

FILTER LIFE ESTIMATION METHODS APPLIED AT LOW ORGANIC CONTAMINANT CONCENTRATIONS

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Abstract

The goal of this work is to apply an estimation method to determine the life time and capacity for various activated carbons based filters. The Wheeler-Jonas (W-J) equation is used as a theoretical model to achieve this goal. Breakthrough measurements of several adsorbents, using both acetone and octane as probe molecules, were performed under dry and humid conditions using different detectors. The adsorption capacity of the different adsorbents were tracked instantaneously throughout the experiment by applying a simple modification to the breakthrough curve results. The experimental results from dynamic testing are compared to those obtained from the Wheeler-Jonas equation. Various activated carbons of different origins and activation methods were studied. The importance of this work, in addition to determining the lifetime and the capacity of activated carbon-based filters, lies in its ability to track the variation in capacity over time. The amount of contaminant adsorbed on each adsorbent is correlated with the chemical and structural features of each adsorbent's surface. Sorption of nitrogen and Boehm titrations were used to characterize the surface of the carbon samples.

Introduction

Most of the known volatile organic compounds (VOCs) are toxic to human health. It is of great importance to reduce their amounts in ambient air to safely acceptable levels. Activated carbons are the most versatile and frequently used adsorbents for such compounds due to their large surface area, high adsorptive capacity, commercial availability and low cost (Bansal et al, 1988; Gregg and Sing, 1982; Puri, 1970).

The breakthrough time in a filter is defined as the time at which the concentration of the compound penetrating the filter reaches an unacceptable level. For many years, contaminant breakthrough in air filters was detected based simply on the "warning" properties of the contaminant that the filter was designed to remove. These warning properties are usually based on the user's senses, such as taste, smell or skin irritation. This method is not reliable and accompanied by low accuracy in the prediction of filter breakthrough. This is due to the fact that the sensitivity toward the warning properties varies from one person to another. Moreover, many contaminants do not have any warning properties or are not intense enough to be sensed at low concentrations.

In the past there have been efforts made to estimate the service life of gas filters using semi-empirical models that are based on theoretical and experimental approaches. Those most commonly used are Langmuir, Dubinin-Radushkevich (D-R), Mecklenburg, Wheeler-Jonas and Yoon-Nelson equations (Do, 1998; Dubinin, 1989; Fletcher and Thomas, 1999; Jonas and Rehrmann, 1972; Mecklenburg, 1925; Mecklenburg, 1930; Lodewyckx and Vansant, 1999, 2000, 2000; Lodewyckx et al., 2004; Yoon and Nelson, 1984). Of these models, the Wheeler-Jonas equation is generally thought to be the most powerful. Many of the modern equations are complex and require the exact knowledge of several, not readily available parameters. Other models do not take into consideration the effect of some static or dynamic parameters on the adsorption process which will be reflected in an inaccurate estimation of filter life or filter breakthrough.

The preference of Wheeler-Jonas equation to predict the service life of air filters is attributed to several factors: its apparent simplicity, readily available parameters from literature or from a carbon manufacturer, it includes both capacity and the dynamic effects, and it yields accurate predictions of the filter breakthrough time. These factors strongly recommend its applicability to different adsorption

applications. The Wheeler–Jonas equation can be used to describe any type of adsorption of a single organic vapor by a suitable adsorbent. Its use has been extended to water-vapour coadsorption by Lodewyckx et al., when they applied the equation to predict the breakthrough times of organic vapors under humid conditions (Lodewyckx and Vansant, 1999, 2000 and 2000; Lodewyckx et al, 2004).

Several papers in the literature have studied the adsorption mechanism of VOC's such as acetone and octane on activated carbons; however, very few have discussed the capacity and the service life of activated carbons based filters when applied to the removal of such compounds from air stream under humid conditions (Cal et al, 1997 ; Díaz et al, 2004; Díaz et al, 2005; Economy et al, 1999 ; Fletcher and Thomas, 1999; Journet et al, 2005 ; Madey et al, 1988 ; Mangun et al, 1998 and 1999; Matsumoto et al, 1997). It is known that both porosity and surface chemistry are expected to play a role in the adsorption process of acetone and octane. The effect of surface chemistry is more pronounced in the case of acetone, especially under humid conditions. In spite of the preferable adsorption of water molecules on the surface polar sites of activated carbons, their presence in the adsorbed form on the surface is expected to enhance acetone adsorption due to the high solubility of acetone in water (Bansal et al, 1988; Cal et al, 1997 ; El-Sayed et al, 2006; Gregg and Sing, 1982) .

The goal of this work is to apply an estimation method to determine the life time and capacity for various activated carbons based filters, especially when exposed to low or even very low concentrations of organic vapours. The effect of porosity and surface chemistry of activated carbons on the adsorption of low concentrations of acetone and octane from a humid air stream will also be studied. Additionally, this work focuses on both the adsorption capacity and adsorption kinetics of these organics on activated carbon. The analysis is based on obtaining breakthrough curves on packed beds of activated carbons under different experimental conditions. The effect of various chemical, structural and experimental parameters on the adsorption rate and capacity will be investigated. The properties considered include: gas/ vapor properties, carbon properties, bed geometry, relative humidity (RH), temperature, airflow rate, and vapor concentrations. This work is part of a project to develop improved equations for estimating filters service times that take into consideration the different parameters mentioned above.

Theoretical

The Wheeler–Jonas equation Eq. (1) is a well-known predictive equation to estimate the breakthrough times of physisorbed organic vapors on activated carbon beds (Bansal et al, 1988; Gregg and Sing, 1982; Jonas and Rehrmann, 1973; Smoot, 1977). The prediction is based solely on the measurable and readily available macroscopic parameters:

$$t_b = \frac{M.W_e}{Q.C_{in}} - \frac{W_e.\rho_b}{k_v.C_{in}} \ln \left[\frac{C_{in} - C_{out}}{C_{out}} \right] \quad (1)$$

where t_b is the breakthrough time to reach C_{out} (min), C_{in} is the vapor inlet concentration in air (g/cm^3), C_{out} is the chosen breakthrough concentration (g/cm^3), M is the weight of the carbon bed (g), W_e is the equilibrium adsorption capacity ($\text{g}/\text{gcarbon}$), Q is the volumetric flow rate (cm^3/min), ρ_b is the bulk density of the carbon bed ($\text{gcarbon}/\text{cm}^3$) and k_v is the overall adsorption rate coefficient (min^{-1}). Two unknown parameters in this equation need to be calculated which are W_e and k_v . In the past several models were derived which allow the calculations of these unknown parameters without any prior breakthrough experiment (Jonas, and Rehrmann, 1974; Jonas et al, 1979; Lodewyckx and Vansant, 2000; Lodewyckx et al., 2004; Wood and Stampfer, 1993). W_e is normally estimated from the Dubinin–Radushkevich given in equation 2.

$$W_e = W_o d_L \exp \left[\frac{-BT^2}{\beta^2} \log^2 \left(\frac{C_s}{C_{in}} \right) \right] \quad (2)$$

W_e is the static adsorption capacity ($\text{g}/\text{gcarbon}$), W_o is the micropore volume (cm^3/g), d_L is the liquid density (g/cm^3), B is a structural constant of the carbon, β - the affinity coefficient of the organic vapor, T is the adsorption temperature (K), C_s is the saturation vapor concentration (ppmv), C_{in} is the vapor inlet concentration (ppmv). The only unknown parameters in this equation are W_o and B , which can be derived from nitrogen adsorption isotherms at 77 K.

Calculation of the overall adsorption rate coefficient k_v is more complex. For organic compounds, k_v is mainly linked to the effects of surface diffusion. The most recent and complete equation to calculate the

value of k_v is by using the following semi-empirical equation

$$K_v = 800 \beta^{0.33} v_L^{0.75} d_p^{-1.5} \sqrt{\frac{W_e}{M_w}} \quad (3)$$

M_w is the molecular weight of the vapor (g/mole), d_p is the average diameter of the carbon particle (cm), and v_L is the linear velocity through the bed (cm/s).

Experimentally, different breakthrough times can be obtained by varying the weight of the carbon bed. A plot of breakthrough time versus carbon bed weight should yield a straight line that allows calculating W_e and k_v using the slope and the intercept. Usually the W-J model is used for medium to high inlet concentrations (500 to 10,000 ppmv) of organic vapour. One of the goals of this work is to evaluate its applicability to low concentrations (< 100 ppmv).

Experimental

1. Materials

Seven activated carbon samples of different origins and methods of activation were selected for this study. They are named D, F, H, I, K, L and N. These materials were chosen based on their diversity in surface chemical and structural properties.

2. Study of Surface Chemistry of Carbon

2.1 Boehm Titration

One gram of carbon sample was added to each of the 50 ml of 0.05N solutions: sodium hydroxide, sodium carbonate, sodium bicarbonate and hydrochloric acid. The vials were sealed and shaken for 24 h. The samples were filtered and then 10 mL of each filtrate was pipetted out and the excess of base or acid was titrated with 0.1N HCl or NaOH, respectively. The number of acidic sites of various types were calculated under the assumption that NaOH neutralizes carboxyl, phenolic, and lactonic groups; Na_2CO_3 carboxyl and lactonic; and NaHCO_3 only carboxyl groups (Boehm, 1966). The total number of acidic and basic groups is calculated based on the amounts of sodium hydroxide and hydrochloric acid reacted.

2.2 pH Measurement

0.4 g of dry carbon powder was added to 20 ml of water and the suspension was stirred overnight to reach equilibrium. Then the samples were filtered and the pH of the filtrates was measured.

3. Characterization of the Pore Structure

3.1 Sorption of Nitrogen

An ASAP 2010 (Micromeritics, Norcross, GA, USA) was used to measure nitrogen adsorption isotherms at 77 K. Before the experiment, the samples were heated at 333 K and outgassed under a vacuum to constant pressure of 10^{-5} torr. Characterization of pore sizes and structure were accomplished using Density Functional Theory (DFT) [31]. Using DFT, specific surface areas, total pore volume, micropore volume, volume of pores smaller than 10\AA ($V_{<10\text{\AA}}$) were calculated (Lastokie et al, 1993). Moreover, the surface areas, S_{BET} , were also calculated using the BET method.

4. Break through Measurements

4.1 Adsorption of Acetone and octane

Acetone breakthrough tests were carried out under dynamic conditions on all carbons at low concentration using packed beds, 1.5 inches in diameter (Dallas et al, 2005). The service life of activated carbon filters was predicted from the adsorbate breakthrough time. The experiments were carried out at room temperature (25°C) with an acetone inlet concentration of 50 ppmv, and a 10% breakthrough (10% t_b) concentration of 5 ppmv was chosen for material performance comparisons. Three different weights of each activated carbon were used to calculate W_e and k_v from the Wheeler-Jonas equation. Sample weights were in the range between 10-40g. The air flow was adjusted to 30 L/min with a relative humidity of 50% at 25 °C. For comparison purposes, octane breakthrough measurements were done on carbon F under the same experimental conditions as acetone. FID detector was used to monitor acetone and octane concentrations.

The influence of water on acetone and octane uptake was studied by measuring their breakthrough tests on carbon F at 0% and 50 % RH while keeping all other experimental conditions constant.

The effect of inlet concentration of acetone on activated carbons capacity was studied under the same experimental conditions described above but varying the inlet concentration. The inlet concentrations of acetone used were: 250ppmv, 135 ppmv, 95 ppmv, 50 ppmv, and 25 ppmv.

To study the effect of temperature on activated carbon capacity, breakthrough measurements for acetone and octane on carbon F were done at three different temperatures for acetone (26°C , 30°C and 35.5°C) and two temperatures (26 °C and 35.5°C) for octane. Acetone and octane inlet concentrations were adjusted to 50 ppmv while keeping all other experimental conditions constant.

Carbon F is a standard carbon for the removal of acetone and octane. This explains the reason for its selection to study the effect of various experimental parameters on the adsorption process

4.2 Capacity Curves

From the breakthrough curves, the capacity of all activated carbons for acetone can be calculated using the following equation:

$$q = \sum_{i=1}^t \frac{(C_s - C_o) * F}{m} \quad (4)$$

Where q is the capacity of activated carbons (g acetone/g of adsorbent), C_s is the outlet concentration (g/L) at the end of breakthrough curve, C_o the outlet concentration (g/L) at time i , F is the flow rate (L/min) and m is the mass (g) of the activated carbon sample in the packed bed. When the outlet concentration reaches the challenge concentration (C_s), the total capacity can be calculated and is referred to as W_e . At this point W_e is equal to q.

Results and Discussion

Understanding the surface chemistry and pore structure of various activated carbon adsorbents is very important in elucidating the mechanisms involved in the removal of small molecules such as acetone and octane in humid conditions. The structural parameters such as surface area (S_{BET}), the volume of pores smaller than 10 Å ($V_{<10\text{\AA}}$), micropore volume (V_{mic}) and the total pore volume (V_t) were calculated from the nitrogen isotherms for all activated carbons. The results are summarized in Table 1. It is clearly shown that carbon F is mainly microporous whereas carbon L has significant contribution of mesopores. Carbon D has the highest surface area, which is due in part to it also having the largest pore volume on this carbon.

Table 1. Structural Parameters Calculated from Nitrogen Adsorption at 77 K.

Sample	$V_{<10\text{\AA}}$ (cm ³ /g)	V_{mic} (cm ³ /g)	V_t (cm ³ /g)	S_{BET} (m ² /g)
D	0.2622	0.5212	0.6423	1908
F	0.261	0.3547	0.3713	1005
H	0.197	0.2666	0.396	761
I	0.2632	0.3656	0.3832	1064
K	0.2069	0.2790	0.3219	747
L	0.2650	0.3261	0.542	879
N	0.2665	0.4509	0.5226	1280

Table 2 presents the results obtained from Boehm titrations along with the pH values of the carbon surfaces. The data reported indicates that the initial carbons differ in their acidity. This is expected due to the differences in the organic precursor and the activation methods. It is clearly seen that the carbon samples differ in the total number of groups and their distribution which result in different values of the surface pH. The high pH value for carbons D, I, L and N indicate that their surfaces are basic. This is

supported by the absence of the strong carboxylic acid groups and by the relatively large number of basic groups on the surface of those carbons. Although F, H and K carbons have similar average neutral surface pH values and a similar total number of groups, the distribution of the surface groups into specific categories is different. The presence of more carboxylic and phenolic groups on the surfaces of carbons H and K results in their having slightly lower surface pH values.

Table 2. Results of Boehm Titration (mmol/g).

Sample	pH	Carboxylic	Lactonic	Phenolic	Acidic	Basic
D	9.61	0.0000	0.0000	0.1254	0.1254	0.8632
F	7.56	0.0250	0.0754	0.1504	0.2508	0.3813
H	7.29	0.1003	0.0252	0.2759	0.4014	0.3813
I	9.44	0.0000	0.0000	0.2006	0.2006	0.5338
K	7.12	0.1254	0.0252	0.3013	0.4516	0.3287
L	8.56	0.0000	0.0000	0.2006	0.2006	0.5836
N	10.38	0.0000	0.0000	0.2560	0.2560	0.7130

Acetone breakthrough curves were measured on three different weights of each of the carbon samples at 50% RH. For comparison purposes, breakthrough curves measured on 15g of each samples are presented in Figure 1. Since the service life of a filter depends on the breakthrough time, the 10% t_b was chosen as an indicator to check the performance of various activated carbons in removing acetone. The results indicate that carbon F has the highest efficiency for removing acetone followed by carbons I and L respectively. Carbon D showed the shortest breakthrough time.

In order to compare the capacities of the various activated carbons for acetone, the breakthrough curves from Figure 1 were converted into capacity curves shown in Figure 2. In addition to providing the total capacity, the curves shown in figure 2 provide simple approach to tracking the available capacity on carbons at different time intervals.

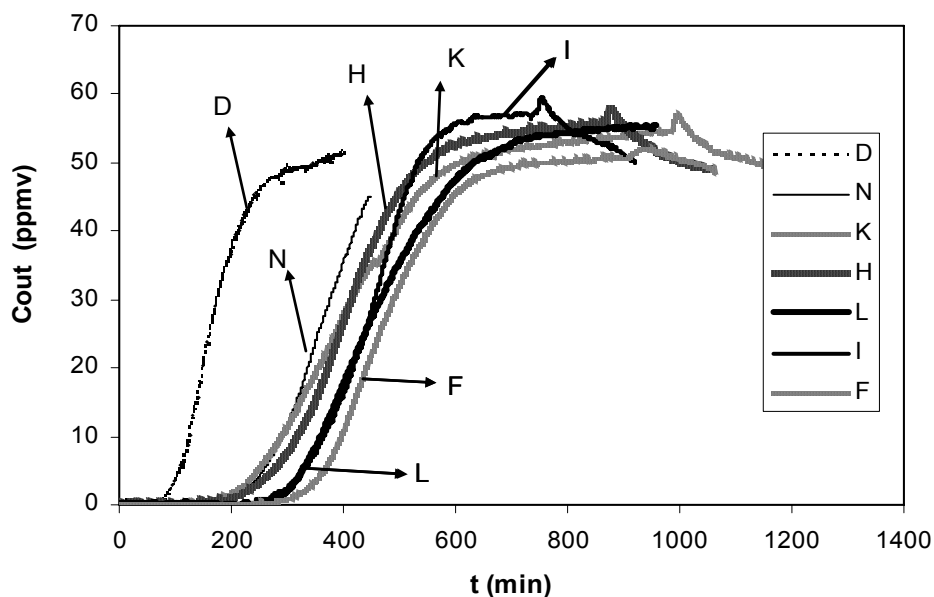


Figure 1. Acetone breakthrough curves on 15g activated carbon packed beds samples at 50%RH, 30LPM and 25C.

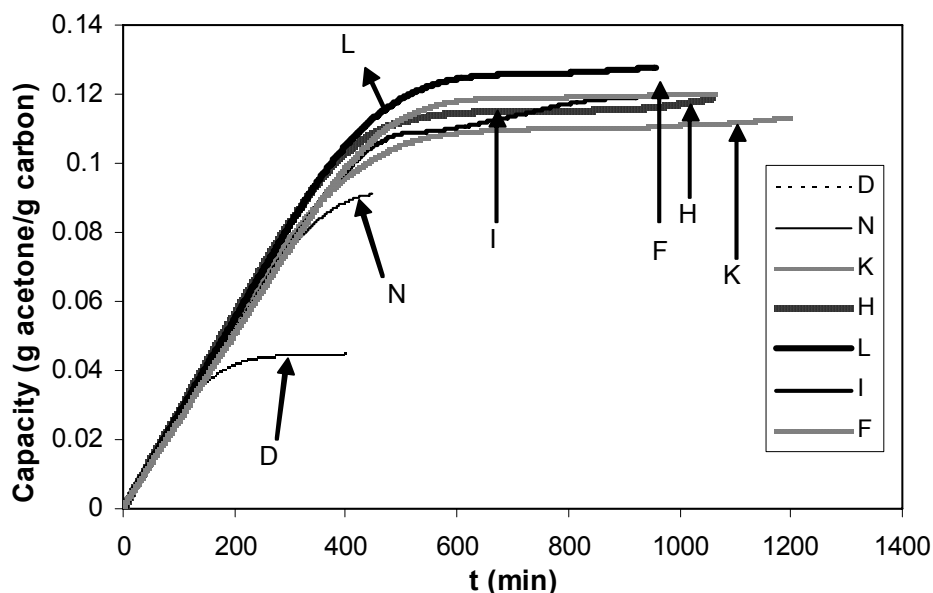


Figure 2. Acetone capacity curves calculated from breakthrough curves on Figure 1 for activated carbon samples.

Figure 2 shows similar trends to the ones observed in Figure 1. Carbons F and L have the highest capacities at full breakthrough. If dispersive interactions were the only forces controlling the adsorption process, the capacity should depend on the volume of micro or super-micro pores. No direct correlation was found between the capacities shown in Figure 2 and the micropore volumes. The adsorption mechanism of acetone on activated carbons is more complex and involves several other types of interactions than dispersive. Support for this is the low capacity for carbon D in spite of its high micropore volume. Therefore, surface chemistry plays a role in the adsorption process of acetone on activated carbons by promoting specific interactions.

Table 3. Experimental (W-J) and calculated values of W_e and K_v for all activated carbons

Sample	Wheeler-Jonas		Calculated	
	W_e	K_v	W_e	K_v
D	0.0621	3202	NA	NA
F	0.1004	14357	0.088	15282
H	0.0754	27683	0.075	14190
I	0.1143	9090	0.104	16680
K	0.0906	8339	0.070	10358
L	0.1065	8011	0.091	15538
N	0.0906	6637	0.092	15658

A plot between the 10% t_b and carbon weight for each carbon was performed. Using the intercept and the slope of the lines connecting the points for each carbon, one can calculate the parameters W_e and K_v of the Wheeler-Jonas equation. Moreover, the values of these parameters were also calculated using equations 2 and 3. The results are grouped in Table 3. It is clear that Carbon I has the highest capacity for acetone followed by L and F while carbon D has the lowest. Although the values presented in Table 3 slightly differ

from the trend observed in Figure 2, they are of the same order of magnitude. It is important to mention that the Wheeler-Jonas equation involve the use of at least three points to calculate W_e which will result in more precise values than those present in Figure 2 (one point calculation). Comparison between the experimental and calculated W_e and K_v and their impact on the calculated service life will be discussed at the end of this paper.

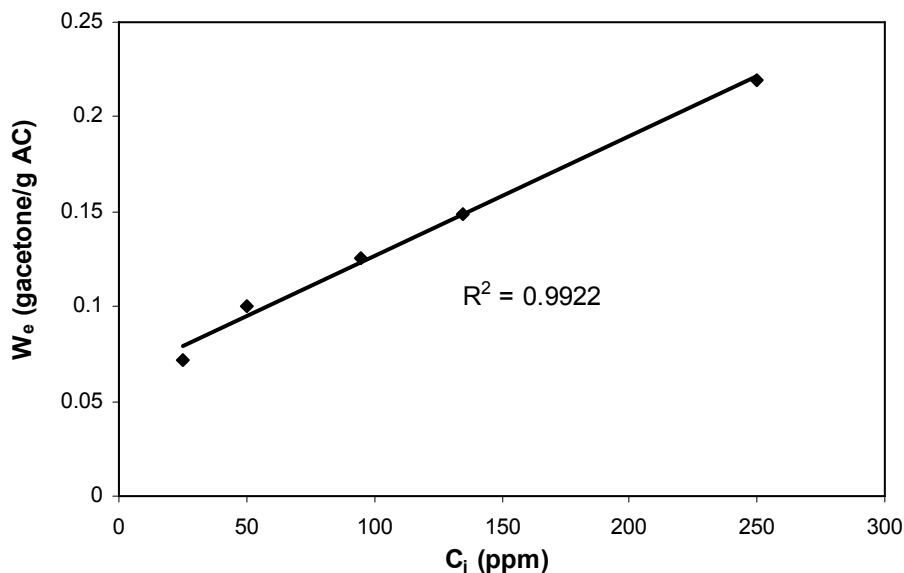


Figure 3. Effect of acetone inlet concentration on the capacity of activated carbon.

The effect of acetone inlet concentration (250, 135, 95, 50, and 25 ppmv) was also studied at 50%RH, 26 °C, and flow rate of 30LPM. Three different weights of carbon F were used at each inlet concentration. The capacity of Carbon F was calculated using Wheeler-Jonas equation. The results are shown in Figure 3. It is clearly seen that the capacity of Carbon F for acetone decreases linearly with the decrease in inlet concentration. There seems to be a change in the slope at a very low concentration (25 ppmv). However, this conclusion has to be treated carefully since at these very low concentrations the experimental errors can have a significant impact on the measurements.

Comparisons of the capacities of Carbon F for acetone and octane along with the effect of temperature and humidity are summarized in Table 4. It is clearly seen that Carbon F has much higher capacity for octane than acetone. The capacity of Carbon F for octane noticeably decreases with 50 % humidity. In contrast, no significant effect on the capacity for acetone is seen. It is important to mention that acetone is highly soluble in water. Although water in the air stream is expected to compete with acetone and octane for the polar sites on the surface and will be preferably adsorbed, acetone can be adsorbed by specific interaction with adsorbed water on the surface or by simply dissolving in the adsorbed water layer. It is clear that temperature has great impact on the adsorption process of both compounds. The results show that the Carbon F capacity for acetone decreases by about 0.3% by weight with every 1°C increase in temperature. The effect is even more pronounced in case of octane (~0.9%).

Comparison of the experimental W_e and t_b for acetone with the calculated values using equations 2 and 3 under the variable experimental conditions discussed before was performed (see Figures 4 and 5). Figures 4 and 5 show that good linear relationships exist for all samples, even when considering t_b . This indicates that the model succeeds in estimating the breakthrough times for all carbon samples under different experimental conditions. It is important to mention that the data showing the temperature effect were excluded from the plots since the calculated values showed deviation up to 44%. Such deviation between experimental and calculated results for t_b and W_e is expected, knowing that, as yet, the impact of temperature effects is not included in the model.

Table 4. Effect of temperature and humidity on acetone and octane adsorption on carbon F.

Contaminant	RH (%)	T (0C)	M (g)	10% t _b (min)	W _e (g/g)	K _v
Acetone	0	26	15	294	0.0993	10399
	0	26	30	692		
	0	26	45	1060		
	50	26	15	325	0.1004	14359
		26	30	730		
		26	45	1068		
	50	30	15	259	0.0889	9579
		30	30	609		
		30	45	945		
	50	35.5	15	202	0.0716	8211
		35.5	30	470		
		35.5	45	754		
Octane	50	26	10	471	0.4114	25548
	50	26	15	743		
	0	26	10	511	0.5347	12927
		26	15	890		
		26	20	1218		
	0	35	10	478	0.4495	16947
		35	15	766		
		35	30	1664		

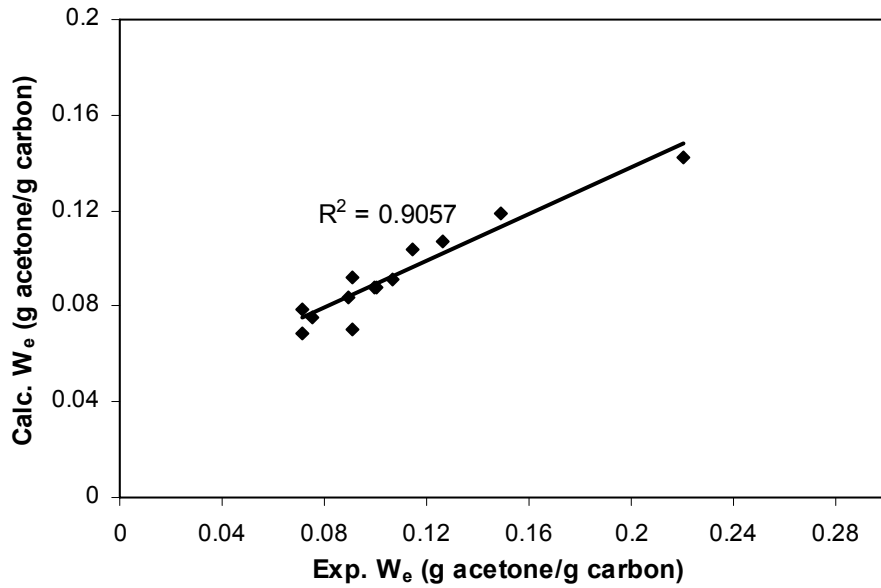


Figure 4. Calculated vs. Experimental W_e (g/g).

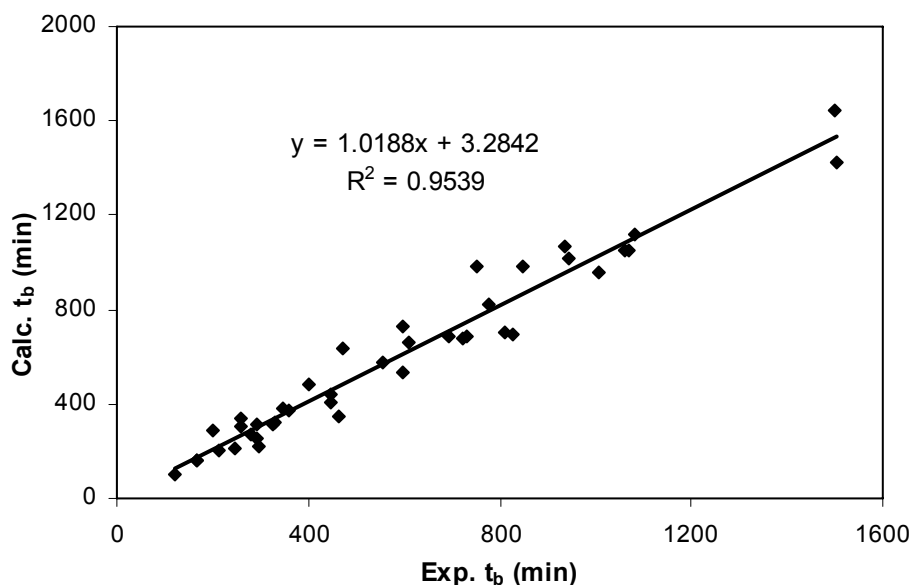


Figure 5. Calculated vs. Experimental t_b (min)

Conclusions

The models used for estimating the overall adsorption rate coefficients and adsorption capacity of the Modified Wheeler-Jonas equation showed great success. The models require only readily available input parameters (particle size, affinity coefficient of the vapour and linear velocity of the air stream). Accurate predictions for the breakthrough time of acetone on activated carbon filters were obtained. This demonstrates the applicability of this model to low inlet concentrations (< 100 ppmv). The least accurate predictions were found at elevated temperatures. Further modifications to the models are required to include the effect of temperature. Temperature showed large impact on acetone and octane adsorption. Humidity mainly decreased octane but not acetone. Acetone adsorbed via dispersive and specific interactions and by dissolving through the water adsorbed on activated carbons.

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