Adsorption (2013) 19:1189–1196
DOI 10.1007/s10450-013-9556-x

Adsorption Henry constants calculated from the entire isotherm

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Received: 10 March 2013 / Accepted: 12 June 2013 / Published online: 23 June 2013

Abstract A method is introduced to derive adsorption Henry constants from experimental isotherm data regardless of whether the measurements extend into the Henry law region. The method uses the intersection between Henry's law and the Gaussian isotherm model. In this way, the Henry constant can be calculated from Gaussian model parameters obtained from a fit of the model to the entire isotherm. In addition, the Gaussian model is shown to closely agree with Henry's law over a wide range. To demonstrate the accuracy of the method, Henry constants are derived from 63 experimental isotherms collected from the literature for supercritical *n* alkanes on 5A zeolite. These Henry constants compare well to values found in the literature.

Keywords Separations · Henry constants · 5A zeolite · Supercritical *n* alkanes isotherms

List of symbols

Variables

C	Concentration (mg/L)
K_H	Henry constant (g/100 g Z kPa)
$K_{H,lit}$	Henry constant reported in the literature (g/100 g Z kPa)
K_H^*	Normalized Henry constant
P	Pressure (kPa)
P*	Normalized pressure P/P_{50}
$P(Z_H)$	Pressure calculated at Z_H (kPa)
P_{50}	Pressure at 50 % loading, same value as the log-mean pressure (kPa)
q	Zeolite loading (g/100 g Z)
$q(Z_H)$	Zeolite loading at Z_H
q_{max}	Maximum zeolite loading (g/100 g Z)
T_r	Reduced temperature
Z	Standard normal probability distribution variable
Z_H	Z-value at the intersection between Henry's law and the Gaussian model

Greek letters

ϵ	Tolerance level
θ	Loading ($= q/q_{max}$)
$\theta(Z_H)$	Corresponding loading at $Z_H = 0.027$
σ_e	Standard deviation for the isotherm when using natural logarithm
σ_{10}	Standard deviation for the isotherm when using base 10 logarithm
$(-\Delta H_0)$	Heat of adsorption at 0 % loading (kJ/mol)
$(-\Delta H_{50})$	Heat of adsorption at 50 % loading (kJ/mol)
Φ	Cumulative distribution function
Φ^{-1}	Inverse cumulative distribution function

1 Introduction

The Gaussian adsorption isotherm model has been shown to fit well the behavior of type I isotherms (Abouelnasr and Loughlin 2010; Loughlin and Abouelnasr 2011). The objective of this paper is to demonstrate that the Gaussian model is also consistent with Henry's law for much of the Henry law region, and that Henry constants may be calculated by the Gaussian model, regardless of whether the experimental data extend into the Henry law region.

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The adsorption of a molecule onto an adsorbent depends on many factors, such as its velocity and whether a site is available. A process which depends upon the probability of many different phenomena is often well-described by a log-normal Gaussian distribution. Indeed, type I adsorption isotherms, when plotted as q or θ versus ln P or ln c, exhibit the shape of the Gaussian cumulative frequency distribution (Abouelnasr and Loughlin 2010; Loughlin and Abouelnasr 2011).

2 Theoretical

In the Gaussian isotherm model, the standard normal probability distribution variable, Z, is first related to the log of the pressure by Eq. 1, where P_{50} is the pressure at 50 % loading, which is also the log-mean pressure for the entire isotherm. The standard deviation for the entire isotherm is specified as σ_{10} when using logarithm base 10, and σ_e when using natural logarithms.

$$Z = log\left(\frac{P}{P_{50}}\right)^{\frac{1}{\sigma_{10}}} = ln\left(\frac{P}{P_{50}}\right)^{\frac{1}{\sigma_e}} \tag{1}$$

Another form of this equation may be obtained through rearrangement.

$$P^* = \frac{P}{P_{50}} = 10^{\sigma_{10}Z} = e^{\sigma_e Z} \tag{2}$$

The two standard deviations, σ_{10} and σ_e, are related by the natural log of 10.

$$\sigma_e = \sigma_{10} \ln 10 \tag{3}$$

The Gaussian isotherm, $\Phi(Z)$, is defined as the cumulative distribution function for the standard normal distribution. It corresponds to θ, the loading.

$$\Phi(Z) = \theta = \frac{q}{q_{max}} = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{Z} e^{-z^2/2} dz \tag{4}$$

Eq. 4 may be rearranged by taking the inverse of the cumulative distribution function:

$$Z = \Phi^{-1}(\theta) \tag{5}$$

For simplicity, the cumulative distribution function is henceforth referred to as Φ, and the inverse function as Φ^{-1}. The values of Φ and Φ^{-1} are commonly determined from tables of the standard normal distribution. In Excel, functions NORMSDIST and NORMSINV can be used to provide Φ and Φ^{-1}.

Rearranging Eq. 5 provides a linear equation for ln P versus $\Phi^{-1}(\theta)$:

$$\ln P = \ln P_{50} + \sigma_e \Phi^{-1}(\theta) \tag{6}$$

If q_{max} is known then linear regression techniques may be used to find P_{50} and σ_e.

The Gaussian isotherm model fits the isotherm data well, as was shown for supercritical *n* alkanes on 5A zeolite (Loughlin and Abouelnasr 2011). The authors have also successfully applied the Gaussian model on other type I systems, including subcritical *n* alkanes on 5A zeolite and several types of hydrocarbons, alkanes, alkenes, aromatics and inorganics on 13X zeolite. This will be transmitted in forthcoming papers (Abouelnasr and Loughlin 2013).

Although the model fits well, in the limit as pressure approaches zero, the gradient of this isotherm also approaches zero. However, this does not limit the utility of the model in the Henry law region, nor does it preclude its ability to determine the Henry constant, as we shall show.

To compare the Gaussian model to Henry's law, we must first restate Henry's law. Adsorption in the Henry's law region is defined by the equation

$$q = K_H P \tag{7}$$

where K_H is usually defined by

$$K_H = \lim_{P \to 0} \frac{dq}{dP} \tag{8}$$

This linear relationship may alternatively be stated using the differential of the logarithms as:

$$\frac{d \ln q}{d \ln P} = 1 \tag{9}$$

which also gives a linear relationship compatible with Eqs. 7 and 8. Equation 9 can be used to find the intersection point between Henry's law and the Gaussian model. This point can then be used to establish the Henry constant from Gaussian parameters. In essence, this allows the Henry constant to be determined from experimental data which might not extend into the Henry law region.

To find the intersection point, we begin by rearranging Eq. 9.

$$\frac{dq}{dP} = \frac{q}{P} \tag{10}$$

Multiplying by (P_{50}/q_{max}) and substituting θ and P* gives the following relation.

$$\frac{d\theta}{dP^*} = \frac{\theta}{P^*} \tag{11}$$

Substituting for P* using Eq. 2 and rearranging provides Eq. 12.

$$\frac{d\theta}{dZ} = \sigma_e \theta \tag{12}$$

Now the Gaussian model is introduced by using the derivative of Eq. 4. Substitution into Eq. 12 and rearrangement yields Eq. 13.

$$\theta = \frac{e^{-Z^2/2}}{\sqrt{2\pi}\sigma_e} \tag{13}$$

This is an implicit equation for Z. It may be solved to yield the value of Z at the intersection, denoted as Z_H, with a corresponding value of fractional loading denoted by $\theta(Z_H)$.

$$\theta(Z_H) = \frac{e^{-Z_H^2/2}}{\sqrt{2\pi}\sigma_e} \tag{14}$$

Upon specifying σ_e, this equation may be solved for Z_H. For example, for supercritical *n* alkane adsorption onto 5A zeolite, σ_{10} has been observed to be 1 (Loughlin and Abouelnasr 2011). Thus, σ_e is 2.3 (from Eq. 3). The subsequent value for Z_H from Eq. 14 is −1.92, and the value for $\theta(Z_H)$ is 2.7 %. Thus the intersection between Henry's law and the Gaussian model is at 2.7 % loading.

An expression for the Henry constant can now be derived from Eq. 14, using the fact that

$$K_H = \frac{q(Z_H)}{P(Z_H)} = \frac{q_{max}\theta(Z_H)}{P(Z_H)} \tag{15}$$

$P(Z_H)$ and $q(Z_H)$ are the pressure and the loading at the intersection point. After substituting for $\theta(Z_H)$ from Eq. 14 and rearranging we obtain,

$$K_H = \frac{q_{max}e^{-Z_H^2/2}}{\sqrt{2\pi}\sigma_e P(Z_H)} \tag{16}$$

Substituting for $P(Z_H)$ from Eq. 2 provides the following.

$$K_H = \frac{q_{max}e^{-\left(Z_H^2/2+\sigma_e Z_H\right)}}{\sqrt{2\pi}\sigma_e P_{50}} \tag{17}$$

This is the defining equation for K_H using the Gaussian isotherm. In this manner, K_H may be found from experimental data that might be far above the Henry's law region. To summarize the process:

1. Determine q_{max}, then fit the experimental data to the Gaussian model (Eq. 6), using linear regression to find P_{50} and σ_e.
2. Calculate Z_H from Eq. 14.
3. Determine K_H from Eq. 17.

The utility of the Gaussian model does not end at the intersection point. It remains in close agreement with Henry's law over a wide region. To delineate the entire region where the two models are in close agreement, we use a fractional difference between the two compared to a tolerance ε:

$$\frac{\theta(\mathrm{Henry'sLaw}) - \theta(\mathrm{Gaussian})}{\theta(\mathrm{Henry'sLaw})} \le \epsilon \tag{18}$$

Substituting and rearranging provides the following equation.

$$\frac{K_H P^* P_{50}}{q_{max}}(1-\epsilon) - \Phi(Z) \le 0 \tag{19}$$

This equation can be rearranged by substituting for P* using Eq. 2 to give Eq. 20 :

$$K_H^* e^{(\sigma_e Z)}(1-\epsilon) - \Phi(Z) \le 0 \tag{20}$$

where K_H^* is a normalized Henry parameter defined as:

$$K_H^* = \frac{K_H P_{50}}{q_{max}} = \frac{e^{-\left(Z_H^2/2+\sigma_e Z_H\right)}}{\sqrt{2\pi}\sigma_e} \tag{21}$$

K_H^* is temperature-invariant due to the presence of the isosteric heat in the P_{50} term, which cancels that in the K_H term.

Equation 20 is used to determine the range of applicability to meet a given tolerance level ϵ. For example, for supercritical *n* alkanes on 5A zeolite, σ_e is 2.3 and Z_H is −1.92, so that K_H^* is 2.28, and Eq. 20 becomes:

$$2.28\ e^{2.3\,Z}(1-\epsilon) - \Phi(Z) \le 0 \tag{22}$$

Solving this implicit equation for a tolerance level of 1 % provides a range for Z of −2.1 to −1.8, satisfying this relationship. This corresponds to a range for θ from 1.8 to 3.9 %. This close agreement between the Gaussian model and Henry's law is discussed in the results section.

3 Modeling results

To demonstrate the potential of the Gaussian model to determine Henry constants, we will apply it to supercritical *n* alkanes on 5A zeolite. All isotherms in this system have $\sigma_e = 2.3$ and $q_{max} = 9$, as shown in Loughlin and Abouelnasr (2009). Henry's law may be written as

$$\theta = K_H^* P^* \tag{23}$$

where K_H^* is 2.28 for supercritical *n* alkanes on 5A zeolite.

The Gaussian isotherm may also be expressed in terms of P* using Eqs. 1 and 4

$$\theta = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{ln(P^*)^{\frac{1}{\sigma_e}}} e^{-z^2/2} dz \tag{24}$$

This is temperature-invariant due to the presence of the P* term, which allows all the isotherms to fit on one characteristic curve as reported in our earlier paper (Loughlin and Abouelnasr 2011). Eqs. 23 and 24 are plotted for a θ range of 0–0.05 in Fig. 1. The two plots practically coincide. The normalized Henry's parameter line is linear but the Gaussian curve exhibits a sigmoidal shape dipping slightly lower for a θ range of 0–0.02, practically coinciding for the range of θ from 0.02 to 0.035 and then dipping slightly lower again for the region 0.035–0.05. The two plots exactly coincide at $\theta(Z_H) = 0.027$.

The per cent difference between the Gaussian model and the normalized Henry's parameter is illustrated in Fig. 2 for a θ range of 0–0.05. The Gaussian model is in excellent agreement with the normalized Henry's parameter over the range for θ from 0.018 to 0.039, or 1.8 to 3.9 % where the tolerance is less than 1 %.

In the lowest region, the % difference for the Gaussian isotherm becomes very large, as may be observed in Fig. 2. This is in agreement with the derivative of Eq. 4 which indicates that the slope in this region approaches zero. This may be a limitation if fundamental measurements are made to measure the Henry constant on the face of a pure crystal surface at extremely low loadings. However, very few measurements are executed this way. Practical measurements for isotherm modeling generally do not include this region; in fact most data tend to be above a loading of 1 or 2 %.

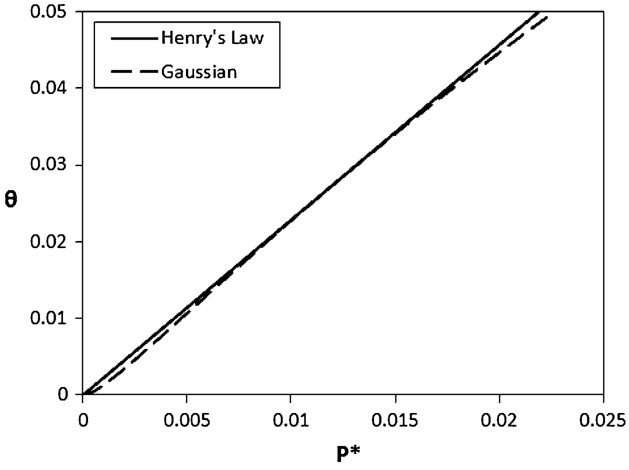

Fig. 1 Expanded plot of θ versus P* illustrating agreement between temperature-invariant normalized Henry parameter K_H^* and Gaussian isotherm at loadings for $\theta < 0.05$

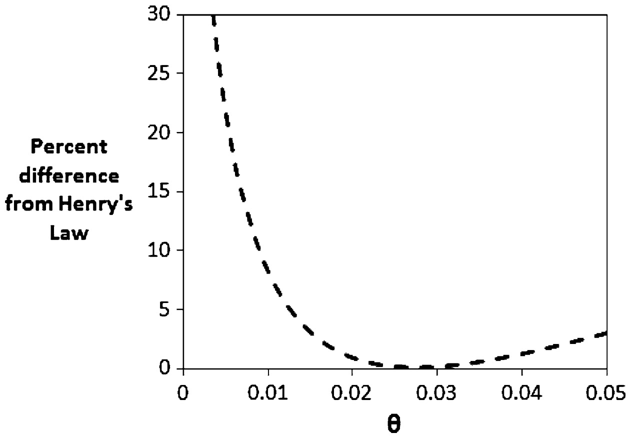

Fig. 2 Plot of per cent difference from Henry's law for Gaussian isotherm for $\theta < 0.05$

The isotherms for methane were fitted in our first paper on the Gaussian isotherm (Abouelnasr and Loughlin 2010). The plot is reproduced from that paper as Fig. 3. The fit was given in the 2010 paper (Abouelnasr and Loughlin 2010) as

$$\theta = 2.28\left(\frac{P}{P_{50}}\right)^{1/\sigma} \tag{25}$$

where σ refers to σ_{10}. For the case of $\sigma_{10} = 1$, this equation may be rearranged as

$$\theta = 2.28\,P^* \tag{26}$$

which is identical to the theoretical equation provided in Eq. 23. This is quite remarkable as at that juncture, the authors had not even considered this manuscript.

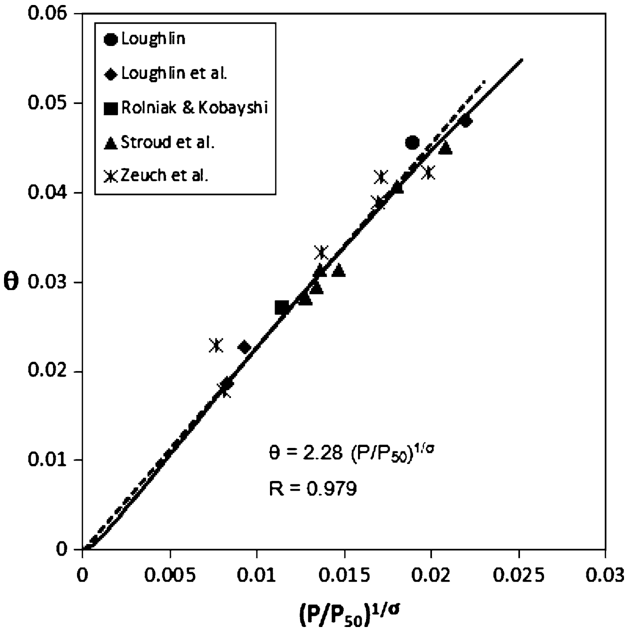

Fig. 3 Low concentration plot of θ versus P* for supercritical methane data on 5A zeolite

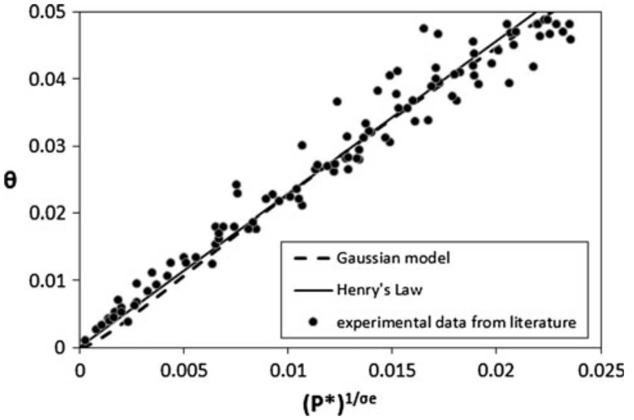

Fig. 4 Low concentration plot of θ versus P* for supercritical *n* alkane data on 5A zeolite

Table 1 Comparison of literature and calculated K_H values for all 63 isotherms

Carbon number	Reduced temperature	θ range	σ	P_{50} (kPa)	K_H in g/100 g Z kPa		
					Literature values	Gaussian model values	
						σ = 1	Actual σ
$n = 1$	0.97	0.60–0.80	0.84	7.25	5.06[a]	2.76	1.48
25 isotherms	1.02	0.05–0.79	0.90	9.81	2.22[a]	2.04	1.38
	1.14	0.08–0.58	0.98	37.5	0.560[a]	0.533	0.50
	1.21	0.11–0.39	0.85	74.1	0.295[a]	0.270	0.15
	1.22	0.06–0.41	1.02	88.2	0.265[a]	0.226	0.26
	1.33	0.03–0.27	1.10	248	0.0742[a]	0.0806	0.13
	1.35	0.26–0.79	1.13	282		0.0703	0.14
	1.43	0.03–0.15	0.99	427		0.0468	0.046
		0.02–0.16	0.97	450	0.0472[a]	0.0468	0.041
		0.33–0.83	0.96	514	0.0365[a]	0.0389	0.033
		0.04–0.14	1.10	654		0.0303	0.050
	1.44	0.10–0.67	0.91	511	0.0283[a]	0.0391	0.028
	1.48	0.15–0.77	1.05	673		0.0295	0.038
	1.51	0.41–0.89	0.96	563		0.0352	0.030
		0.03–0.11	1.10	946		0.0210	0.035
	1.54	0.06–0.47	0.96	1,280		0.0155	0.013
	1.56	0.35–0.85	1.00	774		0.0284	0.026
		0.02–0.42	0.92	990	0.0222[a]	0.0202	0.015
					0.0182[a]		
		0.08–0.70	1.04	1,240		0.0161	0.020
	1.57	0.05–0.59	0.88	1,100	0.0299[a]	0.0182	0.011
		0.02–0.09	1.07	1,310	0.0135[a]	0.0152	0.022
	1.62	0.29–0.80	1.06	961	0.0145[a]	0.0208	0.028
		0.03–0.38	0.89	1,230	0.0300[a]	0.0162	0.011
	1.71	0.04–0.51	0.87	1,820	0.00809[a]	0.0110	0.0066
	1.84	0.02–0.39	0.92	3,500	0.00476[a]	0.00571	0.0043
$n = 2$	0.96	0.08–0.76	0.79	14.8	0.371[a]	1.30	0.63
5 isotherms	0.98	0.21–0.76	0.84	15.6	0.417[a]	1.28	0.71
	1.01	0.001–0.85	0.88	30.2	0.771[a]	0.649	0.42
	1.06	0.08–0.41	0.73	38.7	0.284[a]	0.516	0.20
	1.13	0.04–0.25	0.81	90.7	0.118[a]	0.220	0.11
$n = 3$	1.08	0.12–0.63	0.80	21.7	0.612	0.921	0.44
$n = 4$	1.00	0.50–0.85	0.83	4.49	4.96[b]	4.42	2.33
6 isotherms		0.21–0.99	0.69	4.39		4.52	1.60
		0.18–0.98	0.72	5.06		3.92	1.48
	1.13	0.10–0.79	0.99	21.5	1.06[b]	0.923	0.92
		0.24–0.78	0.99	21.5	0.978[b]	0.923	0.92
	1.17	0.16–0.46	0.94	37.3		0.532	0.42
$n = 5$	1.00	0.27–0.88	0.93	4.38		4.53	3.43
9 isotherms	1.01	0.28–0.88	0.87	5.45		3.64	2.21
	1.11	0.04–0.51	0.80	10.6	0.995[c]	1.87	0.92
		0.09–0.70	0.88	20.0		0.992	0.62
	1.21	0.01–0.32	0.99	44.2	0.404[c]	0.449	0.45
		0.05–0.93	0.77	49.4		0.402	0.18
	1.22	0.07–0.51	0.91	56.8		0.349	0.25
	1.31	0.02–0.18	0.98	110	0.182[c]	0.180	0.17
	1.43	0.003–0.10	1.16	439	0.0819[c]	0.0452	0.10

Table 1 continued

Carbon number	Reduced temperature	θ range	σ	P_{50} (kPa)	K_H in g/100 g Z kPa		
					Literature values	Gaussian model values	
						σ = 1	Actual σ
$n = 6$ 6 isotherms	1.03	0.34–0.70	0.99	4.13	5.0[d] 6.76[d]	4.80	4.69
	1.13	0.16–0.50	1.19	15.6	1.54[d] 1.67[d]	1.27	3.47
	1.17	0.01–0.11	0.98	26.3		0.754	0.71
	1.20	0.04–0.11	1.02	22.6		0.878	0.98
	1.23	0.004–0.18	1.19	53.7		0.369	0.99
	1.26	0.01–0.14	1.10	58.2		0.341	0.58
$n = 7$	1.06	0.22–0.80	0.99	9.05	3.038[d]	2.27	2.13
$n = 8$ 7 isotherms	1.00	0.10–0.53	0.93	1.42	12.2[c] 9.9[d] 9.02[d]	14.0	10.7
	1.04	0.03–0.26	1.08	7.13		2.78	4.21
	1.07	0.05–0.26	1.03	9.73		2.04	2.45
	1.08	0.06–0.29	0.96	5.60	3.56[c] 2.9[d] 2.31[d]	3.54	3.06
	1.10	0.06–0.29	1.11	17.3		1.16	1.99
	1.12	0.02–0.17	1.05	20.0		0.992	1.27
	1.17	0.007–0.12	1.04	26.4	1.01[c] 0.82[d] 0.707[d]	0.751	0.93
$n = 10$	1.01	0.08–0.39	1.28	0.705		28.1	128
	1.08	0.22–0.82	1.22	3.52		5.64	17.9
$n = 12$	1.01	0.26–0.90	0.94	0.404		49.1	39.9

[a] Loughlin et al. (1990)

[b] Moeller et al. (2006)

[c] Vavlitis et al. (1981)

[d] Silva and Rodrigues (1999)

Data measurements in the lowest region probably provide the best estimate of the Henry constant for the simple reason that the loading is less than 1 % and hence is clearly in the Henry law region. However, if the pressure is less than 1 or 2 mbar, there may be thermal transpiration effects which may invalidate the results if due care is not exercised. For the supercritical *n* alkane isotherms available in the literature, there are only two isotherms that are in this region; these are the ethane isotherm of Glessner and Myers (1969) and the n pentane isotherm of Vavlitis (1978). The isotherm of Glessner and Myers was measured at extremely low concentrations with the objective that the data would clearly fit in the Henry law region. As both these low concentration isotherms have σ_{10} different from 1, consideration of them will be deferred at this time. They will be discussed with the subcritical data in a forthcoming paper (Abouelnasr and Loughlin 2013).

Data measurements over portions of the entire isotherm are more frequent and exist for 63 isotherms from 18 studies for supercritical *n* alkanes on 5A zeolite (Loughlin and Abouelnasr 2011). Twenty-six of the 63 isotherms included data points at loadings of less than 5 %. A loading of 5 % is generally accepted to be in the Henry's law region but is probably too high. A more appropriate maximum loading for the Henry law region is probably $\theta < 0.03$ but as this excludes so much data, it is generally not adhered to, with the result that 0.05 is approximately the maximum acceptable value. These experimental data are shown in Fig. 4. The data appear strongly linear especially in the highest region and are correlated by the equation

$$\theta = 2.22P^{*} \tag{27}$$

with a regression coefficient of 0.96. The value of 2.22 is within 3 % of the theoretical value of 2.28. It should be

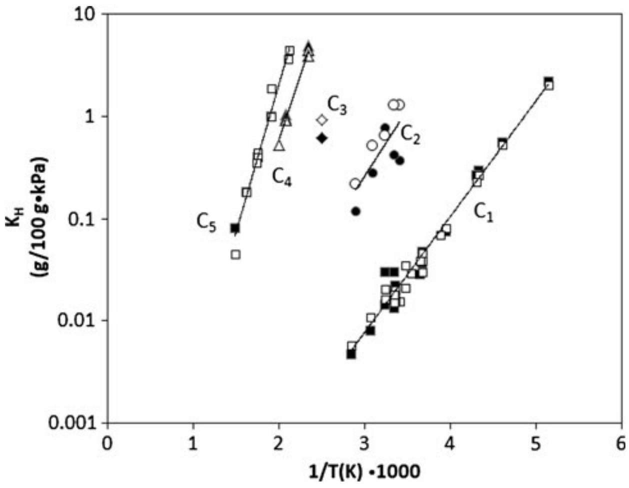

Fig. 5 Plot of ln K_H versus 1/T for the first five *n* alkanes, illustrating literature values (*full symbols*) and values calculated from the Gaussian model (*open symbols*). Lines are from a linear regression of all points for each alkane

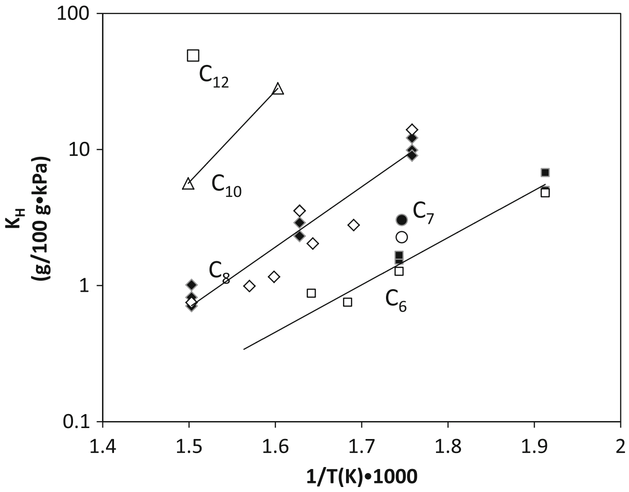

Fig. 6 Plot of ln K_H versus 1/T for C_6, C_7 C_8 C_{10} and C_{12}, illustrating literature values (*full symbols*) and values calculated from the Gaussian model (*open symbols*). Lines are from a linear regression of all points for each alkane

noted that the coefficients 2.28 for methane and 2.22 for all the alkanes are strongly consistent with each other.

Literature and calculated values of K_H are presented in Table 1 for all 63 isotherms. The first five columns contain the carbon number, the reduced temperature, the range of theta for the experimental data, σ, and P_{50} from the Gaussian fit of the isotherm. Published values of K_H have been abstracted from the literature and are Tabulated in column 6. Only those values which were reported as being calculated using the virial isotherm or multi-site Langmuir are included in the Table. Values calculated using the Langmuir isotherm are not included as they were observed to be inconsistent with the other two methods. K_H values calculated using Eqs. 14 and 17 are tabulated in column 7 (using a value of 1 for σ) and in column 8 (using the value of σ from the Gaussian fit of the isotherm). There is a remarkable agreement between the literature values and the calculated values. There is not a large difference between the K_H values calculated using the two values of σ.

A plot of ln K_H versus 1/T is given in Figs. 5 and 6. In these Figures the literature data are shown as full symbols, and values calculated from isotherms as open symbols. An excellent fit can be observed between $K_{H,lit}$ values, and the calculated values of K_H. In particular the lower *n* alkanes are observed to fit well. The higher alkanes such as n octane exhibit more scatter; this may be due to the differences between the heat of adsorption at zero loading, $(-\Delta H_0)$, as opposed to that at 50 % loading, $(-\Delta H_{50})$, or the difficulty of measuring the Henry constants for higher *n* alkanes.

4 Conclusions

Henry constants can be calculated from the Gaussian fit of an experimental isotherm, regardless of whether the experimental data extend into the Henry law region. The calculated values are in excellent agreement with reported experimental values. In addition, the Gaussian model has been shown (Loughlin and Abouelnasr 2011) to be in excellent agreement with Henry's law within much of the Henry law region.

Acknowledgments The authors wish to acknowledge the support of the American University of Sharjah, Sharjah during the course of this work.

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