

Adsorption of methyl *tert*-butyl ether using granular activated carbon: Equilibrium and kinetic analysis

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ABSTRACT: The adsorption of methyl *tert*-butyl ether by granular activated carbon was investigated. The experimental data were analyzed using the Freundlich isotherm and the Langmuir isotherm. Although equilibrium data were found to follow Freundlich isotherm model, it were fitted better by the Langmuir model with a maximum adsorption capacity of 204.1 mg/g. The kinetic data obtained at different concentrations were analyzed to predict the constant rate of adsorption using three common kinetic models: pseudo-first-order, pseudo-second-order equation and intraparticle diffusion equation. The pseudo-second-order model was suitable for describing the adsorption kinetics for the removal of methyl *tert*-butyl ether from aqueous solution onto granular activated carbon. Both the Lagergren first-order rate constant k_1 and pseudo-second-order rate constant k_2 decrease with increasing initial concentrations of methyl *tert*-butyl ether and the intraparticle diffusion rate constant k_p shows the reverse characteristic. Analysis of sorption data using a boyd plot confirmed that external mass transfer is the main rate-limiting step at the initial stage of adsorption. Results illustrate that granular activated carbon is an effective adsorbent for methyl *tert*-butyl ether and also provide specific guidance into adsorption of methyl *tert*-butyl ether on granular activated carbon in contaminated groundwater.

Keywords: Adsorption equilibrium; Intraparticle diffusion model; Mechanism; Modeling

INTRODUCTION

Methyl *tert*-butyl ether (MTBE) is a kind of fuel additive used as a replacement for lead to promote combustion efficiency and to reduce air pollution (Squillace *et al.*, 1996). However, with the widespread use of MTBE, it has become one of the most frequently detected underground water pollutants caused by gasoline leaking from underground storage tanks, pipelines and other components of gasoline distribution systems (Kharoune *et al.*, 2001). MTBE has been classified as a possible human carcinogen by the U.S. Environmental Protection Agency (Johnson *et al.*, 2000). Moreover, MTBE is reported to induce a variety type of deoxyribonucleic acid (DNA) damage such as single-strand breaks, double-strand breaks and oxidative base modification (Chen *et al.*, 2008). Now, water resources are severely threatened by MTBE due to its low odor and taste threshold of about 2~2.5 µg/L (Fiorenza and Rifai, 2003). To date, MTBE-related problems have spread

worldwide including USA, Europe and Asia (Squillace *et al.*, 1996; Achten and Puttmann, 2000; Hsieh *et al.*, 2006; Ayotte *et al.*, 2008). Therefore, there has been a growing concern regarding the treatment of MTBE-contaminated groundwater in recent years (Suidan *et al.*, 2005; Chen *et al.*, 2008, 2009). Given its high aqueous solubility, MTBE removal by traditional treatment technologies, such as air stripping has been expected to be less effective (Chang and Young, 2000; Soltanali and Shams Haghani, 2008). The byproducts of advanced oxidation process have prevented this technique from general application though it is quite effective in removing MTBE (Acero *et al.*, 2001). Granular activated carbon (GAC) adsorption is a widely used technology for treating water contaminated with many taste- and odor-causing organics and synthetic organic chemicals, either independently or coupled with biological degradation (Suffet and Wable, 1995; El Diwani *et al.*, 2009). Some researches have reported that GAC could successfully remove MTBE from contaminated water (Wilhelm *et al.*, 2002; Shih *et al.*,

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2003; Sutherland et al., 2004) mainly due to its well-developed porous internal structure and large surface area (Yang and Al-Duri, 2005). The systemic investigation on the kinetic and mechanism of MTBE adsorption has not reported. In order to optimize the design and operation of an adsorption system for wastewater treatment, it is essential to study the adsorption equilibrium and kinetics which could supply the fundamental information of adsorption (Abdel-Ghani et al., 2009). Although various models were used to describe the adsorption kinetics, most of them need many parameters which require lots of experiments and massive calculation time. Therefore, several simplified models using lumped parameter were developed, which could also explain the whole adsorption process for many adsorption systems. Two widely used models are the Lagergren first-order kinetic model and pseudo-second-order model which assume the driving force of adsorption is the difference between the equilibrium concentration q_e and the adsorbate uptake q (Chang et al., 2003; Chang and Juang, 2004; Ozcan et al., 2004). Another well-known lumped kinetic model is the intraparticle diffusion model which assumes that the film diffusion is negligible and intraparticle diffusion is the only rate controlling step (Wu et al., 2001; Chang and Juang, 2004).

The objective of this research is to study the adsorption mechanism of MTBE from aqueous solution onto GAC and to further evaluate the validity of common isotherm models and kinetic models. The research was carried out in the laboratory (Hangzhou, China) during the period of March to December in 2008.

MATERIALS AND METHODS

MTBE with purity greater than 99.8 %, supplied by Tedia Company (USA), was employed as adsorbate. The adsorbent used in this study is GAC (Shanghai Activated Carbon Corporation, China) which was washed thoroughly in distilled water to remove the suspension residues and dried at 105 °C for at least 24 h. Its physical properties were as follows: particle size, 18-30 mesh fraction; particle density, 0.69 g/cm³; true density, 1.17 g/cm³; particle porosity computed from particle density and true density, 0.41.

To determine the adsorption isotherms, MTBE of different initial concentrations was added to the sealed serum bottles with 200 mL aqueous solutions containing fixed concentration of GAC (1 g/L). Then the bottles were shaken in a rotary shaker (28.0 ± 0.5

°C, 160 r/min) for eight days to attain equilibrium. Each experiment was carried out in triplicate. The effect of the initial MTBE concentrations on adsorption kinetics was investigated. The adsorption was carried out in 250 mL sealed serum bottles with the same concentration of GAC (1 g/L) and MTBE at initial concentrations of 57.2, 126.3, 180, 235.4 mg/L, respectively. The solution was stirred by a rotary shaker (28.0 ± 0.5 °C, 160 r/min) and samples from three parallel replicates were taken at predetermined time intervals. The concentration of MTBE in liquid phase was determined by a gas chromatography (Agilent 6890N, USA) equipped with a silica HP-Innowax capillary column (30 m × 0.32 mm × 0.5 μm, J & W Scientific, USA) and a flame ionization detector. The operating conditions were as follows: injector, 250 °C; oven, 80 °C for 4 min and then programmed to 160 °C at the rate of 30 °C/min and detector, 300 °C. The amount of MTBE adsorbed was determined as:

$$q = (C_0 - C_t)V / W \quad (1)$$

RESULTS AND DISCUSSION

Adsorption isotherm in water solution

Adsorption isotherm study provides fundamental physiochemical data for evaluating the adsorption capacities of an adsorbent, one of the most important criteria in selecting a suitable adsorbent. In the present investigation, the isotherm data were analyzed using the Langmuir and Freundlich isotherm equations (Figs. 1 and 2). The Langmuir sorption isotherm (Kim et al., 2004) is the best and the Freundlich isotherm (Chang et al., 2003) is an empirical equation most frequently used to describe the adsorption of inorganic and organic components in solution, respectively expressed as:

$$\text{Langmuir: } q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (2)$$

$$\text{Freundlich: } q_e = K_f \times C_e^{1/n} \quad (3)$$

The linearized form of above equations can be written as follows:

$$\frac{1}{q_e} = \frac{1}{q_m K_L C_e} + \frac{1}{q_m} \quad (4)$$

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (5)$$



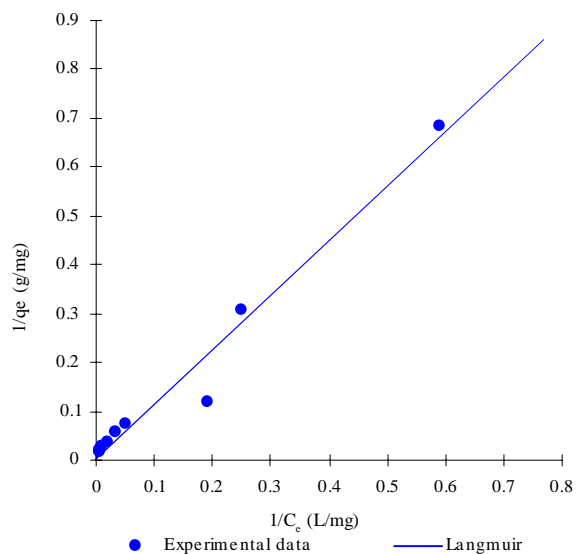


Fig. 1: Experimental data and predicted Langmuir isotherm of MTBE in water solution onto GAC. (Sorbent dose: 1 g/L; shaker speed: 160 r/min; temp.: 28.0 ± 0.5°C; contact time: 8 days)

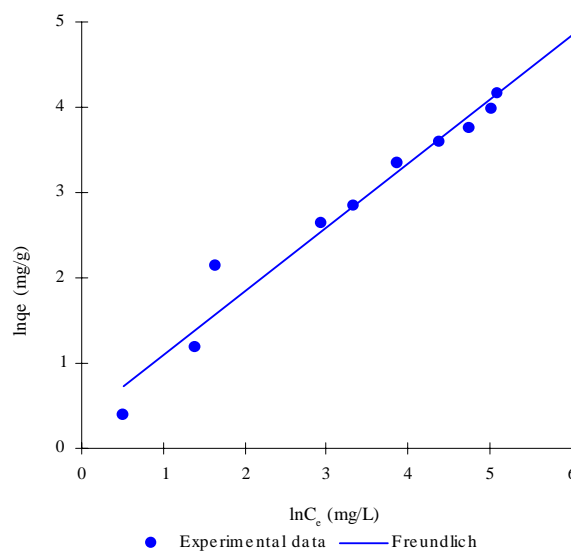


Fig. 2: Experimental data and predicted Freundlich isotherm of MTBE in water solution onto GAC. (Sorbent dose: 1 g/L; shaker speed: 160 r/min; temp.: 28.0 ± 0.5°C; contact time: 8 days)

Table 1: Parameter values from fitting the adsorption equilibrium data with isotherm models

q_m (mg/g)	Langmuir		Freundlich		
	K_L (L/mg)	R^2	K_f (mg/g) (L/mg) ^{1/n}	n	R^2
204.1	4.39	0.9722	1.39	1.33	0.963

If the adsorption follows the Langmuir equation, a plot of $1/q_e$ versus $1/C_e$ should be a straight line. q_m and K_L can be determined from the intercept and slope. Similarly, from a plot of $\ln q_e$ against $\ln C_e$, K_f and n can be computed. Table 1 shows the results of the Langmuir and Freundlich isotherm, indicating the reasonably good correlation between the experiment and both the two models, as reflected by correlation coefficients (R^2) in the value of 0.972 and 0.963. The fact that the Langmuir isotherm fits the experimental data well may be due to the homogenous distribution of active sites on GAC. The maximum sorption capacity q_m of GAC for MTBE attained from the intercept is 204.1 mg/g, reflecting that GAC has good capacity adsorbing MTBE. Moreover, the value of n was computed as 1.33 between 1 and 10, which also display the good sorption capacity of GAC for MTBE (Ben Hamissa et al., 2008). Ji et al. (2009) claimed that MTBE adsorption on the nonpolar porous polymer and chloromethylated polymer followed Langmuir adsorption model, which is somewhat in

accord with the phenomenon of present work. The adsorption capacity of GAC for MTBE adsorption may be dependent on both pore structure and surface chemistry of the adsorbents (Ji et al., 2009).

Adsorption kinetics

The effect of MTBE at different initial concentrations on the adsorption kinetics is shown in Fig. 3. It reveals that the adsorbate uptake q increases with increasing initial concentrations. In the diagram, the slope at each point of the curve denotes the transient adsorption rate (dq/dt). Fig. 3 shows that at all initial MTBE concentrations, the adsorption rate decreases with the contact time until it gradually approaches a plateau because the driving force ($q_e - q$) continuously decreases with time.

In the present study, the instantaneous adsorption of the batch process at different initial concentrations of MTBE was investigated using the Lagergren first-order kinetic model and pseudo-



Adsorption of MTBE using GAC

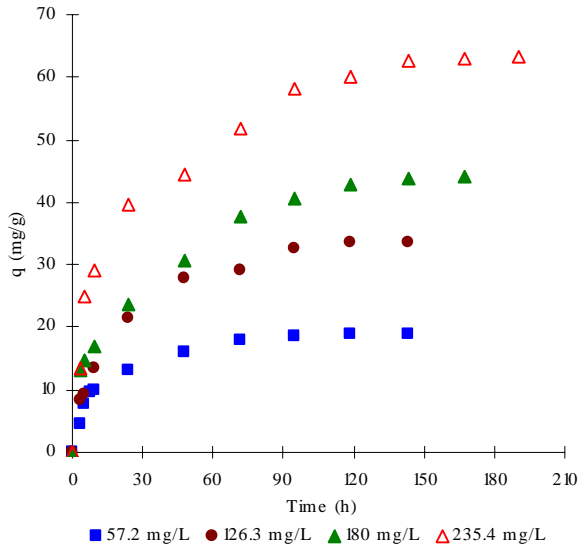


Fig. 3: The adsorption of MTBE at different initial concentrations onto GAC. (Agitation speed: 160 r/min; sorbent dose: 1 g/L; temperature: 28.0 ± 0.5 °C.)

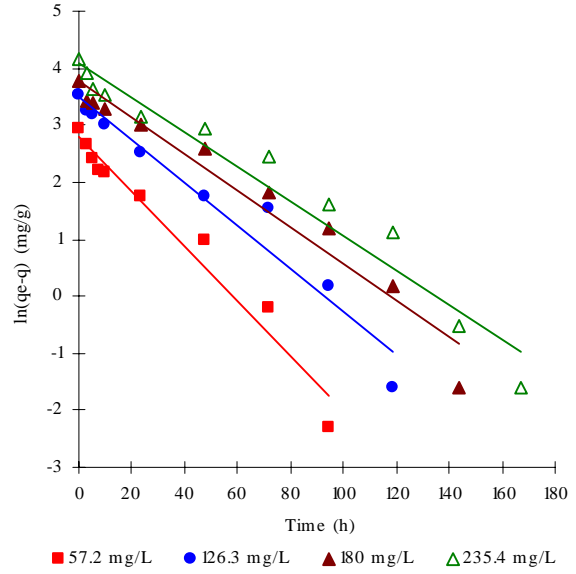


Fig. 4: Lagergren plot for MTBE sorption onto GAC

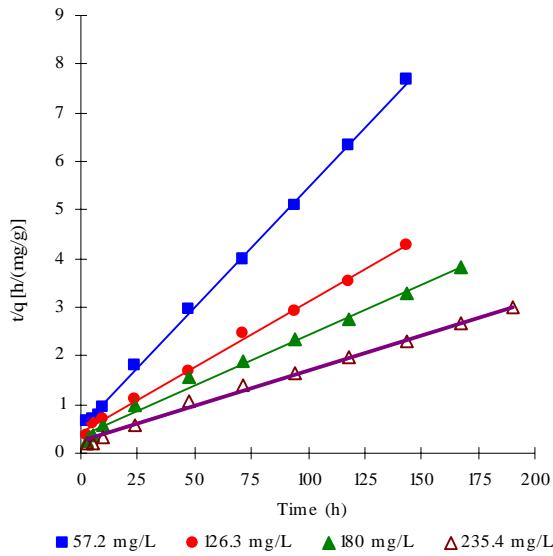


Fig. 5: Pseudo-second-order plot for MTBE sorption onto GAC

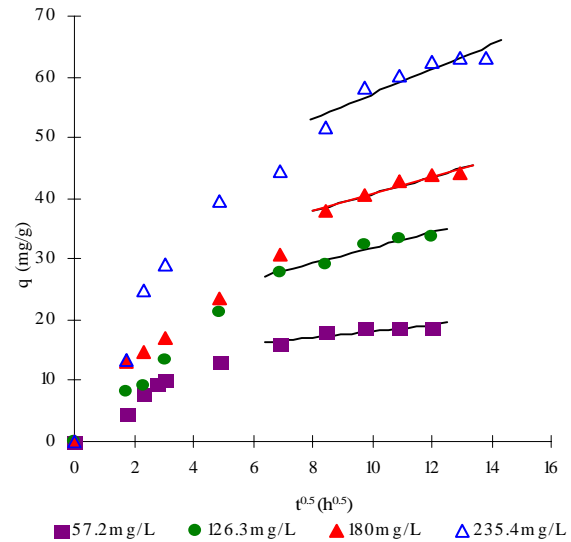


Fig. 6: Intraparticle diffusion plot for MTBE sorption onto GAC

second-order model (Yang and Al-Duri, 2005), respectively represented as:

$$\frac{dq}{dt} = k_1(q_e - q) \quad (6)$$

$$\frac{dq}{dt} = k_2(q_e - q)^2 \quad (7)$$

Integrating the above equations for the boundary conditions $t = 0$ to $t = t$ and $q = 0$ to $q = q$ gives

$$\ln(q_e - q) = \ln q_e - k_1 t \quad (8)$$

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (9)$$



Constant k_1 and q_e can be calculated from the plot of $\ln(q_e - q)$ versus time (t). Similarly, the plot of t/q against t gives the value of k_2 and q_e .

Figs. 4 and 5 show that at all initial MTBE concentrations, the experimental data are both well represented by the Lagergren model and the pseudo-second-order model. The values of rate parameters and correlation coefficient R^2 have been obtained and listed in Table 2. It is observed that the pseudo-second-order model yields a somewhat better fit than the Lagergren model by comparing the results of correlation coefficients. It could be concluded that the mechanism of adsorption was pseudo-second-order reaction. This conclusion is consistent with those studies demonstrating that the pseudo-second-order kinetic model is reasonably good fit of data over the entire adsorption process (Wu *et al.*, 2001; Chang *et al.*, 2003; Chang and Juang, 2004;

Ozcan *et al.*, 2004). Clearly, it is seen that when the initial MTBE concentration increases, the equilibrium concentration q_e increases accordingly. Furthermore, Table 2 illustrates that both the rate constants k_1 and k_2 decrease with increasing initial MTBE concentrations, which reveals the fact that it is faster for an adsorption system with MTBE at lower initial concentrations to reach equilibrium. This trend is compatible with those observed for other adsorption systems (Wu *et al.*, 2001; Chang *et al.*, 2003; Rengaraj *et al.*, 2004). Because the above two equations could not definitely reveal the adsorption mechanism, another kinetic model named intraparticle diffusion model was used in the present study (Yang *et al.*, 2005). The formation of this model is as follows:

$$q = k_p t^{0.5} \tag{10}$$

$$k_p = \frac{6q_e}{R} \sqrt{\frac{D}{\pi}} \tag{11}$$

If the adsorption process follows the intraparticle diffusion model, a plot of q against $t^{0.5}$ should be a straight line. k_p can be calculated from the slope of the straight line. Nevertheless, it is common to observe multilinearity on the $q-t^{0.5}$ plot, which reveals the different stages in adsorption involving external mass transfer and intraparticle diffusion (Yang *et al.*, 2005).

The intraparticle diffusion plot for the adsorption of MTBE onto GAC is shown in Fig. 6. It is observed that there are two linear portions which elucidate the two adsorption stages: external mass transfer at initial period, then followed by intraparticle diffusion of MTBE onto GAC. The slope of the second linear portion suggests the rate of intraparticle diffusion listed in Table 2 which shows that k_p increases with increasing initial MTBE concentration. From Eq. 11,

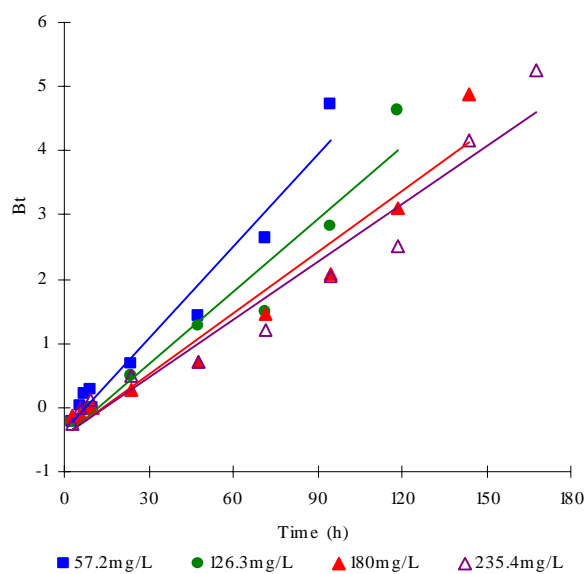


Fig. 7: Boyd plot for MTBE adsorption onto GAC

Table 2: Kinetic parameters for MTBE sorption onto GAC

C_0 (mg/L)	Experimental q_e (mg/g)	Lagergren first-order			Pseudo-second-order			Intraparticle diffusion
		q_e (mg/g)	k_1 (h^{-1})	R^2	q_e (mg/g)	k_2 (g/mg/h)	R^2	k_p (mg/g/h ^{0.5})
57.2	18.7	16.5	0.0482	0.9621	20.16	0.00497	0.9989	0.51285
126.3	33.7	32.8	0.0377	0.9545	37.4	0.00168	0.9973	1.27812
180	44	43.61	0.0322	0.9550	48.36	0.00118	0.9916	1.40141
235.4	63.2	60.19	0.0302	0.9564	68.12	0.00094	0.9955	2.02965



it can be seen that k_p is proportional to both q_e and $D^{1/2}$. The equilibrium concentration q_e increases accordingly with the increasing initial MTBE concentration. Moreover, some studies have reported that the intraparticle diffusion, D , increases with the increasing initial concentration (AIDuri and Mckay, 1991; Chatzopoulos et al., 1993; Yang et al., 2003). Consequently, the constant k_p increases with increasing initial concentration due to the increasing q_e and $D^{1/2}$. In order to interpret the rate-controlling step during the adsorption process, the experimental data are further analyzed by the model given by Boyd (Boyd et al., 1947).

$$F = 1 - \frac{6}{\pi^2} \exp(-Bt) \quad (12)$$

Since F is equal to q/q_e , Bt could be represented as follows:

$$Bt = -0.4977 - \ln\left(1 - \frac{q}{q_e}\right) \quad (13)$$

The value of Bt against time (t) is plotted in Fig. 7 employed to characterize the particle diffusion control mechanism (Kumar et al., 2005). It is shown that the plots are linear but do not pass through the origin, signifying that external mass transfer is the main rate controlling step at the initial stages. A similar observation was previously reported by Kumar et al. (2005). When the amounts of organic molecules which have reached external activated carbon increase to a certain degree, the sorption process becomes intraparticle-diffusion-controlled.

CONCLUSION

The adsorption equilibrium and kinetics of MTBE onto GAC have been studied in the present work. The equilibrium data could be fitted by both the Langmuir isotherm model and the Freundlich isotherm model which reflect that GAC has good capacity adsorbing MTBE. The adsorption kinetic data have been respectively analyzed by the Lagergren first-order model, the pseudo-second-order model and the intraparticle diffusion model. The results indicate that the adsorption of MTBE onto GAC could be best described by the pseudo-second-order model. Both the rate constants k_1 and k_2 decrease with increasing initial concentrations of MTBE and k_p shows the reverse characteristic due

to the increasing q_e and $D^{1/2}$. Analysis of mechanism reveals that the adsorption process of MTBE onto GAC includes two stages: external mass transfer at initial period, then followed by intraparticle diffusion. At the initial stage of adsorption, external mass transfer is the main rate controlling step. When the amounts of organic molecule which have reached external activated carbon increase to a certain degree, the sorption process becomes intraparticle-diffusion-controlled.

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Nomenclature

- C_0 the initial concentration of the MTBE solution (mg/L)
- C_t the liquid phase concentration of the MTBE solution at time t (mg/L)
- C_e the equilibrium liquid-phase concentration of MTBE (mg/L)
- V the volume of the MTBE solution (mL)
- W the mass of the dry adsorbent (g)
- q the amount of MTBE adsorbed onto the solid phase at any time (mg/g)
- q_e the amount of adsorption to GAC at equilibrium (mg/g)
- q_m a constant related to the monolayer coverage of adsorbate onto the adsorbent which reflects the adsorption capacity (mg/g)
- t time (h)
- K_L Langmuir isotherm constant (L/mg)
- K_f Freundlich isotherm constant [(mg/g)(L/mg)^{1/n}]
- n Freundlich exponent related to adsorption intensity.
- k_1 the Lagergren first-order adsorption rate constant (h⁻¹)
- k_2 the pseudo-second-order adsorption rate constant (g/mg/h)
- k_p the intraparticle diffusion rate constant (mg/g/h^{0.5})
- R the particle radius (cm)
- D the intraparticle diffusivity constant
- F the fractional uptake at time t
- B a mathematical function of F
- R^2 squared regression correlation coefficient



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