

Polyaniline coated on the Sawdust Nanocomposites (PAn/SD) for the Removal of Hg (II) ions from Aqueous Solutions

Mazyar Sharifzadeh baei, and Hossein Esfandian

Abstract—The aim of this research work is to investigate sorption characteristic of polyaniline coated on the sawdust (PAn/SD) for the removal of Hg (II) ions from aqueous solutions. The sorption of Hg (II) ions by batch method is carried out. The optimum conditions of sorption were found to be: a PAn/SD dose of 0.4 g in 100 ml of Hg (II), contact time of 25 min, pH and temperature 10 and 20 °C respectively. It was found that temperature has a negative effect on the removal efficiency. Three equations, i.e. Morris–Weber, Lagergren and pseudo second order have been tested to track the kinetics of removal process. The kinetic data indicated that the adsorption process was controlled by pseudo-second-order equation. The Langmuir, Freundlich and D-R are subjected to sorption data to estimate sorption capacity, intensity and energy. The data is fitted with Freundlich. For the desorption experiments, several solvents (alkaline, bases and water) have been used. It was achieved 75 % desorption efficiency using HCl.

Keywords—Hg (II), polyaniline, sawdust, removal, isotherm, thermodynamic.

I. INTRODUCTION

ENVIRONMENTAL contamination due to mercury is caused by several industries, petrochemicals, mining, painting, and also by agricultural sources such as fertilizers and fungicidal sprays [1,2]. The toxicity of mercury depends strongly on its redox state. Mercury and its compounds are cumulative toxins and in small quantities are hazardous to human health [3]. The major effects of mercury poisoning manifest as neurological and renal disturbances as it can easily pass the blood brain barrier and affect on the brain. High concentration of Hg(II) cause impairment of pulmonary function and kidney. Consequently removal of mercury in water and wastewater assumes importance [1]. A number of methods for removal of mercury (II) from aqueous solution are available such as biological treatment [4], electrocoagulation [5] and membrane filtration [6-8].

It is known that adsorption is one of the most efficient methods for removal of heavy metals from water and wastewater and adsorption of Hg (II) from aqueous solutions on adsorbents has been investigated [9-19]. The

ion exchange capacities of conducting polymers were well understood and it was found to depend on the polymerization conditions, the type and size of the dopants incorporated during the polymerization process as well as on the ions present in the electrolyte solution, the polymer thickness and ageing of the polymer [20]. Review of the literature revealed that polypyrrole synthesized in solutions with small dopants such as Cl^- , ClO_4^- , NO_3^- , etc., mainly exhibits anion-exchanger behavior due to the high mobility of these ions in the polymer matrix. While under certain conditions cation exchange was also found to take place with large dopants like polyvinylsulfonate and polystyrenesulfonate, due to immobility of these ions in the polymer matrix [21]. Adsorption of metal ions by several functionalized polymers based on amines derivatives such as polyacrylonitrile fibers, ethylenediamine, polyacrylamides, poly-4-vinylpyridine, polyethyleneimine, aniline formaldehyde condensate, etc., have been reported [22–26]. Chakraborty and coworkers have investigated one amine-based polymer, short-chain PAn coated on jute fiber for the removal of chromium in batch mode and Fixed-bed column [27-29].

The effect of polyaniline and polypyrrole composites and the influence of type and concentration of stabilizer, pH of solution, and type of adsorbent on lead salt removal from aqueous solution were studied [30]. In other paper, fixed bed column was used for the removal of some anions and heavy metals from cotton textile industry wastewater using PAn and its nanocomposite containing nanometer-size Fe_3O_4 [31].

In this study, removal of Hg(II) by PAn/SD is studied. The effects of dosage of adsorbent, contact time, temperature, and pH value on sorption of Hg(II) are investigated in detail, also adsorption isotherms, thermodynamic parameters and adsorption kinetics are obtained.

II. MATERIALS AND METHODS

Materials

A stock solution of Hg(II) (1000 mg/L) was prepared in milli-Q water with HgCl_2 . It was then diluted to prepare solutions of the desired concentrations. All the chemicals used throughout this study were of analytical grade either from Merck. pH of the solution monitored by adding 0.5 M HCl and 0.5 M NaOH solution as per required pH value. Aniline (Merck) was purified by vacuum distillation for polymerization.

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III. RESULTS AND DISCUSSION

Effect of contact pH

The pH value of the aqueous solution is an important controlling parameter in the adsorption process. These pH values affect the surface charge of adsorbent, the degree of ionization and speciation of adsorbate during adsorption. Thus the effect of pH (H^+ ion concentration) in the solutions on the adsorption percentage of mercury ions was studied at different pH ranging from 2 to 10 (Fig.1)

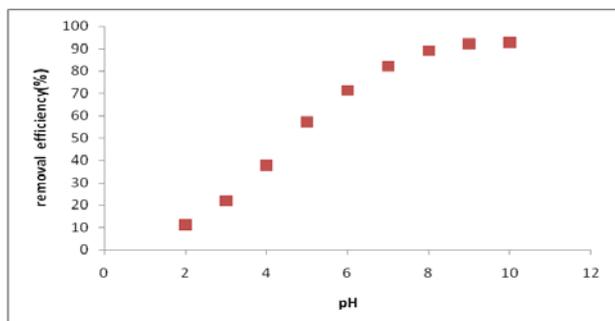


Fig. 1. The effect of pH on the removal efficiency (the initial concentration, rotating speed, contact time, volume of solution and amount of adsorbent was 50 mg/l, 400 rpm, 25 min, 100 ml and 0.4 g respectively).

As our results show, with increasing the pH of solution, sorption of mercuric ion is increased and in acidic media ($pH < 4$) the sorption of metal ions is decreased substantially. With increasing the pH of treated solution, the polymer is changed into undoped form, then free amine or imine groups in the polymer will be available for metal chelating, so the sorption of Hg^{2+} ion is increased considerably. At acidic pH values, polyaniline is changed into acid doped state ($-N$ groups are protonated), so the polymer can not function as a ligand or chelating agent, therefore, the metal uptake is not occurred [33].

Effect of contact time

Fig.2 shows the effect of contact time on sorption of $Hg(II)$ by PAn/SD. For these cases, initial $Hg(II)$ concentration was of 50 mg/L, pH of 10 was used for $Hg(II)$.

Also PAn/SD dose of 0.4 g in 100 ml were used. For $Hg(II)$ sorption rate reaches up to 93.12 when contact time is 25 min, and then little change of sorption rate is observed.

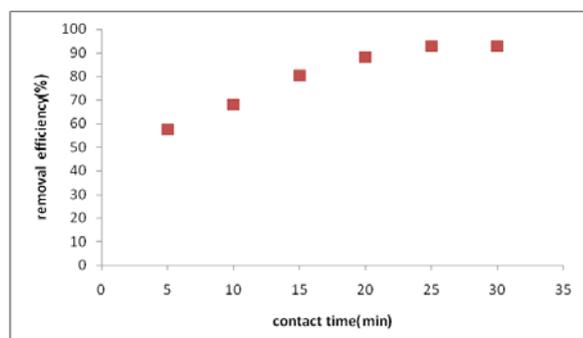


Fig. 2 The effect of contact time on the removal efficiency(the initial concentration, rotating speed, pH, volume of solution and amount of adsorbent was 50 mg/l, 400 rpm, 10, 100 ml and 0.4 g respectively).

This result revealed that adsorption of $Hg(II)$ is fast and the equilibrium was achieved by 25 min of contact time. Taking into account these results, a contact time of 25 min was chosen for further experiments.

Kinetics of sorption

Most of the adsorption transformation processes of various solid phases are time dependent. To understand the dynamic interactions of nitrate with PAn/SD and to predict their fate with time, knowledge of the kinetics of these processes is important [34].

To investigate the change in the concentration of sorbate onto sorbent with shaking time, the kinetic data of $Hg(II)$ ions sorption onto PAn/SD were subjected to Morris-Weber Eq. (6) [35]:

$$q_t = K_{id} (t)^{0.5} + C \quad (6)$$

where q_t is the sorbed concentration of $Hg(II)$ ions at time 't'. The plot of q_t versus $t^{1/2}$ is given in Fig. 3. The value of rate constant of Morris-weber transport, K_{id} , calculated from the slope of the linear plot are shown in Fig 3. The rate constant $K_{id} = 1.698 \text{ min}^{-1}$ was calculated from the slope of the straight line with a correlation factor of 0.991.

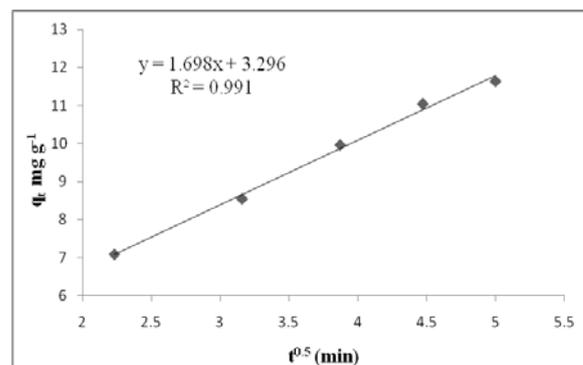


Fig.3. Morris-Weber plot of $Hg(II)$ sorption onto PAn/SD (the initial concentration, rotating speed, pH, volume of solution and amount of adsorbent was 50 mg/l, 400 rpm, 10, 100 ml and 0.4 g respectively).

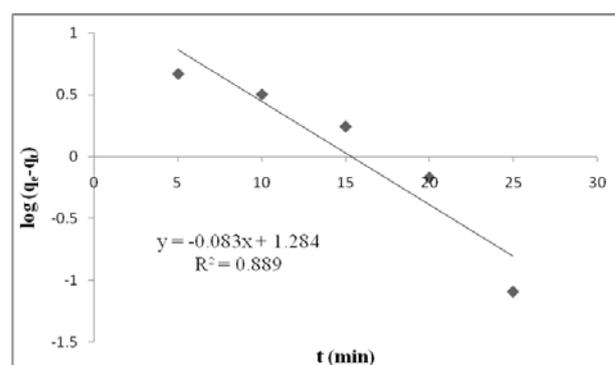


Fig. 4 Validation of Lagergren plot of $Hg(II)$ sorption onto PAn/SD (the initial concentration, rotating speed, pH, volume of solution and amount of adsorbent was 50 mg/l, 400 rpm, 10, 100 ml and 0.4 g respectively).

The first step stage can be attributed to the diffusion of adsorbate through the solution to the external surface of the adsorbent or the boundary surface diffusion of the sorbate molecules. The second stage describes the gradual sorption, where intraparticle diffusion is rate-limiting, and the third stage is attributed to the final equilibrium due to extremely low sorbate concentration left in solution and the reduction

of interior active sites. The three stages in the plot suggest that the sorption process occurs by surface adsorption and intraparticle diffusion.

In 1898, Lagergren [36] suggested a pseudo-first-order equation for the sorption of liquid/solid system based on solid capacity. It assumes that the rate of change of sorbate uptake with time is directly proportional to the difference in the saturation concentration and the amount of solid uptake with time. The Lagergren equation is the most widely used rate equation in liquid phase sorption. The general equation is expressed as:

$$\log(q_e - q_t) = \log q_e - \left(\frac{K}{2.303}\right)t \tag{7}$$

where q_e is the sorbed concentration at equilibrium and K is the first order rate constant. The linear plot of $\log(q_e - q_t)$ against time 't' (Fig. 4) demonstrates the applicability of the above equation for Hg (II) ions sorption onto PAn/SD. The rate constant $K = 0.184 \text{ min}^{-1}$ was calculated from the slope of the straight line with a correlation factor of 0.889.

The kinetic data of Hg (II) ions sorption onto PAn/SD was subjected to pseudo second order Eq. (8) [37]:

$$\frac{t}{q_t} = \frac{1}{Kq_e^2} + \frac{t}{q_e} \tag{8}$$

The rate constant was calculated from the slope of the straight line (Fig.5). The rate constant $K = 0.034 \text{ min}^{-1}$ was calculated from the slope of the straight line with a correlation factor of 0.996. The kinetic data indicated that the adsorption process was controlled by pseudo-second-order equation.

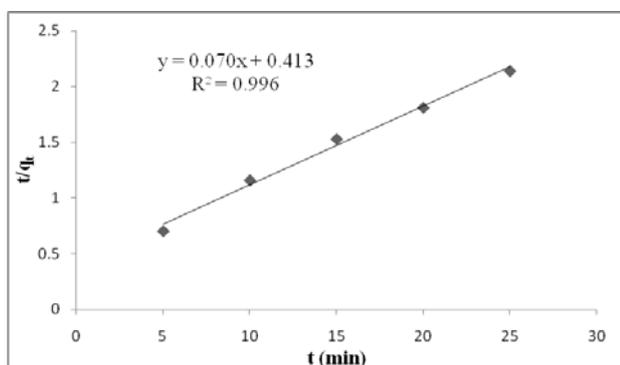


Fig. 5 Pseudo second order plot of Hg (II) sorption onto PAn/SD (the initial concentration, rotating speed, pH, volume of solution and amount of adsorbent was 50 mg/l, 400 rpm, 10, 100 ml and 0.4 g respectively).

Effect of PAn/SD dosage on sorption of Hg (II)

To assess the effect of sorbent dose, different amounts (0.1–0.5 g) of sorbent was suspended in 100 mL Hg (II) solution (50 mg/L) under optimized conditions of pH and contact time. The effect of adsorbent dose on the removal efficiency is shown in Fig. 10. It was observed that the adsorption percentage of Hg (II) onto the PAn/SD increased rapidly with the increasing of adsorbent concentration (Fig. 6). This result is expected because the increase of adsorbent dose leads to greater surface area. When the adsorbent dose was increased from 0.05 g to 0.5g, the percentage of Hg (II) ions adsorption increased from 55.31 to 93.14. At higher Dose of PAn/SD, the equilibrium uptake of Hg (II) did not increase significantly with increasing PAn/SD. The data of Fig. 10 were fitted to Langmuir, Freundlich and Dubnin-

Randkovich (D-R) models in order to examine the models constants adsorption isotherms.

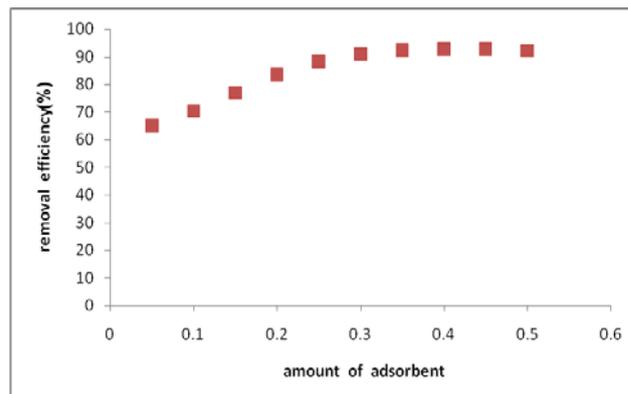


Fig.6. The effect of amount of PAn/SD on the removal efficiency (the initial concentration, rotating speed, pH, volume of solution and contact time was 50 mg/l, 400 rpm, 10, 100 ml and 25 min respectively).

The isotherm model

The adsorption isotherm is based on the assumption that every adsorption site is equivalent and independent of whether or not adjacent sites are occupied. Isotherms show the relationship between metal concentration in solution and the amount of nitrate sorbed on a specific sorbent at a constant temperature.

The Langmuir isotherm model

The Langmuir isotherm model is valid for monolayer adsorption onto surface containing finite number of identical sorption sites which is presented by the following equation (9):

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \tag{9}$$

where q_e is the amount of metal adsorbed per specific amount of adsorbent (mg/g), C_e is equilibrium concentration of the solution (mg/L), and q_m is the maximum amount of metal ions required to form a monolayer (mg/g). The Langmuir equation can be rearranged to linear form for the convenience of plotting and determining the Langmuir constants (K_L) as below. The values of q_m and K_L can be determined from the linear plot of C_{eq}/q_{eq} versus C_{eq} :

$$\frac{C_e}{q_e} = \frac{1}{q_0 \cdot K_L} + \frac{1}{q_0} C_e \tag{10}$$

The equilibrium data were analyzed using the linearized form the Langmuir adsorption isotherm Eq. (10). The Langmuir constants, K_L and monolayer sorption capacity, q_m were calculated from the slope and intercept of the plot between C_e/q_e and C_e (Fig.7).The results and equations are indicated in Fig7 and Table 1. As can be seen, the correlation factor of this equation is low, and it is not suitable for this process.

The Freundlich isotherm model

While Langmuir isotherm assumes that enthalpy of adsorption is independent of the amount adsorbed, the empirical Freundlich equation, based on sorption on heterogeneous surface, can be derived assuming a logarithmic decrease in the enthalpy of adsorption with the increase in the fraction of

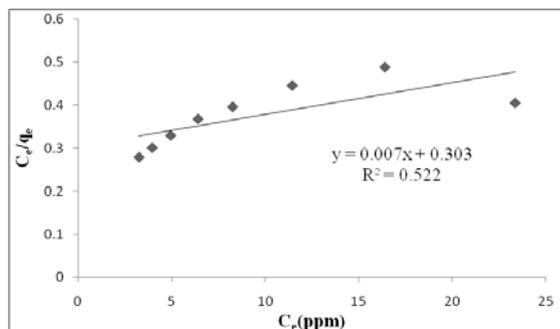


Fig.7. Langmuir sorption isotherm (the initial concentration, rotating speed, pH, volume of solution and contact time was 50 mg/l, 400 rpm, 10, 100 ml and 25 min respectively).

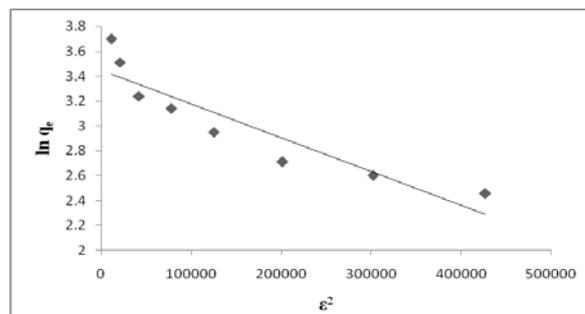


Fig. 8. D-R sorption isotherm (the initial concentration, rotating speed, pH, volume of solution and contact time was 50 mg/l, 400 rpm, 10, 100 ml and 25 min respectively).

TABLE I

THE ISOTHERM PARAMETER FOR THE HG ADSORPTION ON THE PAN/SD

langmuir equation		r^2
$y = 0.007x + 0.303$		0.522
Freundlich parameter		
K_F	N	r^2
4.65	1.36	0.989
D-R parameter		
β	q_m	r^2
3×10^{-6}	8.32	0.849

occupied sites. The Freundlich equation is purely empirical based on sorption on heterogeneous surface and is given by:

$$q_e = K_F (C_e)^{1/n} \tag{11}$$

K_F and $(1/n)$ are the Freundlich constant and adsorption intensity, respectively. Equilibrium constants evaluated from the intercept and the slope, respectively, of the linear plot of $\log q_e$ versus $\log C_e$ based on experimental data. The Freundlich equation can be linearized in logarithmic form for the determination of the Freundlich constants as shown below:

$$\log(q_e) = \log(K_F) + \frac{1}{n} \log(C_e) \tag{12}$$

The slope and the intercept correspond to $(1/n)$ and K_F , respectively. It was revealed that the plot of $\log q_e$ and $\log C_e$ yields a straight line (Fig.8). The results are indicated in Table 1.

The parameter k related to the adsorption density increased with a decrease of adsorbent amount. The meaning of $n > 1.0$ indicates that nitrate was adsorbed favorably by adsorbents at different adsorbent amounts.

The Dubinin–Radushkevick isotherm model

The Dubinin–Radushkevick (D–R) [38-40] isotherm was used to determine the nature of the adsorption process viz. physisorption or chemisorptions.

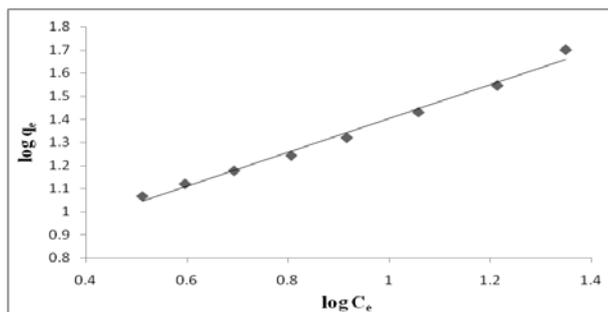


Fig. 8. Freundlich sorption isotherm (the initial concentration, rotating speed, pH, volume of solution and contact time was 50 mg/l, 400 rpm, 10, 100 ml and 25 min respectively).

The linear form of this model is expressed by:

$$\ln(q_e/q_m) = \ln(q_m) - \beta \epsilon^2 \tag{13}$$

Where q_e is the amount of Hg (II) adsorbed per unit dosage of the adsorbent (mg/g), q_m the monolayer capacity, and β is the activity coefficient related to mean sorption energy and ϵ is the Polanyi potential described as

$$\epsilon = RT \ln \left[1 + \left(\frac{1}{C_e} \right) \right] \tag{14}$$

From the plots of $\ln q_e$ versus ϵ^2 (Fig. 9) the values of β , q_m were determined by the slope and intercept of the linear plot. The statistical results along with the isotherm constants are also given in Table 2. As our results show, adsorption of Hg (II) by PAN/SD can be fitted using Freundlich equation also the D-R equation shows considerable correlation factor. Although the Freundlich isotherm provides the information about the surface heterogeneity and the exponential distribution of the surface and their energies, it does not predict any saturation of the surface of the adsorbent by the adsorbate. Hence, infinite surface coverage could be predicted mathematically. In contrast, D–R isotherm relates the heterogeneity of energies close to the adsorbent surface. The quantity can be related to the mean sorption energy, E , which is the free energy for the transfer of 1 mole of metal ions from the infinity to the surface of the adsorbent [41]. The mean free energy of adsorption (E , kJ/mol) can be calculated as follows [42]:

$$E = (2\beta)^{-0.5} \tag{15}$$

TABLE II

THE EFFECT OF TEMPERATURE ON THE REMOVAL EFFICIENCY

Temperature $^{\circ}\text{C}$	Removal efficiency of Hg(II)
20	93.12
30	92.25
40	89.35
50	86.34

It is known that the magnitude of E is useful for estimating the type of adsorption and if this value below 8 kJ/mol the adsorption type can be explained by physical adsorption, and between 8 and 16 kJ/mol the adsorption type can be explained by ion exchange. The values of E found in this study were between 8 and 16 kJ/mol, indicating that the adsorption of nitrate onto PAN/SD was ion exchange in nature. This confirms that anionic exchange and electrostatic attraction are the main mechanisms responsible for the adsorption of nitrate onto PAN/SD [43].

IV. CONCLUSIONS

The PAN/SD showed considerable potential for the removal of Hg (II) from aqueous solutions. The optimum conditions of sorption were found to be: a sorbent dose of 0.4 g in 100 ml of solution. The optimum contact time and pH were 25 min and 10 respectively. The results gained from this study were well described by the theoretical Freundlich. The kinetic data indicated that the adsorption process was controlled by pseudo-second-order equation. The results show the exothermic nature of the adsorption. A regeneration study of the material was also carried out and was found that the PAN/SD can be regenerated and reused using HCl solution.

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