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Isotherm and Thermodynamic Analysis of Carbon Dioxide on Activated Carbon

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Abstract

In this study, commercial Norit® SX2 activated carbon has been evaluated as a solid adsorbent for carbon dioxide (CO₂) capture at the post-combustion conditions, which are at an ambient pressure and low temperatures (< 120°C). The CO₂ equilibrium adsorption capacity is assessed through a static volumetric technique. This solid adsorbent is characterized in terms of elemental analysis, nitrogen physisorption, as well as surface morphology. The CO₂ adsorptive property of the commercial Norit® SX2 demonstrates a reduction in the amount of CO₂ adsorbed at an elevated temperature, and accordingly, it indicates that CO₂ adsorption is a physical adsorption process and demonstrates a behavior of an exothermic reaction, which consistent with the thermodynamics analysis. Different isotherm models are applied to mathematically model the CO₂ adsorption, and on basis of the regression coefficient (R²), the Freundlich model provides a perfect fit to the experimental data, owing to closeness of the R² to unity. The result obtained in this study can serve as a benchmark while searching for inexpensive and superior activated carbon production in future studies.

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Keywords: Activated carbon; CO₂ adsorption; Isotherm; Physisorption; Thermodynamics.

1. Introduction

The climatic change is undeniably the main environmental problem in the 21st century that faces the mankind today. By now, Malaysia has experienced harsh weather events such as drought and flooding which results in economic losses and worsens the human health. Tangang et al. [1] projected that the global warming contributes to increase in surface temperature to 3-5°C by end of the 21st century. This warmer atmosphere drives ice and glacier melting, thus increases sea level to 95 cm, and may influence the rainfall pattern too [2]. These ecological damages are primarily contributed from the anthropogenic carbon dioxide (CO₂) emission from industrial sectors i.e. power plant, gas processing, refinery, chemical and petrochemical, iron and steel, as well as in cement manufacturing [3]. Due to rapid industrialization activity and prolong CO₂ atmospheric lifetime, the CO₂ concentration increases from 280 ppm in pre-industrial period to almost 400 ppm today, despite its recommended limit is at 350 ppm [4]. Since the estimated CO₂ concentration is beyond the upper limit, mitigating the CO₂ emission needs to be considered. Several methods have been introduced by researchers in tackling this problem, which include an improvement in energy efficiency, shift to lower carbon-fuel material and renewable energy, promote afforestation and reforestation, along with carbon capture and sequestration (CCS) [5]. Amongst these options, CCS is promising since it is capable to minimize the CO₂ emission from large point emission sources such as power sector, up to 85-90% [5, 6]. Capturing CO₂ from these power plants occurs in three different routes, viz.,

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post-combustion, pre-combustion, and oxy-fuel combustion. Between these techniques, the post-combustion route is extensively employed in industries for the CO₂ removal from an exhaust gas of fossil fuel combustion. Wide acceptance of this technology is associated with an easiness of retrofitting the existing power plant, and allows the combustion system to remain unchanged [5, 7]. Presently, conventional method for the post-combustion CO₂ capture is via amine scrubbing system. Throughout this technology, the flue gas which is at 40-60°C passes through an absorber column and reacts with aqueous amine solutions. Upon equilibrium, the CO₂-loaded amine solution is drained from the absorber and is transported to a stripper, whereby the CO₂ is released at higher temperature (> 100 °C) [8, 9]. Despite being extensively applied at the industrial scales, consumption of amine based solvent such as monoethanolamine has its own limitations, owing to high energy requirement for absorbent regeneration, equipment corrosion, and potential of solvent degradation [10]. Moreover, evaporation and amine loss to ambient air is a big challenge, as atmospheric degradation of the amine-based solvent produce a wide range of byproducts such as amide, aldehyde, nitrosamine, and nitramine, that jeopardizes both health and environmental aspect [11].

Due to numerous side effects of the amine scrubbing, alternative to this conventional technology should be pursued. Since the condition of post-combustion is only at atmospheric pressure, membrane separation and cryogenic distillation is impractical for the CO₂ removal. Due to low pressure of the flue gas, driving force for the membrane separation is low as well [12]. Meanwhile, cryogenic distillation that includes a cooling step and enables liquefied CO₂ production is unfavorable for the post-combustion CO₂ capture, as it requires high energy requirement for the refrigeration stage [5, 12, 13]. Due to limitation of these technologies, solid adsorption has been proposed due to its plenteous advantages such as low energy requirement for regeneration, better CO₂ adsorption capacity, high selectivity, low equipment cost, and easy to handle. Lower energy penalty in the adsorption-desorption process is owed to absence in water and low heat capacity of the solid adsorbent [14]. Amongst the low-temperature adsorbents, activated carbon presents several benefits such as hydrophobic in nature, inexpensive, and less regeneration energy [15]. In this present study, performance of the CO₂ adsorption onto the commercial activated carbon is evaluated at an ambient pressure and at temperature range of 25-100 °C. Besides, the CO₂ adsorption isotherm is assessed through several models. This isotherm analysis is significant as it defines the gas-solid adsorption behavior and to assist in the process optimization and design [6, 16]. The typical isotherm of gas adsorption onto solid adsorbent is illustrated in Figure 1.

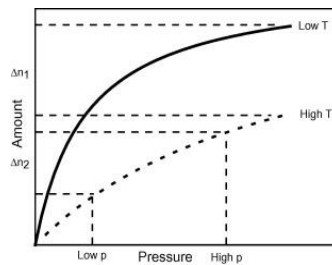


Fig. 1: Typical gas-solid physisorption isotherm [16]

2. Experimental

2.1. Material and Characterization

In this study, the commercial multi-purpose Norit[®] SX2 activated carbon supplied by Sigma Aldrich (M) Sdn. Bhd. was used. This activated carbon which is in powdered form had been produced from peat-based resources, and was steam-activated and acid washed. The textural properties of the activated carbon in terms of surface area and porosity were evaluated by nitrogen physisorption at -196°C by using a Micromeritics Tristar II apparatus. Prior to the measurement, carbon sample was outgassed at 350°C for four hours, to remove the physically adsorbed moisture and contaminant from the surface. Elemental structures of this solid adsorbent were identified through an ultimate analysis, using a CHNS/O analyzer (Perkin Elmer PE2400). Moreover, the surface morphology was analyzed via Field Emission Scanning Electron Microscopy (FESEM). In the FESEM analysis, a small portion of the sample was mounted to the sample holder by using a double-sided adhesive tape [17].

2.2. CO₂ Adsorption Measurement

The volumetric adsorption of purified CO₂ (99.8% purity) was performed by using a High Pressure Volumetric Analyzer from the Particulate System. Prior to CO₂ adsorption, about 0.3-0.4 g of the carbon sample was placed inside a 2 cm³ sample cylinder, and degassed at 150°C for 8 hours under a vacuum condition, to ensure all impurities were removed from the carbon structures. A filter gasket of size 60 μm was placed on top of the sample cylinder, to prevent the fine particles to enter the valve [18]. Upon completion of the degassing step, the samples were cooled to ambient temperature and readied for subsequent adsorption stage at temperature range of 25-100°C. At low temperature of 25-50°C, the sample temperature was regulated by Julabo re-circulating water bath, whereas at elevated temperature (100 °C), a furnace was used to control the temperature. The CO₂ adsorption process

was initiated by dosing the gas adsorbates into the system, by allowing the valve between the loading and sample cylinder open, and let the CO₂ adsorbate in contact with the adsorbent. The holding time at each pressure interval was fixed at 45 min to ensure equilibrium of the adsorption process [18]. Once the equilibrium stage at a certain pressure was achieved, the adsorption process was repeated until the stipulated maximum pressure was attained [18, 19]. The volumetric CO₂ sorption capacity was determined by the difference between the amounts of dosed gas into the system and the amounts of gas left in the system upon adsorption process, and data was directly acquired from the Comprehensive Data Analysis Package using Microsoft Excel macros (v.22.0.6) software. Then, each of the resulting points of volumes adsorbed and equilibrium pressures were plotted to produce the isotherm. Further, the volumetric data are used to compute the maximum adsorption capacity that can be obtained by the activated carbon, through several types of two-parameter isotherm models such as Freundlich, Langmuir, Dubinin Radushkevich, and Temkin. The non-linear and linearized equation of these isotherm models are summarized in Table 1.

Table 1: Non-linear and linearized isotherm models

Isotherm	Non-linear equation	Linear equation	Eq.
Langmuir	$q_e = \frac{q_m k_L P_e}{1 + k_L P_e}$	$\frac{P_e}{q_e} = \frac{1}{k_L q_m} + \frac{P_e}{q_m}$	(1)
Freundlich	$q_e = k_F P^{1/n}$	$\log q_e = \log k_F + (1/n) \log P_e$	(2)
Dubinin Radushkevich	$q_e = q_m e^{-\lambda \omega^2}$	$\ln q_e = \ln q_m - \lambda \omega^2$	(3)
Temkin	$q_e = B (\ln k_T P_e)$	$q_e = B \ln k_T + B \ln P_e$	(4)

Whereby P_e is equilibrium pressure (bar); q_e is amount of CO₂ adsorbed (cm³/g); q_m is the maximum CO₂ adsorption capacity (cm³/g); k_L is Langmuir constant (1/bar); k_F (cm³/g·bar^{1/n}) & n is Freundlich constant; λ (mol²/J²) is D-R constant; ω (J/mol) is the Polanyi potential (equivalent to $RT \ln(1/(1+P))$); $B = RT/b_T$; b_T (J/mol); and k_T (cm³/g·bar) is Temkin constant. The validity of these models is evaluated by the correlation coefficient (R^2), which is within the range of 0-1, in which R^2 closer to unity implies the best fitting towards the particular isotherm model.

3. Results and Discussions

3.1. Characterization of Activated Carbon

The chemical compositions of the commercial activated carbon that are measured from the CHNS analysis are summarized in Table 2. Based on Table 2, it is noticeably shown that this adsorbent is rich with carbon content, thus, is favorable for the CO₂ adsorption process due to van der Waals interaction force [6]. Aside from the chemical analysis, the pore characteristic in term of the surface area is significant, as high value indicates a greater adsorption capacity [20]. The pore characteristics of the adsorbent are listed in Table 2. In Figure 2, the shape of gas isotherm that illustrates the adsorbed amount at the specific relative pressures depicts the pore characteristic of the adsorbent. Based on Figure 2, the shape of the N₂ adsorption-desorption isotherm obeys the Type IV, as is classified by The International Union of Pure and Applied Chemistry (IUPAC), and accordingly, this Norit® SX2 is identified as a mesoporous material [21]. The result is consistent with the average pore size (4.05 nm) which is shown in Table 2, therefore, conforms to the IUPAC standard that fixed the pore width of mesoporous material to be within the range of 2-50 nm. In addition, FESEM image of this activated carbon that is presented in Figure 3 proves that the exterior structure consists of some pores that can facilitate the transport process of the CO₂ molecules towards the inner side of the porous activated carbon.

Table 2: Chemical compositions of Norit® SX2

Characteristics	Values
BET surface area (m ² /g)	660.73
Pore volume (cm ³ /g)	0.67
Average pore size (nm)	4.05
Elemental analysis (wt %)	
Carbon	77.48
Hydrogen	1.61
Nitrogen	0.33
Sulfur	0.05
Oxygen (By differences)	20.53

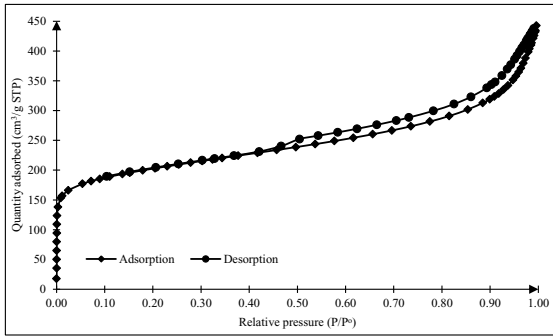


Fig. 2: N₂ adsorption-desorption isotherms

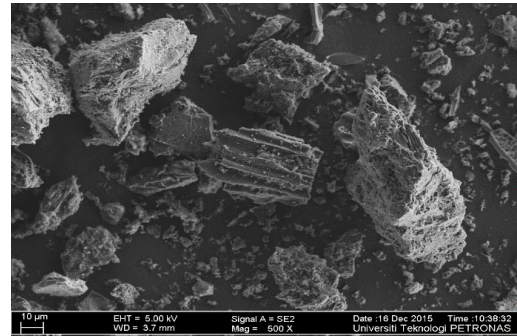


Fig. 3: FESEM image of Norit® SX2

3.2. CO₂ Adsorption and Isotherm Modeling 5.12-8.62

Figure 4 depicts the CO₂ adsorption profile at an ambient pressure and temperature of 25-100°C. From Figure 4, it evidently shows that the CO₂ adsorption capacity decreases with respect to temperatures, due to the reduction in binding strength between the CO₂ adsorbate and activated carbon [6]. Besides, reduction in the CO₂ adsorption capacity at an elevated temperature proves that the adsorption process is exothermic, in which it defines an occurrence of the physical adsorption (physisorption) process [22]. Unlike chemisorption, the physisorption involves a weak van der Waals force, which tends to be broken at high temperature, and accordingly, decreases the adsorption capacity [23]. In addition, higher surface adsorption energy and molecular diffusion at elevated temperatures results in instability of the CO₂ adsorbates on the carbon surface, and thereby, promotes desorption process [24]. Hauchhum and Mahanta [25] agreed that an increment in bed temperature accelerates the internal energy of the adsorbent, and consequently, releases the CO₂ molecules from the surface. Accordingly, reduction in the adsorption capacity with respect to the temperature implies an exothermic process, whereby the adsorption is controlled by physisorption [22].

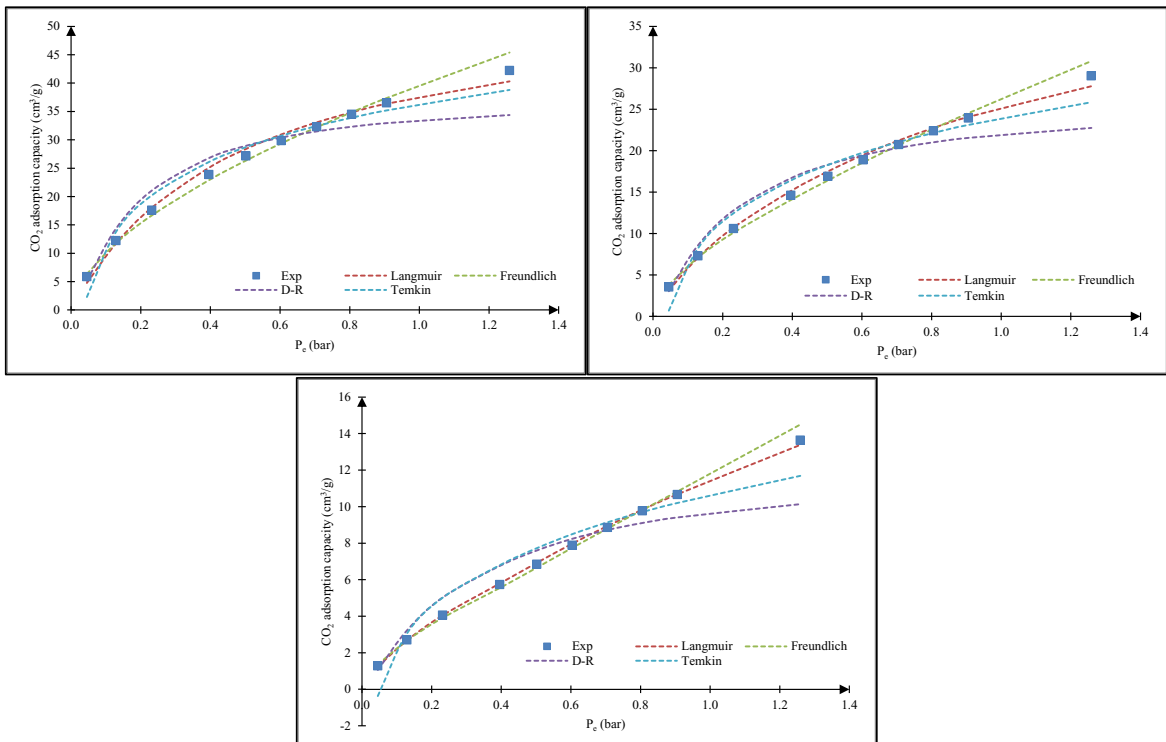


Fig. 4: Experimental equilibrium data and isotherms by linear regression method for CO₂ adsorption at (a) 25°C, (b) 50 °C, and (c) 100 °C

The predicted isotherm constants for the CO₂ adsorption and the corresponding R² value from the linear regression method are shown in Table 3. Based on Table 3, the Langmuir constant, k_L and Freundlich constant, k_F that relates to the adsorption affinity decreases at high temperature, therefore, implies a physisorption behavior. The reduction in CO₂ adsorption capacity is explained by the Le Chatelier's principle, whereby endothermic desorption is favorable at elevated temperatures. The exothermic behavior of the CO₂ adsorption is in parallel with the q_m value that tends to decrease with increased in the adsorption temperature. Further, favorability of the adsorption process can be indicated by a separation factor, R_L, whereby the calculated R_L at different pressure and temperature is shown in Figure 5. The R_L value which is within the range of 0-1 suggests that the CO₂ adsorption is favorable [26]. Furthermore, favorability of the CO₂ adsorption can be supported by the Freundlich constant, n which is within the range of 1-2 as shown in Table 3. In addition, the Dubinin Radushkevich and Temkin isotherm will provide a useful information related to the energy parameters, in terms of E (mean free energy of adsorption) and b_T (heat of adsorption) [27]. The calculated E values which are within the range of 3-4 kJ/mol suggests that the CO₂ adsorption is physical in nature, as the magnitude of E is below 8 kJ/mol, whereas value of 8 < E < 16 is an indicator of the chemical adsorption [28]. On the basis of R² value, the Freundlich model gives the best fit towards the experimental data over the entire temperature range. Therefore, it implies that the surface of the activated carbon is heterogeneous and a multi-layer CO₂ adsorption occurs and does not restrict to monolayer, as proposed in the Langmuir-type. Moreover, Perez et al. [29] suggested that the Langmuir model best describes the chemisorption reaction due to restriction to monolayer formation, whereas the Freundlich primarily represents the physisorption process, since it allows these adsorbate molecules to form a successive layer onto the surface of the Norit® activated carbon.

Table 3: Two-parameter isotherm models at different temperatures via linearized technique

Type	25 °C	50 °C	100 °C	Type	25 °C	50 °C	100 °C
Langmuir				Freundlich			
q _m	55.8659	45.6621	34.0136	k _F	39.6096	26.2482	11.7490
k _L	2.0575	1.2303	0.5043	n	1.6895	1.4548	1.1975
R ²	0.9818	0.9791	0.9913	R ²	0.9942	0.9959	0.9976
Dubinin Radushkevich				Temkin			
q _m	36.8001	24.8088	11.3305	B	10.9830	8.2613	4.2537
λ	3.2592E-8	3.5392E-8	4.6637E-8	k _T	27.1774	18.0917	12.1332
E	3.9168	3.7587	3.2743	b _T	225.5824	325.0605	729.0411
R ²	0.9540	0.9502	0.9435	R ²	0.9626	0.9481	0.9223

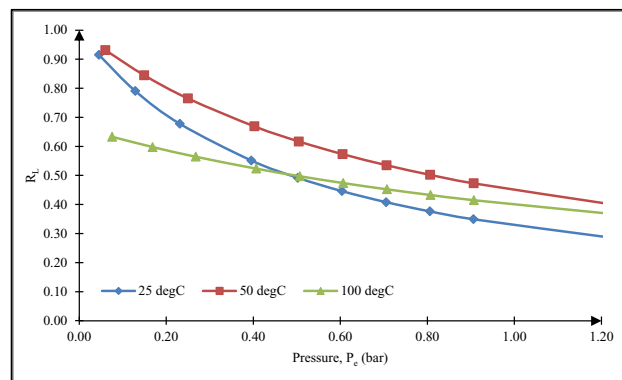


Fig. 5: Langmuir separation factor, R_L as a function of pressure and temperature

3.3. Thermodynamics Analysis

The thermodynamic parameters of the CO₂ adsorption process in terms of the change in Gibbs free energy (ΔG°), change in enthalpy of reaction (ΔH°), and change in entropy of adsorbate and adsorbent interaction (ΔS°) are estimated by applying the van Hoff's formulation, as is represented by Eq. (5) – (6) [30, 31].

$$\ln k_L = \Delta S/R - \Delta H/RT \quad (5)$$

$$\Delta G = - RT \ln k_L \quad (6)$$

Based on Eq. (5) – (6), R represents the universal gas constant (8.314 J/mol·K), T is adsorption temperature in Kelvin, and k_L is the Langmuir constant (1/bar) deduced from the Langmuir isotherm fit. The estimated values of the thermodynamic parameters are tabulated in Table 4. Based on the experimental findings, the negative sign of ΔH° value indicates an exothermic nature of the CO₂ adsorption process, whilst negative ΔS° value suggests high orderliness of the adsorbate molecules upon adsorption. Zhao et al. [32] claimed that the negative ΔS° can be interpreted by the behavior of the CO₂ molecules upon the adsorption process, which is from randomized to an ordered form on the surface of the adsorbent. The reduction in the entropy value upon the adsorption process is due to a lesser degree of freedom of the gas molecule, owing to minimum free space on the carbon surface. In addition, the magnitude of ΔH° denotes the type of CO₂ adsorption process, whether it belongs to the physical adsorption or chemical adsorption. It has been reported that the magnitude of ΔH° for the physisorption reaction is < 20 kJ/mol, whilst for the chemical adsorption, the value is within 80-200 kJ/mol [32, 33]. Therefore, the calculated ΔH° which is about 20 kJ/mol suggests that the CO₂ adsorption is physisorption, and consistent with the reduction in amount of CO₂ adsorbed at an elevated temperature (as shown in Figure 4). On the other hand, the ΔG° values are divided into two regions, in which one at low temperature (25-50 °C) with a negative value and the other one is at high temperature range (75-120 °C) with a positive value. The negative sign of ΔG° at low temperature indicates an occurrence of the favorable and spontaneous adsorption, whilst positive value at an elevated temperature implies a non-spontaneous adsorption reaction [30, 34]. In addition, decreased in negative ΔG° value with increasing temperature implies that the CO₂ adsorption process is more favorable at 25°C rather than at 50°C [32].

Table 4: Thermodynamics parameters of CO₂ adsorption onto Norit® SX2 and comparison with other work

Sample	ΔH° (kJ/mol)	ΔS° (kJ/mol·K)	ΔG° (kJ/mol)					Ref.
			298 K	323 K	348 K	373 K	393 K	
Norit® SX2	-19.657	-0.059	-1.788	-0.557	0.855	2.123	4.171	Present work
Coconut fiber AC	-14.980	-0.024	-7.653	n/a	n/a	n/a	n/a	[34]
Zeolite 4A	-12.852	-18.499	-7.299	n/a	n/a	n/a	n/a	[34]
RF resin	-4.200	0.020	n/a	-10.600	-10.9	-11.600	n/a	[35]

3.4. Comparison with Other Activated Carbons

Table 5 shows a comparison of the CO₂ adsorption capacities onto various types of activated carbons at temperature ranges of 25-100 °C. From this comparative study, it shows that the synthesized activated carbons from waste material have a comparable or considerably higher adsorption capacity compared to the commercial Norit®, wherein is considered as a benchmark. Thereby, it reflects further studies on the waste materials conversion to the carbonaceous adsorbent, and applied in the power plants as a medium for CO₂ separation. The utilization of these renewable waste materials is feasible as it overcomes the current problem that is depletion of the non-renewable precursors i.e. peat, which is being used for the Norit® SX2 formation. Besides, Khalili et al. [36] agreed that these activated carbons should be synthesized from the renewable feedstock in order to ensure a long-term sustainability of this industry. Furthermore, using the wastes as raw precursors is beneficial due to probability of low production cost, given that these waste materials are widely abundance in the country and can be freely obtained, or just incur small amounts of money. In terms of adsorption, the other works shows a similar trend whereby the adsorption capacity is the highest at the lowest bed temperature. In context of the post-combustion CO₂ capture whereby the flue gas temperature is around 40-60 °C, the adsorbent should have high sorption capacity with respect to temperature, and accordingly, surface modification may be needed. In terms of the modification process, the mesoporous carbon is preferred since it allows better distribution of the amine polymers during the impregnation and accordingly, it facilitates the CO₂ uptake at elevated temperature and improves the CO₂ selectivity too. On the other hand, textural properties and micropore structures play a dominant role in the CO₂ uptake at room temperatures (physisorption) [37]. Nevertheless, referring to Table 5, escalating CO₂ uptakes by bean dregs despite the mesoporous structures are owed to their physicochemical properties, which are predominantly composed of nitrogen, thus, enhances the CO₂ uptake via an acid-base interaction.

Table 5: Comparison study of maximum CO₂ adsorption capacity (mmol/g) on various adsorbents

Adsorbent	Types of activation	Porosity	Adsorption capacity, cm ³ /g (mmol/g)			Reference
			25-30 °C	50-75 °C	100-120 °C	
Norit® SX2 (peat)	Steam activation	Mesoporous	1.88	1.29	0.61	This work
Algae	KOH activation	Micro/mesoporous	1.39	0.41-0.21	n/a	[38]
Bean dregs	KOH activation	Mesoporous	4.24	1.80	n/a	[39]
Empty fruit bunch AC	KOH activation	Microporous	2.63	1.74	n/a	[40]
Coconut AC	CO ₂ activation	Microporous	1.79	1.27	0.43	[41]

4. Conclusions

Throughout the study, CO₂ adsorption at an ambient pressure and at different operating temperatures (25-100°C) has been measured by using a static volumetric instrument. Based on the experimental findings, it is concluded that the CO₂ adsorption onto the commercial activated carbon follows the physisorption behavior, whereby the CO₂ adsorption capacity increases with respect to pressure and decreases at elevated temperature. Various isotherm models such as the Freundlich, Langmuir, Dubinin Radushkevich, and Temkin are used to fit the experimental adsorption data. According to the analysis, the Freundlich isotherm is found to give the best fit to the experimental data over the whole temperature range, due to the highest of regression coefficient (R²) and closeness to unity, thus, implies a perfect fit to the experimental data. Besides, thermodynamic analysis proves that the CO₂ adsorption is a spontaneous process at low temperature, physisorption, and exothermic in nature. The isothermal data of this commercial activated carbon is significant, and can be considered as a baseline for a comparison with the synthesized activated carbons in future.

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