

THE PENNSYLVANIA STATE UNIVERSITY  
SCHREYER HONORS COLLEGE

DEPARTMENT OF CHEMICAL ENGINEERING

ADSORPTION OF CARBON DIOXIDE USING THE MOLECULAR BASKET SORBENT

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A thesis  
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of the requirements  
for a baccalaureate degree  
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with honors in Chemical Engineering

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## ABSTRACT

The Molecular Basket Sorbent (MBS) is a novel material for adsorbing carbon dioxide. MBS consists of a polyethylenimine sorbent on a mesoporous silica support that has proven to have high capacity, regenerability, selectivity, and energy efficiency for adsorbing CO<sub>2</sub>. The purpose of this paper is to describe attempts at increasing the MBS capacity through changing characteristics of the sorbent and to assess the viability of applying this technology to help decrease the amount of CO<sub>2</sub> released into the atmosphere. This is demonstrated through determination of isothermal capacities of several different samples, literature review, and comparison to the literature.

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## CHAPTER 1

### Introduction

#### Part I: Carbon Dioxide Adsorption

Adsorption is a process in which one or more components of a fluid stream, the adsorbate, are held in a thin film by a solid, the adsorbent. This adsorbate-adsorbent interaction can be called either chemical adsorption or physical adsorption. Chemical adsorption, or chemisorption, is caused by chemical bonding interactions. Physical adsorption is mediated by molecular interactions called Van der Waal forces. Van der Waal forces are intermolecular forces that do not arise due to covalent bonding or electrostatic interactions between molecules. Instead, they come from dipole interactions between two molecules. These dipoles can be created by many different methods, one of which is electronegativity. Electronegativity is a property of an atom that measures its affinity to pull electrons toward itself, specifically when it is in a covalent bond with another atom. This is particularly relevant for carbon dioxide ( $\text{CO}_2$ ), as oxygen has a very high electronegativity, while carbon has a relatively low electronegativity. This causes a dipole to form within the  $\text{CO}_2$  molecule, with the carbon atom having a partial positive charge and both oxygen atoms having a partial negative charge. This dipole and acidic property make  $\text{CO}_2$  a prime target for capture via adsorption.

Science has taken advantage of this knowledge and developed methods to effectively capture  $\text{CO}_2$ . One of the most popular methods of doing so is that of metal-organic frameworks (MOFs).

MOFs are a polymeric material that consist of metal ions linked together via organic bridging ligands. These structures can have many large mesopores, leading to a high surface area to volume ratio. This high ratio allows them to have high adsorption capacities and therefore ideal for CO<sub>2</sub> capture.<sup>1</sup> Capacities of MOFs have been reported to be up to 1500 mg CO<sub>2</sub> per mg adsorbent at room temperature and high CO<sub>2</sub> partial pressures.<sup>2-3</sup> However, despite their high capacity, MOFs have a few disadvantages that prevent them from being viable beyond the laboratory: (1) they require a very difficult synthesis process and (2) their structure has poor reproducibility due to partial self-assembly, and interpenetration of pores.<sup>1</sup> All of these lead to an inability to mass-produce a consistent CO<sub>2</sub> adsorbent. Furthermore, most published data on MOFs is at both high CO<sub>2</sub> partial pressure (typically greater than one bar) and low temperature (at or below room temperature). Unfortunately, the industrial processes that produce CO<sub>2</sub> are often above room temperature and below one bar partial pressure. Because of this, there is a clear need for a CO<sub>2</sub> sorbent that successfully operates under low-pressure and high-temperature conditions.

## **Part II: The Molecular Basket Sorbent (MBS)**

In the Song lab, a novel method for CO<sub>2</sub> adsorption was created in the Molecular Basket Sorbent (MBS). The MBS is created by loading a CO<sub>2</sub>-philic polymer adsorbent, polyethylenimine (PEI), onto a mesoporous silica, SBA-15. The structure of PEI can be found in Figure 1 below.

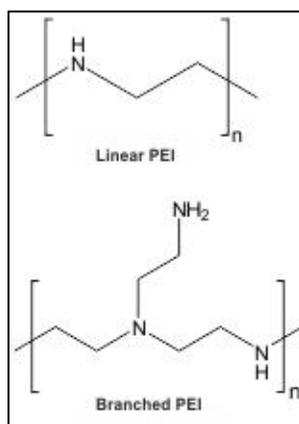


Figure 1. The structure of linear and branched PEI.<sup>4</sup>

Using a fixed-bed flow system that allows for control over both CO<sub>2</sub> partial pressure and temperature, MBS using a 50 weight % loading of PEI on MBS was determined to be 90 mg CO<sub>2</sub>/g MBS.<sup>5</sup> MBS has also proved to have a high regenerability, as studied through a temperature-programmed desorption method with a 110°C regeneration temperature. Further characterization proved that MBS has not only high capacity and regenerability, but high selectivity and energy efficiency as well. It is believed that these are largely due to the interactions present between SBA-15 and PEI. The surface of SBA-15 cooperates with the amine groups of the PEI to form Si-O<sup>-</sup> N<sup>+</sup>H<sub>3</sub>R and/or Si-O<sup>-</sup> N<sup>+</sup>H<sub>2</sub>R electro-static interactions. It is these interactions that help thermally stabilize the PEI, decrease its fluidity, and overall increase the adsorption capacity. At the time of its conception, the MBS showed a capacity above any other known technique at high temperatures and low pressures.<sup>5</sup>

This past work, along with the ever-present possibility for improvement on current technologies, provides the basis for this experiment's attempt at creating an improved MBS.

### Part III: Applications

Due to human activities, greenhouse gas emissions have seen a significant worldwide increase during the last couple of decades. This has resulted in a notable increase in the concentration of these gases in the atmosphere, resulting in levels higher than any recorded for hundreds of thousands of years. These high levels have resulted in global climate change that can cause serious adverse effects such as rising seas, incredible storms, and other forms of extreme weather.<sup>6</sup> The commonly accepted safe concentration of CO<sub>2</sub> is 350 parts per million, and having surpassed the 400 parts per million mark globally in February of 2015, it is clear that something must be done in order to not only prevent a further rise in CO<sub>2</sub> concentration, but to also ensure its decline.<sup>7</sup>

As an ultimate goal for this project, it is the desire of the Song lab to provide the MBS as this solution – with this paper providing further basis for improvement towards that goal. A very large majority of the CO<sub>2</sub> released into the atmosphere is due to electricity production via coal-fired power plants, so naturally they are a prime target for CO<sub>2</sub> reduction methods.<sup>6</sup> The Molecular Basket Sorbent could be applied to a power plant's effluent gases in order to strip them of their CO<sub>2</sub>, thereby preventing its release into the atmosphere. Currently, liquid amine scrubbing techniques are used, but they have the severe drawbacks of amine degradation, slow adsorption and desorption, and very energy intensive operation.<sup>8</sup> After being sequestered in the MBS, CO<sub>2</sub> is easily released through the MBS regeneration process described earlier and further discussed by Ma, Wang, and Song.<sup>5</sup> This cycle will be able to significantly reduce the amount of CO<sub>2</sub> released into the air by coal-fired power plants, but will ultimately create an additional cost

for the plants operation. This increased cost of operation will be assessed in greater depth later in this paper.

#### **Part IV: Research Problem and Hypothesis**

My present hypothesis is that a novel MBS can be created to improve on its current performance through the testing of different mesoporous supports, molecular weights of polyethylenimine, and different PEI loadings. Currently in its second generation, the MBS has already seen significant improvement by changing the mesoporous silica used as support, although the effects of different PEI molecular weights have not been thoroughly examined. The successful completion of this experiment will provide the basis for further research on advancing MBS technology so that is suitable for industrial use.

## CHAPTER 2

### Materials and Methods

#### Part I: Preparation of Mesoporous Supports

The standard support for the Molecular Basket Sorbent is the mesoporous silica SBA-15. The SBA-15 used to prepare the samples tested in this experiment was prepared in house. Approximately 26 g of P-123, a triblock copolymer, 120 mL of HCl, and 600 mL of deionized H<sub>2</sub>O were added to a 1 L beaker and stirred until all of the P-123 dissolved. The beaker was then heated to 40°C and approximately 50 g of tetraethyl orthosilicate (TEOS) was added drop-wise. The TEOS functions as a crosslinking agent for the P-123. The contents were then covered and stirred for 20 hours at 40°C. The solution will go from slightly opaque in appearance to completely white. After 20 hours, two autoclaves with Teflon inserts were prepared and the contents of the beaker were poured in. The autoclaves were placed in a vacuum oven at 100°C for 24 hours. Upon completion of the 24 hours, the contents were allowed to cool to room temperature. The SBA-15 that precipitated upon cooling was recovered using vacuum filtration to separate it from the leftover fluids and then covered and left in a vacuum oven at 100°C overnight. Finally, the sample was then calcined at 550°C for 6 hours using a ramp rate up to temperature of 1°C per minute. The other two mesoporous supports used in this experiment, activated carbon and CAB-O-SIL M-5 fumed silica, were purchased from Sigma-Aldrich and Cabot Corporation respectively.

## **Part II: Preparation of MBS**

Each MBS sample, regardless of support or polyethylenimine molecular weight, was prepared following the same basic wet impregnation procedure. The appropriate amount of PEI was massed in a beaker according to the samples desired loading capacity on a basis of 1.0000 g of support (0.4286 g for 30% and 1.0000 g for 50% loading). Approximately 50 g of methanol was then added to the beaker and stirred with a Teflon stir bar while covered under ambient conditions for 15 minutes. 1.0000g of support was then added to the beaker and stirred while covered under ambient conditions for 5 hours. The sample was then stirred overnight under 40°C in the fume hood. The sample was then placed in a vacuum oven at approximately 40°C to further evaporate any methanol left in the beaker. The sample was then ground into a fine powder using a mortar and pestle, massed, and placed into a glass vial for storage. Note: the 50% PEI/SBA-15 samples were previously prepared using a different batch of SBA-15 than the 30% PEI/SBA-15 samples.

The polyethylenimine used in this experiment was purchased from Sigma-Aldrich. Linear PEI with molecular weights of 600, 1200, and 1800 g/mol were used.

## **Part III: Determination of Isothermal Capacity of MBS**

Each Molecular Basket Sorbent sample was tested to determine its isothermal CO<sub>2</sub> adsorption capacity. Data were obtained at 0°C, 22°C, and 50°C for all samples. Because of the desire to employ the MBS in a coal-fired power plant, it is imperative that the MBS be tested at temperatures relevant to power plant effluent temperatures. Before data collection, each sample

was pre-treated under a nitrogen stream at 100°C for one hour at a ramp rate of 15°C per minute.

To maintain isothermal surroundings during data collection, temperature baths were used to submerge the sample tubes. At 0°C, ice and water were placed in a dewar and allowed to equilibrate. For 22°C, room temperature water was placed in a dewar. For 50°C, a glycol bath was used. Figures 2-9 below summarize the obtained results.

## CHAPTER 3

### Results and Discussion

#### Part I: Isothermal CO<sub>2</sub> Adsorption Capacities

The following data were obtained using a TriStar 3020 to determine isothermal CO<sub>2</sub> adsorption capacity. Note: due to time constraints in obtaining data, experimentation was not carried out for all samples at all temperatures, but trends can still be observed.

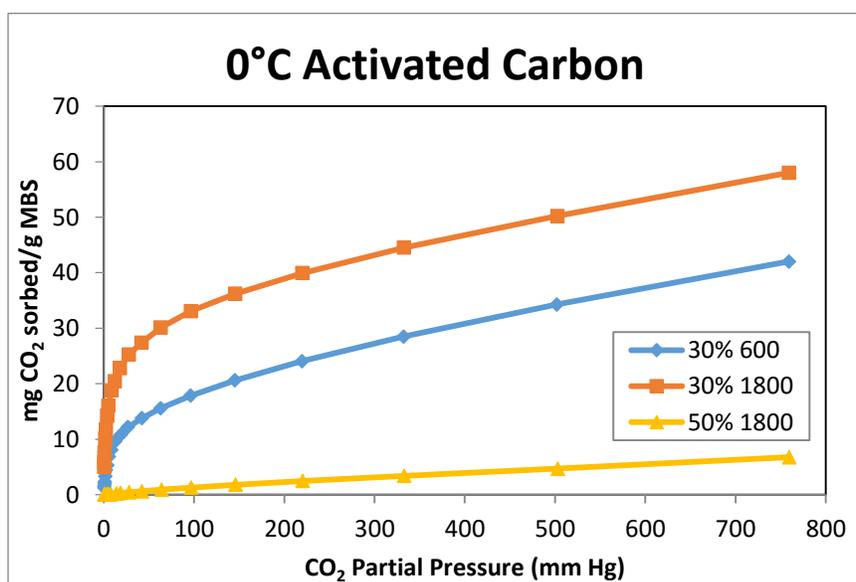


Figure 2. CO<sub>2</sub> adsorption capacity for different PEI loadings using activated carbon support at 0°C.

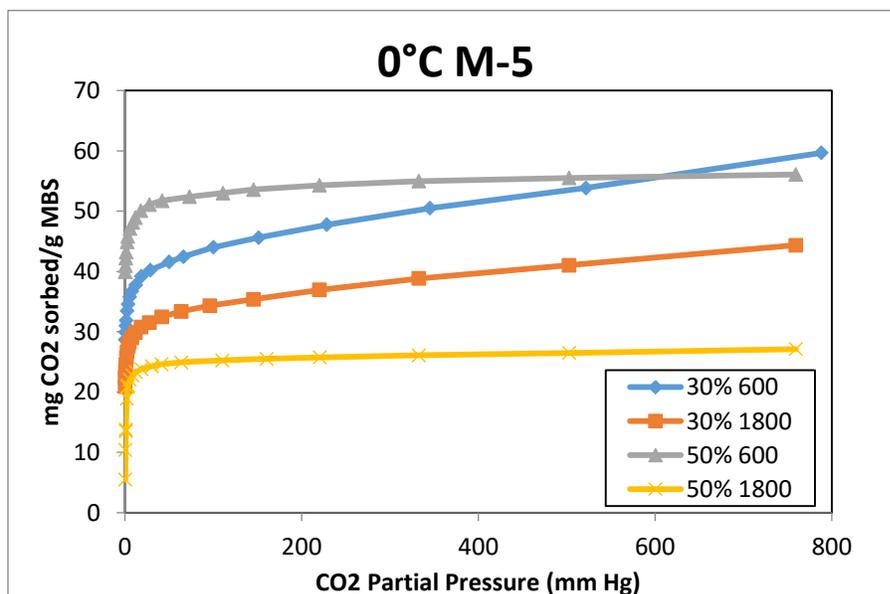


Figure 3. CO<sub>2</sub> adsorption capacity for different PEI loadings using M-5 support at 0°C.

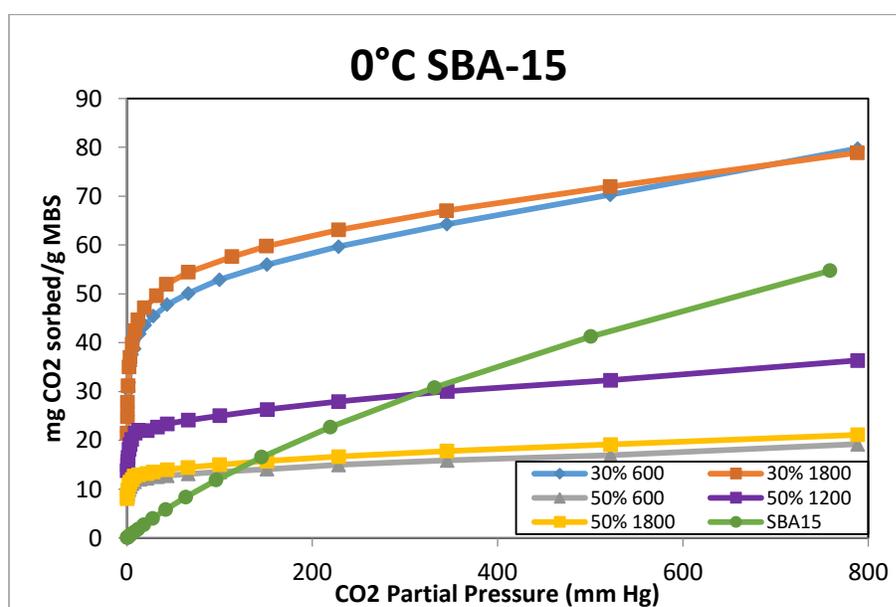
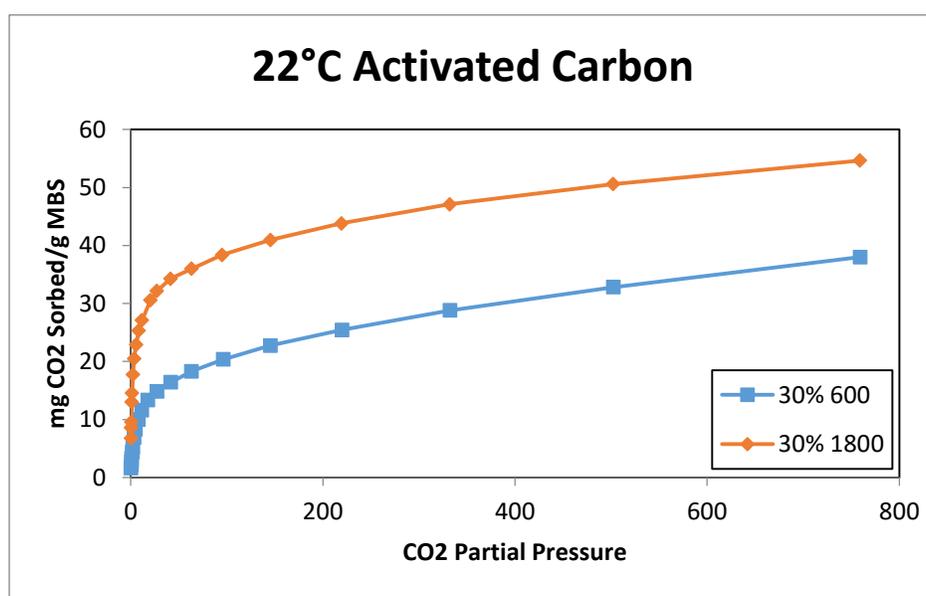


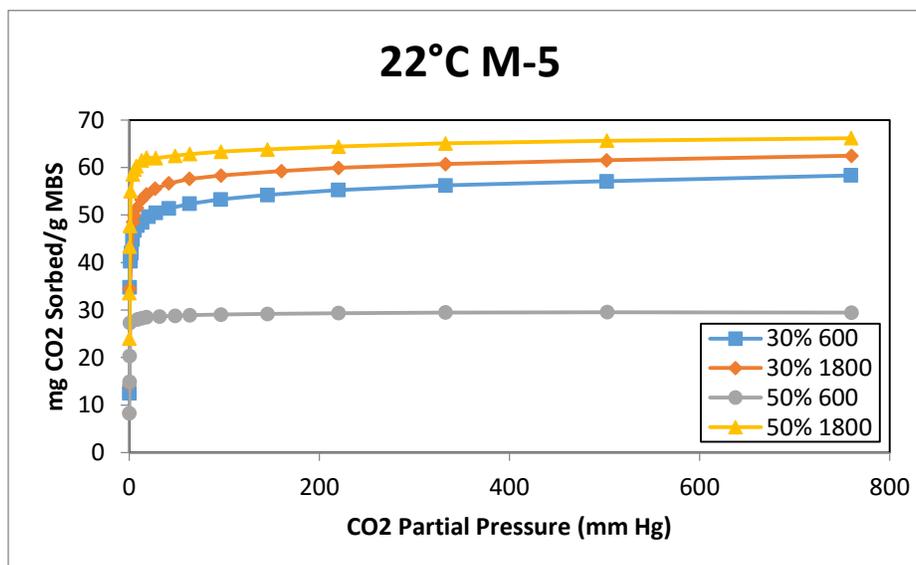
Figure 4. CO<sub>2</sub> adsorption capacity for different PEI loadings using SBA-15 support at 0°C.

At 0°C it is easy to tell that MBS with 30% PEI loadings outperformed those with 50% loadings.

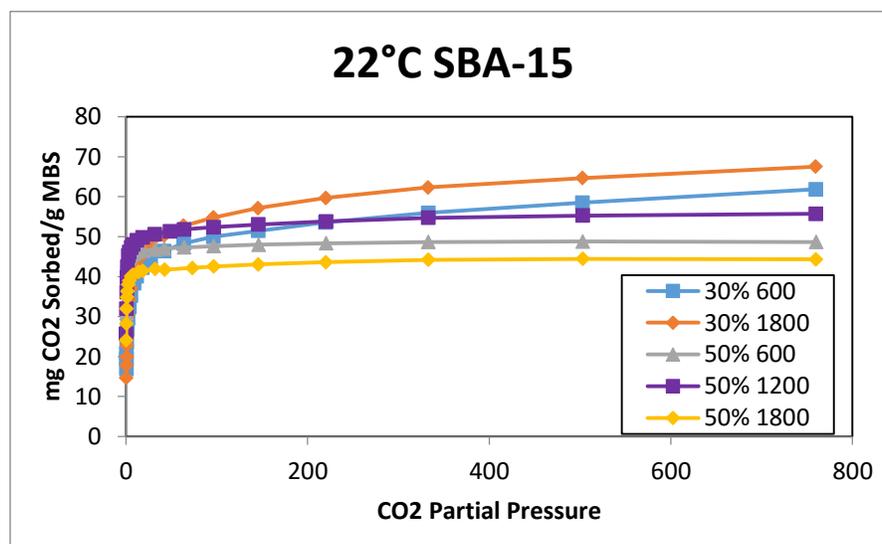
A possible reason behind this is that having a loading of 50% or more could oversaturate the pores in the support, causing the overall surface area to decrease and therefore decrease the capacity. However, there is no apparent trend between PEI molecular weight and CO<sub>2</sub> capacity. The highest capacity obtained of approximately 80 mg CO<sub>2</sub> per g MBS is on par with the current generation of MBS, but most samples remain at or below 60 mg/g.



**Figure 5. CO<sub>2</sub> adsorption capacity for different PEI loadings using activated carbon support at 22°C.**



**Figure 6. CO<sub>2</sub> adsorption capacity for different PEI loadings using M-5 support at 22°C.**



**Figure 7. CO<sub>2</sub> adsorption capacity for different PEI loadings using SBA-15 support at 22°C.**

At 22°C, the general trend of 30% loading having a higher capacity than 50% loading continues. However, MBS with a higher PEI molecular weight are starting to perform better than those with lower molecular weights. This could possibly be due to the higher molecular weights providing

some extra thermal stability at higher temperatures. Overall, the adsorption capacities have increased with temperature. This is unexpected, as adsorption is an exothermic process and it would therefore be reasonable to presume that it would become less favorable as temperature increases. However, this is not the case and therefore suggests behavior that is more complex than simple adsorption is occurring. To explain this, the adsorption-diffusion-absorption mechanism has been suggested. This states that the CO<sub>2</sub> is first adsorbed onto the PEI surface, but then diffuses through the PEI into the bulk of the support, effectively being absorbed into the PEI.<sup>9</sup> This is able to explain the increase in adsorption with increase in temperature because the increase in thermal energy allows the CO<sub>2</sub> to diffuse into the bulk more favorably than it will desorb.

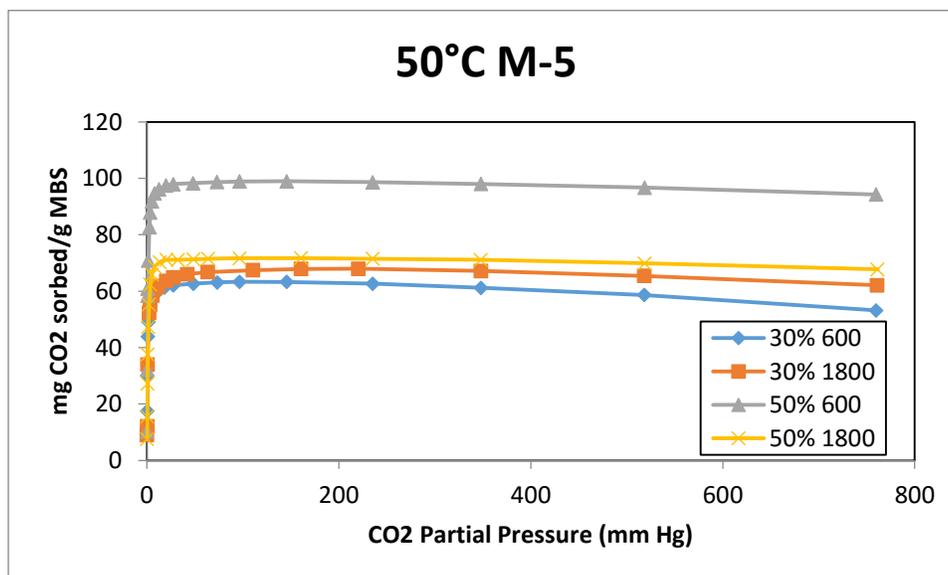


Figure 8. CO<sub>2</sub> adsorption capacity for different PEI loadings using M-5 support at 50°C.

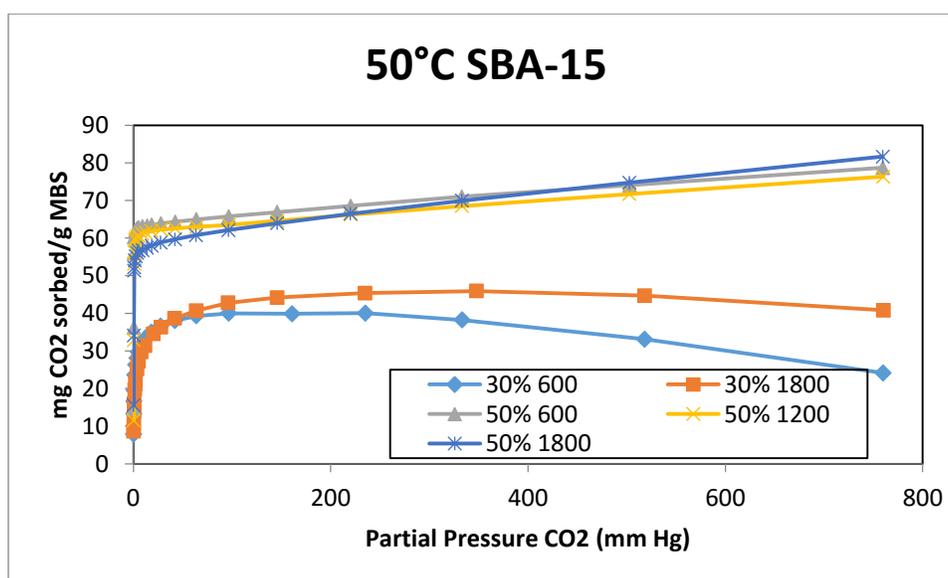


Figure 9. CO<sub>2</sub> adsorption capacity for different PEI loadings using SBA-15 support at 50°C.

Finally, we can see that increasing the temperature further to 50°C encouraged the diffusion of CO<sub>2</sub> into the MBS bulk and increased the adsorption capacity, having now reached capacities of nearly 100 mg/g. The heavier PEI samples have now obtained higher capacities than their lighter

counterparts, which can also be attributed to the adsorption-diffusion-absorption mechanism. PEI-1800 has a viscosity 10 to 20 times that of PEI-600, which causes the heavier PEI to have lower diffusivity at lower temperatures. As the temperature increases, the CO<sub>2</sub> can more easily diffuse through it, but not so easily that desorption begins to become more favorable, which lowers the capacity as seen in the PEI-600 samples. This decrease in capacity by the lighter PEI samples suggests that there is an optimal operating temperature and associated time frame for not only MBS made with PEI-600, but for MBS of all molecular weights.

## **Part II: Assessment of Industrial Use**

To assess MBS suitability for industrial use, a stoichiometric analysis of a coal-fired power plant was performed using Microsoft Excel. Assuming a 500 MW-producing plant with a 35% efficiency using a coal with a heating value of 32,029 kJ/kg, it was determined that nearly 7500 kg/min CO<sub>2</sub> was released from the burning of coal. Assuming a sorbent CO<sub>2</sub> load capacity of 100 mg/g, 50 minute cycling time, and the standard 19.3m tall scrubber, this 500 MW plant would require 30 scrubbers running to achieve a somewhat reasonable diameter of 1.92 m per scrubber in order to capture only 10% of the effluent CO<sub>2</sub>. This would also generate a large amount of heat, due to the fact that adsorption is an exothermic process. Therefore, part of the plant's energy produced would be used to cool the stripping towers, and thereby reducing the plant's effective output. This cost would certainly not go unnoticed, and would undoubtedly be absorbed by the customers of the power plant. Based on this simple mass and energy based model, and with the average price of electricity in Pennsylvania being \$0.1275/kWh, the average price would see a 3.3% price increase if only 10% of effluent CO<sub>2</sub> were captured and a 29.5%

price increase if 90% of the effluent CO<sub>2</sub> were to be adsorbed.<sup>10-12</sup> Calculations can be found in the spreadsheet shown in the appendix.

## CHAPTER 4

### Conclusion

This thesis work led to the successful characterization and determination of certain optimizing characteristics of the Molecular Basket Sorbent. Although it is not currently ready for industrial use due to the large scrubbing tower size and potential increase in cost of electricity, the MBS's capacity can be manipulated in many different ways. In this paper, it was found that M-5 fumed silica could be a support used to replace SBA-15 in order to improve MBS technology as M-5 samples had similar or better CO<sub>2</sub> adsorption capacities as the SBA-15 samples. It was also found that polyethylenimine molecular weight had an effect on capacity; in general, a higher molecular weight provided a higher capacity at higher temperatures. However, in order to find the optimum conditions and sorbent/support pairing, more experimentation is needed.

This research was completed in the Summer of 2013 and Spring of 2015. Since then, the influence of the solute used during impregnation (water vs. methanol vs. ethanol vs. propanol vs. butanol) has begun to be studied in order to better optimize the MBS. All attempts to maximize the CO<sub>2</sub> adsorption capacity will be useful in the efforts to create a viable industrial sorbent to help combat the increasing atmospheric CO<sub>2</sub> levels.

## APPENDIX

### 500 MW Power Plant

Fuel composition	As received	Coal rate=	1,429 MW(chemical)	Stoich O2=	190.3 kmol/min		
C (mass%)	76%	=	4.9E+09 BTU/h	=	6090.351034 kg/min		
H	4.00%	=	2.676 kg/min				
O	8.00%						
S	0.50%						
N	1.50%						
H2O	0.00%						
Ash	10.0%						
Total	100.0%						
Power=	500 MW(e)						
HHV=	32029 kJ/kg						
=	13770 BTU/lb						
Efficiency=	35%						
Excess air=	10%						
Higher Heating Values							
Coal Grade	Heating Value (Btu/lb)						
Anthracite	12910						
Semi-Anthracite	13770						
Low-volatile bituminous	14340						
Medium-volatile bituminous	13840						
High-volatile bituminous A	13090						
High-volatile bituminous B	12130						
High-volatile bituminous C	10750						
Subbituminous B	9150						
Subbituminous C	8940						
Lignite	6900						
		IN (kmol/min)	FORMED	CONSUMED	OUT (kmol/min)	MOL FRAC	
O2	209.4	0.0	190.3	19.0	0.0186	O2	624.7
N2	779.1	0.0	0.0	779.1	0.7609	N2	25569.9
CO2	0.0	169.3	0.0	169.3	0.1654	CO2	5557.9
H2O	0.0	53.1	0.0	53.1	0.0519	SO2	13.7
SO2	0.0	0.4	0.0	0.4	0.0004		
NO2	0.0	2.9	0.0	2.9	0.0028		
H	106.2	0.0	106.2	0.0	0.0000		
S	0.4	0.0	0.4	0.0	0.0000		
C	169.3	0.0	169.3	0.0	0.0000		
O	13.4	0.0	13.4	0.0	0.0000		
N	2.9	0.0	2.9	0.0	0.0000		
Total	1264.4	225.7	466.3	1023.8	1.0000		
		IN (kg/min)	FORMED	CONSUMED	OUT (kg/min)	MASS FRAC	
O2	6699.4	0.0	6090.4	609.0	0.0196	O2	31.9988
N2	21824.4	0.0	0.0	21824.4	0.7040	N2	28.0134
CO2	0.0	7452.5	0.0	7452.5	0.2404	CO2	44.0095
H2O	0.0	956.6	0.0	956.6	0.3309	H2O	18.01528
SO2	0.0	26.7	0.0	26.7	0.0009	H	1.008
NO2	0.0	131.8	0.0	131.8	0.0041	S	32.065
H	107.0	0.0	107.0	0.0	0.0000	C	12.0107
S	13.4	0.0	13.4	0.0	0.0000		
C	2033.9	0.0	2033.9	0.0	0.0000		
O	214.1	0.0	214.1	0.0	0.0000		
N	40.1	0.0	40.1	0.0	0.0000		
Total	30932.3	8567.7	8244.6	31001.1	1.0000		

**Figure 10. Mass balance for a 500 MW coal fired power plant.**

This mass balance was used to determine stripping tower sizing and heating balance. The full spreadsheet (which allows for value manipulation after downloading) can be downloaded from this [link](#).

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