ORIGINAL PAPER

Hexamine adsorption study on activated carbon from aqueous solutions for application in treatment of hexamine industrial wastewater

M. Taghdiri · N. Zamani

Abstract The adsorption of hexamine onto powdered activated carbon from aqueous solutions was studied in a fixed bed system. Langmuir, Freundlich, Redlich–Peterson and Toth isotherm models were used to fit the experimental data and isotherm parameters were determined. The results revealed that the adsorption isotherm models fitted the data in the order of Langmuir > Toth > Redlich–Peterson > Freundlich. Lagergren pseudo-first order kinetic model was found to correlate well with the experimental data. The effects of solution pH, temperature, initial hexamine concentration and added salts concentration on the adsorption capacity and the rate of adsorption were studied. The results indicate that the rate of adsorption increases and then decreases as temperature of the hexamine solution increases, however, the adsorption capacity decreases. The addition of low concentration of salt significantly increases the adsorption capacity of activated carbon. The results showed that the activated carbon has potential for the adsorption of hexamine from industrial hexamine wastewater.

Keywords Amine · Carbon powder · Adsorption isotherms · Kinetic

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Introduction

Activated carbon is the most common solid phase extraction material used for the removal of organic and inorganic compounds (Bansal and Goyal 2005; Cabal et al. 2009; Gur-Reznik et al. 2008; McDonough et al. 2008; Onundi et al. 2010; Yapsakl et al. 2009; Yu et al. 2009). Activated carbon is known to have a heterogeneous physical and chemical structure. It is characterized by the existence of micro, meso, and macro pores of different sizes, while its surface indicates hydrophobic characteristics. Activated carbon structure consists of graphene layers between which slit-shaped pores are formed. The acidic, basic, neutral or oxidative/reductive nature of the activated carbon surface is the result of the presence of heteroatoms such as H, S, O, and N, which usually exist in the form of organic functional groups (Boehm 1994). Adsorption of amines on activated carbon in aqueous phase has been less studied (Cooney and Wijaya 1987; Seidel and Radeke 1990). A number of studies related to the adsorption of amine gases such as ammonia, methyl amine, dimethyl amine and trimethyl amine have been conducted using activated carbon (Tanada et al. 1985; Tanada et al. 1988; Tsutomu et al. 2004). Hexamine or methenamine or urotropine, $(CH_2)_6N_4$, is a non-aromatic heterocyclic amine with a symmetrical three-dimensional molecular structure, as shown in Fig. 1. It is a white, crystalline powder with a slight amine odor, soluble in water (867 g/L at 25 °C), alcohol, and chloroform, but it is insoluble in ether and has a melting point of 280 °C. The acidic dissociation constant (K_a) of hexamine is about 10^{-5} (Strom and Jun 1986) and pH of 10 % aqueous solution is 8–9. Hexamine is used for the production of plastic materials, explosives, phenolic resins, antibacterial pharmaceutics (as a urinary tract antiseptic), disinfecting materials, in the rubber industry as an additive,
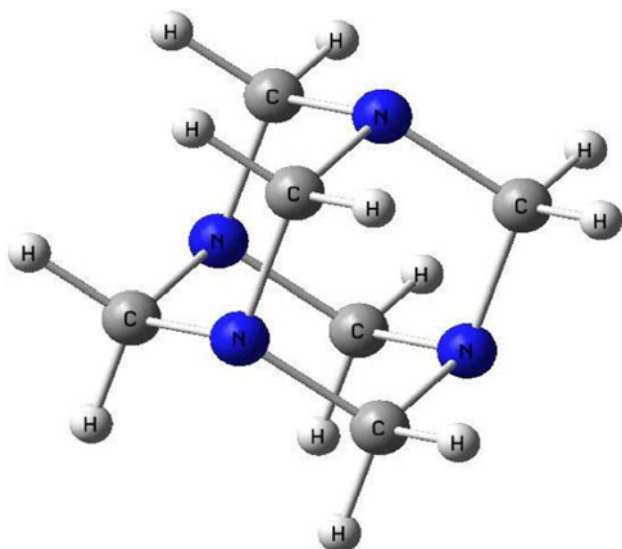
Fig. 1 The molecular structure of hexamine

etc. The most common production method of hexamine in industrial scale is the reaction of formaldehyde with ammonia or ammonium salts. This reaction produces considerable amount of heat and water (Alamdari and Tabkhi 2004). Hexamine wastewater that is formed in the production of hexamine usually contains residual amounts of hexamine and ammonium. The wastewater is initially dosed with sodium hydroxide to raise the solution pH to 11 or higher to convert ammonium ions to ammonia gas. The steam stripper separates the ammonia from the solution. Hexamine has a very stable structure which made the molecule recalcitrant to oxidation via methylotrophic pathway reactions so that very few microorganisms in soil can break it to utilize as sole source of carbon, nitrogen and energy (Middelhoven and van Doesburg 2007).

Therefore, hexamine is resistant to biodegradation (Kaszycki and Koloczek 2002), even when sludge has been acclimated for a long period (Hutnan et al. 2005) and it must be considered as a real wastewater pollutant and should not be allowed to enter water resources and soils. The high strength hexamine-containing wastewater is efficiently removed from industrial wastewater by means of a cation exchange resin (Rafati et al. 2010; Saadatjou et al. 2010). This method requires an expensive cation exchanger resin and remains the trace amounts of hexamine in wastewater. Therefore, it is necessary to adsorb the remaining hexamine with a complement method.

This work addresses the adsorption of hexamine solution on powder-activated carbon under a wide range of operating conditions with a combination of experimental and modeling studies. Equilibrium isotherm parameters are used to describe the experimental sorption data. Isotherm parameters are determined using the method of non-linear

least squares. Kinetic parameters are then evaluated and are correlated with process variables such as solution pH, temperature of solution, initial concentration and added inorganic salt concentration.

It is the object of the present study to evaluate adsorption of hexamine by means of activated carbon which is available at an industrially low cost for possible application in treatment of hexamine industrial wastewater. Hexamine wastewater is formed in the production factories of hexamine. It is also produced from the petrochemical factories (Ting et al. 2007).

This research was carried out in Research and Development Department of Sina Chemical Industries Company, Shiraz, Iran, and was completed in October 2010.

Materials and methods

Apparatus

Weighing of materials was performed using an analytical balance model Sartorius MCBA 100 with precision of ±0.0001 g. A Shimadzu spectrophotometer model 160U was used for spectrophotometric measurement of hexamine. For pH measurements, a Metrohm type 691 pH-meter was used.

Chemicals

Ethyl acetoacetate, *n*-propanol, sodium chloride, potassium iodide and sodium phosphate buffer were purchased from Merck and were of analytical grade. The hexamine powder (99.5 %) was from Sina Chemical Industries Co. (Iran). It was recrystallized from ethanol and dried at 60 °C to constant weight and used for preparation of standard solutions of hexamine. The activated carbon powder type YL-200-II was used for adsorbing of hexamine. Characteristics of activated carbon are shown in Table 1. The usable fraction of activated carbon was prepared by boiling in distilled water for 30 min, cooling and settling then washing three times in distilled water to remove fines and impurities that may have desorbed from the activated carbon. All activated carbon samples were then placed in glass drying pars and dried in an oven at 105 °C for 24 h and were used after cooling.

Table 1 Properties of activated carbon used in the investigation

Type	Surface area (m²/g)	Average pore diameter (nm)	Particle size 200 mesh (%)	Iodine number
YL-200-II	920	2.0	≥90	900

Procedures

Adsorption studies

Aqueous-phase adsorption of hexamine on activated carbon was conducted by placing 50 mL hexamine solution in contact with 1 g activated carbon packed in a fixed bed glass column having an inner diameter of 2 cm under different operating conditions of inlet solution concentration, period of contact time of hexamine and activated carbon, temperature of hexamine solution, hexamine solution pH and added salt concentrations. In all experiments, a control sample of hexamine solution was used without activated carbon to eliminate possible adsorption of hexamine on glass column. Then, the remaining hexamine concentration in the eluate was measured spectrophotometrically and was compared with the amount obtained for control sample.

In the equilibrium studies, a constant adsorbent dose was used while the initial solution concentration was varied. The equilibrium isotherms were determined at pH 13 and at temperature of 5 °C. The equilibrium time for adsorption of hexamine was determined to be 60 min. The procedures of kinetic experiments were identical to those of equilibrium tests. The aqueous samples were taken at preset time intervals, and the concentrations of hexamine were measured. The detail conditions of experiments are shown in the figure legends.

Determination of hexamine

An accurately weighed quantity of hexamine was dissolved in distilled water to prepare a stock solution (1,000 mg/L). Experimental solutions of the desired concentrations were obtained by successive dilutions. Determination of hexamine was performed based on the absorption measurement of Hantzsch ethyl ester, formed upon reacting ethyl acetoacetate with hexamine, using a UV-spectrophotometer at 370 nm (Balasubramanian et al. 2003). The calibration curve for hexamine was linear from 1 to 130 mg/L ($R^2 = 0.99$). The remaining concentration of hexamine in the eluate was determined based on the prepared calibration curve.

The amount of hexamine adsorbed onto the sorbent in mg/g at time, t was calculated using the following equation:

$$q_t = \frac{(C_0 - C_t)V}{m}, \tag{1}$$

where C_t is the concentration of hexamine in the solution at a given time in mg/L, C_0 is initial hexamine concentration in mg/L, m is the amount of sorbent mass in grams, and V is the solution volume in L.

Mathematical modeling

The isotherm and kinetic models were used to describe the sorption data generated from hexamine sorption by activated carbon. The isotherm and kinetic models parameters were determined by non-linear error functions minimizing using the solver add-in with Microsoft's spreadsheet, Excel. Chi-square statistic and the residual root mean square error (RMSE) were used to evaluate the fitness of isotherm and kinetic equations to the experimental data. The Chi-square test statistic is the sum of the squares of the differences between the experimental data and data obtained by calculating from models. The equivalent mathematical statement is:

$$\chi^2 = \sum_{i=1}^{N} \frac{\left(q_{e,\exp.} - q_{e,calc.}\right)^2}{q_{e,calc.}}, \tag{2}$$

where $q_{e,\exp.}$ and $q_{e,calc.}$ are the equilibrium capacity obtained by experimental data and calculating from the model in mg/g, respectively. If data from the model are similar to the experimental data, χ^2 will be a small number; if they are different, χ^2 will be a large number. It is better to also analyze the data set using RMSE to confirm the best fit isotherm for the sorption system. RMSE can be defined as follows:

$$\text{RMSE} = \sqrt{\frac{1}{N-2} \sum_{i=1}^{N} \left(q_{e,\exp.} - q_{e,calc.}\right)^2} \times 100. \tag{3}$$

Results and discussion

Adsorption isotherm models

Equilibrium isotherm equations are used to describe the experimental sorption data. The equation parameters and the underlying thermodynamic assumptions of these equilibrium models often provide some insight into the sorption mechanisms and the surface properties and affinities of the sorbent. The most common isotherms for describing solid–liquid sorption systems are the Langmuir, the Freundlich, the Redlich–Peterson and the Toth isotherms.

The Freundlich isotherm (Freundlich 1906), the earliest known relationship describing the sorption equation, was applied for the adsorption data at equilibrium time. This empirical isotherm based on sorption onto heterogeneous surface is expressed by the following equation:

$$q_e = K_F C_e^{1/n}, \tag{4}$$

where C_e is the equilibrium concentration in mg/L and q_e is the amount of hexamine adsorbed per unit mass of the adsorbent in mg/g. K_F is considered as the capacity factor
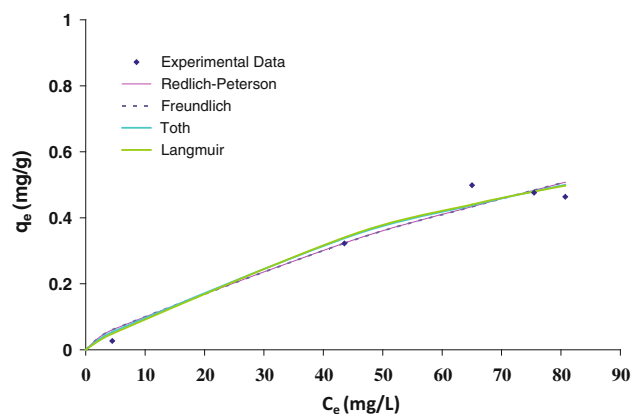
Fig. 2 Langmuir, Freundlich, Redlich–Peterson and Toth isotherms for the adsorption of hexamine by activated carbon ($V = 50$ mL, sorbent dosage = 1 g, pH = 13 and $T = 5$ °C)

Table 2 Parameters of the isotherm models for the adsorption of hexamine onto activated carbon

Model	Parameter	Value	$\chi2$	RMSE
Langmuir	q_m	1.080	0.021	3.678
	K_a	0.011		
Freundlich	K_F	0.020	0.033	4.318
	$1/n$	0.736		
Redlich–Peterson	A	0.129	0.032	4.295
	B	5.569		
	g	0.285		
Toth	K_t	0.066	0.026	3.893
	a_t	23.193		
	$1/t$	0.509		

of the adsorbent in (mg/g) (mg/L)^n while the exponential term $1/n$ represents the heterogeneity of the adsorption surface and is related to the magnitude of the adsorption driving force (El-Sayed and Bandosz 2003).

Figure 2 shows the experimental aqueous-phase adsorption isotherms for hexamine/activated carbon system at temperature solution of 5 °C along with the curve fitted by Freundlich isotherm model. The Freundlich parameters are also shown in Table 2.

The Langmuir (Langmuir 1916) equation which is valid for monolayer sorption onto a surface with a finite number of identical sites is given by Eq. (5):

$$q_e = \frac{q_m K_a C_e}{1 + K_a C_e},\tag{5}$$

where q_m is the maximum amount of adsorbed hexamine per unit mass of sorbent when the saturation is attained, K_a is the Langmuir constant related to the energy of adsorption in L/mg. Figure 2 shows the experimental data and the

non-linear curve fitting by Langmuir model. The parameter of the model, q_m and K_a, is listed in Table 2.

The Redlich–Peterson isotherm (Redlich and Peterson 1959) contains three parameters and involves the features of both the Langmuir and the Freundlich isotherms. It can be described as follows:

$$q_e = \frac{AC_e}{1 + BC_e^g}\tag{6}$$

Three isotherm parameters, A, B and g ($0 < g < 1$), were evaluated using solver tool of Excel software and are presented in Table 2. Figure 2 shows the experimental data and the non-linear curve fitting by Redlich–Peterson model.

Toth isotherm has proven useful in describing sorption in heterogeneous systems such as phenolic compounds on carbon (Ho et al. 2002). It can be represented by the following equation:

$$q_e = \frac{K_t C_e}{(a_t + C_e)^{1/t}}\tag{7}$$

Examination of isotherm plots (Fig. 2) shows Langmuir and Toth isotherm more accurately describe the sorption behavior of hexamine on activated carbon. Since the method used to derive the isotherm parameters minimizes the Chi-square statistic and RMSE, the Langmuir and Toth isotherms exhibit low values that they produces a better fit.

The values of maximum adsorption capacity (q_m) and K_a determined using Langmuir model are 1.080 mg/g and 0.011 L/mg, respectively. The value of K_a is the constant representing the affinity of sorbate to sorbent. The adsorption data obeyed both Langmuir and Toth models exhibiting heterogeneous surface conditions and monolayer adsorption. The Langmuir model generally represents chemisorption on a set of well-defined localized adsorption sites having the same adsorption energy, independent of surface coverage and with no interaction between adsorbed molecules (Behera et al. 2008; Ncibi et al. 2007). It seems that the chemisorption process is dominant over the physical process in the adsorption of hexamine onto activated carbon since there is a sharing of the electron pairs of the nitrogen atoms of hexamine with the surface of activated carbon.

The Freundlich exponent, $1/n$, should have values lying in the range of 0.1–1 for classification as favorable adsorption (Freundlich 1906) and it has been shown that this value between 0.1 and 0.5 indicates beneficial adsorption (Mckay et al. 1982). The value of $1/n$ for the adsorption of most organic compounds by activated carbon is less than 1. When $1/n$ is close to 1 (steep slopes) (in the case of hexamine is 0.736), it indicates a high adsorptive capacity at high equilibrium liquid phase concentrations and rapidly diminishing capacities at lower equilibrium

Table 3 Pseudo-first order sorption kinetic model constants at various pH conditions

pH	8	11	13	14
q_e (mg/g)	0.123	0.175	0.192	0.193
k_1 (min^{-1})	0.038	0.084	0.133	0.209
χ^2	0.0044	0.0017	0.00001	0.000003
RMSE	1.346	1.216	0.117	0.054

Table 4 Pseudo-first order sorption kinetic model constants at different temperatures of hexamine solution

Temperature (°C)	5	10	15	20	40	60
q_e (mg/g)	0.276	0.241	0.216	0.191	0.139	0.127
k_1 (min^{-1})	0.121	0.125	0.169	0.142	0.078	0.035
χ^2	0.0008	0.0006	0.0002	0.003	0.0003	0.0001
RMSE	0.104	0.822	0.463	0.551	0.475	0.235

Table 5 Pseudo-first order sorption kinetic model constants at different initial concentrations of hexamine

Initial concentration of hexamine (mg/L)	5	20	50	75	85	90
q_e (mg/g)	0.027	0.114	0.315	0.512	0.494	0.480
k_1 (min^{-1})	0.128	0.143	0.112	0.133	0.093	0.100
χ^2	0.00003	0.0003	0.0016	0.0041	0.0015	0.0043
RMSE	0.069	0.434	1.564	3.247	1.897	3.199

concentrations. The adsorption system approaches to chemical process and distances from physical process whatever the value of n approaches to unity and below unity (Behera et al. 2008). The value of n in this system (1.36) represents the adsorption process is combination of chemisorption as dominant and physical adsorption.

As it is observed from Fig. 2 and Table 2, the value of g in the Redlich–Peterson isotherm is much smaller than 1, which means the isotherm has been approached the Freundlich but not the Langmuir isotherm. The Chi-square statistic and RMSE values are presented in Table 2, indicating that the Freundlich and Redlich–Peterson models showed lower correlation with the experimental adsorption data compared to the Langmuir model.

Adsorption kinetic model

For evaluating the adsorption kinetic of hexamine, the Lagergren pseudo-first order kinetic model was used that is expressed as Eq. (8) (Annadurai et al. 2002):

$$q_t = q_e - q_e 10^{\frac{-k_1 t}{2.303}}, \tag{8}$$

where q_t and q_e are the amount of hexamine adsorbed (mg/g) at any time and equilibrium time, respectively, and k_1 is the rate constant in min^{-1}. q_e and k_1 were determined in various hexamine solution pH, temperature of hexamine solution, initial solution concentration and added salt concentration.

Adsorption kinetic study of hexamine shows that Lagergren equation could fit well over the range of contact times and describe the rated of hexamine sorption onto activated carbon in various conditions. As can be seen from Tables 3, 4, 5 and 6, the values of Chi-square statistic and RMSE of the pseudo-first order Lagergren model are very low.

Effect of solution pH

To investigate the role of surface chemistry of activated carbon on the adsorption capacity of hexamine, the effect of solution pH was studied. The experiments were carried out by varying the initial pH from 8 to 14, under a constant
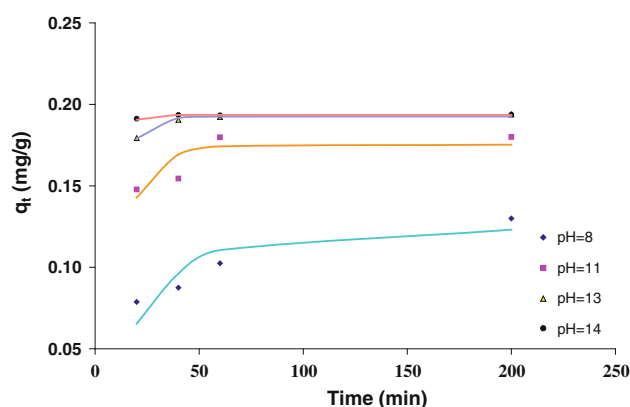
Table 6 Pseudo-first order sorption kinetic model constants at different concentrations of added salts

Concentration of added salt (M)	1×10^{-4}	2×10^{-4}	3×10^{-4}	4×10^{-4}	5×10^{-4}
q_e (mg/g)					
NaCl	1.072	1.043	1.021	0.896	0.679
KI	1.073	1.034	1.011	0.899	0.670
k_1 (min^{-1})					
NaCl	0.186	0.170	0.129	0.139	0.122
KI	0.184	0.158	0.130	0.144	0.110
χ^2					
NaCl	0.0003	0.00006	0.0018	0.0016	0.0007
KI	0.00009	0.00008	0.00225	0.00133	0.00008
RMSE					
NaCl	1.285	0.585	3.061	2.661	1.505
KI	0.717	0.654	3.368	2.449	0.536

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Fig. 3 Pseudo-first order sorption kinetics of hexamine onto activated carbon at various pH conditions. The *solid lines* indicate the fit to Lagergren kinetic model ($C_0 = 50$ mg/L, $V = 50$ mL, sorbent dosage $= 1$ g and $T = 25$ °C)

initial hexamine concentration of 50 mg/L, and activated carbon dosage of 1 g. The acidic pHs were not studied because hexamine is hydrolyzed to formaldehyde and ammonia under acidic conditions (Hutnan et al. 2005). The experimental data and fitted experimental data by kinetic model are shown in Fig. 3. The kinetic model parameters and their corresponding Chi-square and RMSE values are shown in Table 3.

The effect of solution pH on the adsorption of hexamine (Fig. 3) shows the amount of adsorption increases when the pH is increased. Similar results of pH effect were also reported for the adsorption of amine bases including aniline ($pK_a = 4.63$), m-phenylenediamine (pK_a's $= 2.65$ and 4.88) and quinine (pK_a's $= 4.2$ and 8.8) on activated carbon (Cooney and Wijaya 1987). The pK_a of hexamine is 5.05 (Strom and Jun 1986). It can be concluded that the neutral form of hexamine adsorbs more extensively than its ionized form. The adsorption of α-picoline ($pK_b = 8.0$) from aqueous solution has been studied at different pHs onto the activated carbon. It was observed that adsorption increased at pH > 8 (Seidel and Radeke 1990). The adsorption of hexamine also increased at pH > 8 that is close to the pK_b of hexamine, 9.0. Therefore, it may be postulated that the more adsorption of weak amine bases can be attained at pHs greater than pK_b of amine. The kinetic study showed increasing the pH value from 8 to 14 increased the adsorption rate of hexamine about 5.5 times (Table 3). Probably, the interactions of Na$^+$ cations from NaOH with hexamine molecules increase the adsorption rate of hexamine toward the negatively charged carbon surface and hence, the adsorption capacity increases. In the real wastewater, the pH of solution after removal of ammonium by NaOH is greater than 11 and is sufficient for the adsorption of hexamine. This explanation is consistent with the chemisorption of hexamine.

Effect of temperature of hexamine solution

The dependence of the rate of adsorption and adsorption capacity on the temperature of hexamine solution is shown in Fig. 4. The values of q_e and k_1 are listed in Table 4.

The effect of temperature study showed a decrease of the adsorption capacity as temperature was increased from 5 to 60 °C and an increase of the rate of adsorption as temperature was increased from 5 to 15 °C (Table 4). Usually, the adsorption process is combination of chemisorptions (most frequently irreversible adsorption) and physical adsorption (most probably reversible adsorption). Physical adsorption capacity decreases with increasing temperature due to breaking of weak bonds. Since the adsorption rate is diffusion limited process, the rate of adsorption of the organic from solution increases as the temperature increases. At higher temperatures, however, high heat energy to the molecule enables to break off the weak adsorption potential.

Effect of initial concentration

The experimental results of the sorption of hexamine from aqueous solutions at various initial concentrations on activated carbon are shown in Fig. 5. The non-linear pseudo-first order Lagergren models are also presented in Fig. 5. The parameters of kinetic model and their corresponding Chi-square and RMSE values are shown in Table 5. The equilibrium sorption capacity of activated carbon increased from 0.027 to 0.512 mg/g with an increase in the concentration of hexamine from 5 to 75 mg/L with an adsorbent dose of 1 g and pH of 13. This is due to the acceleration of hexamine diffusion from solutions at higher concentrations (Ozacar 2003; Sun and Xu 1997).

Effect of added salts concentrations

The effect of adding 1×10^{-4}–5×10^{-4} M NaCl and KI salts to solutions of hexamine at pH 13 prior to adsorption onto activated carbon are shown in Figs. 6 and 7, respectively. The values of q_e and k_1, obtained from the fitting of experimental data by kinetic model (Eq. 8) and their corresponding Chi-square and RMSE values, are listed in Table 6.

The results found from the effect of added salt showed the presence of salt significantly increased the adsorption capacity and rate of adsorption of hexamine onto activated carbon as the salt was added in the concentrations of less than 3×10^{-4} M to hexamine solution (Table 6). At the high pH of 13, at which the adsorption studies were conducted, the carbon surface is negatively charged (Arafat et al. 1999) and hexamine ($pK_a = 5.05$) is neutral but containing partial negatively charged nitrogen atoms (due
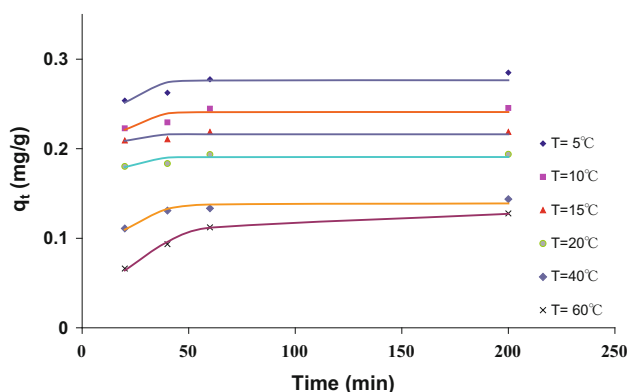
Fig. 4 Pseudo-first order sorption kinetics of hexamine onto activated carbon at different temperatures of hexamine solution. The *solid lines* indicate the fit to Lagergren kinetic model ($C_0 = 50$ mg/L, $V = 50$ mL, sorbent dosage = 1 g and pH = 13)
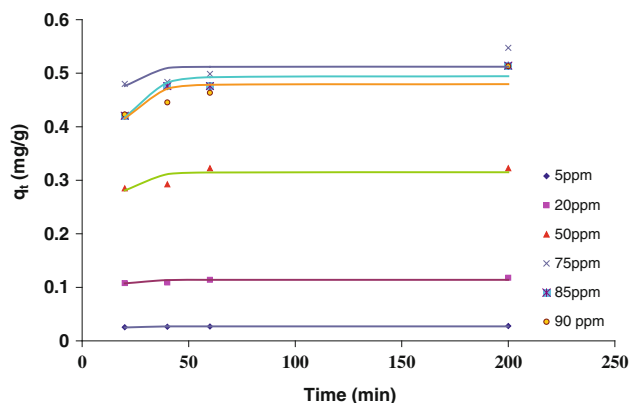

Fig. 6 Pseudo-first order sorption kinetics of hexamine onto activated carbon at different concentrations of NaCl as added salt. The *solid lines* indicate the fit to Lagergren kinetic model ($C_0 = 75$ mg/L, $V = 50$ mL, sorbent dosage = 1 g, pH = 13 and $T = 5°$)

Fig. 5 Pseudo-first order sorption kinetics of hexamine onto activated carbon at different initial concentrations of hexamine. The *solid lines* indicate the fit to Lagergren kinetic model ($V = 50$ mL, sorbent dosage = 1 g, pH = 13 and $T = 5$ °C)

Fig. 7 Pseudo-first order sorption kinetics of hexamine onto activated carbon at different concentrations of KI as added salt. The *solid lines* indicate the fit to Lagergren kinetic model ($C_0 = 75$ mg/L, $V = 50$ mL, sorbent dosage = 1 g, pH = 13 and $T = 5$ °C)

to the existence of the non-bonding electron pairs). The presence of cations in solution causes a partial neutralization of the negative charges on the carbon surface subsequently enabling it to adsorb more molecules of hexamine since the attractive forces are increased between the adsorbed cations and the partial negatively charged nitrogen atoms of hexamine. This explanation is consistent with the Langmuir model that implies the adsorption process is chemical. The adsorption capacity decreases with increasing salt concentration. This phenomenon has also been observed for adsorption of ibuprofen on activated carbon on adding NaCl salt at pH 3 the adsorbate of which is neutral. The reason of this has been explained based on the reducing all the electrostatic interactions, both attractive and repulsive by increasing the ionic strength (i.e. increasing the salt concentration) of the solution (Behera et al. 2012).
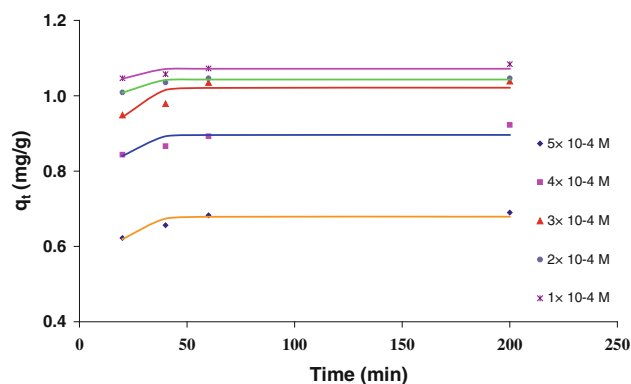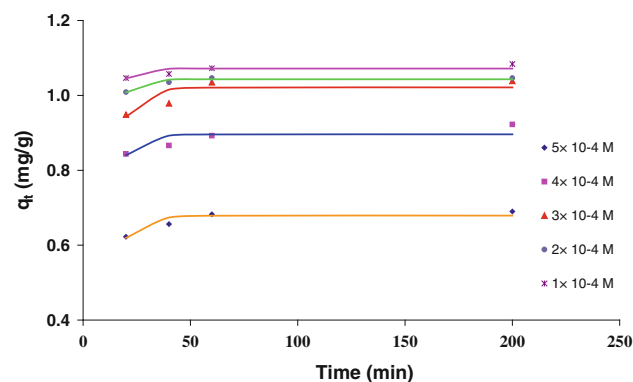
Conclusion

The results of the present investigation showed that the activated carbon has potential for the adsorption of hexamine and can be a low-cost sorbent for removing low concentrations of hexamine from industrial hexamine wastewater. The present work suggests that the temperature, pH solution and added salt concentration ranges during practical use can be changed accordingly on good or poor performances of the adsorbent. The addition of low concentration of salt significantly increases the adsorption capacity of activated carbon in adsorbing hexamine from aqueous solutions.

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