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Amine (Polyethyleneimine)-modified solid adsorbent for CO_2 capture

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Abstract. The increase in the concentration of carbon dioxide (CO_2) gas in the atmosphere has led to various severe negative consequences. There are numerous methods for the reduction of CO_2 that have been introduced such as chemical and physical absorption, organic liquid scrubbing, amine-based absorption, etc. Adsorption by using solid adsorbents is one of the promising methods that has been widely studied by researchers. The performance of the adsorbents can be enhanced by functionalized with diverse types of promoters. This review is discussing the performance of polyethyleneimine (PEI) as a promoter towards the adsorption of CO_2. To achieve high effective PEI-adsorbents, the percentage of PEI amine loading, type of porous support, temperature, and different flow conditions are among the important parameters that need to be considered. The chemical stability of PEI can be improved through modification crosslinking of PEI. Hence in this review, the effect of amine loading, porous support, temperature, slow condition and crosslinking of PEI to its CO_2 adsorption performance is observed.

1. Introduction

Global warming has been one of the serious worldwide threats for decades as it could contribute to a rising level of seawater [1] and various effects on human health [2]. The growing level of global industries and urbanization have inevitably led to the increasing of CO_2 emissions to the atmosphere [3]. Thus, an effective and low-cost carbon capture and storage (CCS) approach to remove tons of CO_2 gas from the environment is needed to overcome these problems [4]. In recent years, the methods involved in CO_2 capture are pre-combustion, post-combustion, and oxy-fuel combustion [5, 6]. Among these methods, post-combustion is considered the promising method since the process is much simpler compared to the other two methods [6].

In the post-combustion method, the adsorption process by using solid adsorbents has gained increasing attention from researchers [7]. However, the performance of solid adsorbents for practical use is still limited by their low CO_2 uptake capacity and selectivity [8]. Amine-functionalized is one of the promising approaches to increase the performance of these adsorbents towards CO_2 adsorption due to several advantages such as good stability, lower heat regeneration, and high adsorption potential [9, 10]. PEI is one of the amine groups that have been widely used due to its high amino contain and good thermal stability [11]. Due to its beneficial behaviors, there are huge numbers of studies have reported on the use of PEI to enhance the CO_2 uptake of various solid adsorbents such as silica [12, 13], graphene oxide [14], metal oxide [15], metal-organic framework [16, 17], etc.

Several interesting reviews are focusing on the CO_2 capture by numerous solid adsorbents, diverse alteration procedures of solid adsorbents, the latest development on amine-functionalized solid adsorbents for post-combustion CO_2 capture, etc. In this respect, the present review is expected to contribute to a better understanding of the recent progress on numerous PEI-functionalized adsorbents for CO_2 capture, several parameters involved in the reaction process, and the modification of PEI by cross-linking.

2. PEI impregnated adsorbents for CO_2 capture

It is well recognized that the presence of PEI as a promoter could significantly improve the CO_2 adsorption of numerous solid adsorbents. In this respect, various works on the PEI-modified solid adsorbents together with the impact of the reaction parameters on the CO_2 uptake performance of the selected adsorbents will be comprehensively discussed in the next sections. Based on table 1, it is shown there are numerous studies of PEI supported to various types of adsorbents. Mainly from the mesoporous silica based due to its properties that are easy to be introduced to functional support [13]. From the data, it clearly showed the presence of impregnated PEI improves the adsorption capacity for all different types of adsorbents. As presented in Table 1, the functionalization of solid adsorbent with PEI has decreased adsorbent textural properties such as surface area and pore volume. It was associated with the coverage of the adsorbent surface, including porous channel by PEI [20]. Thus, adsorbent with good textural properties such as larger surface area and pore volume will require more PEI to reach optimum quantity and, finally, exhibit greater adsorption capacity. Nevertheless, some other parameters that affect the CO_2 adsorption capacity such as amine loading, porous support, adsorption temperature, and different flow conditions. There is also a study of modification PEI through crosslinking by jeon and coworkers that resulting in improve on CO_2 adsorption in terms stability [18].

Table 1. The summary of studies on PEI functional adsorbents.

Adsorbents	Synthesis method	BET surface area (m²/g)	BJH Pore Volume (cm³/g)	Adsorption flow condition (CO₂%: inert @ other gas %)/H₂O	CO₂ uptake (mmol/g)	Stability (mmol/g)	Ref
			Metal-organic framework-based adsorbent				
Mesoporous MOF 177	Sono-chemical	2784	1.34	(100:0)/-	0.86 at 298 K	-	[16]
10wt% PEI-MOF 177	Sono-chemical and wet impregnation	690	0.393	(100:0)/-	2.84 at 298 K	-	
Mesoporous PCN-777	Hydrothermal	2008	2.16	(25:75 N₂)/-	1.13 at 298 K	-	[19]
20% PEI-PCN-777	Hydrothermal and wet impregnation	843	0.37	(25:75 N₂)/-	1.41 at 298 K	1.40 (5th cycle)	
Zn/Co ZIF	Mixing and stirring	163	0.72	(100:0)/-	1.07 at 298 K	-	[20]
40wt% PEI-Zn/CoZIF	Mixing and stirring and Wet Impregnation	11	0.05	(100:0)/-	1.82 at 298 K	1.75 (10th cycle)	
MIL-101	Hydrothermal	3324	1.75	(10:90 He)/-	0.80 at 348 K	-	[21]
70wt% PEI-MIL-101	Hydrothermal	923	0.53	(10:90 He)/-	3.81 at 348 K	3.71 (6th cycle)	
			Silica-based adsorbent				
Mesoporous SBA-15	Cooperative	604	1.192	(44.6: 55.4 N₂)/-	0.11 at 313 K	-	[12]
Mesoporous SBA-11	Cooperative	356	0.792	(44.6: 55.4 N₂)/-	0 at 313 K	-	
Disordered porous SiO₂	Cooperative	256	0.460	(44.6: 55.4 N₂)/-	0.03 at 313 K	-	

40wt% PEI-SBA-15	Cooperative and wet impregnation	248	0.698	(44.6: 55.4 N_2)/-	0.90 at 313 K	0.62 (4th cycle)	
40wt% PEI-SBA-11	Cooperative and wet impregnation	16	0.116	(44.6: 55.4 N_2)/-	0.60 at 313 K	-	
20wt% PEI-SiO$_2$	Cooperative and wet impregnation	65	0.175	(44.6: 55.4 N_2)/-	0.60 at 313 K	-	
HMS-A	Hydrolysis	736	0.66	(100:0)/-	0.53 at 318 K	-	[22]
50wt% PEI-HMS-A	Hydrolysis and wet impregnation	11	0.02	(100:0)/-	2.40 at 318 K	-	
HMS-C	Calcination	1181	0.96	(100:0)/-	0.31 at 318 K	-	
70wt% PEI-HMS-C	Calcination and wet impregnation	2.1	0.01	(100:0)/-	2.19 at 318 K	-	
SFM-0.83-100-5.2	Modified-microemulsion templating	526.2	1.4	(15:85 N_2)/-	1.14 at 348 K	-	[23]
50wt% PEI-SFM-0.83-100-5.2	Modified-microemulsion templating and impregnation	-	-	(15:85 N_2)/-	2.48 at 348 K	2.11 (15th cycle)	
Mesoporous Monolithic MCM550	Gel-casting and Impregnation method	1088.32	1.00	(100:0)/-	0.65 at 348 K	-	[24]
				(12 v%:88 v% N_2)/-	-	-	
60wt% PEI-MCM550	Gel-casting and Impregnation method	12.48	0.01	(100:0)/-	2.32 at 348 K	2.30 (5th cycle)	
				(12 v%:88 v% N_2)/-	1.89 at 348 K	1.91 (5th cycle)	
Mesostructured cellular foam silica	-	458	2.07	(100:0)/-	-	-	[18]
50wt% PEI-Silica	Drying and dry impregnation	103	0.58	(100:0)/-	3.20 at 353 K	-	
				(15:85 N_2)/-	2.86 at 353 K	0.48 (40th cycle)	
50wt% Cross-linked PEI-Silica	Drying and dry impregnation	151	0.79	(100:0)/-	2.74 at 353 K	-	
				(15:85 N_2)/-	2.06 at 353 K	1.08 (40th cycle)	
Mesostructured silica KIL-2	Two-step (aging and solvothermal)	702	1.61	-	0.6 at 363 K	-	[25]
PEI-KIL-2	Two-step and wet impregnation	127	0.35	-	3.6 at 363 K	-	
Mesoporous Si-MCM-41	Hydrothermal and wet impregnation	994	1	(100:0)/-	-	-	[26]
50wt% PEI-MCM-41	Hydrothermal and wet impregnation	6	0	(100:0)/-	2.26 at 373 K	1.82 (6th cycle)	
Metal Oxide-based adsorbent							
MgO	Sol-gel	350	0.414	(99.9:0.01 N_2)/-	0.68 at 303 K	-	[15]
PEI-MgO	Sol-gel and Impregnation	72	0.178	(99.9:0.01 N_2)/-	0.54 at 303 K	-	
Titania-based adsorbent							
Mesoporous TNTs	Hydrothermal	326	0.756	(15: 85 N_2)/-	0.46 at 313 K	-	[27]
30wt% PEI-TNTs	Hydrothermal and wet impregnation	67	0.168	(15: 85 N_2)/-	1.01 at 313 K	0.96 (10th cycle)	
				(15:5 O_2:80 N_2)/-	0.99 at 313 K	0.88 (10th cycle)	
Clay-based adsorbent							
Porous Palygorskite	Hydration	137	0.32	(15 vol%: 80 vol% N_2: 5 vol% O_2)/ 5 vol%	0.27 at 318 K	-	[28]
PEI-Palygorskite	Hydration and wet impregnation	0.11	0.11	(15 vol%: 80 vol% N_2: 5 vol% O_2)/ 5 vol%	1.53 at 318 K	-	
Porous Sepiolite	Hydration	274	0.42	(15 vol%: 80 vol% N_2: 5 vol% O_2)/ 5 vol%	0.925 at 318 K	-	

PEI-Sepiolite	Hydration and wet impregnation	-	-	(15 vol%: 80 vol% N_2: 5 vol% O_2)/ 5 vol%	1.270 at 318 K	1.10 (3rd cycle)	
Sepiolite	-	42.74	0.08	(60 vol%:40 vol% N_2)/-	0.27 at 348 K	-	[29]
MgO-SiO_2 Nanowire's sepiolite MSep	Acid leaching	168.96	0.14	(60 vol%:40 vol% N_2)/-	0.41 at 348 K	-	
50wt% PEI- MSep	Acid leaching and wet impregnation	16.88	0.03	(60 vol%:40 vol% N_2)/-	2.48 at 348 K	2.31 (10th cycle)	
				Carbon-based adsorbent			
Multi-walled CNTs	-	259.51	2.86	(15:85 N_2)/-	-	-	[30]
				(15:5O_2:80 N_2)/-	-	-	
				(15:85 N_2)/(RH=50%)	-	-	
50wt%PEI-CNTs	Wet impregnation	33.67	0.33	(15:85 N_2)/-	4.75 at 333 K	4.5 (20th cycle)	
				(15:5O_2:80 N_2)/-	4.42 at 333 K	-	
				(15:85 N_2)/(RH=50%)	4.97 at 333 K	-	
				Zeolite-based adsorbent			
Mesoporous ZSM-5	Hydrothermal	383.503	0.244	(99.99:0.01 N_2)/-	0.4 at 393 K	-	[31]
30wt% PEI-ZSM-5	Hydrothermal and physical impregnation	6.066	0.027	(99.99:0.01 N_2)/-	1.96 at 393 K	1.81 (10th cycle)	

ZIF: Zeolitic imidazolate frameworks　　　　　　MCM-41: Mobil Composition of Matter-41
MCM-41: Mobil Composition of Matter-41　　　　 PCN-777: Polymers-loaded mesoporous metal organic framework
TNTs: titanate nanotubes　　　　　　　　　　　 MIL-101: Matériaux de l'Institut Lavoisier
HMS-A: Hydrolysed mesoporous silica materials　 CNTs: carbon nanotubes
ZSM-5: Zeolite Socony Mobil–5　　　　　　　　 HMS-C: Calcined-HMS
SFM: Siliceous foam materials

2.1. Effect of amine loading

As previously mentioned, the PEI-impregnated approach could expressively improve the CO_2 adsorption capacity of numerous solid adsorbents. In general, the higher the amine loading will lead to increased CO_2 adsorption capacity. This is because the PEI incorporation could offer more additional active sites to ease the CO_2 adsorption of the solid adsorbents [32]. Based on table 1, Cheng et al. have conducted a study to investigate the effect of amine loading (20-60 wt.%) on Zn/Co zeolitic imidazolate frameworks (Zn/Co ZIF) adsorbents at pure CO_2 gas flow [20]. It was observed that the loading of 20% PEI has led to an increased CO_2 adsorption capacity from 1.07 mmol/g to 1.27 mmol/g and continuously increased up to 1.82 mmol/g for 40wt.%. However, when the PEI loading was further increased to 60 wt.%, the CO_2 uptake capacity was decreased to 1.35 mmol/g, most probably due to the superfluous amount of PEI that might block the main active sites of the adsorbent. Heo and co-workers also have reported the impregnation of PEI to titanate nanotubes (PEI-TNTs) that has successfully increased the CO_2 uptake capacity from 0.21 mg/g to 44.5 mg/g at 30 wt.% loadings. As expected, further addition of PEI to 40 wt.% has led to a decreased CO_2 uptake capacity. Based on both observations, it can be concluded that the excess amine content could suggestively reduce the CO_2 uptake capacity and adsorption rate of the PEI-modified adsorbents. These were due to the increase in CO_2 diffusion resistance caused by the presence of thicker PEI species at high amine loading [33]. The excess PEI loading also tends to block the pores and reduce the surface area and pore volume hence reduces the effective and active sites of the adsorbents [34]. Thus, it is important to recognize the optimum amine loading for that particular support to achieve the maximum CO_2 adsorption capacity.

2.2. Effect of the porous support

The pore structure of the support adsorbents undoubtedly plays an important impact in their CO_2 capture performances. Generally, the adsorbent that has large surface area and high microporosity are favorable for CO_2 adsorption [35]. In this regard, Henao et al. have studied the effect of pore structure of the support towards CO_2 adsorption performance of the hybrid adsorbents [12]. As reported in this study, the performance of the PEI-functionalization on the different porous and morphological structure of silica-based adsorbent were investigated. The morphological properties of studied mesoporous SBA-15, SBA-11 and SiO2 were in 2D hexagonal, 3D cubic and disordered porous structure, respectively. Among those silica supports, it was perceived that the SBA-15 offered the highest surface area and pore volume. Due to these advantages, PEI-SBA-15 gave the highest CO_2 uptake (61.6 mg/g), followed by SBA-11 (32.9 mg/g) and SiO_2 (26.4 mg/g). The largest surface area and pore volume of SBA-15 undeniably have improved its CO_2 uptake performance [36], by providing a lower resistance for CO_2 diffusion [12, 37]. Meanwhile, the SBA-11 that has an interconnected three-dimensional porous system was reported to have the highest resistance for CO_2 diffusion. That clearly explained the reason for a lower CO_2 uptake capacity of PEI-SBA-11 than that of PEI-SBA-15 at the same PEI loading (40 wt.%). As result, the SiO_2 with a disordered structure has demonstrated the lowest CO_2 adsorption performance. Nevertheless, there is a study in table 1 that shows the large surface area support resulting in low CO_2 adsorption performance [28]. If we analyze the study by Gómez-Pozuelo and coworkers, in the beginning it was found out that the CO_2 adsorption uptake by pristine sepiolite adsorbents is high due to its greater surface area that is available for adsorption process. However, after impregnation with PEI, the CO_2 adsorption is still increasing but at a slower rate. This scenario happens because of the textural sepiolite that causes the blockage in the inner spores and reduces the number available active sites [28, 35]. Thus, it can conclude the type of support that needs to be selected should be favorable to be used with PEI for achieving high adsorption uptake capacity.

2.3. Effect of adsorption temperature

The other crucial factor that needs to be considered in the adsorption process is temperature. In general, the CO_2 adsorption could be reduced by the increase in temperature. Cheng et al have examined the CO_2 adsorption of 40wt.% PEI- Zn/Co ZIF at elevated temperatures from 25 °C to 90 °C [20]. The result has shown that the CO_2 adsorption has decreased gradually from 1.82 mmol/g (25 °C) to 1.24 mmol/g (90 °C).

   However, some papers in table 1 reported in the increase in CO_2 adsorption performance at increasing temperatures for the PEI functionalized adsorbents [12, 22]. For example, Sanz-Pérez and co-workers have conducted a study on the effect of increasing temperature to 50wt.% PEI-hydrolysed mesoporous silica (PEI-HMS) at pure CO_2 adsorption flow [22]. The result demonstrated that the CO_2 uptake was considerably increased from 1.9 mmol/g at 30 °C to 4.19 mmol/g at 90 °C. Nevertheless, further increase in temperature to 105 °C has resulted in decreasing CO_2 uptake to 3.92 mmol/g. It was previously reported that the PEI becomes more flexible at the higher temperature, and thus more amino active sites were exposed [38]. As a result, the diffusion of CO_2 through the pore of the adsorbent becomes faster [39] and the CO_2 adsorption performance is increased. As the temperature exceeds the optimal temperature, the CO_2 adsorption then was controlled by the thermodynamics [40], at which all the amino sites were fully utilized [12, 41]. However, it is worth mentioning that the optimal temperature for each PEI-functionalized adsorbent is different based on their pristine structures. For instance, a study by Ouyang et al has reported that the CO_2 uptake of 50wt.% PEI-MgO-SiO_2 nanofibers sepiolite was at maximum capacity at 75°C [29]. While Henao and co-workers have described that 60 °C is the optimum temperature for a maximum capacity of 20wt.% PEI-mesoporous SBA-15 [12].

Interestingly, there is also a case in which the CO_2 uptake of PEI-magnesium oxide (PEI-MgO) based adsorbents are lower than the pristine MgO [42] but the performance was enhanced at higher temperature and exceed the CO_2 uptake of the pristine at 50 ℃. This showed that the PEI-MgO is not capable of performing at low temperatures and it seems unusual behavior for the adsorption process. Thus, the type of adsorbent support that is selected is crucial based on their practical use which is either for low, medium, or high temperature.

2.4. Effect of different flow conditions toward adsorption performance
The flow conditions are one of other vital parameters that need to be observed. This is because the flue gas that was produced from the industry not only contains CO_2 but also contains other impurities. From table 1, there are several studies that are related to the difference of flow conditions towards CO_2 adsorption that conduct the presence of nitrogen gas (N_2) [18, 24], oxygen gas (O_2) [27, 30] and water (H_2O) [30]. Zhou and coworkers have studied the presence of N_2 in flow conditions as much of 12 volume percentage (12v%CO_2:88v%N_2) of PEI-Mobil Composition of Matter-550 (PEI-MCM-550) have reduced the adsorption performance from 2.32 mmol/g to 1.89 mmol/g. In addition, jeon at al have also conduct the comparison between the CO_2 adsorption of PEI-silica (Si) in pure CO_2 and 15% CO_2 with balance N_2, which have been observe the CO_2 uptake capacity in 15% CO_2 is lower (2.86 mmol/g) compared to pure CO_2 gas flow (3.20 mmol/g) at temperature of 353K. Hence, it can be said the drop concentration of CO_2 will reduce the CO_2 adsorption uptake capacity.

For the presence of O_2 in gas flow, Heo et al [27] have reported the presence of 5% O_2 in flue gas has no significant effect on the adsorption performance of PEI- titanate nanotubes (PEI-TNTs) during the first cycle. However, after the 10th cycle, the adsorption uptake capacity was reduced by 10% lower than the binary mixture in the presence of O_2. In addition, Wang et al, have observed the reduction of CO_2 adsorption capacity of PEI- PEI-multi-walled carbon nanotubes (PEI-CNTs) in the presence of 5% O_2 in flue gas from 4.75 mmol/g to 4.42 mmol/g [30]. This can be explained by the fact that the amino group of PEI-adsorbents is occupied and oxidized by O_2 that leads to the reducing number of available active sites for CO_2 diffusion, thus decreasing the CO_2 adsorption performance [30].

In contrast, the presence of water vapor has led to an increased CO_2 adsorption performance as compared to the binary mixture [43, 44]. For instance, Wang and co-workers [30] have achieved 4.42 mmol/g of CO_2 adsorption performance by using PEI-multi-walled carbon nanotubes (PEI-CNTs) under a humid gas mixture (15% CO_2, 85% N_2, RH = 50%), a slightly higher CO_2 uptake than that of performed under 15% CO_2, 5% O_2 and 80% N_2 condition (4.22 mmol/g) [30]. It is believed that the enhanced CO_2 adsorption performance is due to the formation of carbamate and bicarbonate in the humid environment, in which the only carbamate is formed in dry conditions [45]. The formation of bicarbonate requires lower amino compared to carbamate during the dry conditions. However, too high amounts of water will cause competition with CO_2 in occupying the active sites of amino [46].

2.5. Effect of crosslinking
One of the problems that countered in the most PEI support technology is the formation of urea during the regeneration process at high contain of CO_2 and high temperature. [47]. The formation of urea has often been produced from the reaction of primary amines and CO_2. This will cause the large drop of CO_2 adsorption uptake at high Adsorption cycle. To overcome these problems jeon et al have studied the effect of modified crosslinking PEI toward the CO_2 adsorption. The modification of crosslinking PEI lead to increasing the surface area but it caused the reduction in the amount of PEI contain. This

can be shown as the CO_2 uptake for PEI-Si is 2.86 mmol/g at 353 K and influence of 85% N_2 whereas the crosslinking PEI-silica (PEI-Si) is 2.06 mmol/g. Hence, it shown the crosslinking contain less amino active sites compared to pristine PEI. Unexpectedly after the 40th cycle, the crosslinking PEI-Si can maintain the greater CO_2 adsorption capacity which is about 61% of its highest value. This performance showed a lot of improvement of chemical stability as compared to the pristine PEI (16.9%). The improvement of chemical stability is due to cross-linkers shape capable of reducing the urea formation by lesser contact of primary amine and CO_2. Thus, reducing the formation of urea. It was also observed that crosslinking PEI with greater alkyl chains shows a lower formation of urea and greater chemical stability.

3. Conclusions

In this work, the recent studies on the PEI impregnated adsorbents for enhanced CO_2 capture have been thoroughly discussed. The summary effects of each parameter on CO_2 adsorption as in Table 2. It is vital to evaluate each parameter in order to achieve effective CO_2 capture technology. It can be concluded that the growing numbers of recent studies on PEI impregnated on various types of adsorbents has been proved could significantly assist to determine the most effective performance and cost for practical applications of CO_2 capture.

Table 2. The summarize the effect of parameters on CO_2 adsorption

Parameters	Effect on CO_2 adsorption
Amine loading	• Achieve maximum CO_2 adsorption capacity at optimum amine loading for those capable of that support. • Too high amount of amine loading will cause a decrease in CO_2 adsorption uptake.
Porous support	• The porous support with better morphological structure tends to perform better in CO_2 adsorption.
Adsorption temperature	• PEI support achieves maximum CO_2 adsorption when it reaches its optimum temperature that depends on their support.
Flow conditions	• Presence of N_2 and O_2 in flow conditions causes negative effects since it causes reduction of the number of amino active sites. • Presence of H_2O in flow conditions causing positive effect due to formation of bicarbonate.
Crosslinking PEI	• This modification causes a drop in CO_2 adsorption uptake capacity but increases the chemical stability of PEI-support.

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