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Water Adsorption on Charcoal - New Approach in Experimental Studies and Data Representation

M. Geynisman and R. Walker

*Fermi National Accelerator Laboratory
P.O. Box 500, Batavia, Illinois 60510*

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**WATER ADSORPTION ON CHARCOAL - NEW APPROACH IN
EXPERIMENTAL STUDIES AND DATA REPRESENTATION**

by

M.G. Geynisman and R.J. Walker

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RÉSUMÉ / SUMMARY:

The experimental apparatus was built to study the H₂O adsorption on charcoal at very low concentrations and collect the data in the form of isosteres. Experimental method is discussed and the global three-dimensional fit is constructed to predict the post-regeneration conditions of charcoal absorbers.

MOTS-CLÉS / KEYWORDS:

Water, Charcoal, Adsorption, Isosteres, Fit

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**XVIII^e
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M.G. GEYNISMAN, R.J. WALKER

Fermi National Accelerator Laboratory*
Batavia, Illinois, USA

1. INTRODUCTION

The extensive application of charcoal absorbers in cryogenic or chemical service at warm gas conditions creates a strong demand for workable mathematical models to predict equilibrium conditions during adsorption and desorption cycles /1/. The optimal combinations of "adsorbate-adsorbent" are generally used in industrial practice, but some adsorbents can be successfully used to adsorb several substances with about equal thermodynamic properties /2/. Charcoal is named here as a good example since it tends to adsorb not only the broad spectrum of hydrocarbons, but water as well. The charcoal absorbers are installed in cryogenic systems to remove contaminants from a process flow at warm conditions and secure equipment operated at low temperatures from plugging up or physical damage. A correctly timed desorption process must be performed with respect to all possible contaminants. Although charcoal absorbers are mostly designed and used for adsorption not of water, but other substances, water is a residual constituent in charcoal and an alien component permeated in cryo process from outside. The water eventually saturates the charcoal and contaminates the process. Therefore the "water-charcoal" combination is a valid subject for adsorption studies at warm gas temperatures.

The Central Helium Liquefier facility at FERMILAB, which is the world largest stand-alone helium reliquefier with the production of 5500 lt/hr of liquid helium, operates its three 4 ton charcoal absorbers along with some smaller ones to remove impurities from helium flow. The operation of these absorbers is critical for the coldboxes' performance since even a 1 ppmv of H₂O concentration in a 1300 g/s inlet helium gas flow will result in 1 kg/day² of H₂O ice brought into the coldboxes' turbines, filters and heat-exchangers. The charcoal regeneration, or so-called bakeout operation, is a desorption process that is carried out as a combination of thermal, pressure and purge cycles. It results in removing H₂O from that zone of the charcoal where it is present in saturation condition thus bringing the "H₂O-charcoal" system to a lower level of overall H₂O load. A well-timed charcoal regeneration should provide an H₂O post-regeneration equilibrium concentration of less than 10 ppbv at room temperature and 1.25 MPa pressure. This guarantees that the coldboxes operate safely within the next 200 days before the on-line charcoal absorber, though it is still far from reaching the breakthrough conditions, is shut down and replaced with the spare charcoal absorber.

By the end of a bakeout operation, when the charcoal bed temperature is 250-300 deg.C, the level of water concentration in the effluent is still high enough to get an accurate reading with commercially available moisture analyzers (>5 ppmv). Completion of the bakeout operation is followed by charcoal isolation and cooldown back to operating temperature. As a cooldown proceeds the H₂O concentration in the effluent drops dramatically beyond the limits of reliable and precise measurements.

* Operated by Universities Research Association, Inc. under contract with the U.S. Department of Energy.

Since the H_2O concentration can be accurately measured before cooldown is started and performed isothermally at constant overall H_2O load, we only need to know how to scale this information to the lower temperature to be able to calculate the H_2O equilibrium concentration after the charcoal absorber has been brought down to its normal operating temperature. Ideally, we need to have a 3-dimensional dependence between H_2O concentration P {ppmv or partial pressure of H_2O , kPa}, load L {weight of H_2O relative to the weight of charcoal, %} and temperature T {deg.K} at the states of adsorption equilibrium within the applicable range of variables.

The experimental data presented in literature /2,3,4,5,6,etc/ were found scarce and not consistent enough to fulfill our specific objectives. Also, none of the herein above referenced experimenters utilized the isosteric method which seemed to be more useful for our particular application. And lastly, none of the experiments resulted in 3-dimensional mathematical model with coefficients defined for the established range of variables.

A concept was discussed by Brunauer /5/, Haseldon /7/ and others which used isosteres to determine the value of the heat of adsorption in a manner quite analogous to the determination of the heat of vaporization from a vapor-pressure curve from the Clausius-Clapeyron relation. This idea presents us with an opportunity to build descriptive empirical equations in the form of the theoretically meaningful expanded Clausius-Clapeyron equation, thus achieving two goals:

- * create a global 3-dimensional fit for practical applications;
- * study fit coefficients and contribute to the discussions on the heat of adsorption.

These goals motivated us to design and conduct an experimental investigation of " H_2O -charcoal" equilibrium conditions with isosteric method.

2. EXPERIMENTAL SET-UP AND PROCEDURE

The experiment was designed to maintain and quantitatively determine the quasi-stationary adsorption equilibrium conditions of " H_2O -charcoal" **fixed mass** composition. We assumed the chemisorption effects being negligible and the van der Waals type of adsorption being predominant within the discussed range of variables, which was later confirmed by the reversibility in adsorption-desorption cycles. Each isostere was determined and experimentally developed as a set of adsorption equilibrium states at constant load with quasi-stationary transitions from one state to another. The transitions were made by changing the temperature of the charcoal bed very slowly while maintaining a constant temperature distribution throughout the bed. Equilibrium conditions were assumed if no changes (within the applied instrumentation accuracy) were noticed in temperature distribution and H_2O concentration in absorber effluent within one hour. The assumption was made that the amount of H_2O adsorbed on water injection chamber and piping components walls was negligible, thus, all water introduced into the system was subject to the adsorption on charcoal. In fact, one layer of H_2O molecules with equivalent diameter of 4 angstrom covering the total "out-of-absorber" surface of 4550 cm^2 can constitute only 0.00012 g of water which is 0.2% of the smallest amount of H_2O injected.

Dry nitrogen gas (dew point < -80 deg.C) at 0.4 kPa from liquid nitrogen 14,000 liter dewar boil off was used as a carrier gas. Since nitrogen has a negligible rate of adsorption on charcoal, compared to water /5/, the latter was considered to be the only adsorbate in the system. On the other hand, since H_2O concentration in nitrogen never exceeded 1000 ppmv, nitrogen could be considered to be the only gas for flow, thermodynamic and heat exchange calculations.

The experimental apparatus was mechanically designed as a vacuum leak-tight ($<10^{-10}$ st.cm³/sec) closed loop piping system (Figure 1), which included as major elements: charcoal absorber (1), oven (2), external pre-heater (3), heat exchanger (4), moisture analyzer (5), circulating pump (6), precision dew point generator (7), water injection chamber (8), nitrogen source (9).

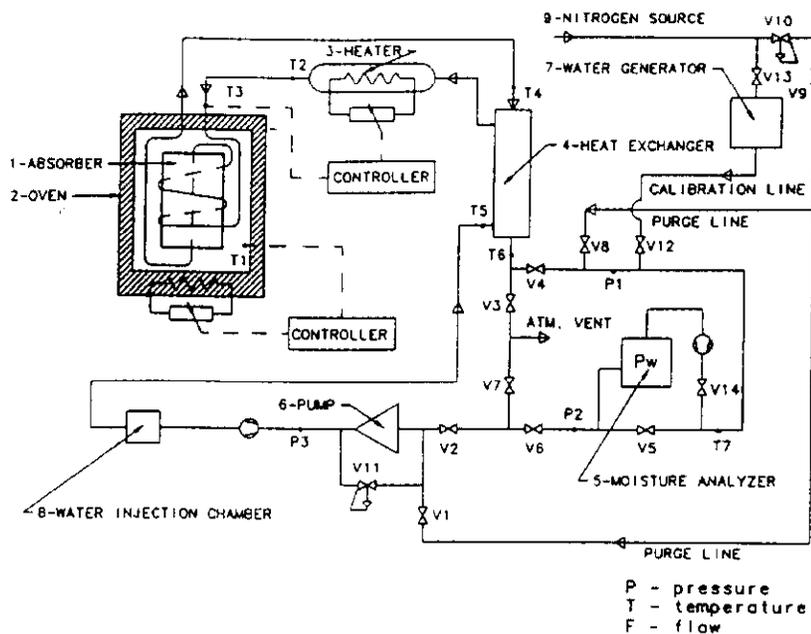


Figure 1 - Simplified diagram of the experimental apparatus

The charcoal absorber was designed as a stainless steel flanged vessel to contain 500.0 g of charcoal within its spring loaded screens. Charcoal was CALGON type CP-IV, 4x6 mesh, 1100 m²/g adsorption capacity and 0.48 g/cm³ apparent density. The absorber vessel was vertically installed inside the 21,000 cm³ high temperature electric oven with the carrier gas flow directed downward. The assumption was made of uniform load distribution within the charcoal volume at a state of equilibrium. Since the objective was to maintain the charcoal bed temperature uniform and at a given level, provisions were made to make adequate temperature measurements and to minimize the difference between the mean temperature of the charcoal bed and measured temperature T1 inside the oven. First, an electric tubular pre-heater was installed to keep the carrier gas temperature T3 at the same given level. Secondly, the charcoal absorber inlet piping was supplied with a 1 m long finned copper tube coiled around the absorber vessel. Calculations showed a maximum difference of 0.25 deg.C between the oven and incoming gas temperatures at no external heat load disturbances. Two PID temperature controllers regulated T1 and T3 by varying the power loads of the oven and the heater.

The heat exchanger was installed to provide extensive heat exchange between charcoal absorber incoming and outgoing gas flows. That let us conduct the pressure and H₂O concentration measurements at ambient temperature level and, on the other hand, facilitate the pre-heating of the charcoal absorber incoming flow. The heat exchanger had a counterflow multiple tube-in-shell design, was made from copper and showed a mean temperature difference of 15 deg.C or less.

The water injection chamber was a flanged cylinder of 300 cm³ volume which had enough space for H₂O evaporation and mixing with nitrogen before the gas was transported along the system. The water was injected while the charcoal was at the maximum temperature so that the water would be distributed uniformly throughout the charcoal more rapidly. The injections were made with a precision syringe through a high vacuum septum. The procedure was to weigh the source vial before and after filling the syringe. The electronic balance resolution was 0.00001 g.

The main operating objective was to keep the carrier gas circulating through the system's closed loop. To accomplish that a diaphragm pump was installed to provide a maximum flow rate of 27,000 st.cm³/min at 1.15 pressure ratio. The flow rate was measured and kept constant with the kickback valve V11. The circulation enabled forced mass transfer with a faster approach to uniform H₂O distribution within charcoal volume and equilibrium conditions. It also allowed us to install the moisture analyzer at the pump suction where the charcoal effluent was already cooled in the counterflow heat exchanger.

The moisture analyzer chosen was a model "Dewlux 20" silicon chip hygrometer manufactured by MCM for the range of 1-1000 ppmv, 1,500 cm³/min recommended flowrate which had a 4-20 mA linearized output signal. This is currently the only commercially available moisture analyzer on the market utilizing "true" silicon chip design /8/. It has been preferred over the broad spectrum of extensively tested aluminum oxide sensors from considerations of repeatability, reliability, accuracy and independence from ambient and carrier gas temperature variations /9,10/. The latter consideration is a must since the temperature of the charcoal absorber effluent varies in time with variations in charcoal bed and room temperatures. In practice this causes significant errors in aluminum oxide sensor readings as the amount of water held by a sensor varies with temperature. Those errors are nonlinear with their corresponding inducements, thereby making automatic corrections difficult. Additionally, after a rise in moisture content occurs the conventional sensors have to be dried for hours/days to bring them back down and often recalibrated as readings tend to drift at low trace levels. Unlike other so-called "silicon chip" sensors in which the silicon is insulated by a non-hygroscopic layer of silicon oxide, but hygroscopic layer is still aluminum oxide or calcium fluoride, the MCM sensor comprises a layer of hygroscopic silicon oxide supported on a substrate of silicon. The 5x5 mm chip sensor is enclosed in a stainless steel housing and connected to a monitor. An attached 1 Watt thermostatically controlled heating element maintains the sensor at constant 40 deg.C temperature which is above normal ambient or sample gas temperatures. Thus it can be operated in a variety of operational modes and environments without loss of accuracy. If the sensor is saturated by a moisture surge, it can be dried in seconds with a push-purge process, which momentarily elevates the sensor temperature and drives off the moisture.

The sensor output signal was converted to a 0-5 V range and the calibration curve provided by the manufacture was checked to be true and stable before and after the data were collected. This was accomplished with a precision gas dilution dew point generator DPG-300 incorporated in the experimental set-up design. The calibration data have been fitted, compared with the factory calibration curve and used to convert the MCM voltage readings into the water concentration values.

The pressures P1, P2 and P3 were measured with calibrated pressure transducers. Temperatures T1,...,T7 were measured with J type and T type thermocouples calibrated with their converters. The moisture analyzer readout and all pertinent pressure and temperature transducers' readouts were continuously recorded.

The experiment consisted of 3 different operational modes:

- **Bakeout.** To achieve the uniform initial conditions each isosteric process was followed with vacuum desorption (1 Pa) and high temperature bakeout of the charcoal (300 deg.C) while purging the system with dry nitrogen via the "V10-V9-V1-absorber-atmosphere" flow path. The moisture analyzer was purged via the "V10-V9-V8-V14-V6-V7-atmosphere" flow path.
- **Calibration,** as discussed above, via the "V13-V12-V14-V6-V7-atmosphere" flow path.
- **Main Run.** The system was initially pressurized with dry nitrogen and isolated. Continuous circulation was initiated while temperatures T1 and T3, flowrates F1 and F2 were controlled and other parameters were measured, as discussed above. The pre-determined amount of H₂O was injected, and constant circulation flow was maintained until equilibrium was attained. Parameters were recorded, and the heater controls were set to bring the charcoal to a new temperature.

The average time-period involved in reaching a new state of equilibrium for a temperature change of 20 deg.C was of the order of 30-48 hours. The temperature range of an isostere was large enough to bring the system's pressure to the sub-atmospheric level, but since it was built and tested to be vacuum leak-tight, no atmospheric moisture was introduced into the system. The adsorption and desorption data were taken along constant mass lines, but as no detectable difference had been found between adsorption and desorption, it was concluded that hysteresis was not a complicating issue.

3. RESULTS AND DISCUSSION

The raw experimental data have been collected as a set of data points spanning a range of partial pressure of water vapor P (in kPa), temperature T (in deg.K) and weight percent loading of water L in the charcoal sample. As a result a set of isosteres has been obtained for loads L from 0.01% to 0.5% (Figure 2).

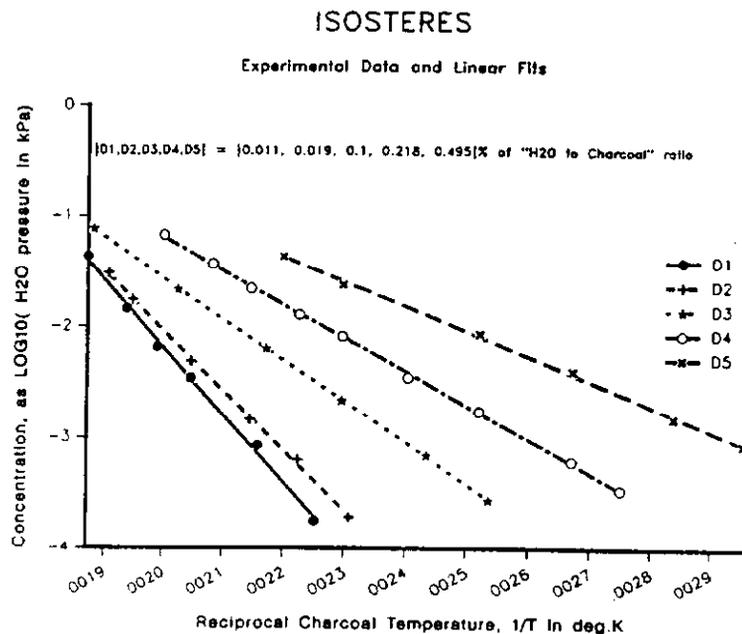


Figure 2 - Experimental Data and Linear Fits.

It was assumed that these data fell on or near a thermodynamically defined surface in the space of these three variables. In order to make the experimental data useful at arbitrary points and to provide a useful extrapolation of the data outside the measured ranges, it is desirable to find an approximate mathematical representation of the thermodynamically defined surface. The approach to this problem is to use the plots shown in Figure 2 to suggest the mathematical form. The data appeared to be very well fitted with linear fits in $[\text{LOG}_{10}(\text{P})-1/\text{T}]$ coordinates. The slopes and intercepts of these lines are evidently a function of load L. This linearity suggests the form:

$$\text{LOG}_{10}(\text{P}) = f_1(\text{L}) + f_2(\text{L})/\text{T} \quad (1)$$

The forms of the intercept function $f_1(\text{L})$ and the slope function $f_2(\text{L})$ were determined by error trial attempting to keep the forms as simple as possible and still produce a good fit to the data. The result of the trials was as follows:

$$f_1(\text{L}) = X_1 + X_2 \text{LOG}_{10}(\text{L}) \quad \text{and} \quad f_2(\text{L}) = X_3 + X_4 \text{LOG}_{10}(\text{L}) \quad (2)$$

where X_1 to X_4 are coefficients to be determined by the fitting program described below. Combining eq.(1) and eq.(2) gives the required mathematical form for the approximation to the surface:

$$Z(\text{L},\text{T}) = X_1 + X_2 \text{LOG}_{10}(\text{L}) + [X_3 + X_4 \text{LOG}_{10}(\text{L})]/\text{T} \quad (3)$$

where $Z(\text{L},\text{T})$ is the surface to be formed as a fit to the data for $\text{LOG}_{10}(\text{P})$.

The values of the parameters X_1 to X_4 are adjusted so that the values of the fitting function lie as close to the data points as possible. This fit is performed in a computer program GEN3 which minimizes the summed square error $E(X_1, X_2, X_3, X_4)$ between the measured values of $\text{LOG}_{10}(\text{P})$ and the values of $Z(\text{L},\text{T})$ calculated using eq.(3). This sum is performed over all measured data points which are weighted equally.

The method of calculation follows a paper by Cziffra and Moravcsik /11/ which develops a linear least squares solution, except that the method is generalized to fit a 3-dimensional surface. Note that eq.(3) is linear in the fitting coefficients. To find a solution, we form a set of 4 linear equations in the coefficients by stating the condition that the minimum error occurs at the point in the 4 dimensional space of the X_k at which the partial derivatives of the error function with respect to the X_k are all zero. With denoting T_i as the temperature in deg.K, L_i as the % loading of the charcoal and P_i as the partial pressure in kPa for the i-th data point, this condition can be stated as follows:

$$E = \sum_{i=1}^N \left[\sum_{k=1}^4 X_k \phi_k(L_i, T_i) - \text{LOG}_{10}(P_i) \right]^2 \quad (4)$$

$$\frac{\partial E}{\partial X_m} = 0 = 2 \times \sum_{i=1}^N \left[\sum_{k=1}^4 X_k \phi_k(L_i, T_i) - \text{LOG}_{10}(P_i) \right] \phi_m(L_i, T_i) \quad (5)$$

where we define for all $i = 1, \dots, 33$ and $m = 1, \dots, 4$:

$$\phi_1(L_i, T_i) = 1 \quad \phi_2(L_i, T_i) = \text{LOG}_{10}(L_i)$$

$$\phi_3(L_i, T_i) = 1/T_i \quad \phi_4(L_i, T_i) = \text{LOG}_{10}(L_i)/T_i$$

Eq.(5) can be seen to be a set of 4 linear equations with the 4 unknown coefficients X_k . These equations can be condensed into a matrix equation:

$$H \times X = G \quad \text{or} \quad X = H^{-1} \times G \quad (6)$$

where X is a 4 dimensional column vector of the unknown coefficients X_k , G is a 4 dimensional row vector formed from the ϕ functions and the data for P as defined below in eq.(7), and H is a 4 x 4 matrix formed from the ϕ functions as defined below in eq.(8).

$$G_m = \sum_{i=1}^N \text{LOG}_{10}(P_i) \phi_m(L_i, T_i) \quad (7)$$

$$H_{km} = \sum_{i=1}^N \phi_k(L_i, T_i) \phi_m(L_i, T_i) \quad (8)$$

The solution X as described above in eq.(6) does not require starting values and gives a unique solution for the X_k . The best four parameter fit to the data with the root mean square error over all the data points as 0.0401205 was found as follows:

$$\begin{aligned} X_1 &= 2.21507 & X_2 &= -3.90706 \\ X_3 &= -1470.31 & X_4 &= 2336.19 \end{aligned}$$

The graphical representation of the surface for the studied range of variables is shown in Figure 3. Cross-sections of the surface made perpendicular to the load, concentration or temperature axis represent isosteres, isobars or isotherms.

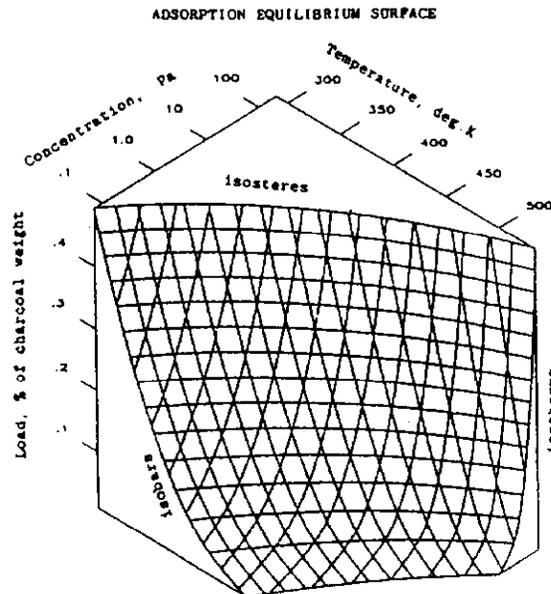


Figure 3 - Adsorption Equilibrium Surface

The analytical expressions for isosteres and isotherms were derived from eq.(3):

$$\text{LOG}_{10}(P) = X_1 + X_2 \text{LOG}_{10}(L) + \frac{X_3 + X_4 \text{LOG}_{10}(L)}{T}, \quad L = \text{const} \quad (10)$$

$$P = 10^{[X_1 + X_3/T + (X_2 + X_4/T)\text{LOG}_{10}(L)]}, \quad T = \text{const} \quad (11)$$

A thermodynamic analysis of an isosteric process /5/ yields the equation:

$$[\partial(\text{LOG}_{10}P)/\partial(1/T)]_{L=\text{const}} = -q_{\text{isos}}/R \quad (12)$$

where q_{isos} is the isosteric heat of adsorption, and $R=0.462$ kJ/kg is the gas constant for H_2O . This equation can be derived from the Clausius-Clapeyron equation /5/ where the specific volume of the adsorbed phase has been neglected. The slope of the plot of $\text{LOG}(P)$ versus $1/T$ at constant L gives q_{isos}/R . Straight lines were obtained for the isosteres (see Figure 2) thus confirming the independence of q_{isos} with respect to temperature over the studied range. Therefore, the integral form of the eq.(12) taken within any temperature range from T_a to T_b was used to calculate isosteric heat for each isostere:

$$q_{\text{isos}} = 2.30255 R T_a T_b \text{LOG}_{10}(P_a/P_b)/(T_a - T_b) \quad (13)$$

The isosteres described analytically with eq.(10) can be expressed as:

$$q_{\text{isos}} = 2.30255 R (X_3 + X_4 \text{LOG}_{10}(L)) \quad (14)$$

Eq.(14) represents the analytical dependence of the isosteric heat on the amount of water adsorbed by charcoal. Figure 4 shows this derived dependence with comparison to the isosteric heat values calculated for the experimental data straight-forward with eq.(13).

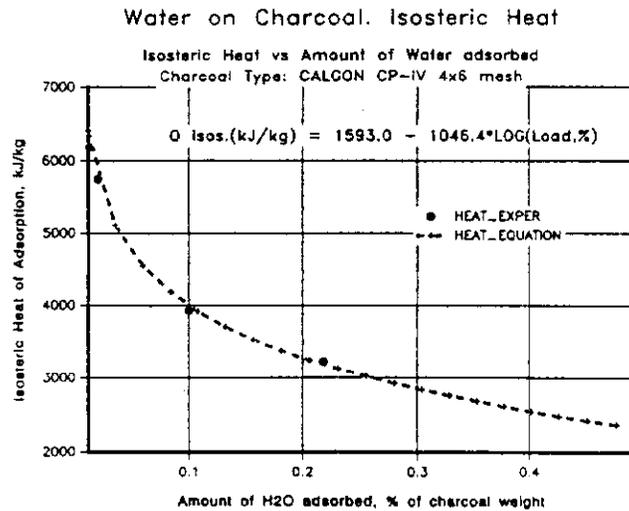


Figure 4 - Isosteric Heat of Adsorption

The isosteric heat values were found to be higher than the heat of liquefaction at 25 deg.C (2441 kJ/kg) or measured /6/ calorimetric heat at 80

deg.C (1930 kJ/kg). This difference is related to the low levels of loads which were studied. Since the isosteric heat varies significantly with the load variations the simplest descriptive forms for adsorption, such as Langmuir or Williams equations, do not describe the data. If chemisorption effects are ruled out and the average distance between adsorbed molecules is too large to permit appreciable interaction, then the large variations in the q_{isost} at low loads should be attributed to the structure of the charcoal itself. This large variation may be explained on the basis of pore structure. It is possible to correlate the energy of adsorption with pore diameter, and the evidence /3/ supports the belief that the smaller the pore diameter, the higher the energy of the sites within the pore. In a pore of molecular dimensions the molecule is probably attracted by surface forces on several sides; that is, there may be a cooperative wall effect assisting adsorption. It is reasonable to suppose that adsorbate molecules are attracted first to regions of the surface which have the highest energy. Consequently, at low concentrations, high energy surface area in fine pores would be the dominant factor determining equilibrium capacity rather than total surface area in all pores.

The typical isotherms plotted as defined with eq.(11) are presented in Figure 5. Inspection reveals that low temperature isotherms exhibit type I, but high temperature isotherms tend to be type III or V adsorption isotherms.

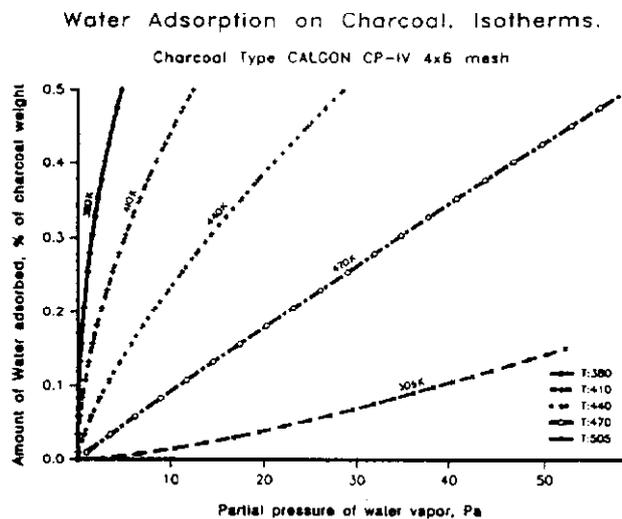


Figure 5 - Adsorption Isotherms

A more precise analysis of an isotherm curvature in "L-P" coordinates shown in Figure 5 can be performed by defining a sign of the $\partial^2 L / \partial P^2$ derived from the function $Z(L, T)$ described with eq.(3):

$$\frac{\partial^2 L}{\partial P^2} = a b (b-1) P^{b-2} \quad (15)$$

where: $a = 10^{-\frac{(X_1 + X_3)}{T}}$ and $b = \frac{T}{X_2 T + X_4}$

The analysis of coefficients a and b for the studied range of temperature shows that the isotherms change their shape from being concave to convex toward the pressure axis at $T=476.087$ deg.K, where $b=1$. The change in

curvature is a direct consequence of the form of eq.(3) since the point $b=1$ is determined by the temperature.

4. CONCLUSION

Consistent experimental data in the form of isosteres have been obtained and analyzed for water adsorption on charcoal at low concentration. A global 3-dimensional fit has been constructed to describe analytically the adsorption equilibrium surface within the studied range of variables and to approximate the equilibrium conditions even beyond that range to predict the post-regeneration conditions of charcoal absorbers. Practical solutions to utilize the isosteric experimental method, including mechanical design, instrumentation selection and computer code writing, have been developed. The analytical fit has been used at FERMILAB to determine the adequacy of the bakeout operation performed for very large charcoal absorbers.

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L'ADSORPTION D'EAU SUR DU CHARBON DE BOIS. UNE NOUVELLE APPROCHE DES TECHNIQUES EXPERIMENTALES ET DE L'ANALYSE DES DONNEES.

RESUME: L'utilisation massive de pieges a charbon par les systemes cryogeniques requiert l'accumulation de donnees experimentales suffisantes pour comprendre l'adsorption d'une grande variete de substances. Peu de resultats experimentaux sur l'adsorption de l'eau par le charbon de bois ont ete publies.

Un dispositif experimental a ete construit. Une recherche a long terme a ete entreprise pour etudier l'adsorption de H₂O et enregistrer les donnees sous forme d'isosteres dans le domaine: [0-500 ppm en concentration, 55-255 deg.C en temperature et 0-0.5% en capacite].

La methode experimentale developpee, les solutions pratiques qui ont ete adoptees en ce qui concerne la selection des instruments et leurs tests, l'ajustement general en trois dimensions des resultats obtenus, et son interpretation, pourraient etre utiles aussi bien aux R&D en cryogenie qu'aux systemes en fonctionnement.