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CO_2 CAPTURE USING GRAPHITE WASTE COMPOSITES AND CERIA

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ABSTRACT

Solid sorbents based on graphite electrode waste and cerium oxide (ceria, CeO_2) have been studied with regard to CO_2 capture. The acid-base properties of cerium oxide produce a sorbent for the capture of CO_2. The aim of the study is to evaluate the performance of CO_2 capture using graphite/CeO_2 composites at different weights of $Ce(NO_3)_3.6H_2O$ (0.5, 1 and 2 g), namely G0.5, G1 and G2, respectively. Volumetric adsorption studies of CO_2 on graphite/CeO_2 composites and ceria were conducted at various pressures (P) of 3, 5, 8, 15 and 20 bar, and temperatures (T) of 303, 308, 318 K. Graphite waste before modification (GBM), activated graphite waste (GA), and CeO_2 for capturing CO_2 were also investigated. By varying the two parameters (P and T), we found that the maximum adsorption capacities of CO_2 at 303 K and 20 bar were 0.0713, 0.0316, 0.1574, 0.0987, 0.1137, and 0.0964 kg/kg respectively, for GBM, GA, G0.5, G1, G2 and CeO_2. The highest adsorption capacity of CO_2 was found in the G0.5 composite. The adsorption performance of CO_2 using ceria was almost similar to the G1 composite. We found that CO_2 adsorption capacity decreases with an increasing temperature from 303 to 318 K. It was concluded that ceria and composite graphite waste/CeO_2 are stable and selective CO_2 sorbents. The work allows us to synthesize a new sorbent which can be effectively applied for CO_2 capture. The adsorption capacity of CO_2 depends significantly on the active site and chemical modifier of the sorbents.

Keywords: Acid-base properties; Ceria; CO_2 Capture; Graphite/CeO_2; Volumetric adsorption

1. INTRODUCTION

Controlling the rising carbon dioxide (CO_2) level in the atmosphere is a major issue, as the gas caused global warming (Hemalatha et al., 2012). Specifically, the accumulation of carbon dioxide (CO_2) emitted into the atmosphere, particularly from fossil fuel combustion in vehicles, industries and power plants, is the cause of this problem (Pham et al., 2016; Zhang et al., 2017). The average CO_2 concentration in the atmosphere was 400 ppm in 2015 (Szczęśniak et al., 2017). Clean energy sources and global warming have been among the major issues and challenges for many years, including a reduction in CO_2 (Krooss et al., 2002). There have been several efforts to reduce emissions of the gas, most centred on a decrease in the use of fuel oil.

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However, natural or bio-gas, which have been explored as replacements for fuel oil, still contain large amounts of CO_2, which decreases the calorific value of the gas combustion. Therefore, a purification process is needed to remove CO_2 from the fuel gas. On the other hand, CO_2 is abundant, non-toxic and easily available, and can also be converted into useful products, such as for renewable carbon resources (Kamimura et al., 2014).

Many technologies for capturing CO_2 using the chemical absorption of amine solution have been reported (Du et al., 2017; Deiana et al., 2017). The reaction is influenced by the temperature of absorption, pressure and the pH value of the amine solution (Deiana et al., 2017). Some conventional primary, secondary, tertiary and polyamines solvents are used for capturing CO_2 from various industrial process (Singto et al., 2016; Deiana et al., 2017; Bernhardsen & Knuutila, 2017; Du et al., 2017). A state-of-the-art process for capturing CO_2 from largely exhausted flue gases has been developed by employing amine-based and regenerative chemical absorption using a primary amine solvent such as aqueous monoethanolamine (MEA) solution (Wang et al., 2011; Singto et al., 2016; Deiana et al., 2017). A primary amine acts as a weak alkaline and forms a stable carbamate when it reacts with CO_2 (Bernhardsen & Knuutila, 2017). Absorption employing amines is an energy-intensive and complex process, requiring a high-energy input for regeneration and handling of the corrosive solvents resulting from the oxidative degradation of the amine-based solutions. The amine solution for capturing CO_2 has disadvantages, such as being toxic, flammable and corrosive for some equipment; a high regeneration energy, having a low absorption rate, and suffering amine loss by degradation and volatilization (Du et al., 2017; Zhang et al., 2017). It is a challenge to develop a solid sorbent for the capture of CO_2 in the form of a promising technology, as the solid sorbent has the advantage of being energy saving with a stable performance (Yoshikawa et al., 2014; Zhang et al., 2017; Wang et al., 2017).

To obtain CO_2 adsorbents with high selectivity, high capacity, high hydrothermality, good mechanical properties, chemical stability, low synthesis cost and also fast adsorption/desorption kinetics has long been an attractive field of investigation (Pevida et al., 2008; Díez et al., 2015; Sarkera et al., 2017; Szczęśniak et al., 2017; Acar et al., 2018). The adsorption process using certain adsorbents is one of the most effective methods for separating or capturing CO_2, due to its easy operation and low energy requirements (Lee & Park, 2015; Pham et al., 2016). This process is widely applied in pollution control, waste management, reaction catalysts, gas storage, cooling systems, and gas purification (Elsayed et al., 2017). The production cost of the adsorbent is also a factor which needs to be considered in order to achieve an economically viable process (Szczęśniak et al., 2017). In this regard, graphite waste is of interest due to its abundance (Kusrini et al., 2018b), low price and the fact that it contains a high level of carbon, so it has porous structure. CO_2 has high polarizability and quadrupole energy, thus is conveniently adsorbed in solid materials at room temperature (Hemalatha et al., 2012).

The main requirement of an adsorbent is that it is composed of a highly porous material such as zeolite, activated carbon, graphite, zeolite modified MgO, graphitic carbon nitride nanosheets, graphite oxide (GO), graphitic nanostructure, or graphene (Mishra & Ramaprabhu, 2011a; Hemalatha et al., 2012; Casco et al., 2014; Díez et al., 2015; Shin et al., 2016; Acar et al., 2017; Sarkera et al., 2017; Ghosh & Ramaprabhu 2017; Zukal et al., 2017; Babu et al., 2017), so that adsorption can occur on the pore walls or at certain locations within the particle. To increase the adsorption capacity of graphite waste, the surface functional group can be modified by using a metal oxide (Li et al., 2017; Slostowski et al., 2017). Some studies have reported that various metal oxides such as Li_2O are capable of adsorbing CO_2 molecules on their surfaces (Baltrusaitis & Grassian, 2005; Mosqueda et al., 2006; Hornebecq et al., 2011).

CO_2 has the capability associated with the surface of oxides such as cerium oxide (CeO_2) due to its acid-base properties (Slostowski et al., 2017). CeO_2 has been widely used in catalysis and adsorption (Yoshikawa et al., 2014; Jamalzadeh et al., 2013). The presence of metal oxides can produce a significant improvement in the CO_2 capture due to the CO_2 interplay (Li et al., 2017). CeO_2 can furnish a favorable catalytic activity, and offer a large area of chemical surface and a high surface reactivity. In addition, CeO_2 represented a very easily to adsorb water and/or carbon dioxide molecules at room temperature, has reported based of the FTIR spectra (Slostowski et al., 2017).

Previously, the applicability of graphite waste and graphite waste/Fe_3O_4 composites as sorbents for capturing CO_2 has been reported (Kusrini et al., 2017), including improved CO_2 adsorption capacities using graphite waste on magnetic nanoparticle Fe_3O_4 impregnation. A study by Kusrini et al. (2018a) has reported the synthesis and development of graphite waste/CeO_2 composites synthesized using pre-treated by mechanical-thermal methods and then followed by chemical modification with Ce salt ($Ce(NO_3)_3.6H_2O$). It is expected that the presence of Ce^{3+} ions on the surface of the graphite waste is able to change the structure of the graphite surface, supply more redox-active sites and elevate the surface area. Thus, new knowledge on the applicability of graphite/CeO_2 composites and ceria (CeO_2) for capturing CO_2 has not been reported. Driven by these findings, in this study we focus on the adsorption of CO_2 using graphite/CeO_2 composites and CeO_2. Their adsorption isotherm was evaluated by a volumetric method.

2. METHODOLOGY

2.1. Materials

The graphite/CeO_2 composites were prepared according to the method reported by Kusrini et al. (2018a), and were used as the adsorbent for CO_2 capture in this study. $Ce(NO_3)_3.6H_2O$ and CeO_2 were purchased from R & M Chemicals (Essex, UK). CO_2 gas with a high purity of 99.9% was used as the adsorbate and helium gas (He) as a relatively unabsorbed substance in the degassing process. The isothermal adsorption of CO_2 on the adsorbents was carried out to obtain the gas absorption capacity data at a pre-determined temperature and pressure.

2.2. Characterization

The surface areas of all the samples were characterized by Brunauer-Emmett-Teller (BET), based on a multipoint adsorption-desorption isotherm of N_2 at 77K using ASAP 2020 V4.02 (Micromeritics, US), an equilbration interval of 5 seconds, no low-pressure dose and analysis bath temperatures at 77K. Prior to BET analysis, the sample was subjected to degassing under high vacuum at 350°C for 4 hours.

2.3. Volumetric Adsorption Isotherms

To determine the adsorbent isotherms including gas flow, volumetric and gravimetric methods are used (Elsayed et al., 2017). The volumetric adsorption technique is more commonly used and has the advantage of simplicity of design and cost effectiveness (Siriwardane et al., 2001; Elsayed et al., 2017). The volumetric measurements are the pressure, volume and temperature when the adsorbate contacts the adsorbent. After equilibrium is reached, the amount of CO_2 adsorbed is calculated from the pressure changes using the ideal gas equation. In this study, we used the volumetric method.

The adsorption capacity of CO_2 by graphite/CeO_2 composites was determined using adsorption isotherms by the volumetric principle. The adsorption experiments were performed over the pressure range of 3–20 bar and each cycle of adsorption was followed by the degassing of CO_2 at 423 K under vacuum conditions.

The adsorption experiments were carried out at different temperatures, 303, 308, and 318 K, and various pressures, 3, 5, 8, 15 and 20 bar. A mass of 2.6 g of graphite waste as sorbent was used. In this study, five types of composites, namely graphite waste before modification (GBM), activated graphite waste (GA), and modified graphite waste with various weights of cerium oxide (CeO_2) (0.5, 1, and 2 g), denoted by G0.5, G1, and G2, respectively, were used. The adsorption capacity of CO_2 was calculated to be the weight (kg) of desorbed CO_2 per weight of sorbent (kg).

3. RESULTS AND DISCUSSION

3.1. CO_2 Adsorption Measurement

The adsorption capacity of CO_2 using the GBM, GA, graphite/CeO_2 composites and also ceria (CeO_2) versus the gas pressure are presented in Figure 1(A-F). The amount of adsorbed CO_2 increased when the temperature was decreased from 31 K to 303 K. A similar phenomeon can be seen in CO_2 adsorption using mesoporous ceria, zeolite and activated carbon, as previously reported (Kamimura et al., 2014). On the contrary, when increasing the pressure from 3 to 8 bar, the adsorption capacity of CO_2 increased. This pressure-dependent adsorption capacity indicates that the speed or flow rate of the CO_2 adsorbed into the surface of graphite/CeO_2 composites increases with CO_2 pressure. The decrease in the adsorption capacity with temperature can be related to its adsorption isotherm; in such a physisorption process the adsorption capacity decreases with temperature due to the condensation of the CO_2 gas on the surface of the adsorbent. The amount of adsorbate adsorbed on the surface is higher at a lower temperature.

For GBM, it appears that the maximum adsorption capacity of CO_2 is 0.0713 kg/kg at a temperature of 303 K and a pressure of 20 bar (see in Figure 1a). The graph shows a linear increase in adsorption capacity after reaching a pressure of 8 bar, especially on the graph line temperature at 318 K. The maximum adsorption capacity of CO_2 using activated graphite waste (GA) is 0.0316 kg/kg (see in Figure 1b). The resulting graph is linear, with increasing adsorption capacity as pressure increases.

In particular, for G0.5 and G1, an increase in adsorption capacity was only observed at high pressure above 8 and 15 bar, respectively. For the pressure of 20 bar and temperature of 303 K, the maximum adsorption capacities of CO_2 are 0.0713, 0.0316, 0.1574, 0.0987 and 0.1137 kg/kg respectively, for GBM, GA, G0.5, G1 and G2. It can be seen that by adding metal salt ($Ce(NO_3)_3.6H_2O$) to the activated graphite waste using the wet impregnation method, the adsorption capacity of CO_2 was slightly different. It is similarly observed that the adsorption capacity did not correlate with the loaded metal oxides, in terms of surface chemistry (Li et al., 2017). The chemically modified graphite waste with CeO_2 tends to have a higher adsorption capacity. Incorporation of CeO_2 on the graphite surface made it become more reactive and have a higher adsorption ability due to the formation of interaction between Ce^{3+} ions and oxygen from the CO_2 molecules to form bridged, bidentate, monodentate and/or polidentate manners (Yoshikawa et al., 2014; Slostowskia et al., 2017). Strong interaction with oxygen from CO_2 molecules occured when introduced the loading metal oxide onto the graphite waste because of the different surface chemistry of oxide to the carbon surface of graphite.

The adsorption performance of CO_2 using CeO_2 as adsorbent was also evaluated in the same operating conditions. Figure 1F shows the adsorption isotherm of CO_2 using CeO_2; the adsorption capacity also decreases with temperature. It is similarly observed that CO_2 adsorption capacity decreases with increasing temperature using graphene and graphene-based materials as sorbents, as reported in a previous study (Szczęśniak et al., 2017).

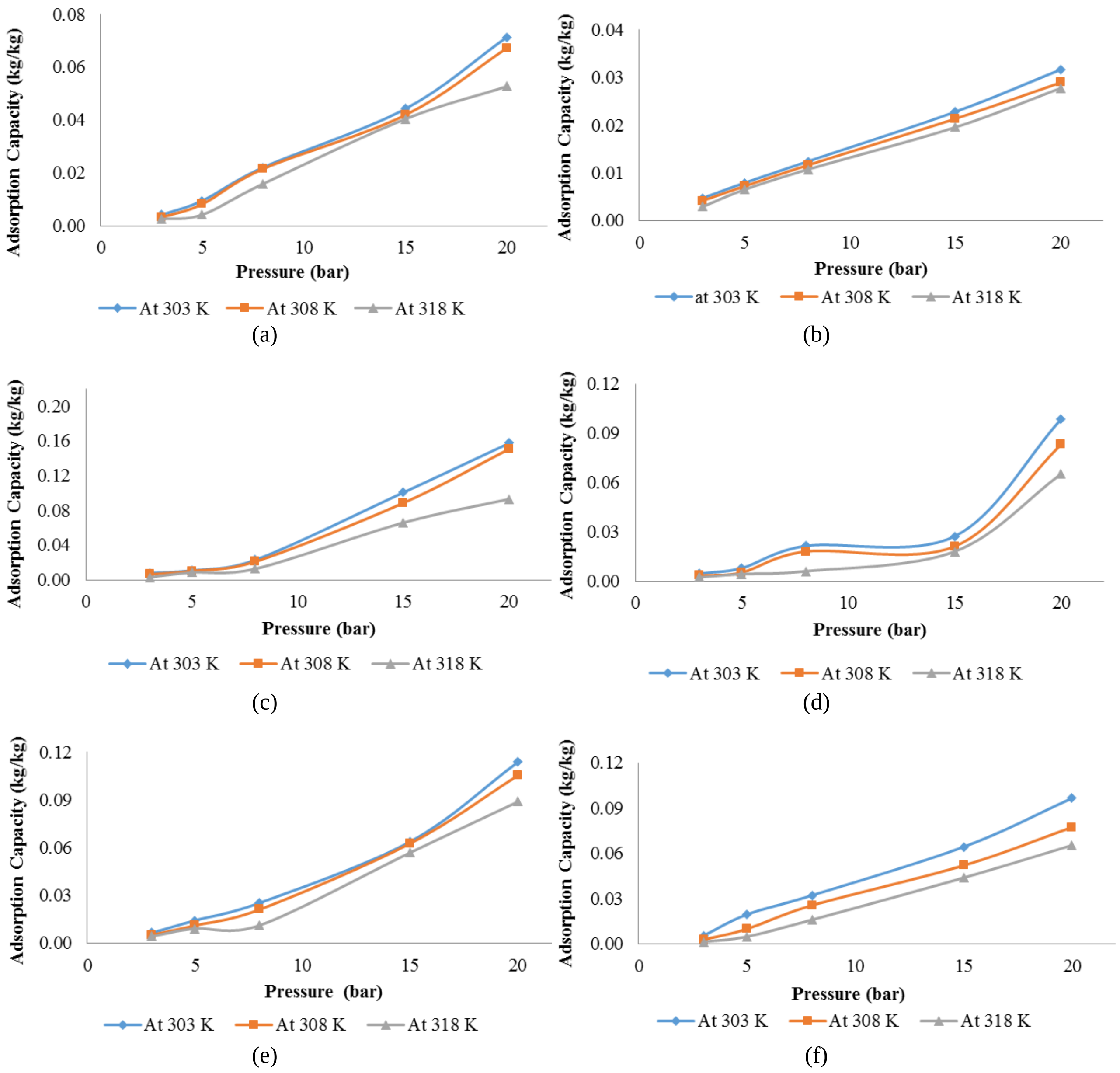

Figure 1 CO_2 adsorption isotherms for the: (a) GBM; (b) GA; (c) G0.5; (d) G1; (e) G2; and (f) CeO_2 with pressure from 3 to 20 bar at temperatures of 303, 308 and 318 K

In this study, the maximum adsorption capacity of CO_2 using CeO_2 is 0.0964 kg/kg at 303 K and 20 bar. This value is comparable to that of the G1 composite (0.0987 kg/kg). This finding may be related to a high affinity of CO_2 to interact with the surface of CeO_2. It can be understood that CO_2 molecules are a Lewis acid, while surface oxygen and/or hydroxyl species in the CeO_2 network act as Lewis basic sites. The CO_2 could also form hydrogencarbonate and bridged carbonate, producing a weak interaction at room temperature (25°C) (Slostowskia et al., 2017), while by increasing the temperature from 573 to 773K a strong interaction with the CeO_2 surface is formed in monodentate, bidentate and polydentate carbonate manners (Yoshikawa et al. 2014; Slostowskia et al., 2017). Although various formations of carbonate species were considered, the interaction of CO_2 with the surface of CeO_2 seems to be energetically low, where the isosteric heat of adsorption of as-made mesoporous CeO_2 was in the range of 30–43 kJ/mol at low coverage of CO_2 (Kamimura et al., 2014). Therefore, physisorption mainly takes place, rather than chemisorption.

In this study, the adsorption performance of graphite waste, graphite waste/CeO_2 composites, and CeO_2 compared to commercial adsorbents such as activated carbon for capturing CO_2, where AC2K-300 (activated carbon washed with HCl, then oxidized with air, followed by 10% K_2CO_3 impregnation and calcination at 300°C) had the highest adsorption capacity, of 110 mg/g at 1000 mbar CO_2 and at 25°C (Acar et al., 2018). This value (0.110 kg/g) at 1 bar is slightly higher than those found in this study for GBM (0.0713 kg/kg), GA (0.0316 kg/kg), G1 (0.0987 kg/kg) and CeO_2 (0.0964 kg/kg). For the other comparison, the conventional activated carbon with a surface area of 1334 m^2/g showed an adsorption capacity of CO_2 of 40 cm^3/mL at 10 bar, and 70 cm^3/mL at 30 bar at 298 K (Kamimura et al., 2014). These values (0.04 and 0.07 kg/kg) are quite similar to the adsorption capacity for GA (0.0316 kg/kg) and GBM (0.0713 kg/kg).

The CeO_2 powder with a specific surface area of 199 m^2/g reported an adsorption capacity of CO_2 of approximately 50 mg of CO_2 per gram of CeO_2 or 1,130 mmol of CO_2/kg CeO_2 (Slostowskia et al., 2017). In other study, the CeO_2 sample was calcined at 773 K with a surface area of 166 m^2/g that was purchased from Daiichi Kigenso Kagaku Kogyo (Yoshikawa et al. 2014), showing an adsorption capacity of CO_2 of about 132 mmol/kg. On the other hand, Kamimura et al. (2014) reported that synthesized mesoporous ceria with the highest surface area of 198 m^2/g had a CO_2 adsorption capacity of 160 cm^3/mL at 10 bar and 226 cm^3/mL at 30 bar at 298K. The activated modified carbon (AMC) with a loading (10%) of metal oxide (CeO_2) had an adsorption capacity of CO_2 of 0.32 mmol/g at 0.1 bar and ~1.85 mmol/g at 1 bar when measured at a temperature of 303 K (Li et al., 2017), while AMC showed an adsorption capacity of CO_2 of 0.32 and 2.25 mmol/g at 0.1 and 1 bar (Li et al., 2017), respectively.

If we compare with the same precursor from graphite electrode waste, only different in the chemical modifier, the adsorption capacity of CO_2 using the graphite waste/Fe_3O_4 20% composite (0.453 kg/kg) (Kusrini et al., 2017) is higher than that found using the G0.5 composite (0.1574 kg/kg) at a similar temperature (30°C) and pressure (20 bar). This finding has been proven to increase the capacity for capturing CO_2 when using magnetite nanoparticles Fe_3O_4 (Kusrini et al., 2017) and that it is much better than that found for cerium nitrate hexahydrate ($Ce(NO_3)_3.6H_2O$) and/or CeO_2 as modifier.

3.2. Surface Area Analysis

Among the three graphite modifications with metal salt ($Ce(NO_3)_3.6H_2O$), the highest adsorption capacity (0.1574 kg/kg) was given by G0.5 composite. From our previous report, the surface area of G0.5 is 20.03 m^2/g (Kusrini et al., 2018a). For comparison, GBM and G2 have a surface area of 26.35 and 26.82 m^2/g respectiely, which is slightly larger than G0.5 composite.

The composites of G0.5, G1 and G2 showed BET surface areas of 20.032, 18.569 and 26.815 m^2/g (Kusrini et al., 2018a). In particular, the as-made graphite/CeO_2 composites have surface areas higher than the non-porous ceria (4 m^2/g and 0.01 cm^3/g) (Kamimura et al., 2014).

In this study, their Langmuir surface areas are in agreement with their BET surface areas determined from multilayer adsorption, as shown in Table 1. The t-plot of the external surface area is more effective than that of the micropore area. This can also be indicative of the non-porous composites. In this case, the external surface area is bigger than the internal one (micropore), so it contributed for effectively in the calculation of the BET surface area. The adsorption of CO_2 by the graphite/CeO_2 composites is most likely to be a physisorption process. It occurs when the intermolecular attraction between the CO_2 gas and adsorbent is greater than the intermolecular forces between the solid molecules themselves. This causes the adsorption to occur as an exothermic process.

Note that a BET surface area of graphite non modified (GNM) of 8.490 m^2/g was reported by Kusrini et al. (2017). This is significantly lower compared to the when GBM in this study (26.348 m^2/g). CO_2 capture using GNM has an adsorption capacity of CO_2 (0.283 kg/kg), which is higher than that found using GBM (0.0713 kg/kg). Increasing the BET surface area of the sorbents did not have a linear correlation for CO_2 capture in this study. Because CO_2 capture is higher by the physisorption process, it therefore depends on the active site and chemical modifier itself, such as $Ce(NO_3)_3.6H_2O$ and magnetite nanoparticles Fe_3O_4 (Kusrini et al., 2017).

Table 1 BET surface areas for graphite before modification (GBM), activated graphite waste (GA), and graphite/CeO_2 composites

Parameter	Type of adsorbent				
	GBM	GA	G0.5	G1	G2
BET surface area (m^2/g) (Kusrini et al., 2018a)	26.348	16.635	20.032	18.569	26.815
Langmuir surface area (m^2/g)	187.036	77.507	95.466	93.841	137.529
t-plot micropore area (m^2/g)	5.775	2.322	2.839	2.362	5.496
t-plot external surface area (m^2/g)	20.574	14.313	17.193	16.206	21.319
Nanoparticles (nm)	227.719	360.689	299.523	323.122	223.757

For comparison, the activated modified carbon (AMC) has a surface area of 2,475 m^2/g (Li et al., 2017), which is very high for mesoporous materials. After wet impregnated treatment with 10% weight loading of metal salt ($Ce(NO_3)_3.6H_2O$) and calcination at 673 K under N_2 flowing, the AMC-CeO_2 (10%) had a surface area of 1,906 m^2/g (Li et al., 2017). In this study, the BET surface area of activated carbon and graphite waste is notably different, so it would influence the capability as sorbent for capturing CO_2.

4. CONCLUSION

CO_2 adsorption has been studied in relation to graphite waste composite, activated graphite waste and CeO_2 using a volumetric adsorption isotherms. The adsorption is physisorption; the adsorption capacity increases with CO_2 pressure, but decreases with temperature. By varying the concentration of Ce salt ($Ce(NO_3)_3.6H_2O$) in the graphite/CeO_2 composites, we found that the maximum adsorption capacity of CO_2 was 0.1574 kg/kg when we used the adsorbent type of G0.5 composite at a temperature of 303K and a pressure of 20 bar. This work allows us to synthesise a new sorbent effective for applicability in CO_2 capture. The adsorption capacity of CO_2 significantly depends on the active site and chemical modifier of the sorbents. Further studies should be carried out on the performance and development of these composites with other types of lanthanides and/or nanoparticles for further commercial applications as adsorbents.

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