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## High Pressure Adsorption Isotherms of Carbon Dioxide and Methane on Activated Carbon From Low-Grade Coal of Indonesia

Awaludin Martin<sup>a</sup>, Muhammad Idrus Alhamid<sup>b</sup>, Nasruddin<sup>b</sup>, Bambang Suryawan<sup>b</sup>, Wai Soong Loh<sup>c</sup>, Azhar Bin Ismail<sup>c</sup>, Wonge Chus<sup>d</sup>, and Kim Choon Ng<sup>c</sup>

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### ABSTRACT

Adsorption isotherms data of methane and carbon dioxide gases on the activated carbons were measured experimentally using a volumetric method with pressure and temperatures ranging from 0 to 3.5 MPa and 27 to 65 °C, respectively. Two types of activated carbons, namely, (1) Kalimantan Timur type activated carbon, which is lab produced from Indonesian low-grade coal and (2) a commercial (Carbotech) activated carbon were used. The adsorption isotherms obtained were found to belong to type 1 of the International Union of Pure and Applied Chemistry classification. The adsorption uptakes for both carbon dioxide and methane on commercial activated carbon are higher than for the Kalimantan Timur activated carbon. This is due to higher Brunauer–Emmet–Teller surface area and pore volume of the former. Langmuir and Tóth isotherm models are correlated to predict the experimental data with acceptable accuracy.

### Introduction

Fossil fuels supply almost 98% of the world's energy consumption; however, they also contribute to emission of greenhouse gases such as carbon dioxide. Recognizing the fact that the major contributor to climate change is the emission of greenhouse gases resulting from human activities, particularly the combustion of fossil fuels, scientists/human beings all over the world are making strides to reduce emissions and capture carbon dioxide. The carbon dioxide is typically captured in dedicated geological reservoirs, in coal seams, or on ocean beds. These geological sequestrations are considered to have great potential for safe, economical, and effective storage of carbon dioxide. There are, however, some concerns, in terms of which the candidate sites should be carefully chosen, normally requiring extensive geological and economic studies.

Furthermore, the instability in oil markets and the increase in environmental concerns (i.e., Kyoto Protocol) have further stimulated research on alternative fuels for transportation in an effort to reduce greenhouse gaseous emissions. Among the alternative fuels being researched, natural gas has emerged as the most promising one. Indonesia has diversified its energy sources to include natural gas because of its great abundance.

Natural gas consists mainly of methane, typically at 80 to 95%, and hydrogen and carbon dioxide as minor impurities. However, some places exist where carbon dioxide contamination is as high as 10%. The maximum amount of carbon dioxide in natural gas should not exceed 2% in order to meet the international quality standard [1]. The carbon dioxide content is

important to prevent equipment and pipeline corruptions. Therefore, suitable gas separation and purification technology has become increasingly important in chemical and petrochemical industries.

The adsorption of gases and vapors by porous solid adsorbents has attracted much attention because of its great practical importance in the fields of gas separation and purification [2]. Three main applications where the adsorption is of interest include treatment of landfill gas (containing about 50% carbon dioxide as an impurity), purification of natural gas (containing up to 12% carbon dioxide), and tertiary oil recovery, where the effluent gas contains variable carbon dioxide/methane ratios.

Adsorption is found to be an effective method for the separation of carbon dioxide from natural gas. The separation processes are based on the differences in adsorption kinetics of the methane and carbon dioxide molecules on solid adsorbents. The carbon dioxide with smaller molecules (0.33 nm) will adsorb more quickly than the methane, which has larger molecules (0.4 nm) [3].

The information concerning the adsorption equilibrium and characteristics of adsorbent and adsorbate are essential for the analysis and design of adsorption separation processes. For practical applications such as gas separation and purification, the adsorption equilibrium must be known over a broad range of operation temperatures [4, 5]. Intensive research was performed to identify the adsorption of carbon dioxide with different adsorbents under different operating conditions [6–10].

In this research, the adsorption isotherms of methane and carbon dioxide with two types of adsorbents, namely, the

lab-produced Kalimantan Timur type activated carbon and the commercial activated carbons, were measured, using the volumetric (i.e., constant-volume-variable-pressures, CVVP) method. Experiments were performed at temperatures ranging from 27 to 65°C and pressures up to 3.5 MPa. These data were correlated with the Langmuir and Tóth isotherm models with fairly good accuracy (within 5.5%); therefore, these models can be used in designing the gas separation system.

## Experimental facility

### Materials

High-purity methane and carbon dioxide were used in the experiment. Two types of solid adsorbent activated carbons (AC), namely, Carbotech (commercial) and lab-produced activated carbon Kalimantan Timur (KT) from Indonesian low-grade coal, were studied. Both the adsorbents are of granular type; Carbotech and KT activated carbons have particle sizes of 0.063 and 0.1 mm, respectively. The KT activated carbon was prepared from a physical activation process. Prior to the activation process, the fresh coal from East Kalimantan, Indonesia, was crushed and sieved to particles of 0.10 mm size. The physical activation of the KT activated carbon was completed in a vertical autoclave activation reactor placed inside a furnace; this includes the carbonization/oxidation and activation processes. The fresh coal was first heat treated by flowing  $O_2$  at 0.1 L  $min^{-1}$  at 300°C for 6 h. Following the carbonization process, the char samples were activated in a stream of  $CO_2$  at 0.15 L  $min^{-1}$ . The samples were activated from room temperature to maximum heat treatment temperatures of 950°C for 6 h. A scanning electron micrograph (SEM) of the lab-produced activated carbon at 500 times magnification is shown in Figure 1. Table 1 presents the structural characteristics (pore volume and surface area) of the aforementioned activated carbons, which were measured using the Autosorb (Quantachrome gas sorption instrument) machine. The surface area was calculated by the Brunauer–Emmett–Teller (BET) method from the nitrogen adsorption isotherm data at 77.3 K. The pore size distributions

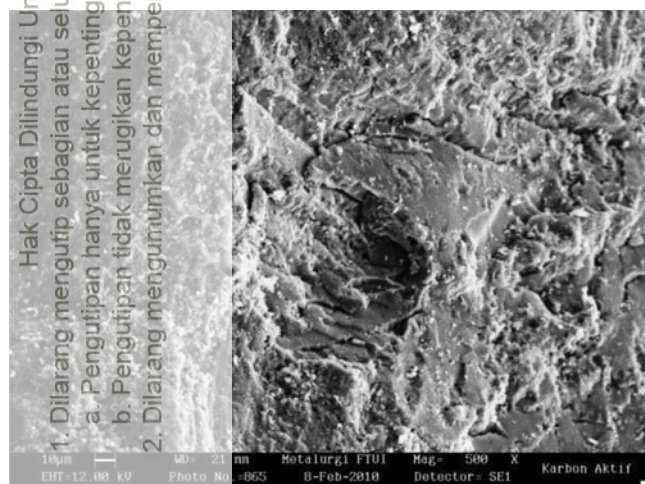
**Table 1.** Structural characteristics of KT and Commercial (Carbotech) activated carbon.

Activated carbon	Micropore volume, $m^3 \cdot g^{-1}$	Surface area, $m^2 \cdot g^{-1}$
Commercial	$0.514 \times 10^{-7}$	885
KT	$0.470 \times 10^{-7}$	668

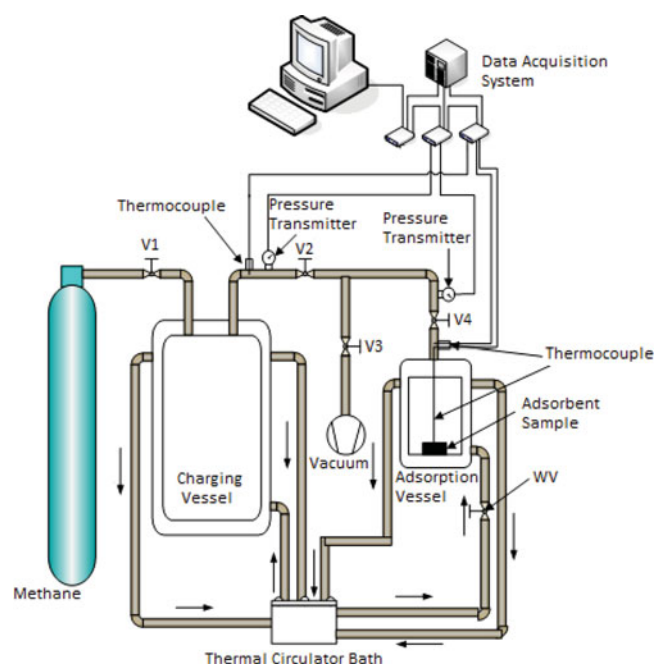
(PSD) of these activated carbons have been obtained by the non-local density functional theory (NLDFT) method for a relative pressure equal to 0.98 [11, 12]. The commercial AC is found to have higher BET surface area than the KT AC, that is, 885  $m^2 \cdot g^{-1}$  and 668  $m^2 \cdot g^{-1}$ , respectively. Similarly, the commercial AC has larger micropore volume ( $0.514 \times 10^{-7} m^3 \cdot g^{-1}$ ) compared to the KT AC ( $0.470 \times 10^{-7} m^3 \cdot g^{-1}$ ).

### Apparatus and procedures

Adsorption isotherms of methane,  $CH_4$ , and carbon dioxide,  $CO_2$ , with activated carbons (KT and Carbotech) were obtained using a volumetric method at temperatures ranging from 27 to 65°C and pressures up to 3.5 MPa. A schematic diagram of the experimental apparatus is shown in Figure 2. The experimental apparatus consists mainly of a stainless-steel measuring or adsorption cell and a charging cell (control volume) with internal volumes of 84.02 ml and 1150.98 ml, respectively. The measuring cell and charging cell are connected through a capillary tube. The solid adsorbents were packed in the measuring cell. A Class-A type K thermocouple probe was inserted into the measuring cell, in contact with the solid adsorbent, to measure the adsorbent temperature directly throughout the isotherm test. All temperature and pressure readings were monitored with a data logging system with sampling intervals of 10 s. The detailed experiment procedures were described previously by Martin et al. [13].



**Figure 1.** Scanning electron micrographs (SEM) photo of KT activated carbon at 500 times magnification.



**Figure 2.** Schematics diagram of the experiment apparatus.



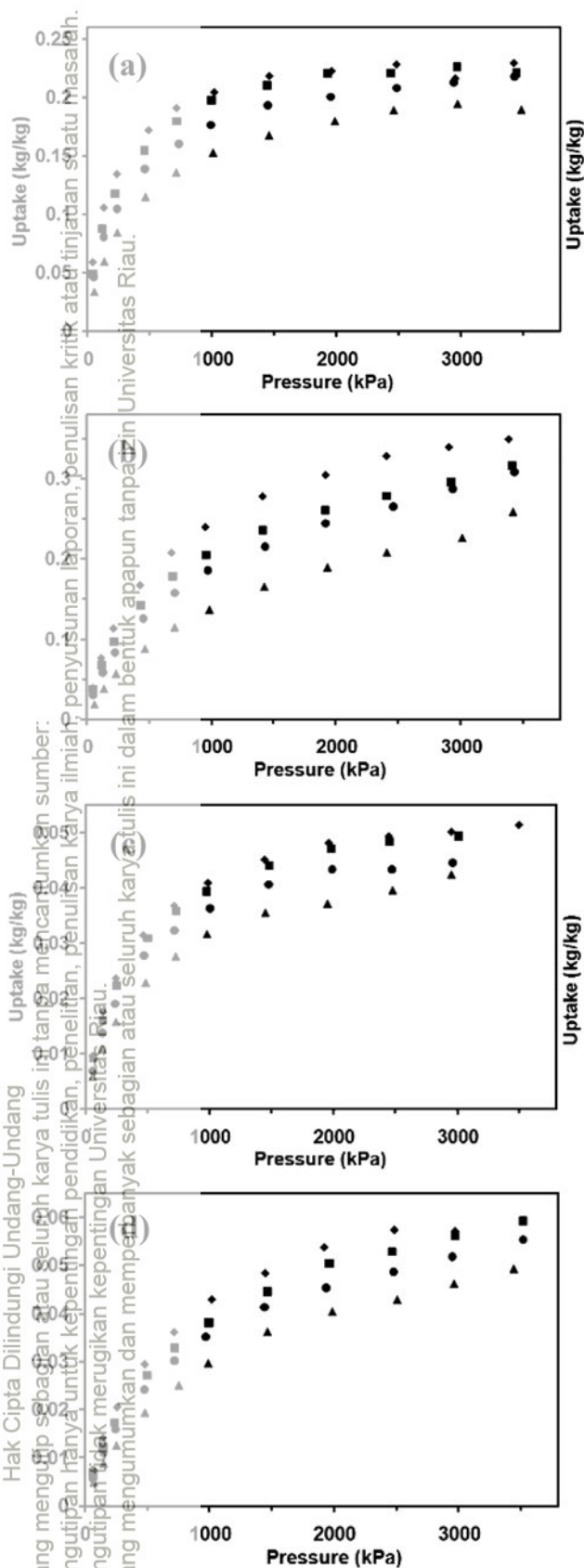


Figure 3. Adsorption isotherms of (a)  $\text{CO}_2$  on KT activated carbon, (b)  $\text{CO}_2$  on commercial activated carbon, (c)  $\text{CH}_4$  on KT activated carbon, and (d)  $\text{CH}_4$  on commercial activated carbon at  $\blacklozenge$  27°C,  $\blacksquare$  35°C,  $\bullet$  45°C, and  $\blacktriangle$  65°C.

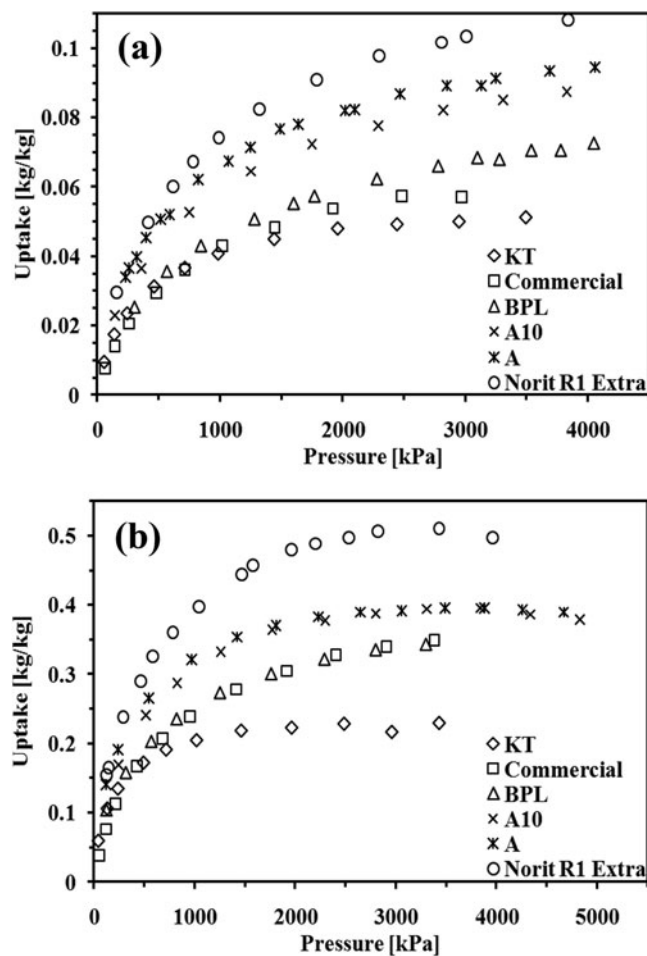


Figure 4. Adsorption isotherms of (a)  $\text{CH}_4$  and (b)  $\text{CO}_2$  with  $\diamond$  KT,  $\square$  commercial,  $\triangle$  BPL,  $\times$  A10,  $\ast$  A, and  $\circ$  Norit R1 Extra activated carbons at 25°C.

### Adsorption isotherm

Adsorption depends mainly on the nature of the solid surface and of the solvent medium. Solid adsorbents with highly developed porous surfaces tend to adsorb gases and vapors. The adsorption capacity under equilibrium condition is the function of both the system temperature and pressure, as well as the thermophysical properties of the adsorbent and adsorbate.

For a given adsorbent–adsorbate system, the adsorption equilibrium data can be expressed as

$$C = C(P, T) \quad (1)$$

where  $C$  is the amount adsorbed per unit mass of adsorbent,  $P$  is the equilibrium pressure, and  $T$  is the equilibrium temperature.

Two adsorption isotherm models, namely, (i) Tóth and (ii) Langmuir, are used to correlate the experimental isotherm data. The Tóth model is commonly used to describe the adsorption behavior of heterogeneous adsorbents such as activated carbon. It can correctly predict the adsorption behavior at both low- and high-pressure regions [7]. However, the Langmuir model has limitations on describing the adsorption behavior at the high-pressure region and material heterogeneity.



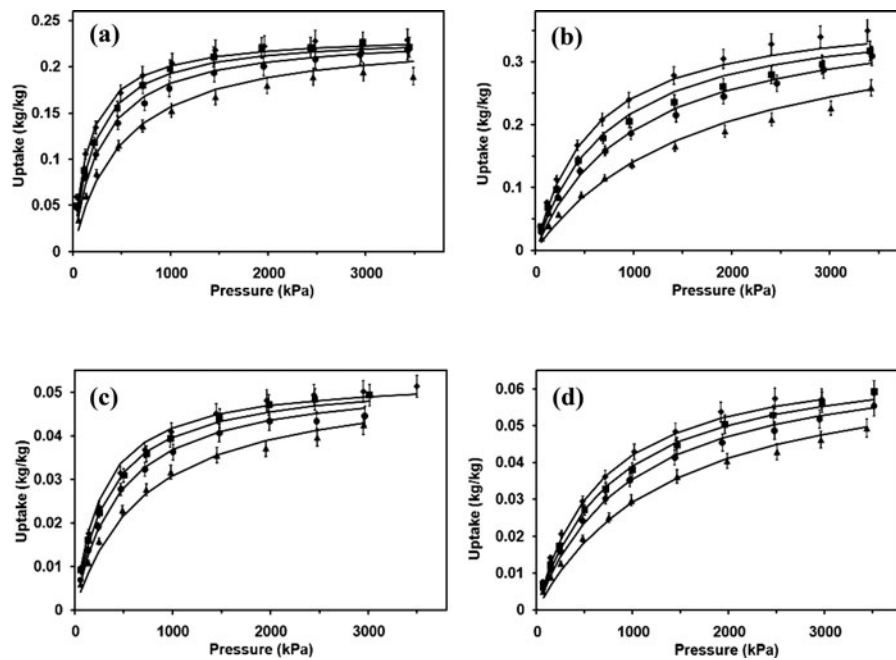


Figure 5. Adsorption isotherms of (a) CO<sub>2</sub> on KT activated carbon, (b) CO<sub>2</sub> on commercial activated carbon, (c) CH<sub>4</sub> on KT activated carbon, and (d) CH<sub>4</sub> on commercial activated carbon at 27°C, 35°C, 45°C, and 65°C. Solid lines represent the Langmuir equation fit.

The Langmuir and Tóth models are presented by Eqs. (2) and (3), respectively:

$$C = \frac{k_0 \cdot \exp(h_{st}/RT) P}{[1 + (k_0 \exp(h_{st}/RT) P)^t]^{1/t}} \quad (2)$$

$$C = \frac{k_0 \cdot \exp(h_{st}/RT) P}{1 + k_0 \exp(h_{st}/RT) P} \quad (3)$$

where  $C$  is the surface coverage or fractional filling of the micropore,  $C_0$  is the adsorbed phase concentration at equilibrium,  $P$  is the limiting adsorption capacity of the adsorbent,  $P$  and  $T$  are the equilibrium pressure and temperature, respectively,  $k_0$  is the equilibrium constant,  $h_{st}$  is the isosteric heat of adsorption,  $R$  is the universal gas constant, and lastly,  $t$  is the parameter that indicates the heterogeneity of the adsorbent. The detailed data were previously presented by Loh et al. [14].

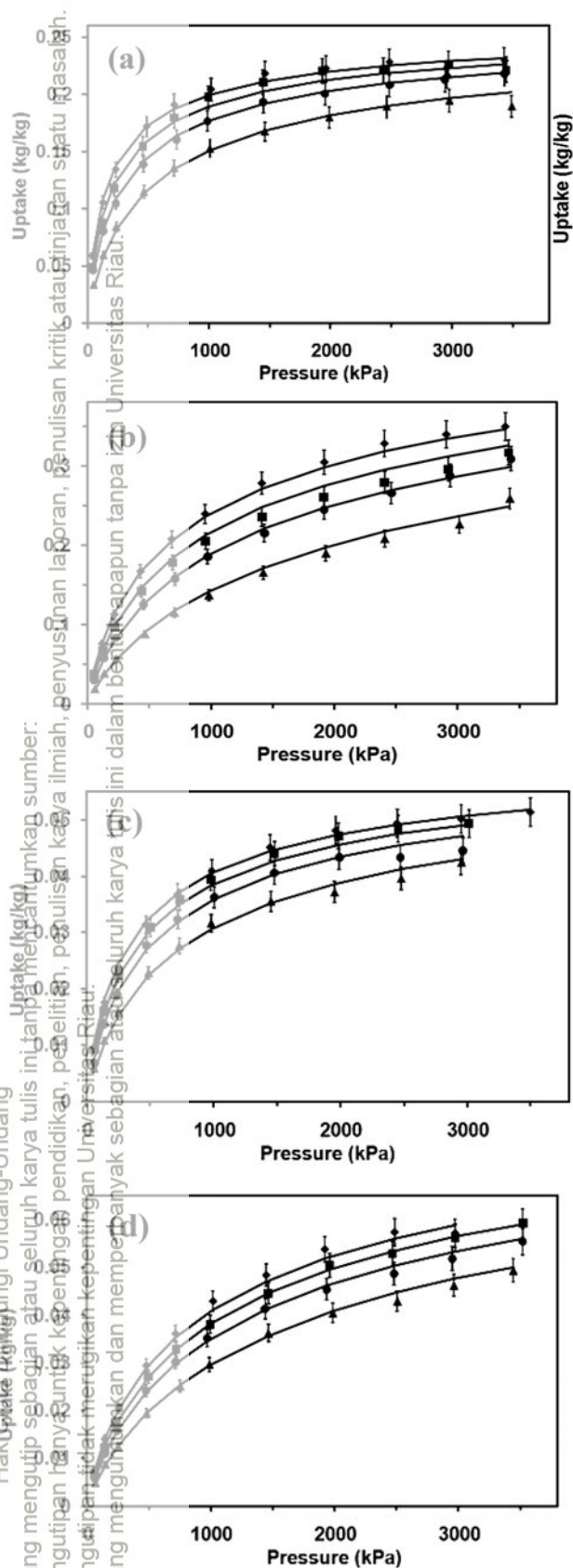
## Results and Discussion

Experimental data for the adsorption of CO<sub>2</sub> and CH<sub>4</sub> with KT activated carbons (lab-produced) and Carbotech were obtained for temperatures ranging from 27 to 65°C and pressures up to 3.5 MPa. The experiments focus on the conditions that are found in typical gas separation or storage processes. The adsorption equilibrium uptakes data are plotted in the pressure-concentration-temperature ( $P$ - $C$ - $T$ ) plane and graphically presented in Figure 3. The adsorption isotherms for KT and Carbotech activated carbons with CO<sub>2</sub> at 27, 35, 45, and 65°C are presented in Figures 3a and 3b, respectively. The equilibrium uptakes of CO<sub>2</sub> on KT and commercial activated carbons at 27°C and 3.5 MPa are measured as 0.23 kg kg<sup>-1</sup> and 0.322 kg kg<sup>-1</sup>, respectively. Similarly, Figures 3c and 3d illustrate the uptakes of CH<sub>4</sub> with KT and Carbotech activated carbons. The equilibrium uptakes for CH<sub>4</sub> with KT and commercial AC at 27°C and at 3.5 MPa are found to be 0.0532 kg kg<sup>-1</sup> and 0.0589 kg kg<sup>-1</sup>.

The equilibrium uptakes of the KT activated carbon are lower compared to the commercial activated carbon owing to a lower BET surface area and pore volume.

The present experimental adsorption uptakes of CH<sub>4</sub> and CO<sub>2</sub> are compared with previous work by Himeno et al. [7]. The equilibrium uptakes at 25°C for CH<sub>4</sub> and CO<sub>2</sub> with assorted activated carbons are shown in Figures 4a and 4b, respectively. All the experiments covered a pressure range up to 3.5 MPa. It is found that the isotherms of the current commercial and KT activated carbons have similar trends with the BPL, A10, A, and Norit R1 Extra activated carbons. The isotherms obtained were found to belong to type 1 of the International Union of Pure and Applied Chemistry (IUPAC) classification [15]. The uptake capacity is lower for both CH<sub>4</sub> and CO<sub>2</sub> of the existing adsorbent (KT and adsorbent), compared to the other activated carbons (BPL, A10, A, and Norit R1 Extra). This is mainly due to the physical characteristics of the adsorbent, since the Carbotech and KT activated carbons have lower micropore volume and BET surface area. The adsorbent with higher microporous structure tends to adsorb more vapors—that is, it has higher equilibrium uptake.

The Langmuir and Tóth isotherm models are used to correlate the experimental data. Figures 5 and 6 depict the Langmuir and Tóth isotherm models of the assorted adsorbent/adsorbate pairs (CO<sub>2</sub>/KT, CO<sub>2</sub>/Commercial AC, CH<sub>4</sub>/KT, and CH<sub>4</sub>/Commercial AC), respectively. The adsorption parameters for Langmuir and Tóth models and associated deviations from experimental data are furnished in Table 2. The Langmuir model gives the highest average regression error for the CO<sub>2</sub>/Commercial AC pair of about 5.93%, presented in Figure 5b. Meanwhile, the Langmuir model predicted the uptake of CH<sub>4</sub> with KT and commercial AC with average regression error of less than 3.87%, as shown in Figures 5c and 5d, respectively. On the other hand, the Tóth model predicted all the equilibrium isotherm uptakes with average regression error of less



**Figure 6.** Adsorption isotherm of (a)  $\text{CO}_2$  on KT activated carbon, (b)  $\text{CO}_2$  on commercial activated carbon, (c)  $\text{CH}_4$  on KT activated carbon, and (d)  $\text{CH}_4$  on commercial activated carbon at  $\blacklozenge$  27°C,  $\blacksquare$  35°C,  $\bullet$  45°C, and  $\blacktriangle$  65°C. Solid lines represent the Tóth equation fit.

**Table 2.** Adsorption parameters of Langmuir and Tóth models.

Model	Parameter	Commercial AC		KT AC	
		$\text{CO}_2$	$\text{CH}_4$	$\text{CO}_2$	$\text{CH}_4$
Langmuir	$C_o$ ( $\text{kg kg}^{-1}$ )	0.385	0.0698	0.235	0.0534
	$h_{st}/R$ (K)	2910.6	1988.096	2888.44	2588.64
	$k_o$ ( $\text{kPa}^{-1}$ )	$1.06 \times 10^{-7}$	$2.01 \times 10^{-6}$	$3.92 \times 10^{-7}$	$2.26 \times 10^{-6}$
	$t$	—	—	—	—
Tóth	Deviation (%)	5.93	3.56	4.166	3.87
	$C_o$ ( $\text{kg kg}^{-1}$ )	0.5439	0.097	0.2627	0.0638
	$h_{st}/R$ (K)	3030.18	1835.79	3131.9	2304.47
	$k_o$ ( $\text{kPa}^{-1}$ )	$1 \times 10^{-7}$	$3.91 \times 10^{-6}$	$3.01 \times 10^{-7}$	$2.15 \times 10^{-6}$
	$t$	0.575	0.614	0.6799	0.6756
	Deviation (%)	3.72	2.4	2.645	2.67

**Table 3.** Heat of adsorption of activated carbons with  $\text{CO}_2$  and  $\text{CH}_4$  derived from langmuir model.

	$h_{st}$ ( $\text{J g}^{-1}$ )		
	Honeycomb monolith [9]	Commercial AC	KT
$\text{CO}_2$	545.71	549.89	545.71
$\text{CH}_4$	1325.69	1030.39	1341.65

than 3.72%. Hence, it is concluded that the Tóth model is more suitable and can accurately predict the uptake behavior of  $\text{CO}_2$  and  $\text{CH}_4$  on the activated carbons in the current study.

Heat of adsorption is a specific combined property of a particular adsorbent/adsorbate pair. It contributes mainly to the internal temperature rise of solid adsorbent during the adsorption process. In this current study, the heat of adsorptions derived from the Langmuir model were compared with previous work [10], as presented in Table 3. The heats of adsorption of  $\text{CO}_2$  with KT activated carbon and honeycomb monolith are about  $545.71 \text{ J g}^{-1}$ , whereas for Carbotech AC the heat of adsorption is calculated as  $549.9 \text{ J g}^{-1}$ . Meanwhile, the heats of adsorption for  $\text{CH}_4$  with KT activated carbon and honeycomb monolith are between 1325 and  $1345 \text{ J g}^{-1}$ . However, for commercial activated carbon, the heat of adsorption is about  $1030 \text{ J g}^{-1}$ . The KT activated carbon and honeycomb monolith have similar heat of adsorption.

## Conclusions

In this paper, adsorption equilibrium of pure carbon dioxide ( $\text{CO}_2$ ) and methane ( $\text{CH}_4$ ) on activated carbon has been measured using the volumetric method at pressures up to 3.5 MPa and temperatures from 27 to  $65^\circ\text{C}$ . Activated carbon derived from East Kalimantan coal has a lower adsorption capacity than the commercially activated carbon. This is due to the former having lower BET surface area and micropore volume. This characteristic of lower porosity is mainly because the KT activated carbon is produced solely by a mechanical crushing process. Additionally, the experimental isotherms data are found to follow trends similar to previous studies. The Langmuir and Tóth adsorption isotherm models are employed to correlate the experimental isotherm data with reasonable accuracy, that is, an average regression error of less than 6%. In this current study, the Tóth equation is found to provide a more accurate correlation than the Langmuir model.



## Nomenclature

AC	activated carbons
C	adsorbed phase concentration at equilibrium
$C_o$	limiting adsorption capacity of the adsorbent, kg/kg adsorbent
$h_{st}$	isosteric heat of adsorption, kJ/kg
$k_o$	equilibrium constant, 1/kPa
KT	Kalimantan Timur
P	equilibrium pressure, Pa
R	universal gas constant, kJ/kg-mol-K
T	equilibrium temperature, °C
t	parameter that indicates the heterogeneity of the adsorbent

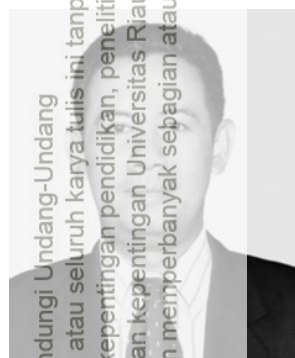
## Greek symbol

$\emptyset$	surface coverage or fractional filling of the micropore
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in various research activities in exploiting potential energy resources such as waste heat and methane hydrate. He has published more than 80 articles in peer-reviewed journals and has served on the editorial board of international journals.



**Kim Choon Ng** obtained the B.Sc. (Hons.) and Ph.D. from Strathclyde University in Glasgow (UK) in 1975 and 1980, respectively. He worked briefly at Babcock Power Ltd. in Renfrew, UK, prior to joining the Department of Mechanical Engineering of the National University of Singapore in 1981, and he is now a tenured full professor. His areas of research are two-phase flows, chiller testing and modeling, electro adsorption chillers, adsorption desalination, and renewable energy systems. He has written more than 90 peer-reviewed

journals, holds patents, and co-authored a book, *Cool Thermodynamics*, printed by CISP (UK) in 2000. He is a member of the IMechE (UK) and the Institution of Engineer Singapore, a chartered engineer (UK), a registered professional engineer (S), and is an associate editor for two international journals.

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