, SD850also give the highest value of 0.30 kg CO_2/kg. The lowest amount of CO_2 captured was observed at temperature 70° C for sample NSD450. These values are comparable with absorption using aqueous monoetanolamine (MEA) that is currently being employed in the industry whereby the amount of removal efficiency is 0.4 kg CO_2/kg MEA [26]. Raw sawdust biochar is seen to give higher adsorption of CO_2 as compared to amine treated biochar sample. Therefore it can be said raw biochar has more potential to be used to capture CO_2 in the flue gas system.

Table 5. Amount of CO_2 captured by using 1 kg of adsorbent

No	Sample	Weight of CO_2 captured (kg) Adsorption at 30 °C	Adsorption at 70 °C
1	SD450	0.23	0.16
2	NSD450	0.22	0.14
3	SD750	0.43	0.27
4	NSD750	0.40	0.20
5	SD850	0.47	0.30
6	NSD850	0.43	0.21

Table 6 shows the comparison of the CO_2 adsorption onto raw and amine treated carbon samples from other experimental works. Most of the carbon exhibit decrement of surface area after amine treatment which may due to the incorporation of nitrogen functionalities onto the carbon surface. The adsorption at temperature 30 °C gives higher CO_2 uptake as compared to adsorption at higher temperature. The physisorption process that takes place at lower temperature is assisted by high surface area in which giving higher CO_2 sequestration. The effectiveness of the biochar used in this study is seen to be compatible when compared with other sorbents and commercial activated carbons.

Table 6.Comparison of adsorption from other studies.

Sample	**Treatment**	**Raw BET Surface Area(m^2/g)**	**Treated BET Surface Area (m^2/g)**	**CO_2 adsorption capacity (mgCO_2/g adsorbent)**	**References**
Fly carbon	Amine	75	241	Temperature 30 °C MEA : 68.6 Temperature 7 0°C MEA : 49.8	[18]
Anthracites	Polyethylenimine (PEI)	1000	< 1	Temperature 75 °C Raw: 16.05,PEI : 26.30	[27]
Palm Shell Activated Carbon (AC)	Polyethylenimine (PEI)	941	1052	Temperature 25 °C Raw: 3.56,PEI: 3.26	[28]
Sawdust 450°	Monoethanolamine (MEA)	8.76	0.61	Temperature 30 °C Raw: 19.7,MEA : 19.1 Temperature 70 °C Raw: 13.5,MEA : 12.1	This study
Sawdust 750°	Monoethanolamine (MEA)	1.36	0.15	Temperature 30 °C Raw: 45.2,MEA : 39.7 Temperature 70 °C Raw: 25.4,MEA : 22.6	This study
Sawdust 850°	Monoethanolamine (MEA)	182.04	3.17	Temperature 30 °C Raw: 47.5,MEA :44.8 Temperature 70 °C Raw: 28.8,MEA : 25.2	This study

5. Conclusions

Gasification residues of sawdust biochar provides the highest CO_2 capture capacity of 0.47 kg CO_2/kg biochar and 0.30 kg CO_2/kg biochar at temperature of 30 °C and 70 °C accordingly. The high uptake of CO_2 is mainly due to the physisorption process that takes place at low temperature in which the adsorption capacity is highly associated to the high values of surface area and pore volume. Amine treated biochar highest CO_2 capture capacity of 0.43 kg CO_2/kg biochar and 0.21 kg CO_2/kg biochar were adsorbed at temperature of 30 °C and 70 °C accordingly. The lower CO_2 uptake adhered by the treated biochar is due to the incorporation of nitrogen functionalities onto the carbon surface. The study performed showed that raw sawdust biochar has the potential to be used as CO_2 sequester in flue gas system in efforts to reduce greenhouse gasses emissions.

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