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## Effect of ligand type on CO<sub>2</sub> adsorption over amine functionalized fibrous adsorbents

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**Abstract.** In this study, ligands originated from glycidyl methacrylate (GMA) and vinyl benzyl chloride (VBC) monomers were grafted onto fibrous polyethylene/polypropylene (PE/PP) substrate and the resulting grafted copolymers were functionalized with ethylenediamine (EDA). The changes in the chemical structure and crystallinity of the aminated adsorbents were evaluated using Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD), respectively. The PE/PP-g-PGMA-EDA adsorbent showed a CO<sub>2</sub> adsorption capacity of 1.73 mmol/g from pure gas, which is 87% lower than that of PE/PP-g-PVBC-EDA (3.24 mmol/g) at 30 bars despite the higher density in the former adsorbent. The effect of temperature, selectivity and CO<sub>2</sub> gas composition was further investigated for the PE/PP-g-PVBC-EDA adsorbent.

### 1. Introduction

Natural gas (NG) is a mixture of a few gas components and it is rich in methane with impurities such as carbon dioxide (CO<sub>2</sub>), nitrogen (N<sub>2</sub>) and hydrogen-sulphide (H<sub>2</sub>S). It can be classified into five categories, such as dry gas, wet gas, sweet gas, sour gas and acid gas. Sour and sweet gas is described as NG with and without significant percentage of H<sub>2</sub>S [1]. Sweet NG reservoirs are depleting worldwide and the remaining sour gas reservoirs has been attracting the interest for exploitation to fulfil the increasing demand on NG [2,3]. However, NG comes with up to 80% of CO<sub>2</sub>, which is highly corrosive in the presence of moisture causing a rapid corrosion in the pipelines and equipment [4]. Hence, removal of CO<sub>2</sub> from NG is mandatory in order to improve the calorific value and meet the typical pipeline specifications of 2-5% of CO<sub>2</sub> [5]. The highest CO<sub>2</sub> content in NG was found in the Platong and Erawan fields in Thailand with CO<sub>2</sub> content of 90%. Moreover, up to 71% to 87% of CO<sub>2</sub> contents have left an estimated 46 to 13 trillion cubic feet of NG reserves in Indonesia and Malaysia undeveloped due to



presence of high CO<sub>2</sub> content. This represents a significant CO<sub>2</sub> capture demand and subsequent storage opportunity in these countries [6].

The large amount of CO<sub>2</sub> from NG should be removed during the offshore NG exploitation especially during the pretreatment to prevent the corrosion to the floating liquefied NG production storage and off-loading platform (LNG-FPSO) equipment. To date, the main technologies for CO<sub>2</sub> removal are absorption, membrane separation and adsorption. Absorption is the best technology for removal of CO<sub>2</sub> from NG [4]. However, it has a number of disadvantages including onshore central plant operation of high cost and corrosion challenges [7]. Moreover, the limited space of LNG-FPSO and the swing caused by waves and typhoons implies that the CO<sub>2</sub> removal plant should be compact, safe and reliable. The limitation in LNG-FPSO has diverted the attention towards membrane separation technology due to their modular installation, smaller footprint, lower weight, easy maintenance and minimum utility requirements [8]. However, membranes cannot cope with NG with high CO<sub>2</sub> contents [9]. On the other hand, adsorption is a promising alternative technology due to their simplicity, low operational requirements, low cost, ease of control and high efficiency [10,11]. Hence, this study explores the development of fibrous adsorbent for CO<sub>2</sub> capture based polyethylene coated polypropylene (PE/PP) non-woven sheets.

The fibrous sheets are converted to adsorbent by imparting chemical ligands using the radiation-induced grafting (RIG) technique and subsequent functionalization [12–15]. The inert polymer substrates were grafted with vinyl or acrylic monomers such as glycidyl methacrylate (GMA) and vinyl benzyl chloride (VBC) [16]. The incorporated epoxy group from GMA and methyl chloride group from VBC provide fibrous substrate the ability to host amine group such as ethylenediamine (EDA) in a post-grafting reactions. Although both final adsorbents have same functional groups, but the impact of having aliphatic and aromatic structure of PGMA and PVBC on the adsorption capacity is not known. Thus, the aims of this study are to investigate the effect of the type of ligands incorporated in PE/PP structure by RIG technique and followed by amination with EDA on the CO<sub>2</sub> adsorption performance.

## 2. Materials and methods

### 2.1 Chemicals and Reagents

The PE/PP nonwoven sheet was grafted with GMA and VBC monomers according to the reported procedure [17]. The synthesis variables were tuned to confer PE/PP substrates with similar degree of grafting in the range of 156–166%. Ethanol (99.6%) was purchased from Chemiz and used for washing of samples. EDA (≥99%) was purchased from Sigma-Aldric. Pure CO<sub>2</sub> gas (99.8%) and Pure CH<sub>4</sub> gas (99.995%) were supplied by Linde Malaysia (Sdn Bhd). Deionized water (DI) was obtained from Barnstead Nanopure Diamond Lab Water Purification System (ThermoFisher, Waltham, USA)

### 2.2 Apparatus and measurements

The changes in the chemical composition after grafting and amination were observed by Fourier transform infrared spectroscopy using a Nicolet iS50 FT-IR spectrometer with spectra obtained in a frequency range of 500–4000 cm<sup>-1</sup> using 32 scans and 4 cm<sup>-1</sup> resolutions. The crystalline structural changes of the samples were investigated by X-ray diffraction (XRD) using a PANalytical Empyrean analyzer at a Bragg's angle in the range of 5–70°. All analysis was carried out using samples of pristine PE/PP, PGMA grafted PE/PP (PE/PP-g-PGMA), PVBC grafted PE/PP (PE/PP-g-PVBC), aminated PE/PP (PE/PP-g-PGMA-EDA) and (PE/PP-g-PVBC-EDA). CO<sub>2</sub> adsorption capacity of the aminated adsorbent were evaluated using a gravimetric sorption analyzers-isoSORP®, Rubotherm-TA instruments operating at a temperature of 30 °C and a pressure range of 5–30 bar.

### 2.3 Amine Functionalization

A 0.1 g of PE/PP-g-X-EDA, where X stands for PGMA and PVBC substrates were placed in a 100 ml round bottom flask containing 30 ml of pure EDA solution. The reaction was carried out under reflux for 6 h. The EDA-functionalized substrates were removed and washed repeatedly with water and ethanol

then dried at 60 °C for 5 h. The weight changes of PE/PP-*g*-X were recorded before and after the reaction. The amination percentage was calculated using eqn 1:

$$\text{Percent of amination (\%)} = \frac{(W_a - W_g) / W_{EDA}}{(W_a - W_o) / W_X} \times 100\% \quad (1)$$

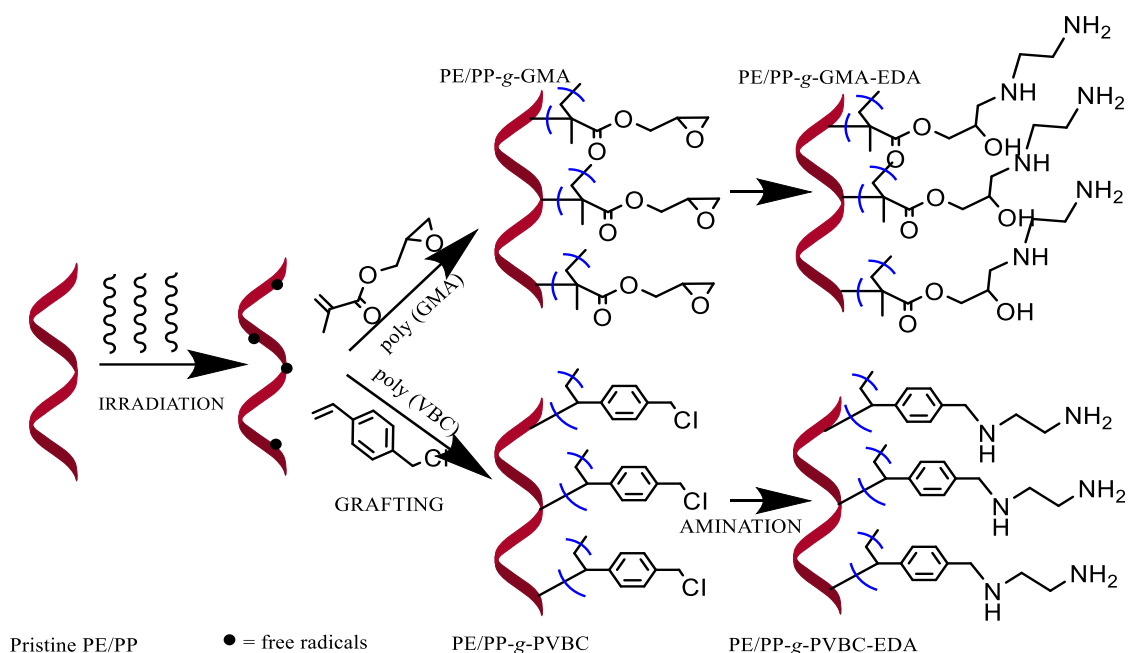
Where,  $W_X$  and  $W_{EDA}$  are the molecular weights of GMA/VBC and EDA (g/mol), respectively.  $W_a$  and  $W_g$  are the weights (g) of the PE/PP-*g*-X substrates before and after amination of both type of ligands, respectively. The  $W_o$  is the weight of PE/PP substrate before grafting.

#### 2.4 CO<sub>2</sub> adsorption tests

0.1 g of PE/PP-*g*-PGMA-EDA and PE/PP-*g*-PVBC-EDA substrates were placed inside the sample container and the adsorption equilibrium of carbon dioxide (CO<sub>2</sub>) and methane (CH<sub>4</sub>) were performed in a magnetic suspension balance (MSB) of gravimetric sorption analyser-isoSORP® from Rubotherm-TA instruments. Details of the basic principles, components and operational procedure of MSB was described elsewhere [18–20]. Prior to the adsorption, the first step in the experiment procedure is the pre-treatment process which carried out to remove moisture content at temperature 80°C for 2 hours under vacuum condition and after that, buoyancy measurements were performed at 30°C from vacuum up to 30 bar using nitrogen gas with 500 ml/min of flow rate. The last procedure was the adsorption equilibrium measurement which measured at 30°C from vacuum to 30 bar with flowrate 500 ml/min for both pure CO<sub>2</sub> and pure CH<sub>4</sub>. The effect of temperature and selectivity study were both conducted at three different temperatures (30°C, 40°C and 50°C) by using the same flowrate and operating pressure. Hence, the best operating temperature was selected to further examine on the performance using different CO<sub>2</sub>/CH<sub>4</sub> composition.

### 3. Results and discussion

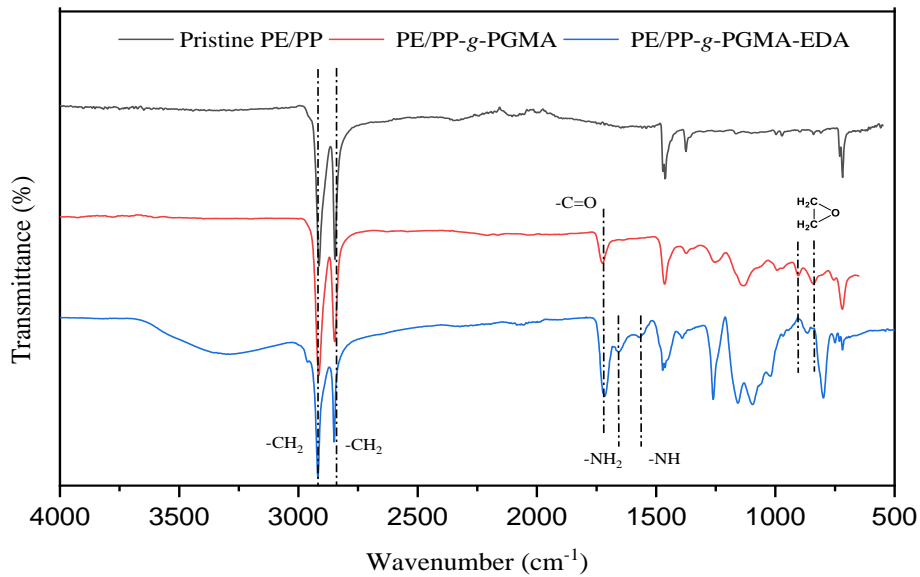
Figure 1 shows reaction scheme for preparation of CO<sub>2</sub> fibrous adsorbent by grafting of GMA or VBC onto PE/PP and subsequent functionalization of PE/PP-*g*-X with EDA, where X referred to PGMA and PVBC ligands which were capable of hosting EDA. The presence of amine groups conferred the basicity needed to capture CO<sub>2</sub> molecules (chemisorption) which is categorized as Lewis acid [21]. The percent of amination obtained for PE/PP sample treated with two different ligands such as PGMA and PVBC was 60% and 47.37%, respectively. The values obtained were calculated based on equation (1). The results show that, PGMA sample contains 21% higher concentration of amine compared to PVBC.



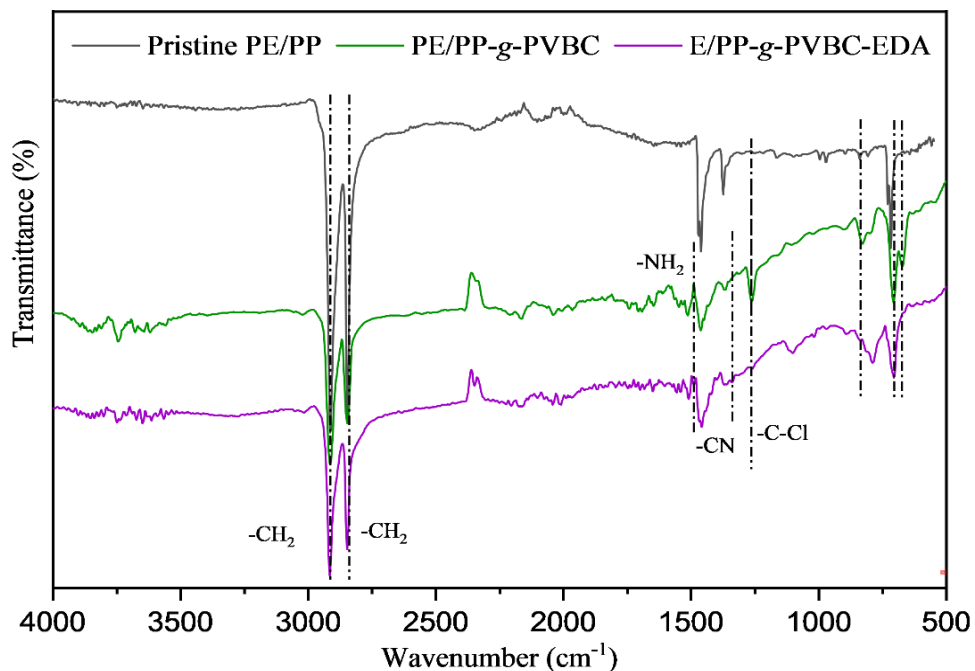
**Figure 1.** Reaction scheme for grafting of GMA/VBC onto PE/PP and subsequent amination with EDA.

### 3.1 Chemical and structural changes

Figure 2a shows FTIR spectra of pristine PE/PP and the corresponding PE/PP grafted with PGMA and the aminated adsorbent. The emergence of new peaks at  $840\text{ cm}^{-1}$ ,  $905\text{ cm}^{-1}$  and  $1740\text{ cm}^{-1}$  confirmed the presence of epoxy group and  $\text{C}=\text{O}$  stretching vibration originated from PGMA chains attached to the PE/PP backbone. This proves that PGMA was covalently bonded to PE/PP substrate [12]. The disappearance of the peaks at  $840\text{ cm}^{-1}$  and  $905\text{ cm}^{-1}$  which is parallel with the appearance of peak at  $3500\text{ cm}^{-1}$  assigned for  $\text{OH}$  confirm the opening of epoxy ring and the introduction of EDA. This was further evidence from the appearance new peaks  $1658\text{ cm}^{-1}$ ,  $1527\text{ cm}^{-1}$  and a broad band at  $3300\text{ cm}^{-1}$  is due to  $\text{NH}$  and  $\text{NH}_2$  [13,22]. Figure 2b shows FTIR spectra of pristine PE/PP, grafted PE/PP (PE/PP-g-PVBC) and aminated PE/PP (PE/PP-g-PVBC-EDA). The bands at  $838$ ,  $702$  and  $676\text{ cm}^{-1}$  are originated from the *p*-substituents on the aromatic ring and the peak at  $1265\text{ cm}^{-1}$  is from  $\text{C}-\text{Cl}$  stretching vibration, which provide a strong evidence of the successful grafting of VBC onto PE/PP substrate [23,24]. The successful incorporation of EDA in PE/PP-g-PVBC was confirmed by the presence of bending vibrations of  $\text{NH}_2$  at  $1490\text{ cm}^{-1}$  and the bending mode of  $\text{CN}$  at  $1330\text{ cm}^{-1}$  [25].

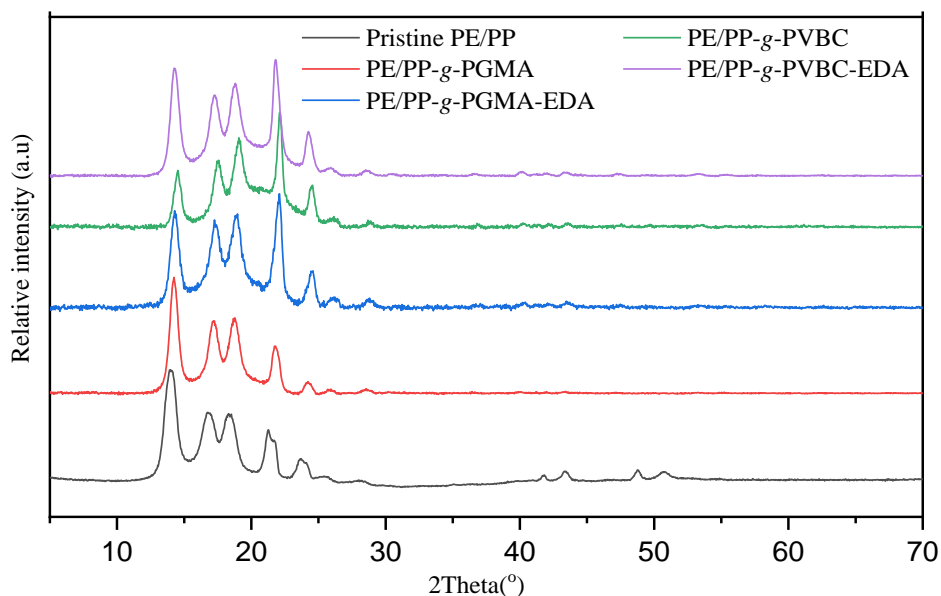


**Figure 2(a).** FTIR spectra of Pristine PE/PP (black), PE/PP-g-PGMA (red) and PE/PP-g-PGMA-EDA (blue).



**Figure 2(b).** FTIR spectra of pristine PE/PP (black), PE/PP-g-PVBC (green) and PE/PP-g-PVBC-EDA (purple)

Figure 3 shows XRD diffractograms of pristine PE/PP, PE/PP-g-PGMA, PE/PP-g-PGMA-EDA, PE/PP-g-PVBC and PE/PP-g-PVBCA-EDA samples. The Bragg's angle at each diffraction peaks occurred at the same positions ( $2\theta$  in the range of  $10-30^\circ$ ), indicating that the crystalline structure of pristine PE/PP was well preserved after grafting and subsequent amination. This suggests that grafting and functionalization took place in the amorphous region and is a complete agreement with literature [26,27].

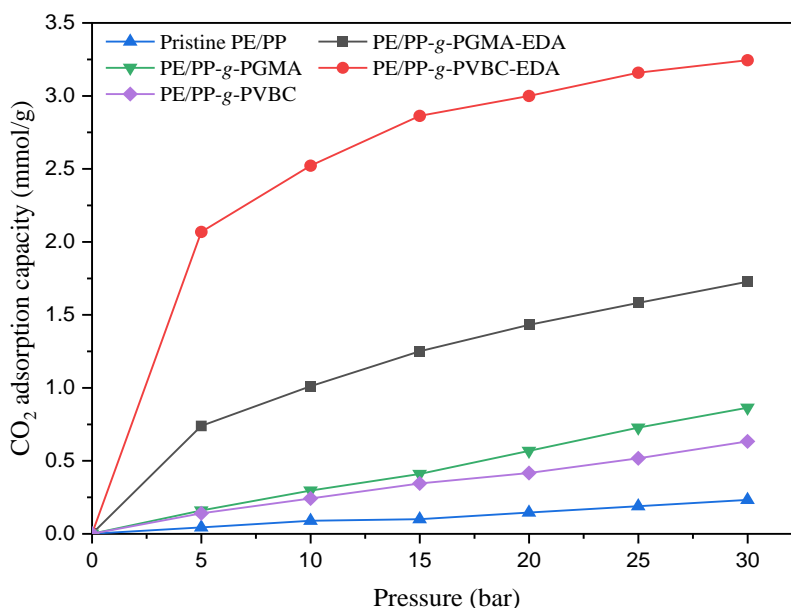


**Figure 3.** XRD diffractograms of pristine PE/PP (black) PE/PP-g-PGMA (red), PE/PP-g-PGMA-EDA (blue), PE/PP-g-PVBC (green) and PE/PP-g-PVBC-EDA (purple).

### 3.2 CO<sub>2</sub> adsorption by two adsorbents

Figure 4 shows the Variation of CO<sub>2</sub> adsorption capacity with pressure for pristine PE/PP, PE/PP-g-PGMA, PE/PP-g-PVBC, PE/PP-g-PGMA-EDA and PE/PP-g-PVBC-EDA. The CO<sub>2</sub> adsorption capacities for pristine PE/PP, PE/PP-g-PGMA and PE/PP-g-PVBC samples were included as references. As can be obviously seen, the adsorbent having aminated PVBC ligand showed an adsorption capacity of 3.24 mmol/g whereas that for the adsorbent containing aminated PGMA ligand was 1.73 mmol/g at 30 bar and 30 °C. Such unexpected result shows that the former adsorbent has 87% higher CO<sub>2</sub> adsorption capacity than the latter despite the early observation revealing that the PGMA ligand hosted 21% EDA more than that of PVBC. This likely to be due to the low degree of flexibility of PGMA, which is highly amorphous in nature and tested below its glass transition temperature compared to that of PVBC, which has a rubbery nature. This allowed more CO<sub>2</sub> adsorption in the latter adsorbent than the former.

Additionally, the high amine content of PGMA-containing adsorbent may have caused a negative impact on the CO<sub>2</sub> adsorption performance by blocking and/or reducing the free volume in the adsorbent, thus decreasing the gas diffusion rate which leading to lower CO<sub>2</sub> adsorption capacity [28].

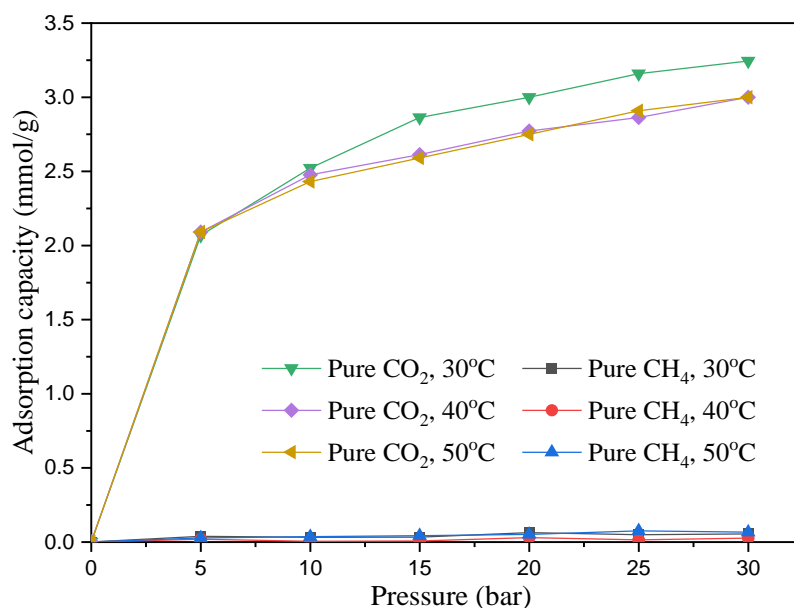


**Figure 4.** Variation of CO<sub>2</sub> adsorption capacity with pressure for pristine PE/PP (blue), PE/PP-g-PGMA (green), PE/PP-g-PVBC (purple), PE/PP-g-PGMA-EDA (black) and PE/PP-g-PVBC-EDA (red).

The effect of temperature the adsorption capacity of the adsorbent containing aminated was carried out and the data is presented in Figure 5. The adsorption performance was reduced by only 7.56% when operating temperature changed from 30°C to 40°C with adsorption capacity dropping from 3.24 to 3.00 mmol/g at 30 bar. Further rise in the temperature to 50°C showed no impact on the adsorption performance. This trend suggests that the adsorbent is stable up to 50°C and can be operated at such temperature despite the minor decrease in the capacity. Figure 5 also shows the adsorption behavior of the adsorbent toward pure CH<sub>4</sub> gas. Obviously, the CH<sub>4</sub> adsorption is extremely low or negligible and this confirms that the adsorbent is highly selective to CO<sub>2</sub>. This is due to the presence of basic group (amine), which is covalently bonded the PE/PP substrate which has a strong affinity to Lewis acids of CO<sub>2</sub> molecules [29,30].

Table 1 provides the data for the selectivity of CO<sub>2</sub> over CH<sub>4</sub> at different pressures and temperatures. The pressure varied from vacuum to 30 bar and the temperature from 30°C to 50°C. Overall, the highest equilibrium selectivity of CO<sub>2</sub> over CH<sub>4</sub> was at 40°C with a huge gap as compared with 30°C and 50°C operating temperatures. Moreover, most of the measured data shows a decrease in the selectivity with the increase in the pressure. This is because at higher pressure, there is only less strong adsorption sites to adsorb CO<sub>2</sub> molecules whereas, at low pressures, more CO<sub>2</sub> molecules occupied the strong adsorption sites [31]. Despite the low CO<sub>2</sub> selectivity at 30°C compared to 40°C, the measured capacity of the adsorbent when pure CH<sub>4</sub> was feed to the system showed negligible capacity at all temperatures. Hence, 30°C was selected as an operating temperature to study the effect of different CO<sub>2</sub> composition on CO<sub>2</sub> adsorption capacity.



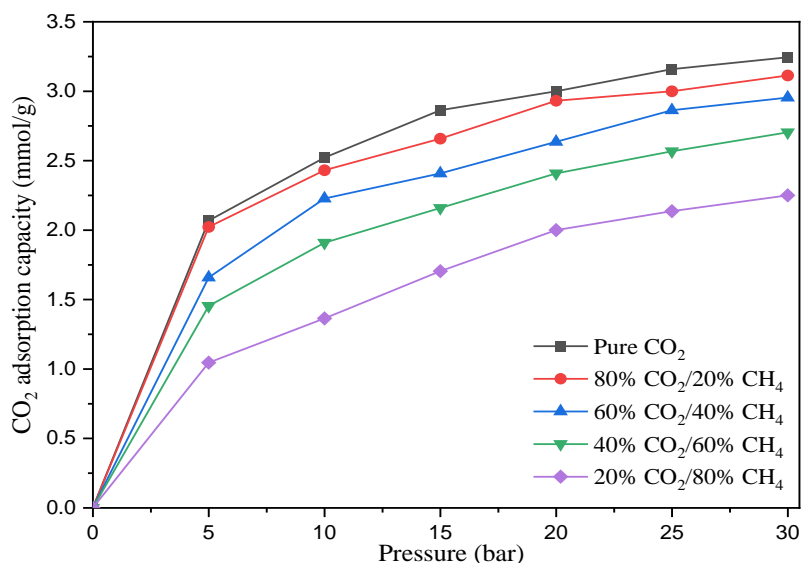


**Figure 5.** Variation CO<sub>2</sub> adsorption capacity with pressure for PE/PP-g-PVBC-EDA adsorbent with pure CO<sub>2</sub> and pure CH<sub>4</sub> at different temperatures

**Table 1.** Selectivity of CO<sub>2</sub> over CH<sub>4</sub> at different pressures and temperatures on PE/PP-g-PVBC-EDA

Sample	Pressure (bar)	Adsorption capacity (mmol/g)(CO <sub>2</sub> )			Adsorption capacity (mmol/g)(CH <sub>4</sub> )			Equilibrium selectivity (CO <sub>2</sub> /CH <sub>4</sub> )		
		Temperature (°C)			Temperature (°C)			Temperature (°C)		
		30	40	50	30	40	50	30	40	50
PE/PP-g-PVBC-EDA	5	2.068	2.090	2.090	0.037	0.018	0.027	55.152	115.000	77.966
	10	2.522	2.477	2.431	0.032	0.005	0.035	79.286	545.000	69.032
	15	2.863	2.613	2.590	0.033	0.007	0.042	86.897	383.333	61.622
	20	2.999	2.772	2.749	0.062	0.030	0.051	48.000	93.846	53.778
	25	3.158	2.863	2.908	0.049	0.014	0.075	64.651	210.000	38.788
	30	3.245	2.999	2.999	0.055	0.026	0.066	59.500	114.783	45.517

Figure 6 shows the variation of CO<sub>2</sub> adsorption capacity with pressure for PE/PP-g-PGMA-EDA with different CO<sub>2</sub>/CH<sub>4</sub> compositions. As can be seen, the adsorption capacity increased when CO<sub>2</sub> composition increased from 20-100% at 30°C and 30 bar. This is mainly due to strong interaction and high selectivity between the CO<sub>2</sub> and the amine anchored in the substrate compared to CH<sub>4</sub>. As the pressure increases, the diffusion of CO<sub>2</sub> through substrate also increased, hence increased the adsorption capacity [8]. From pure CO<sub>2</sub> to 20% of CO<sub>2</sub> gas composition, the performance was degraded in an increasing order of 4.06% < 5.11% < 8.46% < 16.81%. This might be due to the decreasing of chances for CO<sub>2</sub> to reach the amine active site which might be subjected to physical attachment from CH<sub>4</sub> molecules. This observation reveals that the present adsorbent performance shows a potential for the removal of CO<sub>2</sub> from NG stream containing CO<sub>2</sub> up to 80%.



**Figure 6.** Variation of CO<sub>2</sub> adsorption capacity with pressure for PE/PP-g-PGMA-EDA with different CO<sub>2</sub>/CH<sub>4</sub> compositions.

#### 4. Conclusion

The effect of the type of ligands (aminated PGMA and PVBC) incorporated to PE/PP fibrous substrates on the CO<sub>2</sub> adsorption capacity was investigated. The percent of amination showed that the PE/PP-g-PGMA-EDA has higher amination value with 60% compared to PE/PP-g-PVBC-EDA with 47.37%. However, the CO<sub>2</sub> adsorption capacity performance displayed an opposite trend in which the PE/PP-g-PGMA-EDA adsorbent showed a capacity of 1.73 mmol/g compared to 3.24 mmol/g for PE/PP-g-PVBC-EDA with pure CO<sub>2</sub> gas at 30 bar. Moreover, the equilibrium selectivity (CO<sub>2</sub>/CH<sub>4</sub>) analysis showed a great value of 114.78 at 40°C and 30 bar. The effect of temperature showed that the optimum CO<sub>2</sub> adsorption capacity was obtained at temperature 30°C. The effect of different CO<sub>2</sub> compositions on the adsorption revealed that the adsorption capacity increased with the increase in the percent of CO<sub>2</sub> in the gas mixture. It can be concluded that the fibrous adsorbent containing aminated PVBC ligands has a strong potential for future application in removal of CO<sub>2</sub> from natural gas.

#### Acknowledgment

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