

Adsorption of Nitrogen on Activated Carbon—Refit of Experimental Data and Derivation of Properties Required for Design of Equipment

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Volumetric method based adsorption measurements of nitrogen on two specimens of activated carbon (Fluka and Sarabhai) reported by us are refitted to two popular isotherms, namely, Dubinin–Astakhov (D–A) and Toth, in light of improved fitting methods derived recently. Those isotherms have been used to derive other data of relevance in design of engineering equipment such as the concentration dependence of heat of adsorption and Henry's law coefficients. The present fits provide a better representation of experimental measurements than before because the temperature dependence of adsorbed phase volume and structural heterogeneity of micropore distribution have been accounted for in the D–A equation. A new correlation to the Toth equation is a further contribution. The heat of adsorption in the limiting uptake condition is correlated with the Henry's law coefficients at the near zero uptake condition.

Introduction

Adsorption of gases on activated carbon is used in several engineering applications such as purification of gases, sorption-based cooling systems, and gas storage. In addition, nitrogen adsorption is also used to verify theoretical models of adsorption. In our case, adsorption measurements were motivated by the need to build a cryocooler that can use onboard spacecraft waste heat. Experimental data were measured using a volumetric method¹ for two specimens of activated carbon, namely, those manufactured by Fluka and Sarabhai Chemicals. A simple Dubinin equation was used therein which accounts neither for the changes in adsorbed phase volume nor for structural heterogeneity of the micropore size distribution. It is well-known that the Dubinin–Astakhov (D–A) equation gives a better representation of gas phase adsorption for IUPAC type I isotherm data than the simple Dubinin equation.^{2–4} Further, this equation enables evaluation of uptake dependence of isosteric heats of adsorption.^{5–7} However, this equation does not have a Henry's law regime,⁸ and hence it is not possible to derive the Henry's law coefficients. On the other hand, the Toth isotherm is better suited for this purpose. Yet, this isotherm presumes constancy of heats of adsorption over the entire uptake regime. Thus, both the isotherms mentioned above have relative merits, and it is common for engineers to use the appropriate data in the design of engineering systems. For example, the uptake dependence of heat of adsorption is required for determining the heat inventories during the desorption process and cooling requirements during the adsorption process in thermal compressors of sorption coolers. The heating process accounts for nearly a quarter of the total heat required for thermally driven refrigeration systems.⁹ Henry's law coefficients are useful in assessing the low uptake limit behavior of sorption gas storage

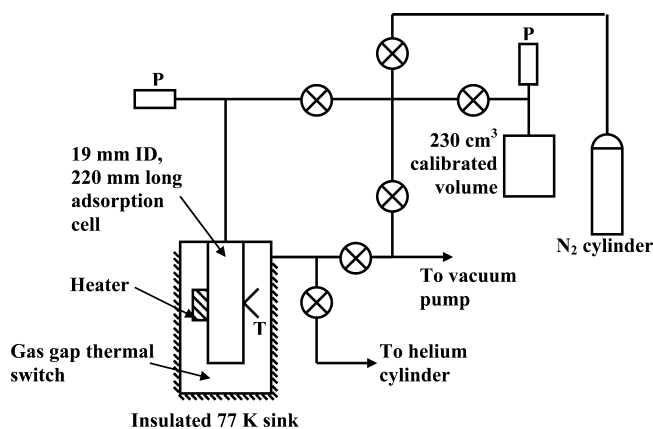


Figure 1. Schematic arrangement of the experimental setup. P, pressure transducer; T, thermocouple.

systems. Recently, Saha et al.^{2,3} have evolved a new method of fitting experimental concentration difference data obtained from desorption measurements to arrive at adsorption parameters. That procedure is now extended to data from an equilibrium volumetric method, and efforts are made to derive the adsorption characteristics for the activated carbon + nitrogen system. In addition to obtaining adsorption parameters, some new derived data are presented for this system.

Experimental Measurements

The raw data for adsorption were obtained through an equilibrium volumetric method which was described in detail by Prasad et al.¹ and Akkimaradi et al.,⁴ and hence only a brief description will be given here. A schematic arrangement is shown in Figure 1. The carbon content of the Fluka and Sarabhai specimens was (98.77 and 97.81) %, respectively. A 99.995 % pure nitrogen sample was used. The calibrated volume is pressurized with nitrogen first. Then the adsorption cell at a temperature of the isotherm is connected to it. After the cell

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Table 1. Experimental Raw Data for Adsorption of Nitrogen on the Fluka Specimen of Activated Carbon

p/MPa	$C/\text{g}\cdot\text{g}^{-1}$	p/MPa	$C/\text{g}\cdot\text{g}^{-1}$	p/MPa	$C/\text{g}\cdot\text{g}^{-1}$
$T = 150 \text{ K}$		$T = 240 \text{ K}$		$T = 300 \text{ K}$	
0.003	0.144	0.015	0.031	0.111	0.028
0.031	0.173	0.111	0.058	0.503	0.047
0.146	0.197	0.289	0.081	0.861	0.066
$T = 180 \text{ K}$		$T = 270 \text{ K}$		$T = 320 \text{ K}$	
0.001	0.032	0.635	0.099	1.451	0.079
0.003	0.062	0.898	0.111	1.864	0.089
0.021	0.091	1.371	0.125	2.391	0.105
0.071	0.119	1.666	0.144	2.922	0.119
0.134	0.138	2.219	0.154	3.533	0.131
0.269	0.163	0.055	0.030	0.159	0.027
0.510	0.181	0.289	0.052	0.610	0.045
$T = 210 \text{ K}$		$T = 270 \text{ K}$		$T = 320 \text{ K}$	
0.005	0.031	0.555	0.074	1.085	0.062
0.033	0.060	1.023	0.089	1.733	0.074
0.114	0.087	1.364	0.100	2.218	0.083
0.289	0.110	1.867	0.114	2.750	0.099
0.458	0.125	2.279	0.131	3.335	0.113
0.710	0.146	2.915	0.140	3.814	0.130
1.044	0.162				
1.458	0.174				

Table 2. Experimental Raw Data for Adsorption of Nitrogen on the Sarabhai Specimen of Activated Carbon

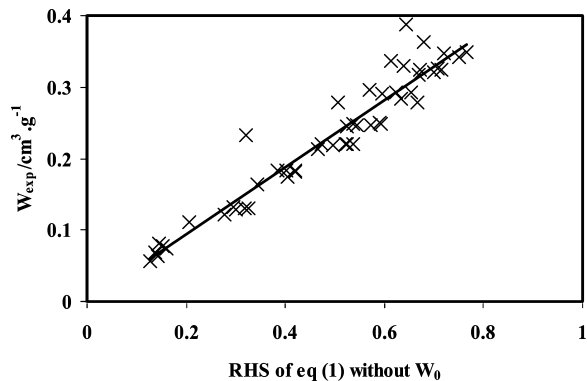
p/MPa	$C/\text{g}\cdot\text{g}^{-1}$	p/MPa	$C/\text{g}\cdot\text{g}^{-1}$	p/MPa	$C/\text{g}\cdot\text{g}^{-1}$
$T = 150 \text{ K}$		$T = 210 \text{ K (contd)}$		$T = 300 \text{ K}$	
0.007	0.029	1.906	0.095	0.193	0.011
0.022	0.055	2.245	0.106	0.452	0.018
0.056	0.080	$T = 240 \text{ K}$		1.064	0.029
0.145	0.102	0.066	0.014	1.759	0.038
0.210	0.125	0.159	0.024	2.375	0.049
0.612	0.134	0.480	0.041	3.102	0.057
0.760	0.150	0.989	0.051	4.143	0.060
$T = 180 \text{ K}$		1.343	0.066	4.708	0.069
0.016	0.029	1.831	0.077	$T = 320 \text{ K}$	
0.087	0.052	2.612	0.081	0.248	0.010
0.210	0.074	3.060	0.091	0.565	0.016
0.428	0.091	$T = 270 \text{ K}$		1.330	0.024
0.626	0.109	0.123	0.013	2.027	0.034
1.232	0.112	0.296	0.021	2.762	0.043
1.450	0.126	0.756	0.035	3.599	0.050
$T = 210 \text{ K}$		1.368	0.044	4.750	0.052
0.067	0.027	1.853	0.057		
0.250	0.047	2.453	0.066		
0.608	0.061	3.390	0.069		
0.860	0.078	3.879	0.079		
1.194	0.092				

pressure and temperature have stabilized, it is isolated, and the final pressure of the calibrated volume is measured. The details of data reduction are given in ref 1. The uncertainties in measurements are 0.5 K for temperature and 0.25 % of full scale (7 MPa) for pressure. Together with uncertainties in the calibrated cell volume and the dead volume in the capillaries, the concentration measurements are expected to have an overall uncertainty of 1 %. The raw data, not presented in ref 1, are given in Tables 1 and 2, which form the bases for the refits derived herein.

The following two isotherm equations are used. The first is the following form of the D–A equation

$$W = W_0 e^{-\left[\frac{RT}{E} \ln\left(\frac{p_s}{p}\right)\right]^n} \quad (1)$$

where $W = Cv_a$ and W_0 , E and n are the adsorption parameters that need to be determined; p , C , and T refer to pressure, concentration and temperature, respectively; and R is the

**Figure 2.** Dubinin–Astakhov equation correlation for the Fluka specimen: ×, experiment; line, best fit.

universal gas constant. The adsorbed phase volume (v_a) is calculated as follows

$$v_a = v_b \exp[\alpha(T - T_b)] \quad (2)$$

α is the thermal expansion coefficient of the adsorbed phase, and v_b is the saturated liquid volume at the normal boiling point (T_b). p_s is the saturation vapor pressure of the adsorbed phase. If the adsorption occurs above the critical point, the pseudo-saturation pressure is determined using the following equation¹⁰ in line with that used for methane²

$$p_s = (T/T_c)^2 p_c \quad (3)$$

where the subscript c refers to the critical point. This is different from the pseudovapor pressure equation used by Prasad et al.¹ which was the extrapolated Antoine equation.

The Toth equation is given below

$$C = \frac{C_0 k_0 \exp\{\Delta H_{\text{ads}}/(RT)\} p}{[1 + \{k_0 \exp(\Delta H_{\text{ads}}/(RT)) \cdot p\}^{1/t}]} \quad (4)$$

In the above equation, ΔH_{ads} is the heat of adsorption. C_0 , k_0 , and t are the adsorption parameters that are analogous to those in eq 1 and need to be evaluated from the experimental measurements. The deviation between experimental and calculated concentration at a given temperature and pressure is defined as the error function as follows

$$\Delta C_i = C_{\text{expi}} - C_{\text{cali}} \quad (5)$$

The least-squares criterion requires that $\sum_{i=1}^n \Delta C_i^2$ be a minimum requiring that

$$\frac{\partial \sum_{i=1}^n \Delta C_i^2}{\partial W_0} = 0; \quad \frac{\partial \sum_{i=1}^n \Delta C_i^2}{\partial E} = 0; \quad \frac{\partial \sum_{i=1}^n \Delta C_i^2}{\partial n} = 0 \quad (6)$$

for the D–A correlation and

$$\frac{\partial \sum_{i=1}^n \Delta C_i^2}{\partial C_0} = 0; \quad \frac{\partial \sum_{i=1}^n \Delta C_i^2}{\partial \Delta h_{st}} = 0; \quad \frac{\partial \sum_{i=1}^n \Delta C_i^2}{\partial t} = 0; \\ \frac{\partial \sum_{i=1}^n \Delta C_i^2}{\partial k_0} = 0 \quad (7)$$

for the Toth equation. The normal equations can then be solved, in principle, to optimize the adsorption parameters in the respective equations. The normal equations are given below for the D–A equation

$$\sum_{i=1}^n \Delta C_i C_{cali} = 0 \quad (8)$$

$$\sum_{i=1}^n \Delta C_i C_{cali} \left[\frac{RT_i}{E} \ln \left(\frac{p_s}{p_i} \right) \right]^n = 0 \quad (9)$$

$$\sum_{i=1}^n \Delta C_i C_{cali} \left[\frac{RT_i}{E} \ln \left(\frac{p_s}{p_i} \right) \right]^n \ln \left[\frac{RT_i}{E} \ln \left(\frac{p_s}{p_i} \right) \right] = 0 \quad (10)$$

For the Toth equation, the normal equations are as follows

$$\sum_{i=1}^n \Delta C_i C_{cali} = 0 \quad (11)$$

$$\sum_{i=1}^n \Delta C_i C_{cali} \left[1 - \left(\frac{C_{cali}}{C_0} \right)^t \right] = 0 \quad (12)$$

$$\sum_{i=1}^n \Delta C_i C_{cali} \left[1 - \left(\frac{C_{cali}}{C_0} \right)^t \frac{1}{k_0} \right] = 0 \quad (13)$$

$$\sum_{i=1}^n \Delta C_i C_{cali} \left[\ln \left(\frac{C_0}{C_{cali}} \right) + \ln \left(k_0 p_i \exp(\Delta h_{st} R/T_i) \left(1 - \left(\frac{C_{cali}}{C_0} \right)^t \right) \right) \right] = 0 \quad (14)$$

However, the normal equations are highly nonlinear which defy a closed form solution. An iterative method was programmed for spreadsheet-based calculations to optimize the adsorption parameters.

Prasad et al.¹ used the following form of equation

$$C = C_0 e^{-\left[\frac{RT}{E} \ln \left(\frac{p_s}{p} \right) \right]} \quad (15)$$

which assumes that the corrections to the adsorbed phase volume and the effect of structural heterogeneity in micropores are negligible.

Results and Discussion

Figures 2 and 3 show plots of the RHS's of eq 1 without W_0 and eq 4 without C_0 for the Fluka specimen. Similar plots were

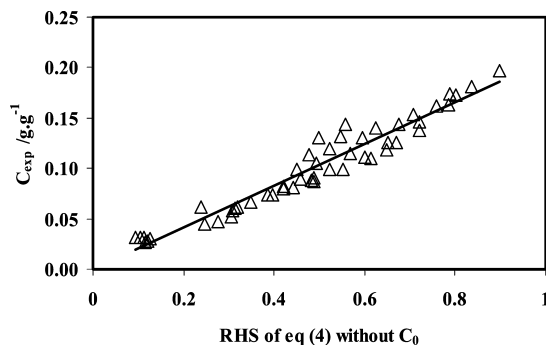


Figure 3. Toth equation correlation for the Fluka specimen. Δ , experiment; line, best fit.

Table 3. Dubinin–Astakhov Adsorption Equation Parameters and Residuals for Adsorption of Nitrogen on the Fluka Specimen of Activated Carbon

parameter	Fluka specimen	Sarabhai specimen
n	1.42	1.42
$E/J \cdot \text{mol}^{-1}$	8260	6120
$W_0/\text{cm}^3 \cdot \text{g}^{-1}$	0.470	0.287
α/K^{-1}	$3.6 \cdot 10^{-3}$	$3.6 \cdot 10^{-3}$
intercept	$-2.19 \cdot 10^{-4}$	$-7.70 \cdot 10^{-4}$
eq 8	$-2.93 \cdot 10^{-3}$	$9.03 \cdot 10^{-4}$
eq 9	$4.03 \cdot 10^{-4}$	$-2.40 \cdot 10^{-4}$
eq 10	$1.70 \cdot 10^{-3}$	$-5.88 \cdot 10^{-4}$
$\sum_{i=1}^n \Delta C_i^2$	$6.62 \cdot 10^{-3}$	$1.34 \cdot 10^{-3}$

Table 4. Toth Adsorption Equation Parameters and Residuals for Adsorption of Nitrogen on the Fluka Specimen of Activated Carbon

parameter	Fluka specimen	Sarabhai specimen
t	0.48	0.52
$\Delta h_{st}/J \cdot \text{mol}^{-1}$	17493	12770
$C_0/\text{g} \cdot \text{g}^{-1}$	0.207	0.176
k_0/MPa^{-1}	$2.49 \cdot 10^{-3}$	$2.73 \cdot 10^{-3}$
Intercept	$-2.425 \cdot 10^{-4}$	$-2.363 \cdot 10^{-4}$
eq 11	$-1.22 \cdot 10^{-3}$	$-4.17 \cdot 10^{-4}$
eq 12	$-1.27 \cdot 10^{-3}$	$-4.31 \cdot 10^{-4}$
eq 13	$-2.09 \cdot 10^{-2}$	$-5.74 \cdot 10^{-3}$
eq 14	$-4.9 \cdot 10^{-3}$	$-1.14 \cdot 10^{-3}$
$\sum_{i=1}^n \Delta C_i^2$	$5.95 \cdot 10^{-3}$	$8.68 \cdot 10^{-4}$

obtained for the other specimen as well. The slope of a linear fit to data in those figures should result in W_0 and C_0 , respectively. In an ideal case, the intercept should be zero. The parameters iterated are n , E to get eqs 8 to 10 satisfied in the case of the D–A equation, and Δh_{st} , k_0 , and t to get eqs 11 to 14 satisfied in the case of the Toth equation. The value of α (in eq 2) was also iterated, although it does not form a part of the normal equations. It is assumed that the adsorbed phase of nitrogen in both specimens behaves identically. The respective parameters and the residuals for each of the normal equations are given in Tables 3 and 4 for the D–A and Toth equations, respectively, for both the specimens. Figures 4 and 5 show the deviation plots for both specimens. In these figures, the correlations proposed in ref 1 are also compared. By and large, the present correlations, in particular, the Toth equation, show an improvement in the interpretation of the experimental data.

The isosteric heat of adsorption is defined as

$$\Delta h_{st} = -R \left. \frac{\partial \ln p}{\partial (1/T)} \right|_C \quad (16)$$

which can be derived from eq 1 as follows

$$\Delta h_{st} = 2RT + E \left[\left(\ln \frac{W_0}{Cv_a} \right)^{1/n} \left\{ 1 + \frac{\alpha T/n}{\ln \frac{W_0}{Cv_a}} \right\} \right] \quad (17)$$

At each measured data point shown in Tables 1 and 2, the isosteric heat of adsorption can be calculated. They are represented in Figure 6 for the Sarabhai specimen, while similar plots for the Fluka specimen were also obtained. Along each isotherm, the isosteric heat of adsorption was fitted to a power law of the following type

$$\Delta h_{st}|_T = \Delta h_{st0} \left(\frac{C}{C_0} \right)^{-x} \quad (18)$$

The temperature dependence of Δh_{st0} and x is shown in Figure 7. The Toth equation values of Δh_{ads} are also drawn in this figure to show that they correspond to the D-A equation based values at (321.3 and 286.5) K for the Fluka and Sarabhai specimens, respectively. Δh_{st0} can be construed to be the isosteric heat of adsorption at saturated loading, that is $C = C_0$. This cannot be derived from eq 17 because the second term on its RHS will be indeterminate. However

$$\text{Lt}_{C \rightarrow C_0} \left[\left(\ln \frac{W_0}{Cv_a} \right)^{1/n} \left\{ 1 + \frac{\alpha T/n}{\ln \frac{W_0}{Cv_a}} \right\} \right] = \frac{\Delta h_{st0} - 2RT}{E} \quad (19)$$

In view of the linear temperature dependence of Δh_{st0} and x , the isosteric heat of adsorption is correlated as follows

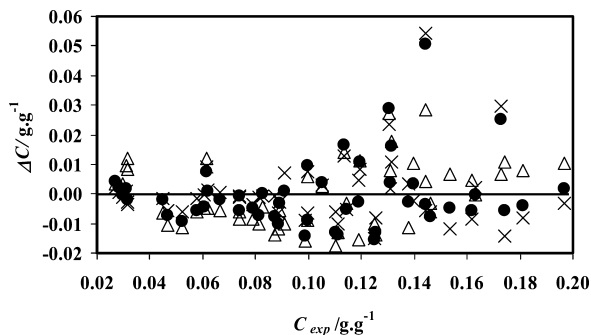


Figure 4. Deviation plots for the Fluka specimen. ×, eq 1; △, eq 4; ●, eq 15.

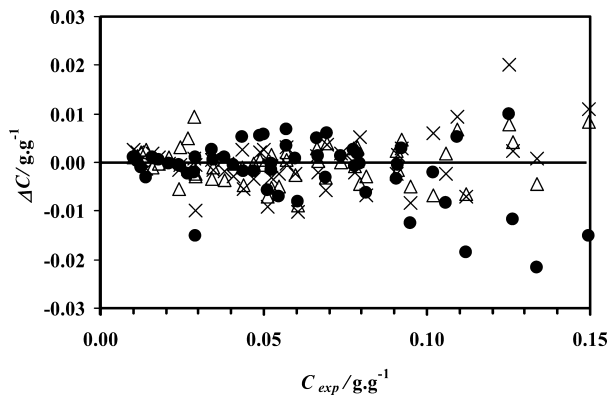


Figure 5. Deviation plots for the Sarabhai specimen. ×, eq 1; △, eq 4; ●, eq 15.

$$\Delta h_{st} = (AT + B) \left(\frac{C}{C_0} \right)^{-(aT-b)} \quad (20)$$

The values of coefficients A , B , a , and b in the above equation are listed in Table 5 for both specimens. The RHS of eq 19 can be calculated from the data so derived. Its temperature dependence is shown in Figure 8. It is seen that this limit is almost the same for both the specimens, and the limiting value is $(3.5 \cdot 10^{-3} T/K + 0.329)$. The treatment accorded to the heat of adsorption here is much more rigorous than that in ref 11, where the adsorbed phase volume effect was not taken into account. From eq 2, it is seen that the adsorbed phase volume varies from about $(1.6 \text{ to } 3) \text{ cm}^3 \cdot \text{g}^{-1}$ in the temperature range of measurements, and hence it cannot be ignored.

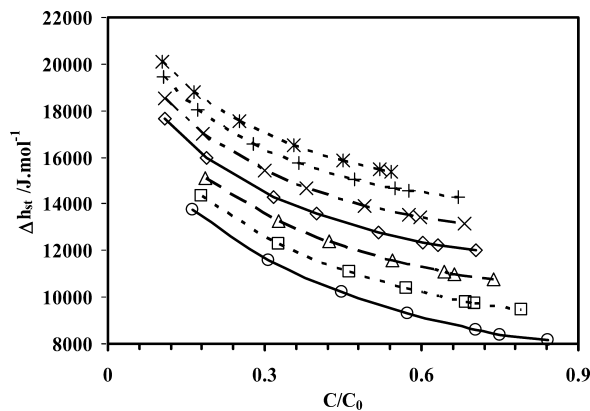


Figure 6. Concentration dependence of isosteric heat of adsorption for the Sarabhai specimen. ○, 150 K; □, 180 K; △, 210 K; ◇, 240 K; ×, 270 K; +, 300 K; *, 320 K.

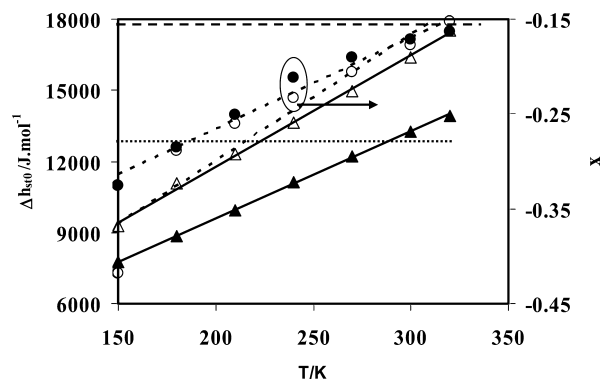


Figure 7. Temperature dependence of Δh_{st0} and index X . △, Δh_{st0} (Fluka) left ordinate; ▲, Δh_{st0} (Sarabhai) left ordinate; ○, index X (Fluka) right ordinate; ●, index X (Sarabhai) right ordinate. The two horizontal lines indicate Δh_{ads} values in the Toth equation.

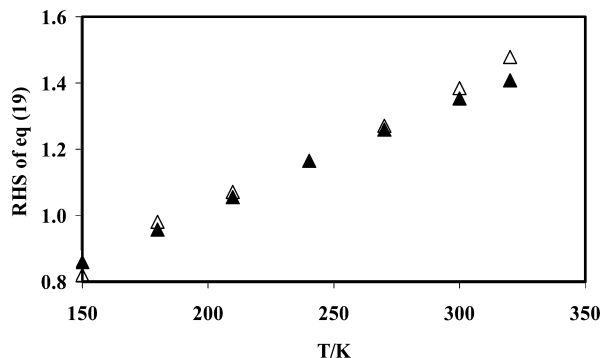


Figure 8. Temperature dependence of RHS of eq 19. △, Fluka; ▲, Sarabhai.

Table 5. Coefficients of Temperature Functions of Δh_{st0} and x in Equation 20

specimen	$A/J \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$	$B/J \cdot \text{mol}^{-1}$	a	b
Fluka	47.05	2377	$1.33 \cdot 10^{-3}$	0.565
Sarabhai	36.67	2265	$0.962 \cdot 10^{-3}$	0.458

The isosteric heat of adsorption determined from the Toth equation can be used to determine the mean pore width of the specimen using the results of ref 12. It is found to be 0.42 nm for the Fluka specimen and 0.58 nm for the Sarabhai specimen. However, the variation of the heat of adsorption predicted by the D–A equation shows that both the specimens have a highly heterogeneous pore size distribution. Its implications are that when a cryocooler is designed, during desorption at high temperature, the heat supplied to the thermal compressor should be steadily enhanced to sustain a meaningful discharge. At the same time, the cooling of the thermal compressor during adsorption needs be stronger at the beginning of the process than at the end.

While the D–A equation does not have a Henry's law regime,⁸ the Toth equation yields the temperature dependence of the Henry's law coefficients straight away.

$$k_H = \lim_{p \rightarrow 0} \frac{\partial C}{\partial p} T = C_0 k_0 \exp\left(\frac{\Delta h_{st}}{RT}\right) \quad (21)$$

The relation between Δh_{st0} , a parameter obtained from the limiting uptake condition from the D–A equation, and the Henry's law coefficient at the zero uptake condition derived from the Toth equation is investigated in Figure 9 for both the specimens. Interestingly, the following power law fits the entities

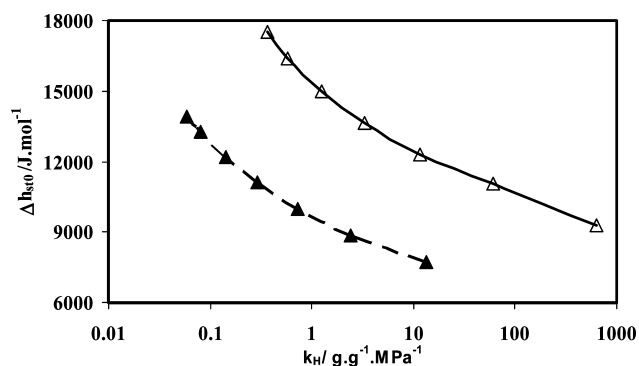


Figure 9. Relation between isosteric heats of adsorption at limiting loading from the D–A equation and Henry's law coefficients. Δ , Fluka; \blacktriangle , Sarabhai.

Table 6. Coefficients in Equation 22

specimen	$\Delta h_{st}^*/J \cdot \text{mol}^{-1}$	q
Fluka	15518	0.083
Sarabhai	9932	0.109

$$\Delta h_{st0} = \Delta h_{st}^* k_H^{-q} \quad (22)$$

The values of Δh_{st}^* and index q are listed in Table 6.

Conclusions

Volumetric method based nitrogen adsorption data for two specimens of activated carbon in the range of (150 to 320) K were refitted to the Dubinin–Astakhov and Toth isotherm equations to improve the accuracy of interpolation. The adsorption parameters in these new equations enable a rigorous treatment of the heat of adsorption and the Henry's law coefficients. The variation of adsorbed phase specific volume is substantial and cannot be ignored. Empirical correlations for several properties of interest are given which can be used in adsorption based gas storage and cryocooler designs.

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