REMOVAL OF QUINOLINE FROM AQUEOUS SOLUTIONS BY LIGNITE, COKING COAL AND ANTHRACITE. ADSORPTION KINETICS

Hongxiang XU[*], Yongtian WANG[**], Gen HUAGN[*], Guixia FAN[***], Lihui GAO[**], Xiaobing LI[**]

[*] School of Chemical and Environmental Engineering, University of Mining and Technology (Beijing), Beijing, China, 100083, xuhongxiang001@163.com

[**] School of Chemical Engineering and Technology, Chinese National Engineering Research Center of Coal Preparation and Purification, China University of Mining and Technology, Xuzhou, Jiangsu, China, 221116.

[***] School of Chemical Engineering and Energy, Zhengzhou University, Zhengzhou, China, 450001

Abstract: Basing on the concept of circular economy, a novel method of industrial organic wastewater treatment by using adsorption on coal is introduced. Coal is used to adsorb organic pollutants from coking wastewaters. After adsorption, the coal would be used in its original purpose, as its value was not reduced and the pollutant was reused. Through the systemic circulation of coking wastewater zero emissions can be achieved. Lignite, coking coal and anthracite were used as adsorbents in batch experiments. The quinoline removal efficiency of coal adsorption was investigated. Both the coking coal and anthracite exhibited properties well-suited for quinoline adsorption removal. The experimental data were fitted to the pseudo-first- order and pseudo-second-order kinetic equations as well as intraparticle diffusion and Bangham models. An attempt was made to find the rate-limiting step involved in the adsorption processes. Both boundary-layer diffusion and intraparticle diffusion are likely involved in the rate-limiting mechanisms. Effect of pH on coal adsorptions by coking coal was investigated. The process of quinoline adsorption on coal was researched. The coal adsorption method for removing refractory organic pollutants is a great hope for achieving wastewater zero emission for coking plants.

Introduction

Coking wastewater usually contains high concentrations of ammonia, sulfide, phenol, quinoline, pyridine indol, and other organic compounds, which makes it difficult to treat because most of those compounds are refractory, highly concentrated mutative and carcinogenic and produce long-term environmental and ecological impacts (Lai et al., 2009; Chen et al., 2009; Fang et al., 2012).

Adsorption is the most convenient and effective technique to remove organic compounds from coking wastewater (Aksu and Yener, 2001; Badmus and Audu, 2009; Lorenc-Grabowska et al., 2013), but there also are some problems including the high cost of adsorbent and adsorbent regeneration, sludge may be produced which may cause other environment problem, and finally adsorbent regeneration loss (Mall and Srivastava, 2006; Ahmaruzzaman, 2008; Belhachemi and Addoun, 2012). Therefore, methods that enhance advantages and avoid disadvantage of adsorption were important in this research. Coal is a complex porous medium and natural adsorbent. After adsorption, the coal may be used in its original purpose and its value was not reduced (Li et al., 2015).

This research investigates the adsorption behavior of quinoline related substances found in simulated coking plant wastewater. The characters of adsorbents are investigated by using FTIR and BET. Lignite, coking coal and anthracite were compared with respect to adsorption capacity and process kinetics by various fitting models. The effect of pH on special surface area and coal adsorption efficiency were also investigated.

Methods and materials

Adsorbate and adsorbents

Adsorbate used in this paper is quinoline, and adsorbents investigated are lignite, coking coal and anthracite which used in this experiment. The characterization of the adsorbent has been previously published (Xu et al., 2016).

Batch adsorption studies

The adsorption kinetics of quinoline on three kinds of coal was investigated in batch sorption experiments and described previously (Xu et al., 2016). The adsorption capacity of coal was calculated using the expression

$$Q_t = \frac{(C_0 - C_t)V}{M} .$$
(1)

The removal efficiency of quinoline was calculated using the expression,

$$E = \frac{C_0 - C_t}{C_0} \times 100\%$$
(2)

where, Q_t (mg·g^{-1}) is the quinoline removed at time t by a unit mass of the adsorbent, C_0 (mg·dm^{-3}) is the initial quinoline concentration, C_t (mg·dm^{-3}) is the quinoline concentration at time t, and M (g) is coal consumption. V (cm^3) is the quinoline solutions volume.

During experiments, the solution pH was carefully adjusted to a range between 1 and 11 by adding small amounts of HCl (0.1 mol·dm^{-3}) or NaOH (0.1 mol·dm^{-3}) solution and it was measured using a pH-meter (S20, Mettler Toledo), while quinoline

solutions contained in 100 cm^3 conical flasks closed with glass stoppers to avoid evaporation were stirred using a mechanical magnetic stirrer. The blank experiments were also carried out to observe the effect of vaporization of quinoline. The amount of vaporization during the experiments was subtracted from the experimental data.

The adsorption experiments, which were conducted at various time intervals and temperatures (283K, 298K and 313K) to determine when the adsorption equilibrium was reached and the maximum removal of naphthalene was attained. After the equilibrium contact time, the samples were filtered and the equilibrium concentrations measured by UV/VIS spectroscopy at the respective standard curve equations, which is 278 nm for quinoline, as described by Lin and Dence (1992).

Adsorption kinetic models

The whole adsorption rate is decided by the slowest step, so to improve that step can increase the whole adsorption rate. To know which step control the rate of adsorption is very important to research (Poot et al., 1978; Rodriguez et al., 2009; Zhang et al., 2010) the adsorption process and improve the adsorption efficiency. Here, kinetic models were used to investigate the mechanism of sorption and potential rate controlling steps.

Pseudo-first-order kinetic equation

The Lagergren pseudo-first-order rate expression is given as (Ho and Mckay, 1999; Lataye et al., 2006 Hwang et al., 2011):

$$\frac{dQ_t}{dt} = k_1(Q_{eq} - Q_t). \tag{3}$$

The integration form of the pseudo-first order kinetic equation is:

$$Q_t = Q_{eq}(1 - e^{-k_1 t}). \tag{4}$$

The line form of the pseudo-first order kinetic equation is:

$$log(Q_{eq} - Q_t) = logQ_{eq} - \left[\left(\frac{k_1}{2.303}\right)t\right]. \tag{5}$$

The boundary conditions $Q_t = 0$ at $t = 0$ and $Q_t = Q_{eq}$ at $t = t$, where, Q_t (mg·g^{-1}) is the amount of quinoline adsorbed at time t (min); k_1 (min^{-1}) is the pseudo-first-order rate constant.

Pseudo-second-order kinetic equation

The pseudo-second-order kinetic equation which was presented by Ho and Mckay, (1988; 2002), Azizian (2004), Chowdury et al. (2011) is expressed as:

$$\frac{dQ_t}{dt} = k_2(Q_{eq} - Q_t)^2 \tag{6}$$

where, k_2 (g·mg^{-1}·min^{-1}) is the pseudo-second-order rate constant.

The integration form of the pseudo-second-order kinetic equation is:

$$\frac{1}{(Q_{eq}-Q_t)} = \frac{1}{Q_{eq}} + k_2 t \tag{7}$$

while linear form of the pseudo-second-order kinetic equation is:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_{eq}^2} + \frac{t}{Q_{eq}}. \tag{8}$$

A special feature of the pseudo-second-order rate expression is that the initial sorption rate at t = 0, sometimes denoted as h (mg·g^{-1} min^{-1}) (Srivastava, 2005; Andersson 2011). The initial sorption rate h can be calculated by:

$$h = k_2 \times Q_{eq}^2. \tag{9}$$

The intraparticle diffusion model

The intraparticle diffusion kinetic equation which was presented by Weber and Morris (1963) (Yu et al., 1996) and is expressed as:

$$Q_t = k_3 t^{0.5} + m \tag{10}$$

where k_3 (mg·g^{-1}·min$^{-0.5}$) is the intraparticle diffusion rate constant, and m (mg·g^{-1}) is the intercept which indicates the boundary layer thickness. The larger the intercept is, the greater the boundary effect is (Weber and Morris, 1963; Kumar et al., 2003).

The Bangham model

The kinetic equation presented by Bangham (Sze and Mackay, 2010) is expressed as:

$$\frac{dQ_t}{dt} = k_4 (C_0 - Q_t m)\gamma t^{\gamma-1}. \tag{11}$$

The integration form of the Bangham model is:

$$Q_t = \left(\frac{C_0}{m}\right) - \frac{C_0}{m\,exp(k_4 m t^\gamma)}. \tag{12}$$

The line form of the Bangham model is:

$$loglog\left(\frac{C_0}{C_0 - Q_t m}\right) = log\left(\frac{k_4 m}{2.3}\right) + \gamma logt. \tag{13}$$

The boundary conditions is $Q_t = 0$ at $t = 0$ and $Q_t = Q_{eqt}$ Q_{eq} at $t = t$, where, m (mg·dm^{-3}) is adsorbent concentration, K_4 is proportionality constant and γ is a constant (Yu et al., 1997; Kaya et al., 2013).

Results and discussion

Chemical composition of lignite, coking coal and anthracite

The chemical composition of the lignite, coking coal and anthracite which was obtained by XRF are shown in Table 1.

Table 1. Chemical composition of the lignite, coking coal and anthracite

Title		Na_2O	TiO_2	K_2O	S	MgO	Fe_2O_3	CaO	Al_2O3	SiO_2
	lignite	0.15	0.20	0.47	0.65	0.69	1.28	1.62	4.68	12.90
Concentration %	coking coal	0.07	0.33	0.21	0.85	0.16	0.73	0.64	5.54	8.07
	anthracite	0.10	0.33	0.06	0.79	0.14	0.63	0.68	4.62	5.48

As shown in Table 1, the sum content of three parts including calcium oxide, alumina and monox in lignite was highest, was 19.91%, those were three main components of ash. The sum content of three parts in coking coal and anthracite were 14.25% and 10.78%, respectively. There may be a large number of silicon and aluminum active adsorption site on lignite surface because that the content of silicon and aluminum are large. So the main gangue mineral in lignite was quartz, which was accord with the XRD previously described Xu et al. (2016).

The special surface area

The special surface area of the lignite, coking coal and anthracite were 6.0876 $m^2 \cdot g^{-1}$, 5.7864 $m^2 \cdot g^{-1}$ and 6.1479 $m^2 \cdot g^{-1}$, respectively, as determined previously (Xu et al., 2016).

Effect of pH on special surface area

The effect of pH on special surface area for coking coal is shown in Fig. 1. As shown in Fig. 1, the special surface area of coking coal decreased as the pH value increased from 2.0 to 6.0, the special surface area remains approximately constant as the pH value increased from 6.0 to 8.0, the special surface area rapidly decreased after pH 8.0. Due to both most of oxygen-containing functional group on the coal surface and hydrochloric acid solutions acidity, there is no chemical reaction, so the coal macromolecular structure has not changed after the coal was treated by hydrochloric acid solutions, and thus the basic structure of the coal sample also has not change (Wang et al., 2012). Because the sulfate and alkali soluble minerals on the coal surface were washed away by solutions of hydrochloric acid and some concave and convex points on the coal surface were formed, the special surface area of the coal increased as shown in Fig. 1. The special surface area of the coal increased slightly because some acidic minerals on the coal surface were dissolved after the coal was treated by solutions of sodium hydroxide solution of known pH value. Meanwhile on the coal surface, the sodium hydroxide may react with the oxygen containing functional groups

and in addition some organic matter and minerals may dissolved, and thus some part of the pore collapsed after the reaction reached a certain point which decreased the special surface area of the coal. The specific surface area increase and reduction is flat around 7.5 pH value. The specific surface area decreased after 8.0 pH value because the reduction part of special surface area was bigger than increase part.
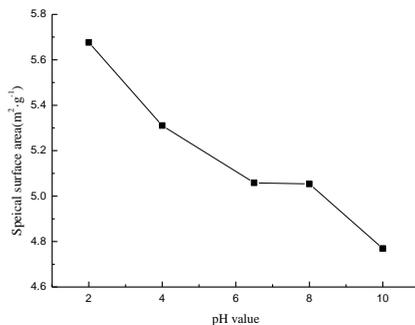

Fig. 1. Effect of pH on the special surface area of coking coal at 25 ℃

Effect of pH on coal adsorption

The effect of pH on the adsorption capacity and the quinoline removal efficiency for lignite, coking coal and anthracite coal are shown in Fig. 2.
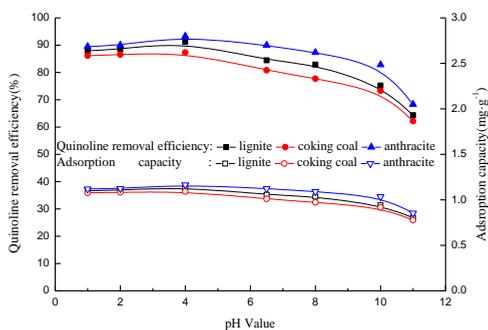

Fig. 2. The influence of pH value to the adsorption capacity and the quinoline removal efficiencies for lignite, coking coal and anthracite

As shown in Fig. 2, the maximum quinoline removal for three kinds of coal was found to beat around pH value of 4.0. Coal adsorption increased at pH values range from 1.0 to 4.0, after that it slightly decreased up to pH = 11 due to that the specific surface area decreased with the PH values from 1 to 11 according to the specific surface area measurements. The optimum adsorption pH value determined by test is 4.

Kinetic models

The equation constants were obtained from the experimental data by applying linear regression analysis to the linear forms of the model equations (Fig. 3) and are reported, together with the correlation coefficients by using Origin 8.5 and 1stOp software, in Table 1.
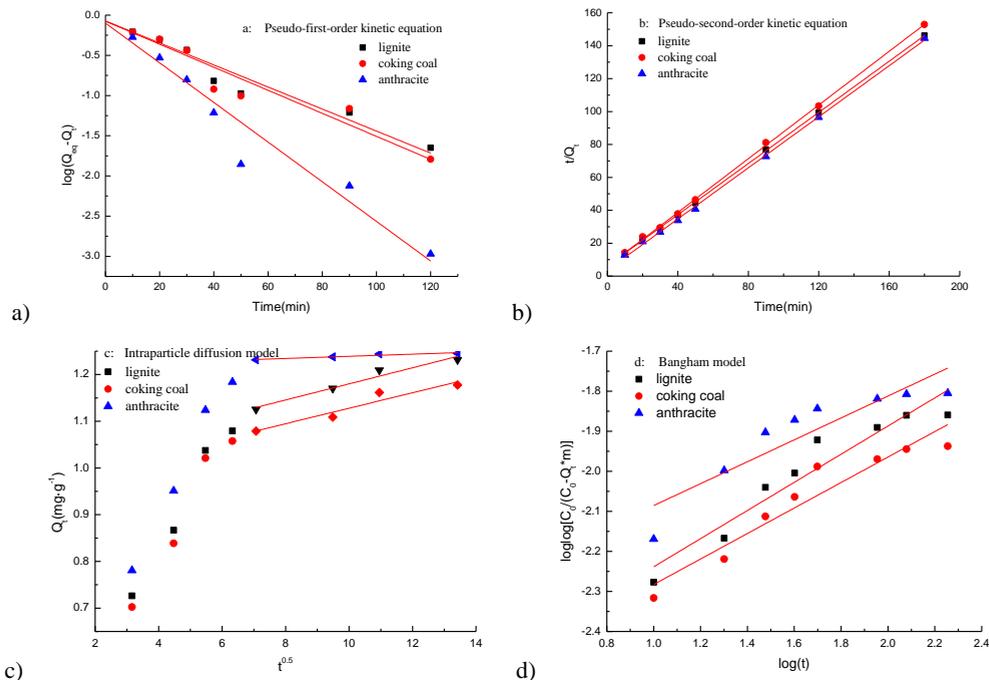

Fig. 3. Adaptation of experimental data on quinoline adsorption onto lignite, coking coal and anthracite using four kinetic models: a) pseudo-first-order equation, b) pseudo-second order equation, c) intraparticle diffucion model, and d) Bangham model

As shown in Fig. 3, it can be seen that the pseudo-first-order rate equation provided a poor fit to the experimental data. However, according to Gerente et al. (2007), this model is generally restricted to only the initial 20~40% of the adsorption capacity and the equation needs further modifications for longer sorption times. Nevertheless, the pseudo-second-order rate expression showed the best fit to the experimental data because all of three R^2 are larger than 0.999.

The pseudo-second-order rate equation also yielded $Q_{eq(calc)}$ values close to the obtained $Q_{eq(exp)}$ results (Table 2). Therefore, sorption of quinoline onto lignite, coking coal and anthracite was more appropriately approximated by the pseudo-second-order rate model. The values of h, representing the initial sorption rate, were obtained from the pseudo-second-order rate expression. The sequence of the obtained

k_2 value was anthracite ＞ coking coal ＞ lignite, implying that sequence of the sorption process was anthracite ＞ coking coal ＞ lignite. The reason is that the special surface area of anthracite is largest and the special surface area of lignite is smallest in three kinds of coal.

Table 2. Constants and correlation coefficients obtained by liner regression

Adsorbents	Pseudo-first-order kinetics model			
	$Q_{eq(exp)}$/mg·g^{-1}	$Q_{eq(calc)}$/mg·g^{-1}	K_1/min^{-1}	R^2
lignite	1.23	0.85	0.0316	0.9480
coking coal	1.19	0.95	0.0268	0.9021
anthracite	1.27	0.79	0.0568	0.9323
Adsorbents	Pseudo-second-order kinetics model			
	$Q_{eq(calc)}$/mg·g^{-1}	K_2/g·mg^{-1}·min^{-1}	h/mg·g^{-1}·min^{-1}	R^2
lignite	1.25	0.1118	0.185	0.9999
coking coal	1.24	0.1014	0.157	0.9998
anthracite	1.28	0.2038	0.330	0.9996
Adsorbents	Intraparticle diffusion model			
	K_3/ mg·g^{-1}·min$^{-0.5}$		intercept	R^2
lignite	0.0172		1.0078	0.9464
coking coal	0.0166		0.9621	0.8872
anthracite	0.0023		1.2159	0.8515
Adsorbents	Bangham model			
	K_4		γ	R^2
lignite	0.0105		0.1825	0.8621
coking coal	0.0104		0.1753	0.8254
anthracite	0.0124		0.1580	0.7314

In the intraparticle diffusion model, if the line goes through the origin, the intraparticle diffusion is the only rate-controlling step. If the line does not go through the origin, the rate-controlling step depends not only on the intraparticle diffusion but also on other steps, such as the boundary-layer mass transfer and internal mass transfer (Gupta et al., 1988; 2003). So the effects of intraparticle diffusion and boundary-layer diffusion on the sorption rate can be determined from the intraparticle diffusion model (Fig. 3-c). The Bangham model yielded relatively low correlation coefficients, which confirmed that intraparticle diffusion is not the only rate-limiting step, and the rate of adsorption could be controlled by several processes, all of which might be operated simultaneously.

Adsorption activation energy calculation

The adsorption activation energy of quinoline adsorption on coking coal was calculated. The quinoline adsorption on coking coal belongs to pseudo-second order kinetic equation according to the results of kinetic model calculation. So the k_2 of pseudo-second order kinetic equation was adsorption rate constant of adsorption reaction. The core of the absolute reaction rate theory is that an activated complex energy barrier is formed when reactants transfer to products. The same may be considered when an adsorption site is activated after overcoming the sorption barrier. So the theory of absolute reaction rate may be transplanted to the study of adsorption kinetics (Kathialagan and Viraraghavan, 2002). The adsorption rate constant k_2 was used to replace the reaction rate constant of Arrhenius equation. Using the assumption that the activation enthalpy and the activation entropy changes affected by temperature were small in the process of adsorption and can thus be ignored, according to the Arrhenius formula the following equation was obtained (Chandra et al., 2007)

$$k_2 = k_0 exp(-\frac{E_a}{RT}) \tag{14}$$

where, k_2 refers to the rate constants of adsorption reaction, $g \cdot mg^{-1} \cdot min^{-1}$; k_0 refers to frequency factor; E_a refers to activation energy, $kJ \cdot mol^{-1}$. R refers to ideal gas constant, 8.314 $J \cdot K^{-1} \cdot mol^{-1}$; T refers to temperature, K.

To calculate the logarithm of the above equation the following equation was obtained:
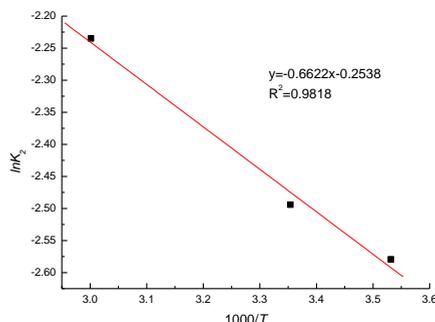
$$lnk_2 = lnk_0 - \frac{E_a}{RT} . \tag{15}$$

Fig. 4. Plot of lnk_2 versus $1000/T$ for quinoline adsorption on coking coal

A straight line was plotted through linear regression for $10^3/T$ and $\ln k_2$ as shown in Fig. 4. The equation of straight line is $\ln k_2 = -0.6622/T - 0.2538$ ($R^2=0.9818$). The activation energy of adsorption E_a ($E_a = 5.51$ $kJ \cdot mol^{-1}$) was obtained from the slope of the linear.

Generally, the activation energy of physical adsorption was 5~40 kJ·mo1^{-1} (Li et al., 2002) and the activation energy of chemical adsorption is greater than 83.72 kJ·mo1^{-1} (Shu and Jia, 2005). Thus, the quinoline adsorption on coking coal belongs to physical adsorption because the activation energy of adsorption was small.

Conclusions

Fitting of experimental data to various kinetic models showed that the process of quinoline adsorption onto three kinds of coal follows pseudo-second-order rate kinetics. The rate of quinoline adsorption on anthracite is largest. The special surface of coking coal increased after the coal was washed by the hydrochloric acid solutions. So the optimum adsorption pH value determined by test is 4. The rate of quinoline adsorption on anthracite is largest. The Bangham model confirmed that intraparticle diffusion is not the only rate-limiting step of the sorption processes, but that boundary-layer diffusion was likely to be involved as well. The activation energy of quinoline adsorption on coking coal E_a was 5.51 kJ·mo1^{-1}, and the quinoline adsorption on coking coal belongs to physical adsorption. The adsorption kinetic research of the coal adsorption method is not only an important investigation for improving the efficiency of organic pollutant removal, but also has significant meanings for achieving wastewater zero emission for coal coking plants in the future.

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