

Carbon Aerogels for High Capacity and Selective Adsorption of Carbon Dioxide

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Abstract:

Carbon aerogels are light, highly porous materials with high surface areas. Their applications have been demonstrated in a wide range of areas, one of which is carbon capture. We have synthesized a new carbon aerogel that specializes in the selective adsorption of CO₂, which was previously synthesized from a new THQ (tetrahydroquinazoline) monomer. This monomer shares a similar structure to Ishida's BO (benzoxazine) monomer, differing in a substitution of the oxygen heteroatom with nitrogen, which succeeded in increasing CO₂ absorption capacity after aerogel synthesis. The THQ monomer was polymerized to form a PTHQ aerogel, which was then oxidized, carbonized, and etched to form a carbon aerogel. This process has several steps, which affect the characteristics of the derived aerogel. PTHQ-derived carbon aerogels were evaluated for their CO₂ adsorption capacity and selectivity towards other gases. CO₂-etched carbon aerogels showed very high CO₂ uptake (11.2 ± 0.9 mmol g⁻¹ at 273 K, 1 bar). The high selectivity of CO₂ versus H₂ in the range of (407 ± 104) is attractive for pre-combustion capture of CO₂ and the high selectivity of CO₂ versus N₂ in the range of (52 ± 18) is attractive for post-combustion CO₂ capture from flue gases.

Introduction:

Global warming has been a major issue during the last few decades. CO₂, CH₄, and NO₂, as the main greenhouse gases in the atmosphere, are the major cause of global warming and atmospheric climate change. Their concentrations in the atmosphere are still increasing due to human activities such as burning of fossil fuels.¹⁻⁵ Consequently, the atmospheric CO₂ concentration has exceeded from the pre-industrial value of 280 ppm to over 415 ppm in 2020, and that of CH₄ has also doubled.^{6,7} Due to COVID-19, there is significant reduction in anthropogenic CO₂ emission⁸ but, a pandemic is not a solution for reducing the concentration of greenhouse gases in atmosphere. Thus, carbon capture and sequestration (CCS) has been considered as an optional approach to reduce CO₂ concentration, which contributes to global warming. CCS refers to capturing CO₂ emission from large scale sources, such as power plants, transferring to a storage and disposing it so that it will not return to the atmosphere.^{9,10}

One method of CCS is through CO₂ absorption in carbon aerogels. Carbon aerogels are microporous carbons which are chemically inert, physiochemically stable with wide pore size distribution and high surface area.^{11,12} They are carbonizable polymeric aerogels synthesized via typical sol-gel chemistry and pyrolyzed to yield carbon aerogel.¹³⁻¹⁵ Chemical transformation during pyrolytic carbonization are responsible for the rigidity and porosity of the carbon aerogel. Surface properties of the carbon aerogels can be modified by doping heteroatom in the polymeric system (e.g., oxygen and nitrogen) to increase interaction with CO₂. Thus, they are viable candidates for CCS due to their template-free synthesis, high thermal stability, tunable surface chemistry, weak physical interaction with CO₂, and high selectivity toward other gases. They are additionally stable at room temperature.

For these reasons, we are creating carbon aerogels with the maximization of absorption of CO₂ along with selective adsorption of carbon dioxide versus other common gases (notably

hydrogen, methane, and nitrogen) so that an effective and efficient CCS mechanism may be found, which can then be used to reduce CO₂ emissions into the atmosphere.

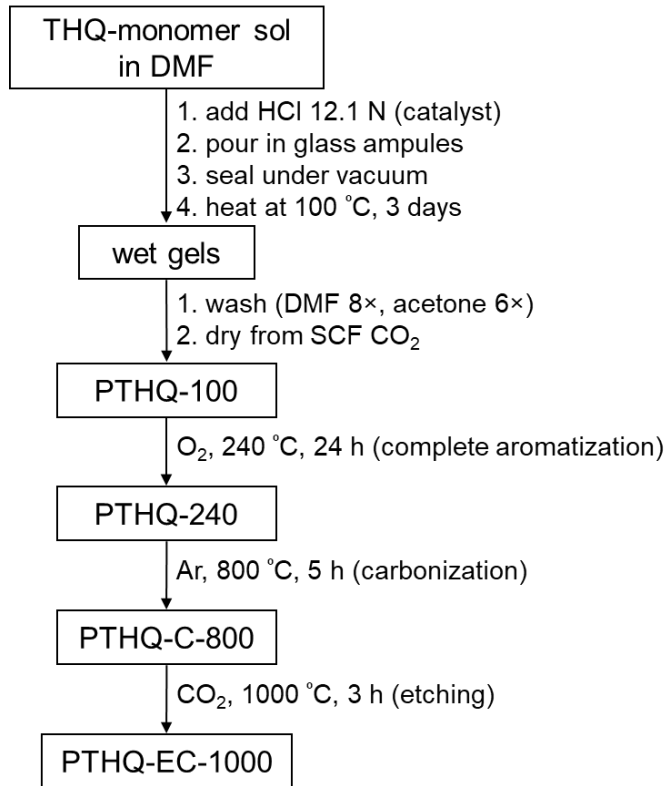
We are working with a base THQ monomer that is structurally similar to Ishida's benzoxazine monomer, differing majorly in heteroatom makeup. It is thought that heteroatoms such as O and N may play a decisive role in the properties of porous carbon materials.¹⁶ In particular, nitrogen-containing functional groups are thought to improve CO₂ interaction for absorption purposes.¹⁷⁻²¹ A comparison can thus be made between aerogels made from THQ and BO to compare N-rich versus O-rich porous carbons as adsorbers.

Experimental:

Preparation of PTHQ Carbon Aerogels:

Scheme 1 summarizes the synthesis of PTHQ carbon aerogels. The THQ monomer was synthesized by using the process from our previous work. The synthesis of polymeric PTHQ aerogels was carried out via HCl catalyzed ring opening polymerization. Gelation was carried out at 100 °C for 3 days. The concentration of THQ monomer in DMF solution was 20% w/w. Gelation solvent was exchanged with acetone, and wet gels were dried into aerogels supercritically using liquid CO₂ in an autoclave which was further vented off as a gas. The aerogels obtained after drying were referred to as PTHQ-100. PTHQ-100 aerogels were subjected to oxidation at 240 °C under flowing oxygen for 24 h and the aerogels obtained were referred to as PTHQ-240. The ring fusion aromatization during oxidation via well-established oxygen/ superoxide/ hydroperoxyl radical chemistry is prerequisite for obtaining sturdy carbons after carbonization. The carbonization of PTHQ-240 aerogels was conducted at 800 °C under flowing high-purity argon and referred to as PTHQ-C-800. The carbonization yield was found to be 61%. Reactive etching of PTHQ-C-800 carbon aerogels was conducted at 1000 °C under flowing CO₂ and referred to as PTHQ-EC-1000 with the etching yield of 35%. Reactive etching removed more than half the mass of carbonized aerogel but it is responsible for the development of high surface area and the formation of micropores.

Scheme 1. Synthetic scheme of PTHQ carbon aerogels.



General Material Properties of PTHQ-C-800 and PTHQ-EC-1000:

In terms of materials properties, PTHQ-C-800 shrunk 45% relative to their parent aerogel PTHQ-100 while PTHQ-EC-1000 shrunk 60%. Overall, total shrinkage is 71% and 85% respectively for carbonized and etched samples relative to the molds. The bulk density, ρ_b , of PTHQ-C-800 is $(0.225 \pm 0.001 \text{ g cm}^{-3})$ which after etching reduced significantly to $(0.152 \pm 0.021 \text{ g cm}^{-3})$ for PTHQ-EC-1000. Skeletal density, ρ_s , of PTHQ-C-800 $(2.197 \pm 0.018 \text{ g cm}^{-3})$ is close to the range of expected densities for amorphous carbons $(1.8\text{-}2.0 \text{ g cm}^{-3})$ while for PTHQ-EC-1000, ρ_s , is $(1.843 \pm 0.012 \text{ g cm}^{-3})$. The porosity of both the aerogels remained high in the range of 90-92% (Table 1).

Porous Nanostructure of PTHQ-C-800 and PTHQ-EC-1000 Aerogels:

Even though SEM images showed meso- and macroporous nature of the skeletal framework, microporosity is not visible in SEM. The CO_2 adsorption capacity of the material is largely dependent on its micropore volume along with surface functionalities. In order to study the carbons obtained from PTHQ as CO_2 adsorber, quantitative evaluation of pore structure is important. Thus, meso- and macroporosity were evaluated with medium pressure N_2 -sorption porosimetry at 77 K. Microporosity was probed with low-pressure N_2 -sorption and with CO_2 adsorption at 77 K and 273 K respectively.

Figure 1 shows N_2 -sorption isotherms for both PTHQ-C-800 and PTHQ-EC-1000 carbon aerogels. They were Type I isotherms with narrow saturation plateau, signifying micro- and macroporosity. Both carbon and etched carbon aerogels show a rapid rise of the volume of N_2 adsorbed at $P/P_0 \ll 0.1$ indicating microporosity. PTHQ-EC-1000 show dramatic rise in volume of N_2 adsorbed after etching as compared to PTHQ-C-800. In fact, the etching process gave rise to high surface area and microporosity which was further conformed by applying density functional theory (DFT) method to CO_2 adsorption isotherm. The open porosity was evaluated by comparing total pore volumes calculated via $V_{\text{Total}} = (1/\rho_b) - (1/\rho_s)$ with V_{max} (the total volume of N_2 uptaken during N_2 -sorption porosimetry as $P/P_0 \rightarrow 1$). The data for both the samples are shown in Table 1.

Table 1. Material properties of PTHQ and PBO derived carbon and etched carbon aerogels.

Sample	Linear shrinkage [%] ^{a,b}	Bulk density, ρ_b [g cm ⁻³] ^a	Skeletal density, ρ_s [g cm ⁻³] ^c	Porosity Π [% v/v] ^d	Specific pore volume [cm ³ g ⁻¹]			Surface area [m ² g ⁻¹]		Average pore diam. [nm] via $4 \times V/\sigma$ ⁱ	
					V_{Total} ^e	$V_{1.7-300nm}$ ^f	V_{max} ^g	BET, σ	Micropore ^h	$V = V_{Total}$	$V = V_{max}$
PTHQ-C-800	46.8 ± 0.1	0.225 ± 0.001	2.197 ± 0.018	90	3.99	0.05	0.19	346	274	46	3.0
PTHQ-EC-1000	47.5 ± 0.6	0.152 ± 0.012	2.184 ± 0.012	93	6.12	0.21	1.02	1650	950	15	2.4
PBO-C-800	36.1 ± 0.4	0.303 ± 0.004	1.658 ± 0.009	82	2.70	0.12	0.29	351	252	22	3.3
PBO-EC-1000	37.8 ± 2.0	0.137 ± 0.023	1.834 ± 0.040	93	6.75	0.35	1.11	1591	1038	17	2.8

^a Average of five samples; ^b All values relative to molds; ^c Single sample, average of 50 measurements; ^d Via $\Pi = 100 \times (\rho_s - \rho_b)/\rho_s$; ^e $V_{Total} = (1/\rho_b) - (1/\rho_s)$; ^f BJH-desorption cumulative pore volume; ^g V_{max} : single point N₂ adsorption at 77 K as $P/P_0 \rightarrow 1.0$; ^h Via the t -plot method; ⁱ For V_{Total} and V_{max} refer footnotes (e) and (f).

Table 2. Micropore analysis and CO₂ uptake of PTHQ and PBO derived carbon and etched carbon aerogels.

Sample	Specific micropore volume [cm ³ g ⁻¹]				Pore diameter [nm] via $4 \times V/VI$ (micropore area) ^e		CO ₂ uptake [mmol g ⁻¹] at 273 K, 1 bar			
	DR (N ₂) ^a	HK (N ₂) ^b	DR (CO ₂) ^c	DFT (CO ₂) ^d	V from DR (N ₂)	V from DR (CO ₂)	Measured	From surface area		From DR (N ₂) ^h
								BET ^f	Micropore ^g	
PTHQ-C-800	0.16	0.15	0.36 ± 0.09	0.10 ± 0.01	2.5	4.7	5.51 ± 0.1	3.02	2.48	3.71
PTHQ-EC-1000	0.98	0.54	0.85 ± 0.05	0.14 ± 0.02	4.1	3.6	11.20 ± 0.9	16.11	9.27	22.66
PBO-C-800	0.20	0.18	0.19 ± 0.02	0.06	2.0	2.2	2.80 ± 0.1	3.42	2.46	4.65
PBO-EC-1000	0.70	0.55	0.39 ± 0.03	0.12 ± 0.00	2.7	1.5	4.60 ± 0.2	15.53	10.13	16.27

^a Via the Dubinin-Radushkevich (DR) method; ^b Via Horvath-Kawazoe (HK) from N₂-sorption data obtained at 77 K using a low-pressure transducer ($P/P_0 \leq 0.01$). Single experiment; ^c Via the DR method from CO₂ adsorption data at 273 K upto relative pressure 0.03. Data are averages from three experiments; ^d As in footnote (b) using DFT method. Pore volumes are for pores < 1 nm in size; ^e Calculated using micropore surface area via t -plot method; ^f Calculated by dividing BET surface area over CO₂ cross sectional area (0.17 nm²), over Avogadro's number; ^g Calculated by dividing micropore area over CO₂ cross sectional area (0.17 nm²), over Avogadro's number; ^h Calculated by assuming that micropore volumes (a) and (b) are filled with liquid CO₂ (density of liquid CO₂ at 273 K, was taken equal to the density of absorbed CO₂ (1.023 g cm⁻³))

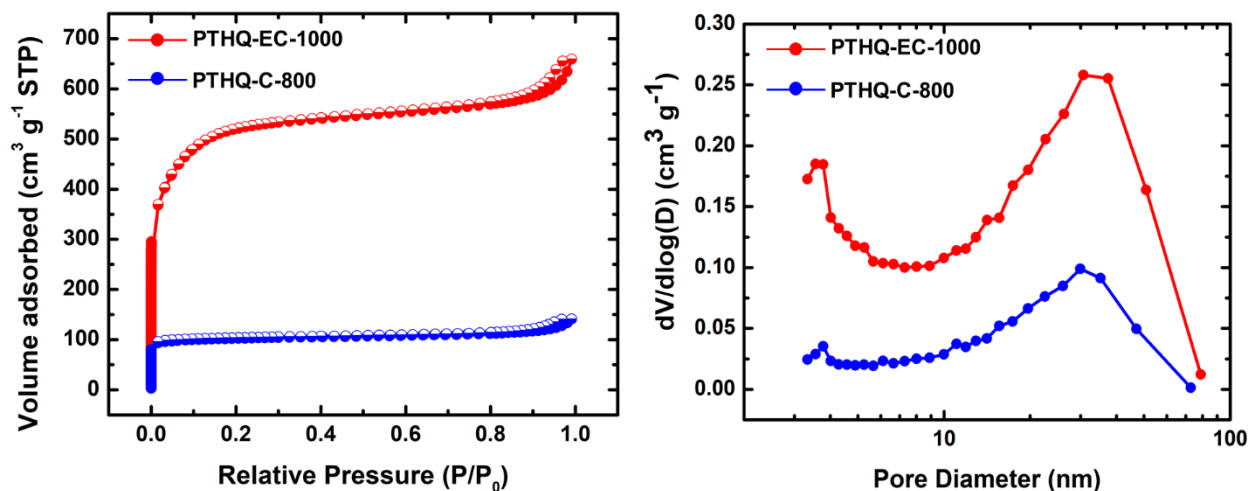


Figure 1. N₂-sorption isotherms for both carbon and etched carbon aerogels (left) and their corresponding pore size distribution from BJH method (right).

BET surface area, σ , was calculated from medium pressure N₂-sorption followed by t -plot analysis of micropore area with the Harkins and Jura method. The surface area of PTHQ-C-800 aerogel is $346 \pm \text{m}^2 \text{g}^{-1}$ which increases more than 4 \times to $1650 \pm \text{m}^2 \text{g}^{-1}$ after etching for PTHQ-EC-1000. BET surface area attributed to micropores was 76% and 42% for C and EC carbon aerogels respectively.

The quantitative evaluation of average pore size was calculated via $4V/\sigma$ method. Two sets of independent data of pore volume was used from independent experiments. Thus, pore sizes were calculated by setting $V = V_{\text{Total}}$ or $V = V_{\text{max}}$. The pore sizes calculated from two methods diverge significantly from one another. In case of PTHQ carbon and etched carbon aerogels, pore size calculated from $V = V_{\text{Total}}$ are much larger for $V = V_{\text{max}}$, indicating a higher contribution from larger macropores not probed by N₂-sorption (>300 nm).

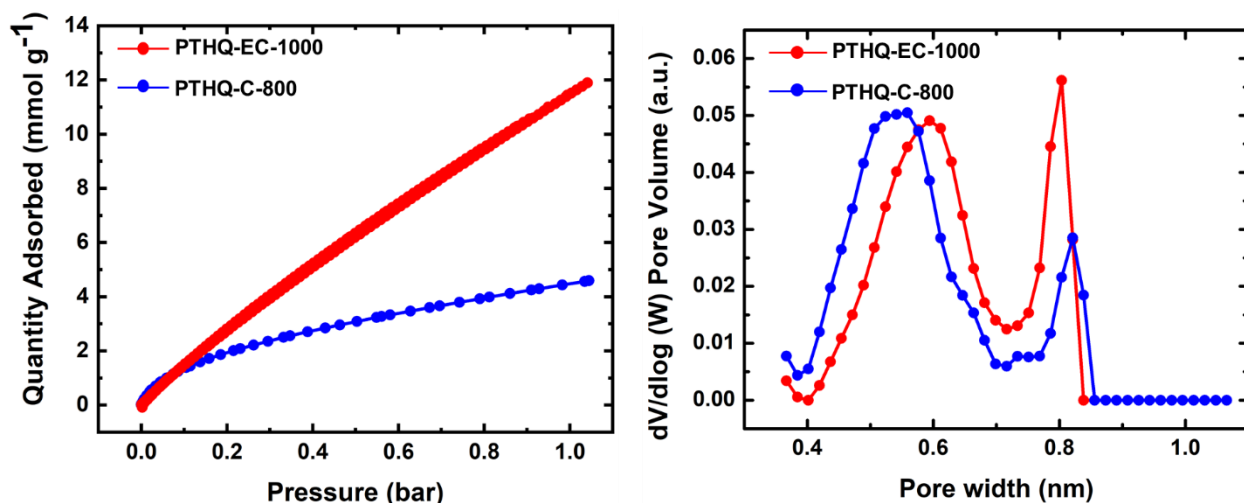


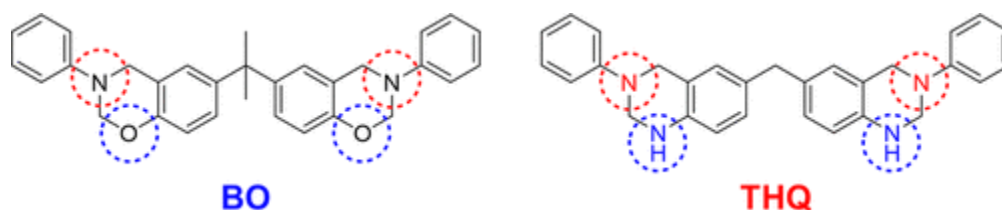
Figure 2. CO₂ adsorption isotherm of PTHQ carbon and etched carbon aerogels at 273 K (left) along with pore size distribution calculated by DFT method assuming slit pore geometry (right).

Micropore size distribution of PTHQ carbon and etched carbon aerogels are shown in Figure 2. The micropore size distribution for both the aerogels looked similar to one another. However, etching process resulted in wider cavities slightly shifting the size distribution towards larger sizes.

Preparation of PBO Carbon Aerogels:

PBO aerogels were synthesized using identical steps to that of PTHQ aerogels, following the process detailed in Scheme 1, with the exception of starting with a base benzoxazine monomer instead of THQ. The critical difference in these monomers is the presence of oxygen heteroatoms in BO, as shown in Scheme 2. Due to the increased amount of nitrogen heteroatoms present in THQ compared to BO, an aerogel made from THQ should have increased CO₂ absorption capacity.

Scheme 2. Chemical structure of BO and THQ monomers.



PTHQ and PBO carbon and etched carbon aerogels as CO₂ adsorbers:

The CO₂ uptake isotherm at 273 K and 1 bar ($P/P_0 = 0.03$) for PTHQ-C-800 and PTHQ-EC-1000 are shown in Figure 2. Significantly high uptake of CO₂ ($11.20 \pm 0.9 \text{ mmol g}^{-1}$) was observed for PTHQ etched carbon aerogels. That value is higher than all the porous CO₂ sorbents, microporous polymers, carbon based adsorbers and carbon nanotubes (CNTs) we are aware of in the literature except etched RF while CO₂ uptake value for PTHQ-C-800 ($5.51 \pm 0.1 \text{ mmol g}^{-1}$) is higher than most of the polymer-based adsorbents and comparable to carbon-based adsorbents.²²

Clearly, etched PTHQ carbon aerogels are more efficient than carbonized aerogels and the uptake amount is significantly large.

The CO₂ uptake of PBO-EC-1000 was found to be $4.60 \pm 0.2 \text{ mmol g}^{-1}$ at 273 K and 1 bar, which is significantly less than the CO₂ uptake found in THQ derived aerogels. This is consistent with the idea that N heteroatoms improve CO₂ absorption capabilities. As they are comparatively poor CO₂ absorbers, PBO aerogels were not analyzed for CO₂ selectivity.

Selectivity towards CO₂ relative to other gases:

It is important for CO₂ adsorber to have a high uptake, but just high adsorption capacity is not enough. The material should adsorb CO₂ preferentially competing with other gases. Ideal adsorbent should have high adsorption capacity and good selectivity towards CO₂ as compared to any other gas for practical application. Thus, for pre-combustion capture, selectivity towards CH₄ and H₂ is important while for post-combustion capture, selectivity towards N₂ is desired. Adsorption isotherms of PTHQ-C-800 and PTHQ-EC-1000 for CH₄, N₂, and H₂ at 273 K are shown in Figure 3.

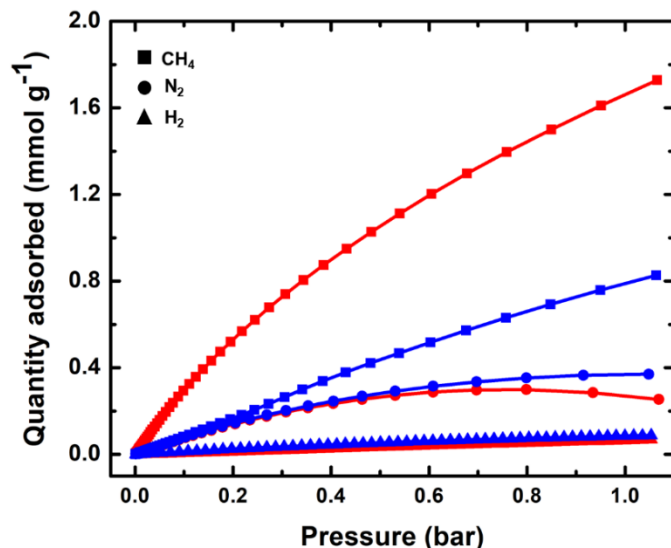


Figure 3. Adsorption isotherm at 273 K up to 1 bar of three gases. Red: PTHQ-EC-1000; Blue: PTHQ-C-800.

Selectivity of both the sample was evaluated by initial slopes of isotherms of two competing gases, assuming that at low coverage the uptake conforms Henry's law. Isotherms at 273 K were fitted with Virial type equation by which Henry's constant K_H was calculated for each gas as described in experimental section. Selectivities were calculated as a ratio of those values and are shown in bar-graph form in Figure 4. Low uptake of N₂ and H₂ at 273 K by both carbonized and etched aerogels resulted in low initial slope from the isotherms leading to high selectivities of CO₂ towards those gases.

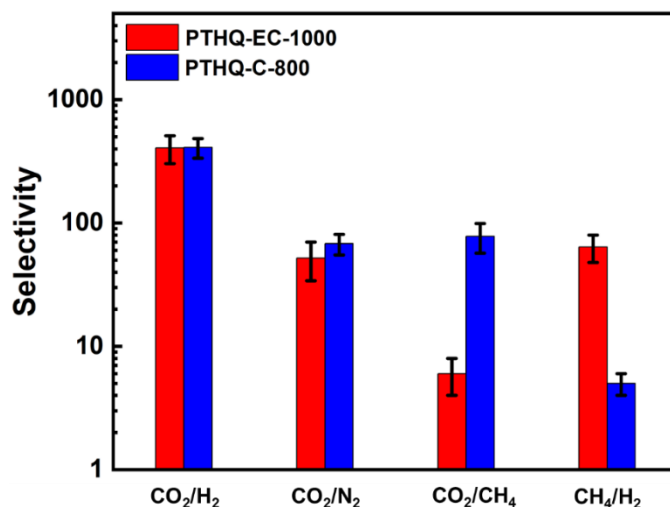


Figure 4. Relative selectivities at 273 K for gases calculated as a ratio of corresponding Henry's constant obtained by virial fitting of the isotherms.

High uptake of CH₄ was observed due to its high polarizability.²³ The adsorption capacity of PTHQ-EC-1000 is higher than PTHQ-C-800 and shows higher selectivity than that of PTHQ-C-800. The trend of high selectivities and low adsorption capacity as in case of CO₂/CH₄ for PTHQ-C-800 has been observed before with many sorbent systems like MOFs, HPCs, BILPs and carbon aerogels obtained from PA, and PBOs. The selectivities obtained from PTHQ carbon and etched carbon aerogels are far superior to their structurally analogues carbon aerogels derived from PBOs.²⁴ Thus, PTHQ carbon aerogels with high CO₂ adsorption capacity and no sign of saturation up to 1 bar and high selectivity towards CO₂ makes it a potential candidate for application in CCS. The high selectivity of PTHQ-EC-1000 for CO₂ versus H₂ (up to 407:1) is attractive for pre-combustion capture of CO₂, and the high selectivity of CO₂ versus N₂ (up to 52:1) is attractive for post-combustion CO₂ capture.

Results and Future Research:

PTHQ-EC-1000 carbon aerogels show the most promise for effective application in CCS due to their high CO₂ absorption (11.2 ± 0.9 mmol g⁻¹ at 273 K, 1 bar) and decent selectivity of CO₂ versus other common gases. Currently, we are working to gather more information on the effect of temperature on the properties of these carbon aerogels to better determine their potential effectiveness in *in-situ* methods of carbon capture.

Nomenclature:

THQ: Tetrahydroquinazoline monomer

PTHQ: Polymerized THQ monomer; preceding letters (if any) denote a carbonized polymer ('C') or a carbon-etched polymer ('EC'); subsequent number denotes temperature in Celsius at which the polymer was formed.

BO: Ishida's Benzoxazine monomer

PBO: Polybenzoxazine; preceding letters (if any) denote a carbonized polymer ('C') or a carbon-etched polymer ('EC'); subsequent number denotes temperature in Celsius at which the polymer was formed.

CCS: Carbon Capture and Storage/Sequestration

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Reflection on the Learning Experience

Reflection on the Learning Experience (On a separate piece of paper):

1. Describe your foundational understanding of how research is conducted in your discipline.
2. How have you expanded your understanding of the informational resources available and how to best use these resources?
3. Describe the knowledge you have gained regarding the fundamentals of experimental design.
4. Describe how you have learned to interpret the results of your research project.

1. [Chemical] Research is typically conducted (as far as I can tell) by first having a problem or question that you would like to answer. You then do research on the topic(s) relevant to the issue to get an idea of how to go about answering the problem/question. After acquiring adequate background knowledge, you can then form good hypotheses and methods to answer the problem/question. After several rounds of testing, data analysis, and possible/probable method revisions, a conclusion should be reached. This conclusion about the problem/question should then be shared and peer reviewed with the greater scientific community.

2. I have gained more experience in navigating the Internet to find relevant information for my project, particularly in the utilization of MST's library repository. For the topic of the OURE project itself, I was able to gain a great understanding of the topic by thoroughly examining the papers that were already published by the graduate students/research advisor that I worked with. In particular, close investigation of gathered data and the experimental sections of the papers I read allowed me to better understand the topics discussed in the papers.

The biggest source of knowledge came from the graduate student that helped me throughout the project. He answered any questions I had from reading the papers and examining the data. It really helps talking to a knowledgeable person on the subject you are studying! I will keep this in mind for the future.

3. Still not exactly sure on how, or if, the "fundamentals of experimental design" differ from the commonly known scientific method. Regardless, the knowledge I have on experimental design is roughly described in my answer to question #1.

I think the most impactful thing I learned over the past year in regards to experiential design is the evolution of goals in an overarching project, or rather the question that should be addressed after the first question in the project is answered. At first, we looked at select aerogels to see if they were good candidates for CO₂ absorption. After we did that, we had to then figure out how to take the best candidates and make them even better CO₂ absorbers,

if possible. After that, we decided to investigate how the aerogels we made would fare in realistic environmental conditions, so we are now looking at water absorption over time and temperature effect on our aerogels. I was a bit uncertain how new research questions were poised after milestones in projects were reached, and now I have more experience in this area.

4. I had fun deciphering the data that was collected on the aerogels from the data we gathered this year and from previous papers on the subject! Figuring out how all the data charts and numbers were gathered/calculated and what they meant felt satisfying. I enjoy having thorough knowledge on a topic that is interesting to me.

As previously stated, I mostly learned how to interpret the data in my project by looking at how it was calculated/gathered and what it ultimately means; this was often accomplished by looking at the experimental section of the papers or asking a knowledgeable graduate student. For specifics, the main objective of this project is to create an aerogel that can effectively absorb CO₂. To do this, the aerogel must have good absorption capacity and good selectivity for CO₂. Good candidates for future use in carbon capture must exhibit both; EC-PTHQ-1000 is a great possible candidate for these reasons.