

**Full GAED Characterization with Aqueous-phase comparisons
for
Sample EE-541
February 22, 2015**

Executive Summary

One sample of granular activated carbon (GAC) EE-541 was fully characterized for aqueous phase comparison using the Gravimetric Adsorption Energy Distribution method (GAED). The sample was compared to four commercially available carbons: Coal-based gas phase, Wood-based, Coal-based liquid phase and Coconut-based carbon of about 1200 iodine number. The Apparent Density (AD) determined by using the ASTM D-2854-96 was 0.478 g/cc and made volume-based comparisons possible. The sample lost about 2½ weight percent on conditioning (heating the sample to 240°C in argon and holding for 25 minutes) indicating it had picked up little from its surroundings like humidity or an oxidizing substance. The cumulative pore volume was about 82% of that of the Coal-base Liquid phase reference and the calculated BET surface area was about ¾ of the Coconut-base Reference material. The structure of EE-541 resembled a lower activity Coal-base Liquid phase reference. However, of the six Application Performance graphs, four of them showed that this material would be equal to or better than the coal-based reference. With Type III (Moderate Loading like Benzene Vapor), the EE-541 would work about as well as the Coal-base Liquid phase reference. With Type IV (Regenerable Trace Loading like Acetone), Type V (Trace Loading like Trichloroethane from Water) and Type VI (Ultra Trace Loading Applications like Vinyl Chloride from Water) this sample out-performed the Coal-based liquid phase material. Microscopic photographs of this sample are available upon request

GAED Results:

Sample EE-541 was fully characterized for aqueous-phase GAED by measuring the entire characteristic curves using the GAED. The Apparent Densities (AD) of 0.478 g/cc was used allowing volume-based results. PACS Laboratories routinely run ASTM D-2854-96 for Apparent Densities before GAED full characterizations. The carbons were then compared to four commercially activated reference samples made from a range of raw materials.

PACS Sample ID

EE-541

Coal-base gas phase

Wood base

Coal-base Liquid phase

Coconut-base

Client Sample Identification

None

The sample was run in as-received, granular form. A summary of the actual test data and conditions used is listed in the data summary table at the end of the report in Appendix A. The EE-541 sample lost 2.49 weight percent on conditioning (heating to 240°C in argon and holding for 25 minutes.) Losses of less than 8 percent indicate a well-stored sample that had been protected from the small amount of moisture pick-up from ambient air

during handling and storage. The EE-541 sample had low weight loss indicating it had been protected, was fresh and not oxidized. All activities and adsorption capacities were calculated on a clean carbon basis. To observe these capacities in the field may require additional processing of the carbon on site.

The GAED run was typical. The difference between the adsorption and desorption curves was minor throughout the experiment, therefore there was no hysteresis present, as was normal for commercially activated carbons. This report extends the comparison of this carbon beyond just the presentation of the characteristic curves. The plots of the differential and cumulative characteristic curve data are presented in Figures 1 and 1b in a volume-based comparison. Weight-based comparisons are also available. The specific run data and results are attached as Appendix A.

GAED Raw Data

The GAED (gravimetric adsorption energy distribution method) measured about 500 adsorption and desorption data points covering seven orders of magnitude in relative pressure (isothermal basis) and three orders of magnitude in carbon loading. The mass adsorbed was also divided by the carbon mass to generate a weight percent loading for easier comparison. The raw data was plotted in Figure 2. At 240°C, the adsorbent gas C134a or 1,1,1,2-tetrafluoroethane was introduced and the loading increased. Note in Figure 2, the mass loading was plotted against temperature but the relative pressure was also changing. There were three variables affecting performance that changed from point to point: vapor pressure, partial pressure, and temperature.

To make comparisons easier, the large data file of adsorption/desorption points at different temperatures and relative pressures was simplified. First the data was interpolated to get 30 evenly spaced points covering the entire data range. Next the adsorption and desorption results were averaged to get the equilibrium values (the difference between adsorption and desorption was minimal for this sample - no hysteresis). The y-axis was converted to pore volume measures, in cc liquid adsorbed or cc pores filled/100grams carbon, instead of weight percent. The average interpolated data for these characteristic curves is presented in Table 1, Figures 1 and 1b.

Performance Prediction Models

These curves are the only carbon related information required to predict physical adsorption performance using Polanyi Adsorption Potential theory. These single and multicomponent, gas and liquid phase, computer models are used to predict carbon performance and are available from PACS. To do performance predictions the following polynomial describes these carbon samples:

Carbon name	Characteristic curve polynomial - 3rd degree
EE-541	$y = 2.2742E-05x^3 - 1.8523E-03x^2 - 2.5493E-02x + 1.5589E+00$
Coal-base gas phase	$y = 5.8955E-05x^3 - 2.8880E-03x^2 - 2.6182E-02x + 1.7029E+00$
Wood base reference	$y = -6.3875E-05x^3 + 2.5948E-03x^2 - 1.1114E-01x + 2.0183E+00$
Coal-base Liquid phase	$y = 3.5299E-05x^3 - 1.8375E-03x^2 - 4.0325E-02x + 1.6682E+00$
Coconut-base	$y = 5.6334E-05x^3 - 3.0968E-03x^2 - 1.3312E-02x + 1.6731E+00$

In the equation, y was the common logarithm of pore volume in cc/100g carbon and x was the $e/4.6V$ adsorption potential in cal/cc. Characteristic curve polynomials are also listed in Appendix A.

Performance in the Six Types of Applications

The simplest comparison of carbon for a specific application is to run the performance prediction calculations for specific conditions, concentrations, and components present in the application. However, our experience with years of carbon optimization and performance comparisons has found that all physical adsorption applications can be placed into six application types. The proof is part of a 16-hour/800 slide-training course on carbon fundamentals given by PACS at least once a year.

The comparative results in Table 2a demonstrate the value of the different carbons for use in the different types of applications on a volume basis. For a given application type, the results are related to the amount of carbon required to get a certain level of performance. Therefore, a carbon with twice the cc/100g adsorption performance in an application type requires half the pounds of carbon to achieve a level of performance in that application type.

Table 2a compares performance on a weight basis and gives the values of the comparative results for the sample carbons versus the performance for the standard commercial carbons for the six application types. These results can also be provided on a weight basis if desired.

A series of two slides are attached as Appendix B, which describe the 6 application types and the classification process to determine what is the application type. Wastewater applications tend to be Type II or Type III. Municipal water purification varies from Type III, Type IV or Type V applications. Removal limits are not low enough and analytical testing is not sensitive enough at this date for Type VI. (Purifying hydrogen of CO and N₂ at room temperature is one of the few current Type VI applications). Municipal plants with surface water sources tend to be Type III or Type IV. Plants with ground water sources tend to be Type IV or V.

Trace Capacity Numbers

The characteristic curves were used to predict the values for the acetoxime trace capacity (TCN), gas-phase trace capacity number (TCNG) and mid capacity number (MCN). These results are presented at the bottom of the summary pages in Appendix A.

Adsorption Isotherms

The characteristic curves are also translated into adsorption isotherms using the programs mentioned above: Figure 3 for MTBE (weakly adsorbed material), Figure 4 for benzene (more strongly adsorbed species) and Figure 5 for phenol at pH=7 (quite strongly adsorbed material).

Pore Size Distributions

The Kelvin equation, modified by Halsey, can be used to convert the characteristic curve data to calculated BET surface areas or pore size distributions. This is not useful in terms of performance evaluations, but some audiences are more comfortable with the concepts of pore radius and a series of capillary sizes when thinking about activated carbon.

Figure 6 shows the cumulative pore size distributions, which we include but find of little use. The single and multi point BET surface area was calculated from these curves and is presented in the Summary Tables in Appendix A.

Interpretation of the GAED results:

1. One sample, EE-541 granular activated carbon (GAC) was fully characterized for aqueous phase comparison by the GAED (gravimetric adsorption energy distribution method.)
2. The sample was compared to four commercially available reference carbons on an aqueous phase basis: Coal-based gas phase, Wood-based, Coal-based liquid phase and Coconut-based carbon of about 1200 iodine.
3. The AD, determined by using the ASTM D-2854-96, was 0.478g/cc and made volume-based comparisons possible.
4. The EE-541 sample lost about 2.5% by weight on conditioning indicating that it was relatively clean and dry. (Data Summary Table Appendix A).
5. Conditioning entailed heating the sample to 240°C in argon and holding for 25 minutes so that all activities and adsorption capacities were calculated on a clean carbon basis.
6. The conditioned sample had about 18% less total adsorption pore volume than the Coal-based Liquid phase reference material (Table 1).
7. The calculated BET surface area indicated that this GAC had a surface area of 643 sq.meters/g, which is about $\frac{3}{4}$ of the coconut-base Reference material (Data Summary Table Appendix A).
8. The Differential Characteristic Curves in Figure 1b showed that the structure of this material resembled the Coal-based Liquid phase reference but with a lower activity.
9. The six Application Performance graphs showed how activated carbon would perform in specific applications. The performance prediction calculation for EE-541 shows that Type III (Moderate Loading Applications like Benzene Vapor from Air) would work about as well as the Coal-base Liquid phase reference. In addition, this sample would out-perform Type IV (Regenerable Trace Loading Applications like Acetone Solvent Recovery), Type V (Trace Loading Applications like Trichloroethane from Water) and Type VI (Ultra Trace Loading Applications like Vinyl Chloride from Water).
10. Microscopic photographs of this sample are available upon request.

Table 1. Carbon Characteristic Curves - Cumulative basis

ADSORPTION POTENTIAL DISTRIBUTIONS

Carbon Pore Volume Data

10/06 CDM

Contour Line Number or Adsorption Potential e/4.6V	EE-541 GAED Aq (EE-541) 42056.00 Auto GAED ver. 10/09 Capacity cc/100g.C	Coal-base gas phase Coal-base gas phase 38082.00 Auto GAED ver. 10/09 Capacity cc/100g.C	Wood base reference Wood-base 38258.00 Auto 4/10/2004 Prgm Capacity cc/100g.C	Coal-base Liquid phase Coal-base Liquid phase 38035.00 Auto 2004 Ramp Prgm Capacity cc/100g.C	Coconut-base Coconut-base 38101.00 Auto GAED ver. 10/06 Capacity cc/100g.C
0	35.93	47.35	108.17	43.87	47.25
1	33.90	45.74	81.89	41.37	45.20
2	31.66	43.32	63.58	38.18	42.96
3	29.31	40.26	50.40	34.61	40.44
4	26.89	36.78	40.62	30.89	37.67
5	24.48	33.06	33.14	27.23	34.68
6	22.12	29.30	27.29	23.76	31.55
7	19.85	25.65	22.60	20.56	28.37
8	17.70	22.22	18.78	17.68	25.24
9	15.69	19.08	15.63	15.14	22.24
10	13.83	16.28	13.02	12.93	19.43
11	12.13	13.82	10.83	11.02	16.86
12	10.59	11.69	8.99	9.38	14.54
13	9.20	9.87	7.46	7.99	12.49
14	7.96	8.33	6.19	6.81	10.69
15	6.86	7.04	5.13	5.80	9.13
16	5.90	5.95	4.25	4.95	7.80
17	5.04	5.05	3.53	4.23	6.65
18	4.30	4.29	2.93	3.61	5.68
19	3.65	3.66	2.44	3.08	4.86
20	3.09	3.14	2.03	2.62	4.16
21	2.61	2.70	1.70	2.22	3.56
22	2.20	2.32	1.41	1.88	3.06
23	1.84	2.01	1.17	1.58	2.63
24	1.54	1.74	0.96	1.32	2.27
25	1.28	1.51	0.78	1.09	1.95
26	1.06	1.31	0.62	0.89	1.69
27	0.87	1.13	0.47	0.71	1.46
28	0.72	0.98	0.34	0.56	1.27
29	0.58	0.85	0.23	0.44	1.11
Density g/cc	0.478	0.516	0.200	0.480	0.454

Table 2a. Performance in the Six Application Types on a Volume Basis

Carbon	EE-541	Coal-base gas phase	Wood base reference	Coal-base Liquid phase	Coconut-base
Application Performance - Volume Basis					
Type	cc/100cc	cc/100cc	cc/100cc	cc/100cc	cc/100cc
Type I	5.64	8.64	10.24	8.46	6.31
Type II	13.14	19.43	8.61	15.27	17.42
Type III	8.97	12.35	4.14	9.18	12.17
Type IV	2.34	2.88	0.91	2.10	3.09
Type V	2.06	2.22	0.59	1.73	2.58
Type VI	0.61	0.78	0.16	0.52	0.89

Type I Regenerable Heavy Loading Applications

Type II Heavy Loading Applications

Type III Moderate Loading Applications

Type IV Regenerable Trace Loading Applications

Type V Trace Loading Applications

Type VI Ultra Trace Loading Applications

Figure 2. Raw Characterization Data
1,1,1,2 tetrafluoroethane at 1 atm pressure
Average of adsorption and desorption data points

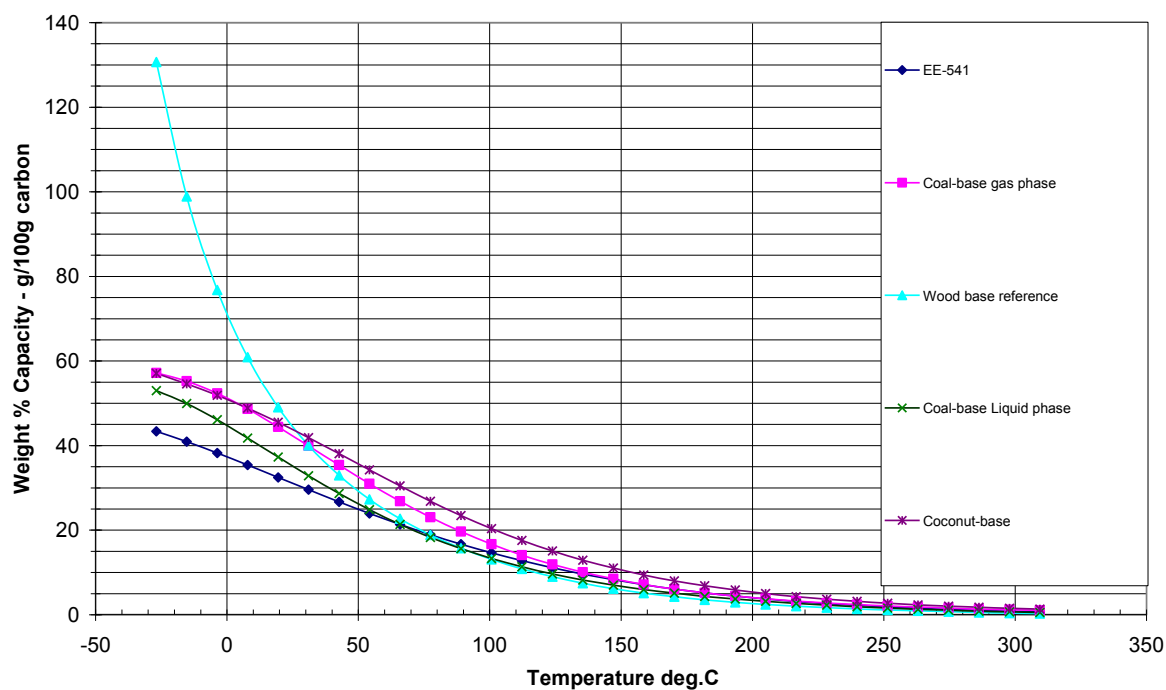
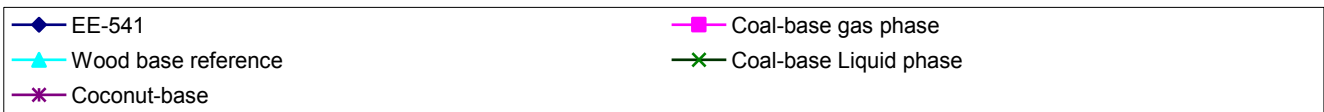
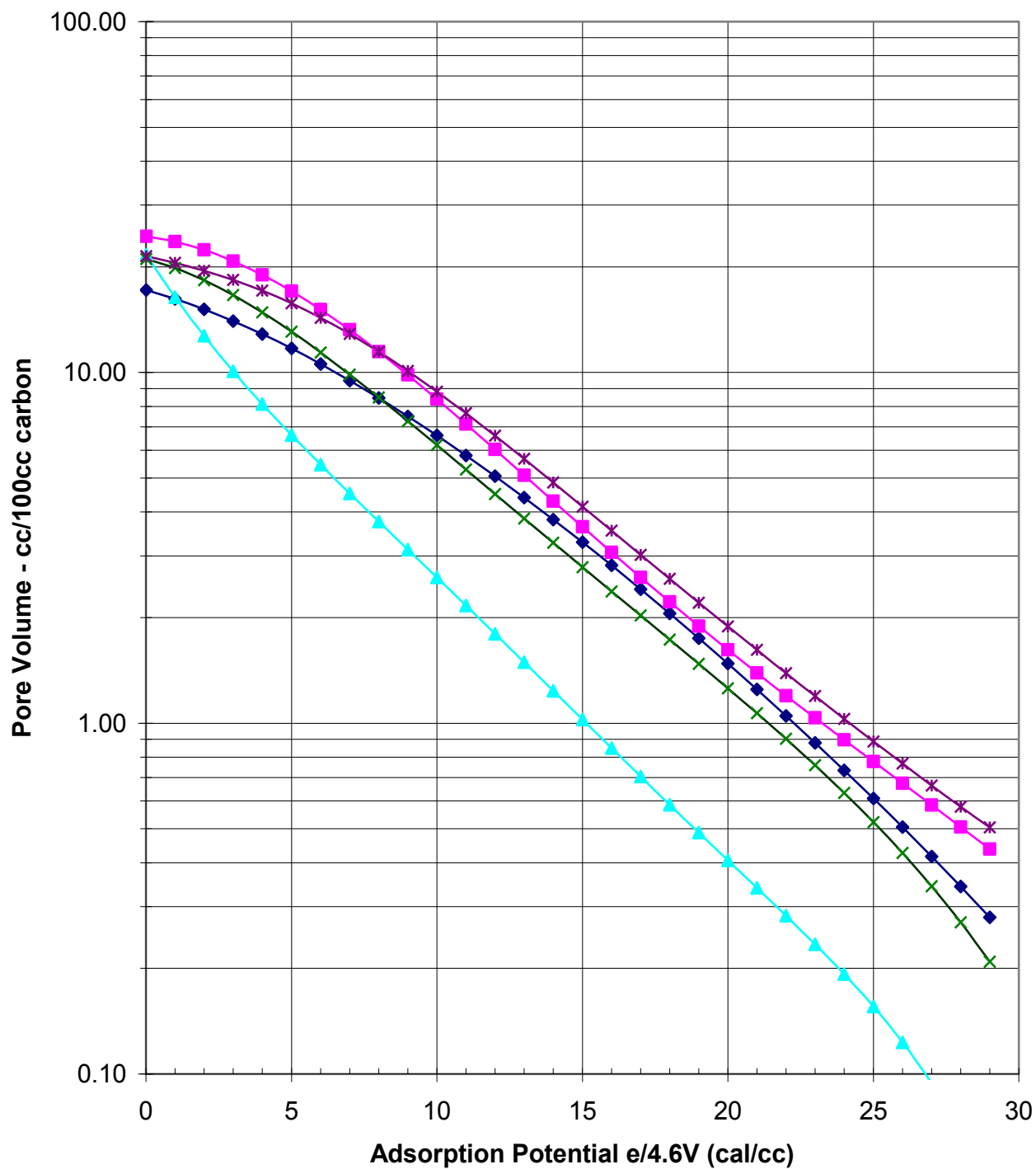


Figure 1. Volume based Carbon Characteristic Curves - Cumulative



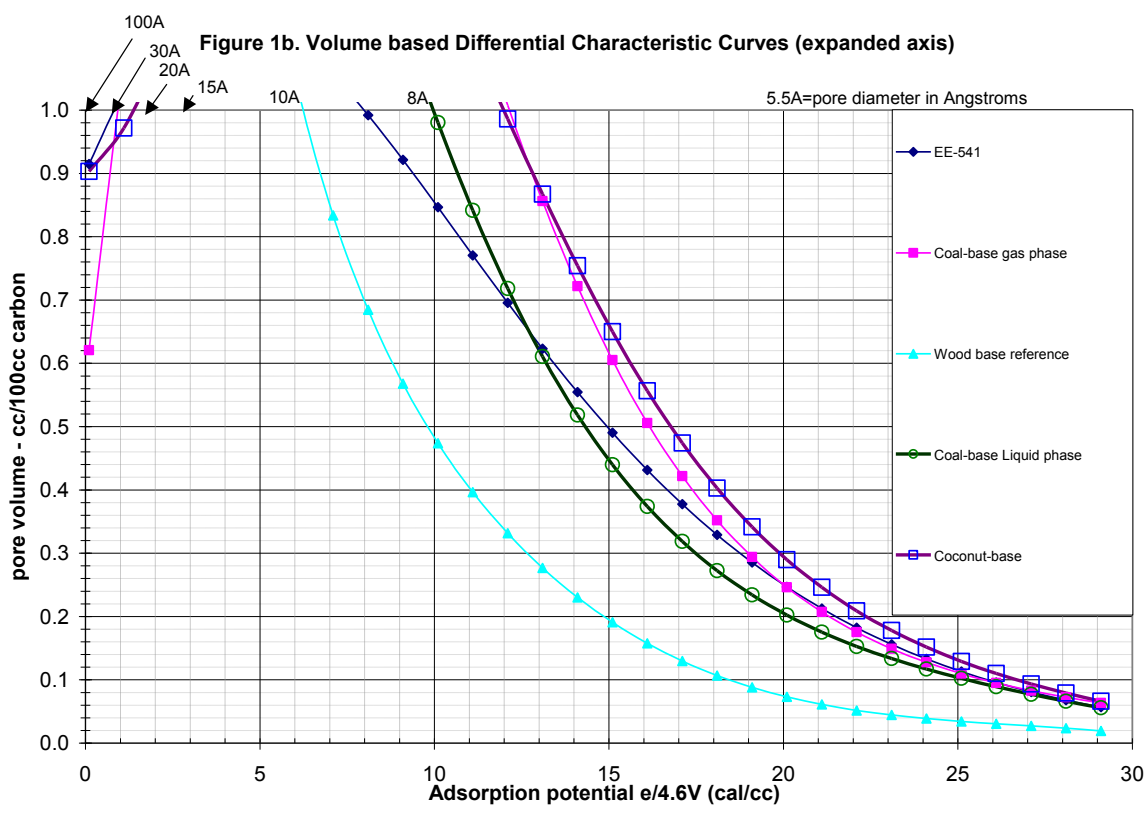
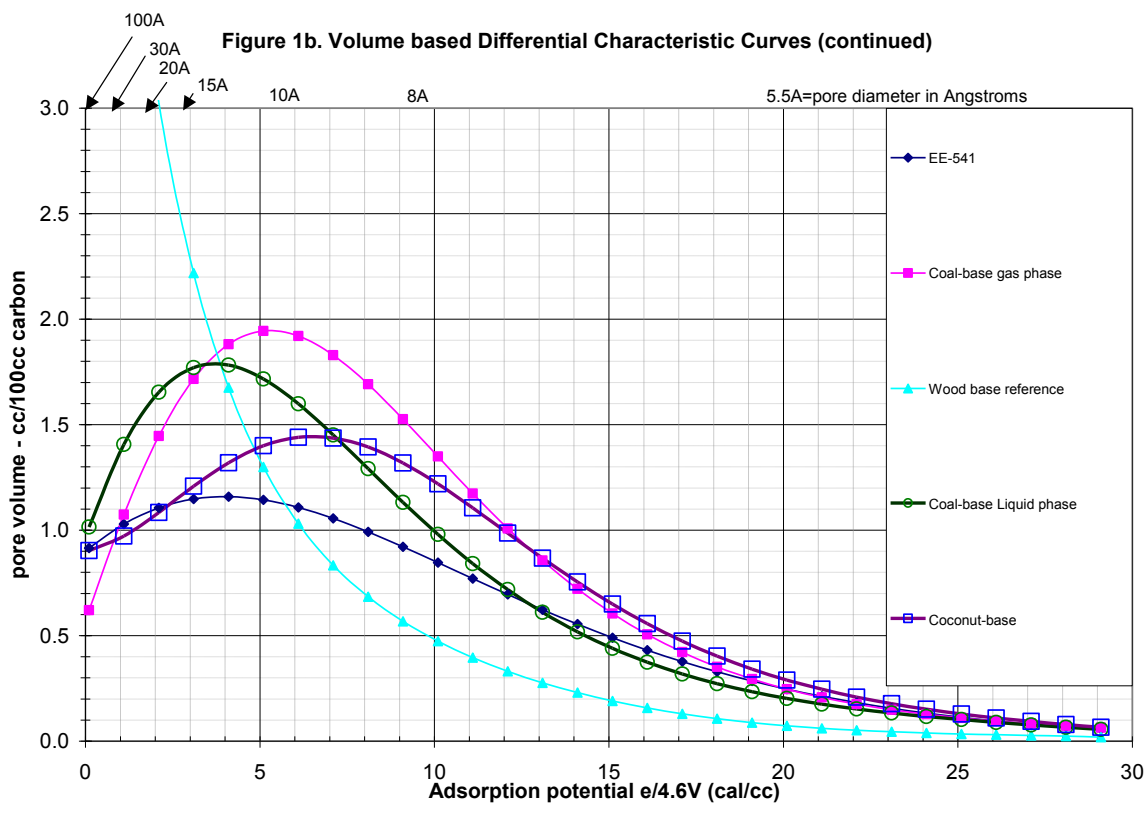


Figure 3. Adsorption Isotherm
MTBE in water

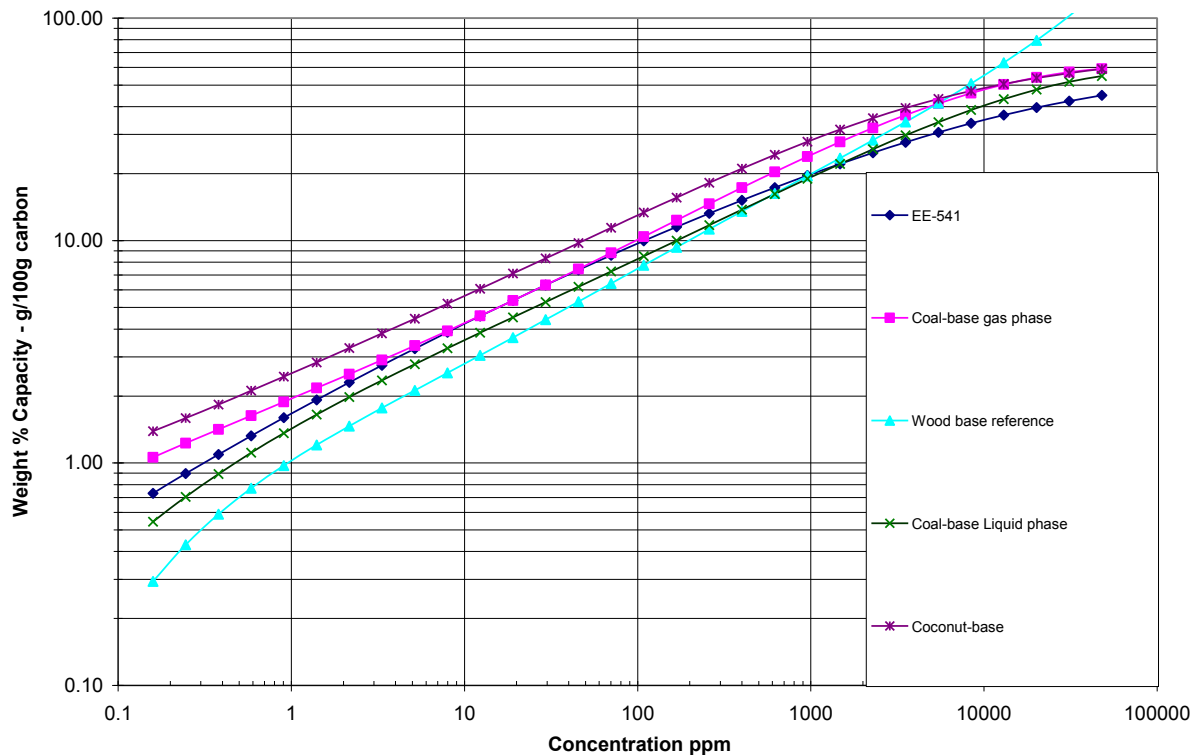


Figure 4. Adsorption Isotherm
Benzene in water

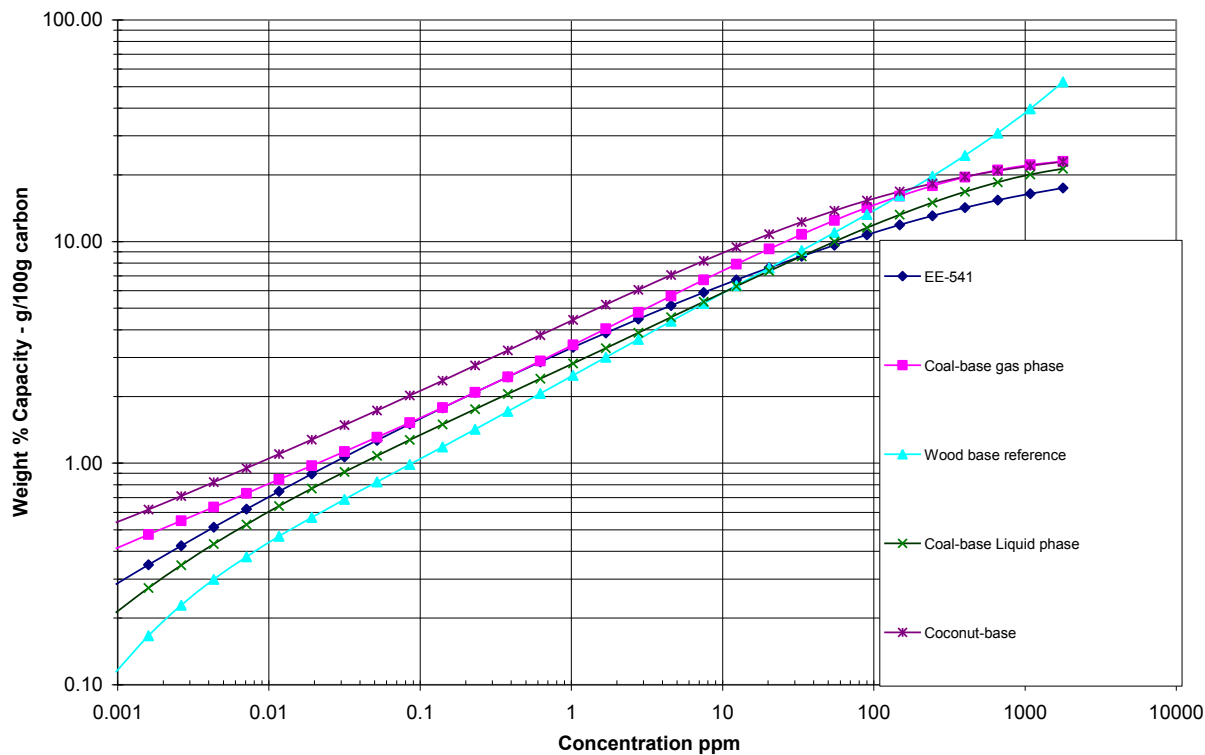


Figure 5. Adsorption Isotherm
Phenol in water

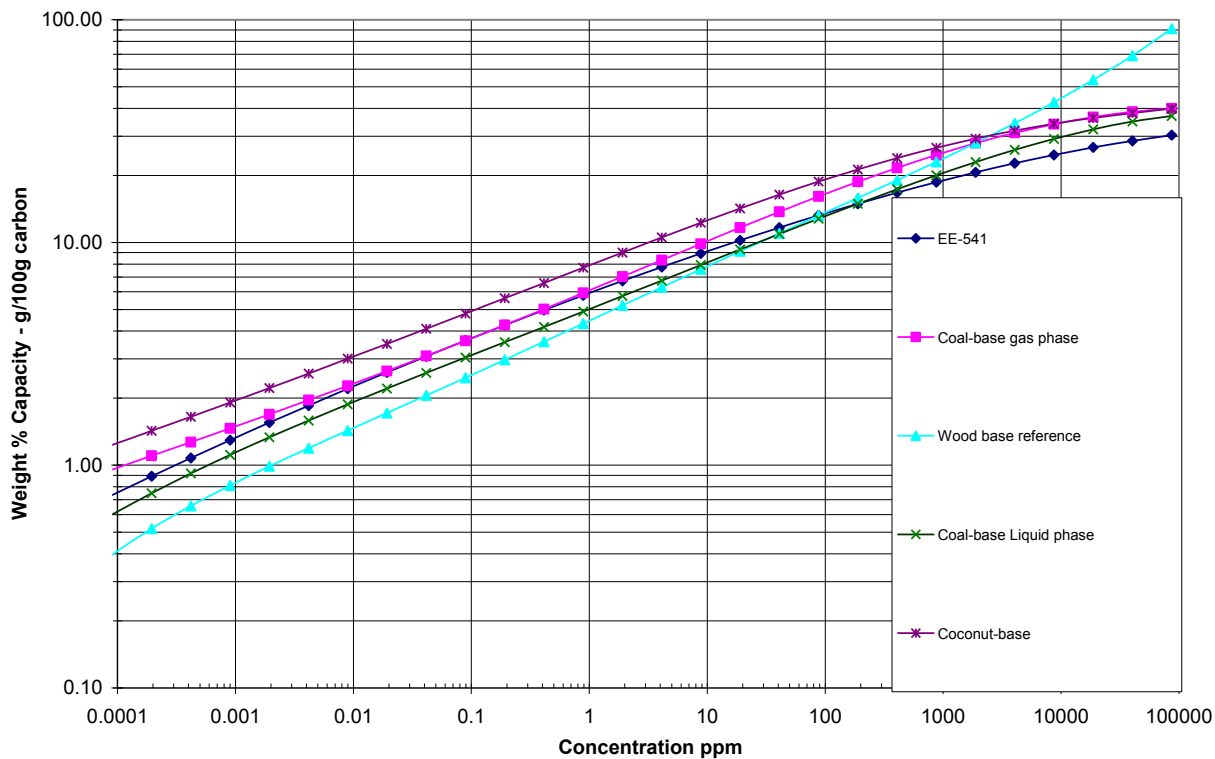
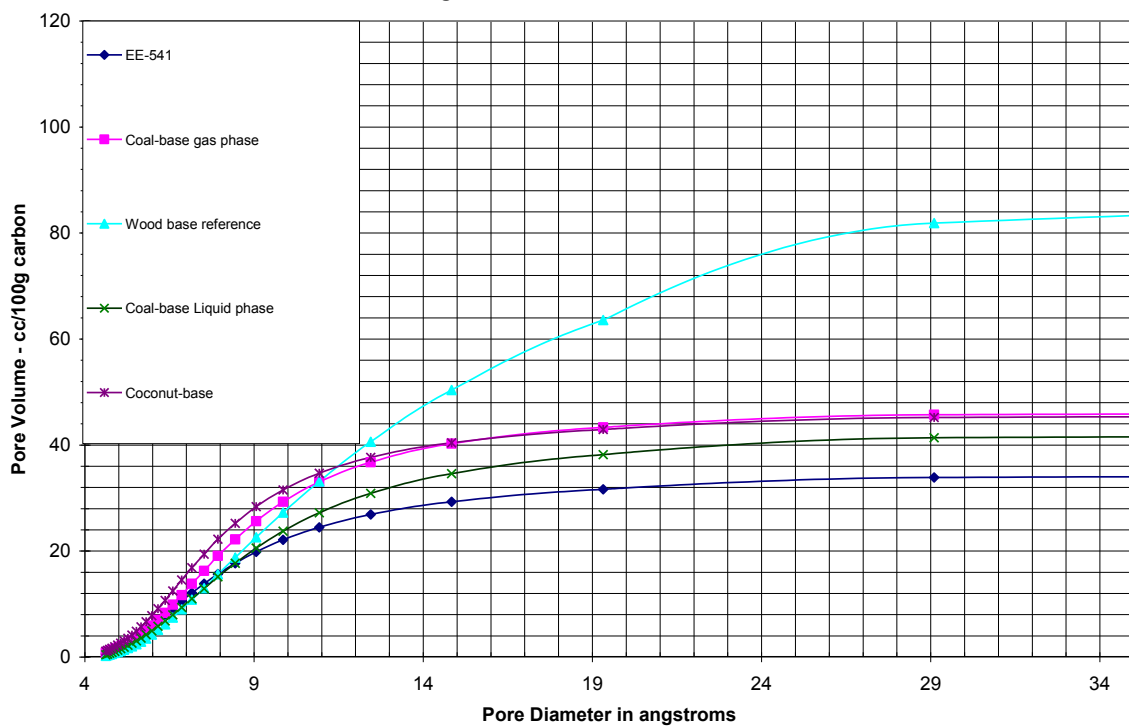
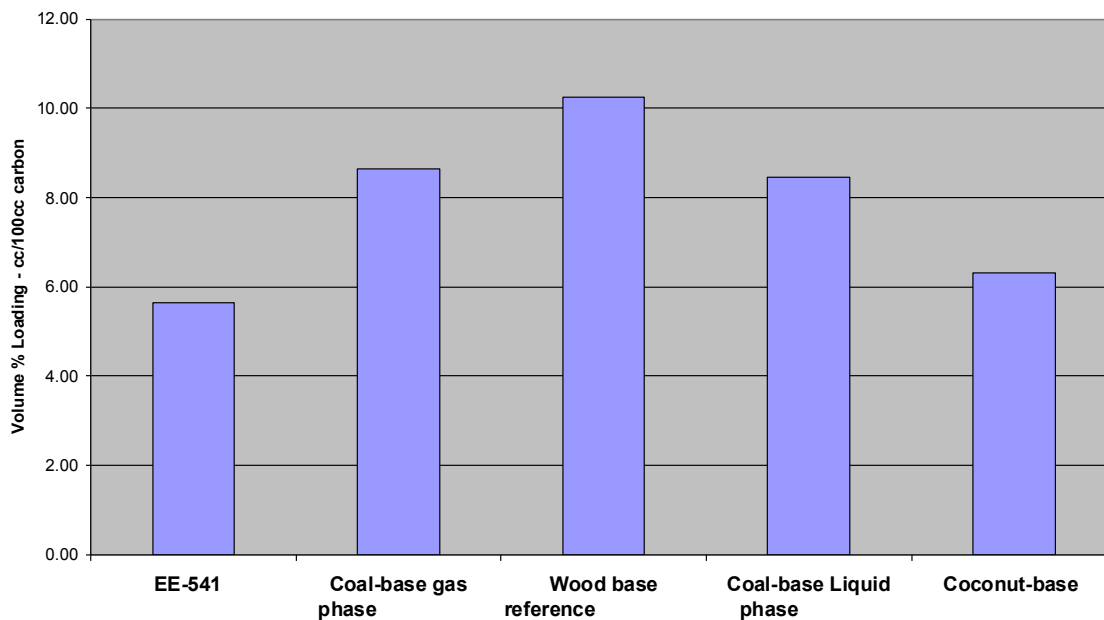


Figure 6. Pore Size Distributions



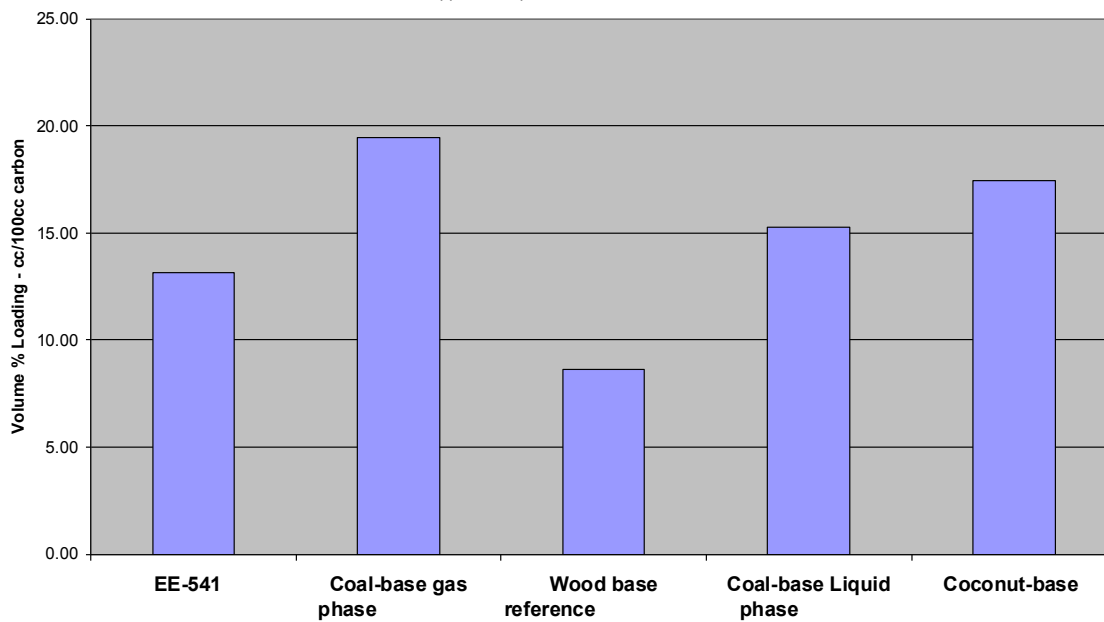
Type I Application Performance - Regenerable Heavy Loading Applications
Example: Butane Working Capacity

1atm Adsorption and 1000 bed volume air purge

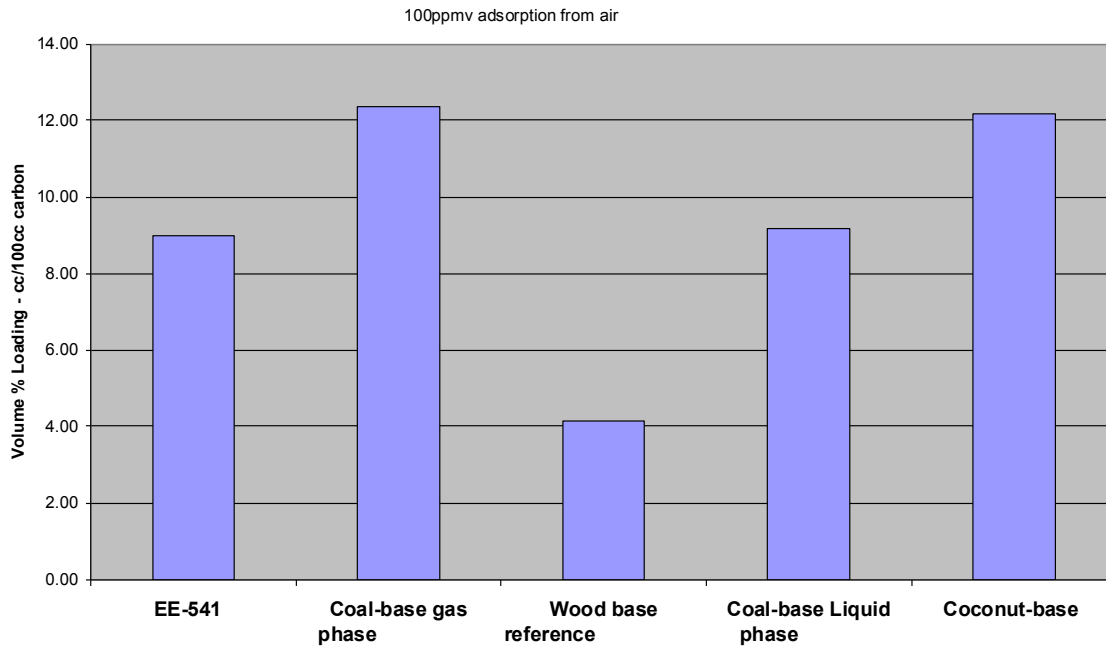


Type II Application Performance - Heavy Loading Applications
Example: p-Nitrophenol from Water

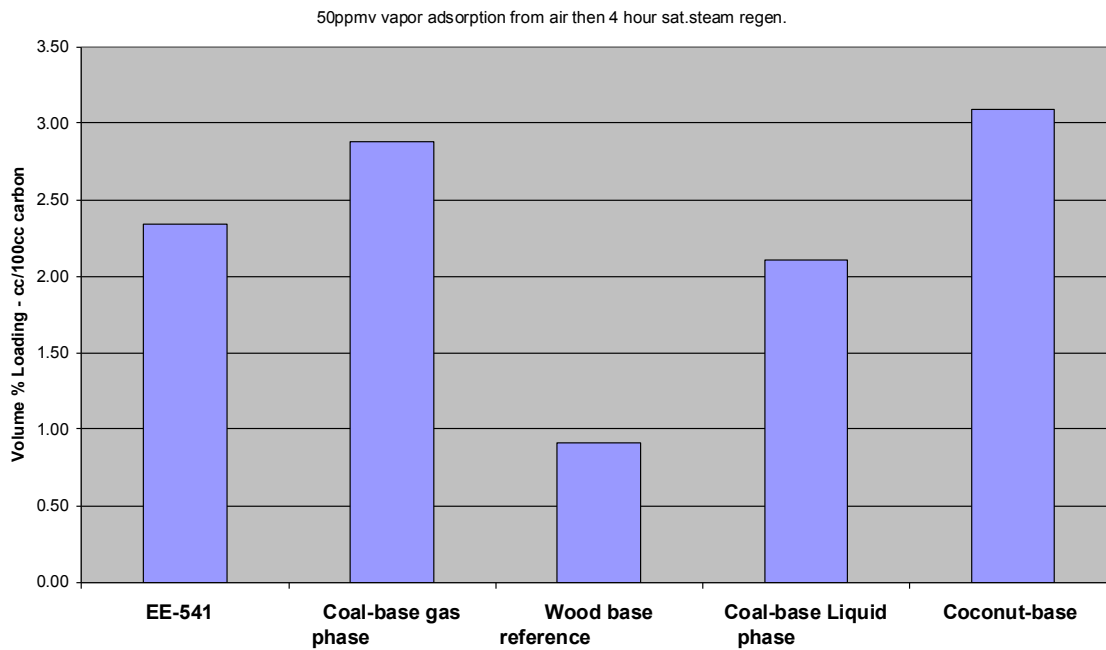
4000ppm adsorption from waste water



Type III Application Performance - Moderate Loading Applications
Example: Benzene Vapor from Air



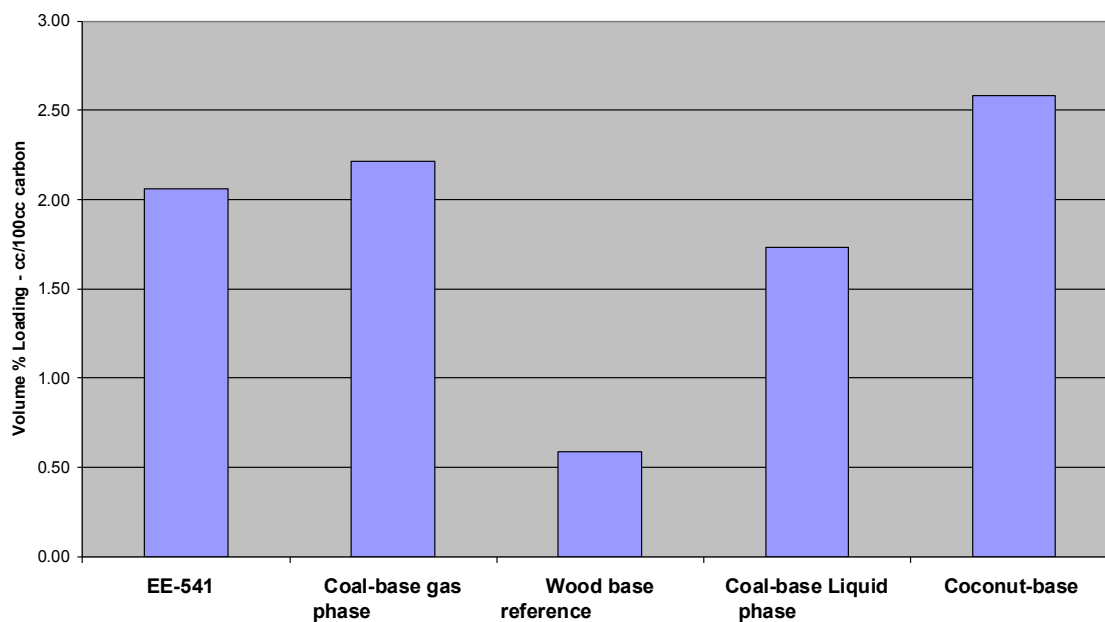
Type IV Application Performance - Regenerable Trace Loading Applications
Example: Acetone Solvent Recovery



Type V Application Performance - Trace Loading Applications

Example: Trichloroethane from Water

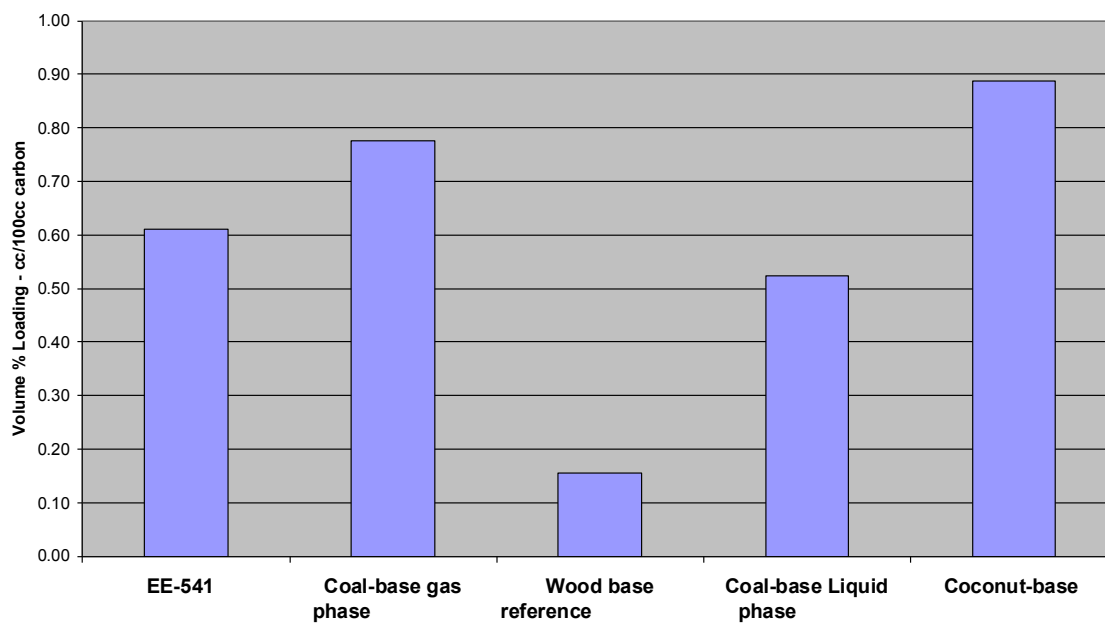
4ppm adsorption from groundwater



Type VI Application Performance - Ultra Trace Loading Applications

Example: Vinyl Chloride from Water

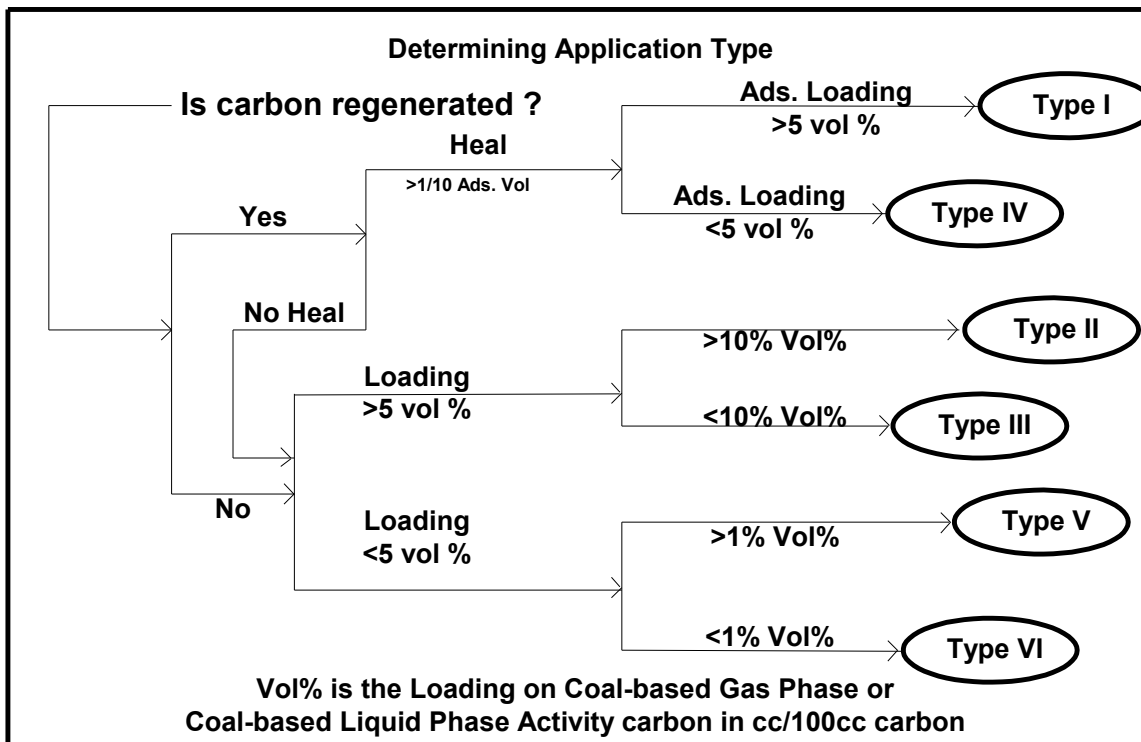
5ppb adsorption from groundwater



Appendix A.
GAED Summary Tables

<u>Sample Description</u>		EE-541		
EE-541		Carbon Characteristic Curve		
GAED Aq (EE-541)		EE-541		
0.478 g/cc AD		Adsorption Potential	Differential Pore Volume	Cumulative Pore Volume
Equipment Information		e/4.6V (cal/cc)	cc/100g	cc/100g
Calculated N2 BET Surface Area				
Operator CDM	BET sq.meters/g= 643	0	1.91	35.93
Analysis Date 2/11/2015	BET C Constant= -90.5323	0.4	2.02	35.14
Start time 6:00:00 AM	Max. P/Po= 0.298	1	2.15	33.90
Procedure Auto GAED ver. 10/09	Min. P/Po= 0.051	1.4	2.23	33.02
File C:\data\PACS	R square= 0.9955	2	2.31	31.66
OrgFile C:\data\PACS	Single point BET sq.meters/g= 639	3	2.40	29.31
Instrument GAED		4	2.42	26.89
Module Mettler		5	2.39	24.48
Xcomment Pan:Al - Gas1:Argon - Gas2:C134a 100cc/min		6	2.32	22.12
Text 500mg Al pan full level - Straight TC		7	2.21	19.85
		8	2.08	17.70
		9	1.93	15.69
		10	1.77	13.83
		11	1.61	12.13
		12	1.46	10.59
		13	1.30	9.20
		14	1.16	7.96
		15	1.03	6.86
		16	0.90	5.90
		17	0.79	5.04
		18	0.69	4.30
		19	0.60	3.65
		20	0.52	3.09
		21	0.44	2.61
		22	0.38	2.20
		23	0.33	1.84
		24	0.28	1.54
		25	0.24	1.28
		26	0.20	1.06
		27	0.17	0.87
		28	0.14	0.72
		29	0.12	0.58
		30	0.10	0.47
Conditioning the Sample				
Pan:Al - Gas1:Argon Conditioning gas				
240.8 C Conditioning temperature in Argon				
1.5729 g Original Carbon wt				
1.5376 g Clean carbon weight				
2.49% wt% loading unconditioned				
Adsorption/desorption experiments				
5 Deg/min adsorption/desorption				
Gas2:C134a 100cc/min Adsorbate gas				
-11.41 C Minimum adsorption temperature				
498 Number of data points				
3 pnts/min Data collection rate				
Polynomial Curve fit of Results				
Comparison	Calads			
<u>Polynomial Coefficients</u>	<u>Polynomial Coefficients</u>			
1.555E+00	1.559E+00			
-2.297E-02	-2.549E-02			
-2.330E-03	-1.852E-03			
5.531E-05	2.274E-05			
-7.236E-07				
R2 = 9.9556E-01	R2 = 9.9555E-01			
Compare Poly	$y = -7.2358E-07x^4 + 5.5311E-05x^3 - 2.3303E-03x^2 - 2.2967E-02x + 1.5554E+00$			
Calads Poly.	$y = 2.2742E-05x^3 - 1.8523E-03x^2 - 2.5493E-02x + 1.5589E+00$			
Calculated Trace Capacity Numbers				
Trace capacity no.Gas-phase TCN-G(g/100cc)= 5.76				
Acetoxime Trace capacity no.TCN(mg/cc)= 13.85				
Mid capacity no.MCN(g/100cc)= 8.25				

Appendix B The Six Application Types and Classifying an Application



Six Categories of Application Types

Based on Effect of Carbon Characteristics Performance and the Optimal Carbon

Application Types

	I	II	III	IV	V	VI
Full e/4.6	1.25	3.5	7.5	13	18	25
Empty e/4.6	6.25	-----	-----	18	-----	-----
Component	Butane	PNP	Benzene	Acetone	TCE	Vinyl Cl
Phase	Vapor	Waste Water	Vapor	Vapor	Ground water	Ground water
Concentration	<u>1atm</u> 1000 BV air purge	4000 ppm	100 ppmv	<u>50 ppm</u> 4hr.steam	4 ppm	5 ppm
Temperature	25/25C	25C	25C	25//100 c	25C	25C