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Sulphur mustard vapor breakthrough behaviour on reactive carbon systems

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Abstract

Breakthrough behaviour of sulphur mustard, the deadliest of persistent chemical warfare agents, on carbon systems such as NaOH/CrO_3/C, NaOH/CrO_3/EDA/C and $RuCl_3$/C has been studied and the data were compared with that of active carbon. Effects of bed lengths of carbons on breakthrough time have also been correlated. Thereafter, the effects of flow rate of air–sulphur mustard mixture, concentration and temperature on the kinetic parameters such as rate constant (k_v) and kinetic saturation capacity (W_e) were analyzed and interpreted by means of modified Wheeler equation. Rate constant was found to be increasing while W_e was found to be invariable with the increase in air flow rate. Both k_v and W_e decreased with the increase of temperature, however, no significant effect on W_e and k_v was observed due to concentration change (0.3–0.6 mg/l). The values of kinetic saturation capacity were used to predict the service lives/breakthrough times of carbon beds (when used in filtration systems).
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Keywords: Active carbon; Sulphur mustard; Breakthrough time; Modified Wheeler equation

1. Introduction

Vapor breakthrough behaviour of carbon beds challenged with contaminated air streams is of great interest in connection with respiratory protection against hazardous vapors. Carbon beds exhibit relatively better adsorptive properties than the other adsorbents such as silica, etc., and make them indispensable for use in respiratory cartridges [1–3]. ASC whetlerite, i.e., active carbon impregnated with salts based on Cu(II), Cr(VI) and Ag(I) ions is one such reactive carbon which has been used in respiratory cartridges for the protection against non-persistent (high volatile and low boiling) chemical warfare agents such as hydrogen cyanide, phosgene and cyanogens chloride [4–7]. Air contaminated by these agents get chemisorbed and get detoxified on the surface of above carbon. However, persistent (high boiling and low volatile) chemical warfare agents are mainly removed by physical adsorption. In such a case, active carbon is suffice enough for the physisorption of the persistent CW agents such as sulphur mustard and nerve agents.

But there are enough possibilities for cross-contamination owing to desorption of the contaminants from the filtration systems when they are disposed off after their use without proper care. In order to avoid this problem, the carbons were modified by impregnation and this imparts chemical reactivity to the active carbon in addition to the physical adsorption potential. Active carbon impregnated with polyoxometallates is one such reactive carbon which was prepared by G.R. Damico et al. [8] and was used for the reactive removal of a thio ether, a simulant of sulphur mustard. Another variant, i.e., active carbon impregnated with NaOH and CrO_3 was successfully used by Prasad et al. [9–11] for the degradation of thiodiglycol, a hydrolysis product of sulphur mustard and then with diethyl sulphide, an analogue of sulphur mustard. Later, they prepared the reactive carbon systems based on NaOH and CrO_3 and EDA, $Mg(NO_3)_2$, $RuCl_3$ and they were proven to be the potential materials for the degradation of sulphur mustard [12,13]. This kind of reactive carbon beds can be utilized in NBC filtration systems for the reactive removal of persistent chemical warfare agents.

The performance of the carbon bed or the service life of NBC filtration system depends upon the adsorption capacity of the adsorbent and the rate of adsorption of the contaminants or adsorption kinetics at breakthrough time. Many equations have

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been derived to correlate the concentration of gas sent into the carbon bed with that of the quantity of gas adsorbed and breakthrough time in order to understand the adsorption processes and to predict the service life of carbon bed [3–5]. Wheeler [14] has proposed a continuity equation which correlates the mass of vapor entered the carbon bed and the mass adsorbed and penetrated it to understand the adsorption kinetics and to predict the service life of carbon bed accurately. Consequently, Jonas et al. have utilized the modified Wheeler equation to predict the service lives of carbon beds accurately and it is [15–21] given below:

$$t_b = \left(\frac{W_e}{C_0 Q}\right)\left[W - \frac{\rho_b Q}{k_v \ln(C_0/C_x)}\right]$$

where t_b is the breakthrough time (min), C_x the exit concentration (g/ml), C_0 the initial concentration (g/ml), Q the volumetric flow rate (liter per minute, lpm), W the weight of adsorbent (g), ρ_b the bulk density of carbon bed (g/ml), W_e the kinetic saturation capacity (g/g) and k_v is the rate constant (min^{-1}).

The values of C_0, W and Q are established by the experimental test conditions. The value of ρ_b depends upon the weight, particle size and shape of the adsorbent and it can be determined experimentally while C_x/C_0 is preselected. In addition to this, the quantity of gas adsorbed under dynamic conditions depends on parameters such as temperature, concentration, bed geometry, flow rate of gas mixture, particle size and reactivity on the adsorbents surface [6]. Of these, flow rate of gas mixture is observed to be one of the important parameters which affect the rate of adsorption prominently. The rate of adsorption increases proportionally with the increase of flow rates of air–gas mixture and these observations are attributed to diffusion of vapor molecules and the same was found to be the rate limiting process. In order to understand this and the effect of above mentioned parameters on adsorption kinetics and service lives, we have attempted to study the breakthrough behaviour of HD vapors on activated carbon, $NaOH/CrO_3/C$, $NaOH/CrO_3/EDA/C$ and $RuCl_3/C$ to determine the adsorption parameters such as kinetic saturation capacity (W_e), and kinetic rate constant (k_v), and service life of the carbon bed for sulphur mustard on the above carbons by using modified Wheeler equation and also to find out the effect of various parameters such as flow rate, concentration and temperature on W_e and k_v.

2. Experimental

2.1. Materials

Active carbon of surface area 1250 m^2/g of coconut shell origin, with a particle size of 12×30 of British sieve size was procured from Active Carbon India Ltd., Hyderabad. NaOH, $RuCl_3 \cdot 3H_2O$, CrO_3 and ethylene diamine (EDA) were obtained from E Merck, India Pvt. Ltd. Carbon tetrachloride (CCl_4) (99.5% purity), XAD-2 were obtained from Lancaster, England and the use of XAD-2 resin is described in Section 2.3.

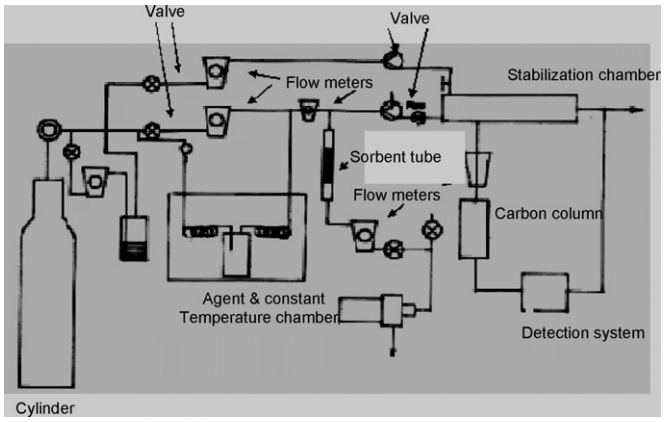

Fig. 1. Gas generation test rig for vapor breakthrough experiments on carbons.

2.2. Characterization

All the carbons were characterized for surface area (N_2 BET) using Gemini 2765 surface area analyzer (Micrometrics, USA).

2.3. Chemicals

Distilled sulphur mustard (2, 2′-dichlorodiethyl sulphide) (>99% GC) (HD) was obtained from Process Technology Development division of our establishment. Carbon tetrachloride (CCl_4) (99.5% purity), XAD-2 were obtained from Lancaster Chemical Company (England).

2.4. Sulphur mustard gas mixture generation

The vapor breakthrough experiments were carried out with a gas generation assembly fabricated by Nucon Engineering (India). Fig. 1 shows the outline diagram of gas generation assembly. HD vapors were generated by using a slightly modified dynamic diffusion system (for low concentration) and purge method (for high concentration) [22]. Moisture free air was used for purging and dilution. Precautions were taken to avoid condensation by putting the heat tape around the gas lining. Concentration of gas mixture was measured by standard reported method, using XAD-2 resin [22,23]. Gas mixture stream with a constant flow rate was drawn through the adsorbent tubes in series containing XAD-2 resin in order to facilitate the adsorption of HD for a particular interval of time. Thereafter, the adsorbed HD was extracted and analyzed by using GC-FID (BP1 column of 30 ft length, 0.33 mm i.d. at isothermal conditions). Under the same situation carbon columns challenged with the HD–air mixture have been examined for breakthrough behaviour and the same procedure has been repeated several times in order to check the reproducibility.

2.5. Vapor breakthrough experiments of HD

Vapor breakthrough experiments of HD were carried out in a column of 1.0 cm diameter using different bed heights of carbons, flow rates and concentrations of HD–air mixture and temperature. The temperature of the carbon was main-

tained by circulating water around the column. The HD vapor breakthrough time on the carbon bed was monitored with an instrument named AP2C containing flame photometric detector (GIAT Industries, France). For this purpose, the gas mixture coming out of carbon column was continuously monitored for the presence of HD. Once the HD concentration in the gas mixture stream coming out of carbon column reaches 0.47 mg/m^3, the AP2C gives a signal (minimum detection limit) indicating the breakthrough.

2.6. Precautions to avoid exposure

Sulphur mustard (HD) is a well-known chemical warfare agent. It effects men and materials and has been documented as mutagenic, carcinogenic and cytotoxic [24]. Skin, eyes and respiratory track are the principle target organs and DNA is the most important molecular target of HD toxicity. Experimental set-up was established in fume hood of high exhaust capacity with scrubber attached. The exhaust air was passed through big carbon filters before sending the exhaust gas to scrubber. Individual protection was taken by wearing NBC suit, gloves, gas mask with a respiratory cartridge, etc. Working environment was checked for every 5 min by chemical agent monitor (AP2C).

3. Results and discussion

Experimental values of breakthrough time (t_b) as a function of bed height for sulphur mustard vapors were measured for active carbon, NaOH/CrO_3/C, NaOH/CrO_3/EDA/C and $RuCl_3$/C systems. Fig. 2 shows the plots of breakthrough time versus bed height for active carbon, NaOH/CrO_3/C, NaOH/CrO_3/EDA/C and $RuCl_3$/C systems at 0.5 mg/l HD concentration and 1.0 lpm flow rate. In the case of active carbon, breakthrough time increased from 8 to 58 min when bed length increased from 0.8 to 1.1 cm. Whereas, in NaOH/CrO_3/C system, breakthrough time increased from 1 to 21 min when bed height increased from 1.05

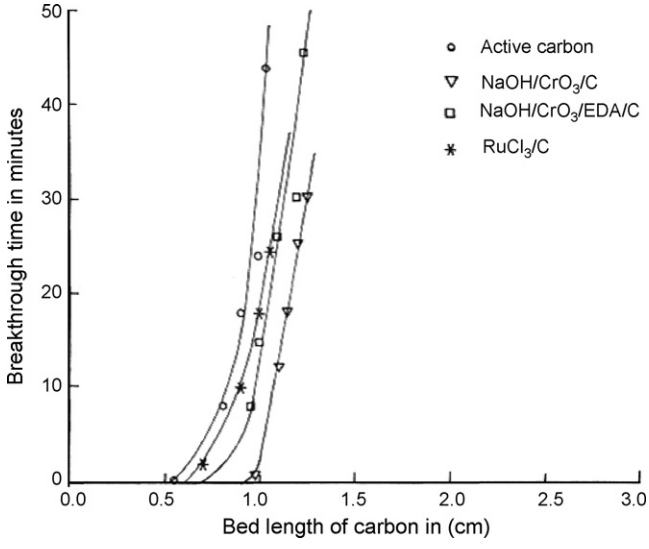

Fig. 2. Effect of bed length/bed height of carbons on sulphur mustard breakthrough time at 0.5 mg/l concentration, 1.0 lpm flow rate and 25 °C.

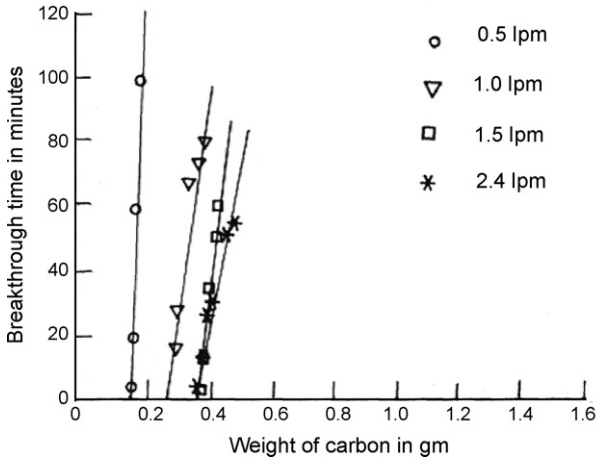

Fig. 3. Sulphur mustard breakthrough time as a function of carbon weight (NaOH/CrO_3/C) at various flow rates, 0.5 mg/l concentration and 25 °C temperature.

to 1.25 cm. And for NaOH/CrO_3/EDA/C system breakthrough time increased from 8 to 44 min when bed height increased from 0.95 to 1.25 cm. $RuCl_3$/C also behaved in a similar manner. In addition to this, at lower bed heights HD breaks through the carbon beds in seconds thus exhibiting insignificant breakthrough time values. This observation clearly indicates that an optimum bed height is needed for getting meaningful breakthrough time values. Initially, the breakthrough time increases non-linearly with the bed height and then after an optimum height it shows a linear increase with the carbon bed height as depicted in the above plots and the same is consistent with the previous reported results by Shilov et al. [25] and Mecklenburg and Kubelka [26]. By using the linear portions of the curves it is possible to predict the breakthrough time of carbon bed or service life of the respirator cartridge containing the same.

In order to study the effect of flow rate on kinetic saturation capacity (W_e) and rate constant (k_v), t_b values were obtained from the breakthrough experiments for different carbon weights using different flow rates ranging from 0.5 to 2.0 lpm for active carbon at 0.250 mg/l (HD concentration) and 25 °C and from 0.5 to 2.4 lpm at 0.5 mg/l and 25 °C for NaOH/CrO_3/C, NaOH/CrO_3/EDA/C and $RuCl_3$/C. Fig. 3 shows the plot of breakthrough time versus carbon weight at various flow rates for NaOH/CrO_3/C. Including this plot, in all the cases of active carbon, NaOH/CrO_3/EDA/C and $RuCl_3$/C, plots of breakthrough time versus carbon weight at various flow rates have given a straight line indicating the adsorption process to be the pseudo first order reaction. The values of kinetic saturation capacity (W_e) were calculated from slope, i.e., (W_e/C_0Q) and rate constant by intercept, i.e., $[\rho_b Q/k_v \ln(C_0/C_x)]$ and the obtained results are furnished in Table 1. In the case of NaOH/CrO_3/C, when the flow rate is increased from 0.5 to 2.4 lpm rate constant increased non-linearly from 7891 to 22,009 min^{-1}, whereas, kinetic saturation capacity (W_e) is found to be invariable. Active carbon, NaOH/CrO_3/EDA/C and $RuCl_3$/C also behaved in a similar manner. Rate constants increased from 12,291 to 19,372 min^{-1}, 8060 to 25,900 min^{-1} and 5808 to 22,009 min^{-1}, respectively. The values of kinetic saturation capacity do not change signifi-

Table 1
Sulphur mustard adsorption capacity and rate constant at various flow rates on carbons at 0.250 mg/l (for active carbon), 0.500 mg/l (other carbons) and 25 °C

Flow rate (lpm)	Active carbon		Flow rate (lpm)	$NaOH/CrO_3/C$		$NaOH/CrO_3/EDA/C$		$RuCl_3/C$	
	W_e (g/g)	k_v (min^{-1})		W_e (g/g)	k_v (min^{-1})	W_e (g/g)	k_v (min^{-1})	W_e (g/g)	k_v (min^{-1})
0.5	0.3	12,291	0.5	0.3	7,891	0.3	8,060	0.3	5,808
1.0	0.3	17,410	1.0	0.3	8,567	0.3	8,953	0.3	9,114
1.5	0.3	18,919	1.5	0.3	13,974	0.3	18,863	0.3	15,840
2.0	0.3	19,372	2.4	0.3	22,009	0.3	25,900	0.3	22,009

cantly. This observation indicates that rate limiting process is the diffusion of vapor molecules on the surface of carbon granules. Most likely, the external diffusion (bulk), internal (inter particle) diffusion and surface diffusion can collectively be manifested as the rate limiting process. Moreover, the value of kinetic saturation capacity was found to be 0.3 g/g for all the carbons at various flow rates from 0.5 to 2.0 lpm (for active carbon) and 0.5 to 2.4 lpm for all other carbons. Though it does not change significantly, it clearly indicates that at all the flow rates ranging from 0.5 to 2.4 lpm, the carbon bed can hold/retain 0.3 g of HD from the contaminated vapor. From this data, it can be calculated that the carbon bed provides protection for 600 min against 0.5 mg/l of HD at 1.0 lpm flow rate and 25 °C temperature. Thus, the above kinetics saturation capacity value is used to predict the service life of carbon bed against HD.

On the other hand, a few measurements with vapor concentration of 0.3, 0.5 and 0.6 mg/l were also made. Rate constant and kinetic saturation capacity for HD were computed by making use of the modified Wheeler equation [26]. Kinetic saturation capacity (W_e) values of HD on $NaOH/CrO_3/C$ system (Fig. 4) for concentration 0.3–0.6 mg/l were obtained as 0.3 g/g to 0.3 g/g and rate constant values varied from 9083 to 9122 min^{-1}. Active carbon, $NaOH/CrO_3/EDA/C$ and $RuCl_3/C$ also behave in a similar manner and the results are shown in Table 2. However, small variations in k_v values may be due to small changes in flow rates. These observations indicate that the concentration variation within 0.3–0.6 mg/l has neither affected kinetic saturation capacity value nor kinetic rate constant value significantly. Hence, it is possible to predict the breakthrough time/service life of the beds of above carbons at the above concentrations by using the kinetics saturation capacity values. The kinetic saturation capacity value is found to be 0.3 g/g for all the carbons at 1.0 lpm flow rate and at concentrations ranging from 0.3 to 0.6 mg/l. From these data, it is observed that the carbon bed (for all carbons) provides protection for 1000 min against 0.3 mg/l of HD, for 500 min against 0.6 mg/l of HD at 1.0 lpm flow rate and 25 °C temperature. Thus, the above kinetic saturation capacity

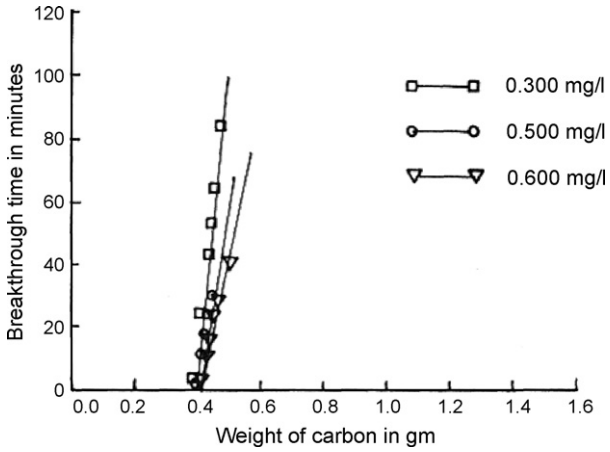

Fig. 4. Sulphur mustard breakthrough time as a function of carbon weight ($NaOH/CrO_3/C$) at various concentrations, 1.0 lpm flow rate and 25 °C temperature.

value obtained from the plots is used to predict the service life of carbon beds against various concentrations.

Moreover, the effect of temperature on W_e and k_v was also studied by varying temperature from 35 to 55 °C and the flow rate was 1.0 lpm and the HD concentration maintained was 0.5 mg/l. Breakthrough time as a function of carbon weight was plotted for active carbon, $NaOH/CrO_3/C$, $NaOH/CrO_3/EDA/C$ and $RuCl_3/C$ systems for various temperatures and the same for active carbon is shown in Fig. 5. The kinetic saturation capacity values and rate constant values for HD for the above carbons at various temperatures are provided in Table 3. Rate constant and kinetic saturation capacity values markedly decrease due to poorer adsorption at higher temperatures. From the obtained values of kinetic saturation capacity, the service lives/breakthrough times of carbon bed are predicted by calculations. From them, it is found that, breakthrough time/service life decreases from 600 to 354 min for active carbon when temperature is increased from 35 to 55 °C, it decreases from 600 to 214 min for $NaOH/CrO_3/C$

Table 2
Sulphur mustard adsorption capacity and rate constant at various concentrations on carbons for 1.0 lpm flow rate and 25 °C

Concentration (mg/l)	Active carbon		$NaOH/CrO_3/C$		$NaOH/CrO_3/EDA/C$		$RuCl_3/C$	
	W_e (g/g)	k_v (min^{-1})	W_e (g/g)	k_v (min^{-1})	W_e (g/g)	k_v (min^{-1})	W_e (g/g)	k_v (min^{-1})
0.3	0.3	9488	0.3	9083	0.3	8654	0.3	9108
0.5	0.3	10239	0.3	8567	0.3	8953	0.3	9114
0.6	0.3	7505	0.3	9122	0.3	8896	0.3	8106

Table 3
Sulphur mustard adsorption capacity and rate constant at various temperatures on carbon for 1.0 lpm flow rate and 0.5 mg/l concentration

Temperature (°C)	Active carbon		$NaOH/CrO_3/C$		$NaOH/CrO_3/EDA/C$		$RuCl_3/C$	
	W_e (g/g)	k_v (min^{-1})	W_e (g/g)	k_v (min^{-1})	W_e (g/g)	k_v (min^{-1})	W_e (g/g)	k_v (min^{-1})
35	0.30	10,135	0.30	9956	0.21	7467	0.18	7456
45	0.23	8,750	0.16	8150	0.15	6930	0.16	8754
55	0.17	8,159	0.10	7217	0.10	6524	0.07	8254

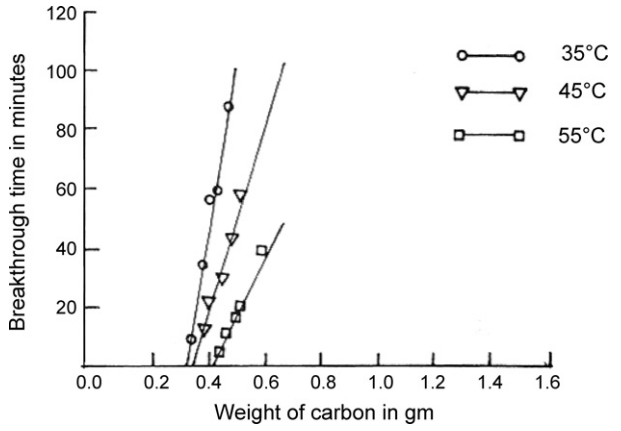

Fig. 5. Sulphur mustard breakthrough time as a function of carbon weight ($NaOH/CrO_3/C$) at various temperatures, 0.5 mg/l concentration and 1.0 lpm flow rate.

system when temperature is increased from 35 to 55 °C, it decreases from 430 to 214 min for $NaOH/CrO_3/EDA/C$ system when temperature is increased from 35 to 55 °C and it decreases from 374 to 150 min for $RuCl_3/C$ system when temperature is increased from 35 to 55 °C. These observations reveal that, in addition to the kinetic saturation capacity values, the breakthrough time/service life values also are ruined at higher temperatures due to poorer adsorption of HD. Although the properties of impregnated carbons such as saturation capacity and service life are ruined at elevated temperatures (35–55 °C), the thermal stability of those remains same [9].

From the above studies and the results obtained, it is understood that the above carbons are capable of holding HD vapors and suitable for providing sufficient respiratory protection against the same. Hence, it is also expected that the same carbons can provide sufficient protection against HD and can be used in NBC filtration systems. However, the breakthrough experiments with the real agent, HD can provide us with enough information which will be useful for confirming the fact that the above carbons can be used without any problem in NBC filtration systems for protection against deadliest agents such as HD.

4. Conclusion

Breakthrough behaviour of sulphur mustard vapors on active carbon, $NaOH/CrO_3/C$, $NaOH/CrO_3/EDA/C$ and $RuCl_3/C$ was studied. Effects of various parameters such as bed height, flow rate, concentration and temperature were interpreted in terms of kinetic saturation capacity and rate constant. Breakthrough time was observed to be increasing with the increase in bed height. Rate constant value increased when the flow rate increased and the value of kinetic saturation capacity was found to be invariable. This indicates that the rate limiting process is controlled by diffusion of HD molecules on the surface of carbon. The concentration variation in between 0.3 and 0.6 mg/l has no significant effect on kinetic saturation capacity or rate constant. Temperature affects the W_e and k_v values adversely due to poorer adsorption at higher temperatures.

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