Investigation on Selective Adsorption of Hg(II) Ions Using 4-Vinyl Pyridine Grafted Poly(ethylene terephthalate) Fiber

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ABSTRACT: In the work, poly(ethylene terephthalate) (PET) fibers were grafted with 4-vinyl pyridine (4-VP) monomer using benzoyl peroxide (Bz_2O_2) as initiator in aqueous media. The removal of Hg(II) ions from aqueous solution by the reactive fiber was examined by batch equilibration technique. Effects of various parameters such as pH, graft yield, adsorption time, initial ion concentration, and adsorption temperature on the adsorption amount of metal ions onto reactive fibers were investigated. The optimum pH of Hg(II) was found 3. The maximum adsorption capacity was found as 137.18 mg g⁻¹. Moreover such parameters as the adsorption kinetics, the adsorption iso-

INTRODUCTION

Environmental pollution with heavy metal ions is a significant problem owing to their tendency to accumulate in living organisms and toxicities in relatively low concentration.^{1–3} The presence of mercury in the aquatic environment is known to cause severe health problems in both animals and humans. The main toxicological effects of mercury include neurological damage, paralysis, blindness, and chromosomes breakage.⁴ The conventional treatments used to remove heavy metals from wastewaters are precipitation, coagulation, reduction, solvent extraction, electrochemical separation through membranes, ion exchange, and adsorption. These methods usually concentrate the metal ions into a smaller volume followed by recovery or secure disposal.^{5–8} Adsorption is considered to be an effective and economical method for removal of pollutants from wastewater.9 There are many types of adsorbents that have been studied for the adsorption of ions from aqueous soltherm, desorption time and the selectivity of the reactive fiber were studied. The adsorption kinetics is in better agreement with pseudo-first order kinetics, and the adsorption data are good fit with Freundlich isotherms. The grafted fiber is more selective for Hg(II) ions in the mixed solution of Hg(II)-Ni(II), Hg(II)-Zn(II), and Hg(II)-Ni(II)-Zn(II) at pH 3. Adsorbed Hg(II) ions were easily desorbed by treating with 1*M* HNO₃ at room temperature. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 1256–1264, 2012

Key words: 4-vinyl pyridine; poly(ethylene terephthalate); adsorption; heavy metal ions; mercury

utions including activated carbon,¹⁰ sawdust,¹¹ sporopollenin,^{12,13} chitosan,¹⁴ peat,¹⁵ cellulose,¹⁶ chelating resins,¹⁷ clay mineral,¹⁸ chelating fibers.¹⁹

Chelating fibers are very useful since they have higher selectivity and larger adsorption capacities than other adsorbents and they are also easy to regenerate. This is mainly attributed to the relatively large external specific surface areas, high adsorption kinetics, introduction of suitable functional groups, and low cost of these polymer fibers.^{20,21}

PET fiber is one of the most important synthetic fibers used in the textile industry. The PET fiber has good resistance to most strong acids, oxidizing agents, sunlight, and microorganisms. However, they are hydrophobic in nature and do not contain chemically reactive groups.²² Certain desirable functional groups can be imparted to the PET surface by grafting with different monomers, such as 4-VP,²³ 4-VP/2-hydroxyethylmethacrylate,^{22,24} *N*-vinyl-2 pyrrolidone,²⁵ acrylic acid,²⁶ methacrylic acid,²⁷ acrylamide,²⁸ acrylonitrile,²⁹ and glycidyl methacrylate.³⁰ It was determined that polymeric materials having functional groups such as carboxylic acid, amine, hydroxyl, and epoxy groups, could be used as complexing agents for the removal of metal ions from aqueous solutions.³¹ Especially, thiol and amide groups have been used in the design of polymeric sorbents for binding mercury ion selectively.³²

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The reactive fibers was prepared by grafting 4-VP monomer onto PET fibers in our previous work.²³ In this study that material is used as an adsorbent for selective removal of Hg(II) from aqueous solution by a batch equilibration technique. The adsorption properties, including effects of grafting yield, pH value, initial ion concentrations, adsorption temperature on adsorption, and selectivity was investigated.

EXPERIMENTAL

Materials

The PET fibers (122 dTex, middle drawing) used in these experiments were provided by SASA (Adana, Turkey). The fibers samples were Soxhlet-extracted until constant weight (for 6 h) with acetone and dried in a vacuum oven at 50°C. 4-VP was purified by vacuum distillation. Bz_2O_2 was twice precipitated from chloroform in methanol and dried in a vacuum oven at 25°C for 2 days. Analytical grade reagent of 1,2-dichloroethane, mercury(II) nitrate, nickel(II) nitrate, zinc(II) nitrate standart solutions, and nitric acid were purchased from Merck. pH values were adjusted with buffer solution of glycine-glycine HCI (pH 1–3), CH₃COOH-CH₃COONa (pH 4–5), and NaH₂PO₄-Na₂HPO₄ (pH 6–8). Other reagents were used as supplied. All reagents were Merck products.

Polymerization procedure

The reactive fibers were prepared by graft copolymerization of 4-VP monomer onto PET fibers by using Bz_2O_2 as an initiator. The fiber samples (0.3 \pm 0.01 g) were dipped into dichloroethane (50 mL) for 2 h at 90°C. After treatment, solvent on the fibers were removed by blotting between a filter paper and put into the polymerization medium. Polymerization was carried out in a thermostated 50-mL tube under reflux. The mixture containing the PET fibers samples (0.3 \pm 0.01 g), appropriate amount of 4-VP and Bz_2O_2 at required concentration in 2mL acetone was made up to 20 mL with deionized water. The mixture was immediately placed into the water bath adjusted to the polymerization temperature. At the end of the predetermined polymerization time, the grafted fibers were taken out. Residual solvent, monomers, and free homopolymers were removed by Soxhlet-extracting the PET fibers in methanol for 96 h. The grafted fibers were then vacuum-dried at 50°C for 72 h and weighed.

The graft yield was calculated from the weight increase in grafted fibers as follows:

$$GY(\%) = \left(\frac{W_g - W_i}{W_i}\right) \times 100 \tag{1}$$

where W_i and W_g denote the weights of the original (ungrafted) and grafted PET fibers, respectively.

Scanning electron microscopy

SEM studies of the original and 4-VP grafted PET fibers coated with gold were performed using a JOEL Model JSM 5600 microscope.

Adsorption procedure

Adsorption experiments were carried out in a batch system at 25°C and 125 rpm by contacting 25 mL heavy metal ion solutions at a specific concentration. The pH of the heavy metal ion solution was adjusted with a suitable buffer solution. The adsorbent dose was fixed as 0.1 g throughout all experiments. The heavy metal solution and 4-VP grafted PET fibers were shaken for a predetermined period of time using orbital shaker. After filtration of the solution the metal ion concentration of the filtrates was measured by a PerkinElmer AAnalyst 400 flame atomic absorption spectrometer model equipped with deuterium lamp background correction, hollow cathode lamps (HCL) and air-acetylene burner was used for the determination of the metals.

The adsorption capacity of the 4-VP grafted PET fibers was evaluated by using the following expression:

$$Q = \frac{(C_{\rm o} - C) \times V}{m} \tag{2}$$

where Q is the amount of ion adsorbed onto unit mass of the 4-VP grafted PET fiber (mg g⁻¹), C_o and C are the concentrations of the ion in the initial solution and in aqueous phase, respectively, after treatment for a certain period of time (mg L⁻¹); V is the volume of the aqueous phase (L); and m is the amount of 4-VP grafted PET fiber used (g), respectively.

Desorption of metal ions

Desorption assays were carried out with the metal ion loaded 4-VP grafted PET fibers. Hg(II) ions were recovered by treating with 1M HNO₃ solution and then analyzed by the method mentioned above. Desorption percent was calculated using the following equations:

% Desorption

$$= \frac{\text{Amount of ions (mg) desorbed}}{\text{Absorbed amount of ions (mg) by adsorbant}} \times 100$$
(3)

RESULTS AND DISCUSSION

4-VP Grafted PET fibers were used as an adsorbent in this study. The scanning electron micrographs of



Figure 1 (a) SEM micrograph of ungrafted PET fibers, (b) SEM micrograph of 4-VP grafted PET fibers.

ungrafted and 4-VP grafted PET fibers are shown in Figure 1. It is clear from the SEM results that the ungrafted PET fiber surface [Fig. 1(a)] has a smooth and relatively homogeneous appearance. At grafted PET fibers, the grafted side chain 4-VP seems to form microphages attached to the PET back-bone and causes a heterogeneous appearance in the graft copolymer [Fig. 1(b)], showing proof of grafting.¹³

Effects of pH on adsorption

The pH of the aqueous solution is an important controlling parameter in the adsorption process. The



Figure 2 The pH dependence of metal ions adsorbed by 4-VP grafted PET fibers: temperature: 25°C; [Hg]: 100 ppm; contact time: 120 min; graft yield: 110%.

effect of solution pH on the adsorption of Hg(II) ions was studied by varying the pH of the solutions between 2 and 6 for Hg(II) and the results were presented in Figure 2. The maximum adsorption of Hg(II) is at pH 3.

The relatively higher uptake of Hg(II) at between 2 and 3 pH values was due to the presence of anion complex such as Hg(NO₃)₄⁻². At low pH, Hg(NO₃)₄⁻² ions are the dominant species.³³ The Hg(II)-4-VP is, however, a labile complex.³³ Thus Hg(NO₃)₄⁻² is attached to with electrostatically interaction to the RNH⁺ (pyridinium) groups (Fig. 3). At above 3 of pH, the adsorption capacity declines. The Hg(II) ions get out of the solution at pH > 4 due to formation of precipitate of Hg(OH)₂.³³

To explain the observed behavior of $Hg(NO_3)_4^{-2}$ adsorption with varying pH, it is necessary to examine various mechanisms such as electrostatic interaction, and chemical reaction which are responsible for adsorption on sorbent surface. The solution is



Figure 3 Adsorption of Hg(II) ions on the 4-VP-grafted PET fibers.



Figure 4 Effect of graft yield on adsorption: temperature: 25°C; contact time: 120 min; [Hg]: 100 ppm; pH: 3.

acidified by hydrochloric acid, surface of the grafted PET of positively charge interface will be associated with Cl⁻ ions (at pH 3). Thereby inhibiting the adsorption of Hg(NO₃)₄⁻². There was competition between Cl⁻ (at low pH, high Cl⁻) and Hg(NO₃)₄⁻² for positively charged adsorption sites. However, at the pH, adsorption cannot be yet occurred. On the other hand, pH around 3 concentration of Cl⁻ is low thus Hg(NO₃)₄⁻² interface with adsorbent instead of Cl⁻. The similar results was observed in the following work.³³

For the adsorption of $Hg(NO_3)_4^{-2}$ on the 4-VP grafted PET fibers at pH 3.0, most of the pyridine groups surface of the sorbent was protonated and possessed positive electric charges. The protonated pyridine groups (pyridinium) can therefore attract the $Hg(NO_3)_4^{-2}$ which carried negative electric charges in the solution through the electrostatic interaction. On the other hand, at pH 3, the protonation of the pyridine groups on the 4-VP grafted PET fibers was probably insignificant and the electrostatic interaction would not play an important role in the adsorption of $Hg(NO_3)_4^{-2}$ on the sorbent.

Effect of graft yield

The effect of the graft yield on the adsorbed amount of metal ions was investigated at 25°C while keeping all other conditions constant. The results are shown in Figure 4. The amount of adsorbed ions increased with grafting yield. Ungrafted PET fibers do not contain suitable functional groups and thus cannot interact with heavy metal ions. Adsorption amount of the PET fiber is increased by grafting of the PET fiber with 4-VP monomer and this is due to the functional groups of 4-VP inserted into the fiber structure. The increase in the adsorption with increasing graft yield may be attributed to a higher surface area and more active sites.

Effect of contact time

The effect of contact time on adsorption of Hg(II) ions by 4-VP grafted PET fibers are shown in Figure 5. It is seen that the adsorption takes place rapidly at first, and then levels off (at high initial concentration). The adsorption equilibrium of Hg(II) ions were attained within 120 min. During the adsorption of metal ions, the ions initially reached the boundary layer and then had to diffuse into the grafted PET fibers surface and finally they had to diffuse into the fibrous structure of the adsorbent. Therefore, this event took a relatively longer contact time. The relation between the nature of the polymer and sorption rate is generally complicated by many possible interactions on the surface. Generally the electrostatic interaction surface binding and chemical reaction may be identified as the major adsorption mechanisms. Thus, those groups of 4-VP grafted PET fibers are responsible for the interaction of ions with the fibers.

To evaluate the kinetic mechanism that controls the adsorption process, pseudofirst order, and pseudosecond order were employed to interpret the experimental data. A good correlation of the kinetic



Figure 5 Effect of contact time on adsorption: temperature: 25°C; graft yield: 140%; [Hg]: 100 ppm; pH: 3.

First-Order and Second-Order Rate Constants										
	First-order rate constants			Second-order rate constants						
$Q_{\rm e}$ (exp.) (mg g ⁻¹)	$\frac{k_1}{(\min^{-1})}$	$Q_{\rm e}$ (teheor.) (mg g ⁻¹)	R^2	$(g mg^{-1}min^{-1})$	$Q_{\rm e}$ (teheor.) (mg g ⁻¹)	R^2				
24.46	0.030	28.07	0.993	0.0001	64.51	0.805				

TABLE I

data explains the adsorption mechanism of the metal ions.³⁴

The pseudofirst-order equation was represent by

$$\log(Q_e - Q_t) = \log Q_e - \left(\frac{k_1}{2.303}\right)t$$
 (4)

where Q_t and Q_e are the amount of ions adsorbed (mg g^{-1}) at any time and equilibrium time, respectively, k_1 is the rate constant (min⁻¹).³⁴ According to the adsorption equation, the experimental result shown in Figure 5 can be converted into the plots of $log(Q_e-Q_t)$ versus t. Value of k_1 was calculated from the linear plot of $log(Q_e-Q_t)$ versus t. Experimental and theoretically calculated Q_e values and coefficients related to Lagergren's plots, are given in Table I. As it can be seen from the results the linear correlation coefficients of the plots are good and experimental and calculated Q_e values are in agreement with each other. Therefore, these results suggest that the adsorption of Hg(II) metal ions on grafted PET fiber, is a first-order reaction. The graphical interpretation of the data for the first-order kinetic model is shown in Figure 6.

pseudosecond-order be The equation can expressed as

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \tag{5}$$

where k_2 (g mg⁻¹ min⁻¹) is the adsorption rate constant of pseudosecond order. By plotting t/Q_t versus t, Q_e , and k_2 can be determined from slope and intercept.³⁴ The rate constants (k_2) , correlation coefficients of the plots together with the experimental and theoretical Q_e values are given in Table I.

Effect of ion concentration on adsorption

The effect of initial metal ion concentration on the adsorption efficiency by 4-VP grafted PET fibers was systematically investigated by varying the initial concentration between 25 and 750 mg L^{-1} . The adsorbed amount of ions as a function of initial concentration at optimum pH was shown in Figure 5. It is clear from the figure that as the concentration of the ions increased, adsorption increased rapidly, then, progressively saturating the adsorbent. The

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maximum adsorption performances for Hg(II) were achieved at 137.18 mg g^{-1} using 750 mg L^{-1} metal ions solution. The relation between the nature of the polymer and sorption rate is generally complicated due to many possible interactions on the surface. Commonly, the electrostatic interaction, surface complexation, and ion exchange mechanisms may be identified as the major adsorption mechanisms.35 In particular, the amin groups of 4-VP on the surface of an adsorbent have been reported to be effective in the adsorption of Hg(II) ions.31,36,37 4-VP grafted PET fibers displayed very high adsorption capacity for Hg(II). Therefore, it seems that the 4-VP grafted PET fibers could be a possible alternative to other adsorption methods and an economical industrial adsorbent.

Adsorption isotherm

Adsorption isotherms describe how adsorbates interact with adsorbents.³⁸ The relationship between the amount of metal ions adsorbed and the metal ions concentration remaining in solution is described by an isotherm. The in Figure 7, it can be seen that the adsorption capacity increased with the equilibrium



Figure 6 Pseudofirst-order plots for Hg(II) ions on 4-VP grafted PET fibers.



Figure 7 Effect of initial concentration of Hg(II) ions on adsorption: temperature: 25°C; contact time: 120 min; graft yield: 140%; pH: 3.

concentration of the metal ion in solution, progressively saturating the adsorbent. For interpretation of the adsorption data, the Langmuir³⁹ and Freund-lich⁴⁰ isotherm models were therefore used.

The linear form of the Langmuir isotherm is given by

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_o} + \frac{1}{Q_o b} \tag{6}$$

where C_e is the concentration of Hg(II) ions (mg L⁻¹) at equilibrium, Q_o the monolayer capacity of adsorbent (mg g⁻¹), Q_e is the amount of adsorption at equilibrium and b Langmuir adsorption constant (L mg⁻¹). The plot of Langmuir isotherm is shown in Figure 8. Thus a plot of C_e/Q_e versus C_e should yield a straight line having a slope of Q_o^{-1} and intercept of $(Q_ob)^{-1}$ The relevant experimental data were therefore treated and it was observed that the relationship between C_e/Q_e and C_e is linear, indicating that the adsorption behavior follows the Langmuir adsorption isotherms. The b, Q_o , and correlation coefficients (R^2) values are presented in Table II.

The Freundlich isotherm equation, the most important multilayer adsorption isotherm for heteroge-



Figure 8 Langmuir isotherm plot for Hg(II).

neous surfaces, is described by the following equation

$$\log Q_{\rm e} = \log K_{\rm F} + 1/n \log C_{\rm e} \, \text{gore} \tag{7}$$

where C_e is the concentration of Hg(II) ions (mg L⁻¹) at aquilibrium, K_F the sorption capacity (mg g⁻¹) and n is an empirical parameter. The plot of Freundlich isotherm is shown in Figure 9. Thus, a plot of log Q_e versus log C_e should give a straight line having a slope of 1/n and intercept of K_F . The K_F , n, and correlation coefficients (R^2) values are presented in Table II.

In terms of R^2 values shown in Table II Freundlich equation represents a better fit to the experimental data than the Langmuir equation. The Freundlich isotherm is derived by assuming a heterogeneous surface with a nonuniform distribution of heat of adsorption over the surface whereas in the Langmuir theory, the basic assumption is that the sorption takes place at specific homogeneous sites within the adsorbent. This result also predicts the heterogeneity of the adsorption sites on grafted PET fibers [see Fig. 1(b)].

TABLE II Langmuir and Freundlich Constants For the Adsorption of Hg(II) Ions on 4-VP Grafted PET Fiber

	Lang	gmuir isoterms		Freundlich isotherms			
Metal	$Q_{\rm o}~({\rm mg~g^{-1}})$	b (L mg ⁻¹)	R^2	$K_F (mg g^{-1})$	п	R^2	
Hg(II)	149.25	0.06	0.978	3.09	0.89	0.987	

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Figure 9 Freundlich isotherm plot for Hg(II).

Effect of temperature

The adsorption of Hg(II) ions on 4-VP grafted PET fibers were studied as a function of temperature. It can be recognized that the temperature has some significance on the adsorption amount. As the temperature rises, the diffusion of the ions becomes much easier into the fibers because of the increase in the degree of swelling and therefore, the adsorption amount of metal ions increases as well. Figure 10 shows the effect of contact time and temperature on adsorption of metal ions by 4-VP grafted PET fibers.



Figure 10 Effect of contact time and temperature on adsorption: graft yield: 140%; [Hg]: 100 ppm; pH: 3.

1.34 3,0 3,1 3,2 T-1x103, K-1 Figure 11 was obtained using the data in Figure 10, and the heat of the adsorption values were found as 1.06 kJ mol⁻¹. The values of the heat of adsorption show that it is physical adsorption that takes place in the adsorption process, compared with those of typical chemical reaction of $65-250 \text{ kJ mol}^{-1.41}$.

Selective adsorption of Hg(II) ions

4-VP grafted PET fibers can be used in selective separate Hg(II) ion from binary and ternary mixed solution of metal ions. Figure 12 shows the results of removal of metal ions by grafted fibers from an equimolar solution of Hg(II), Ni(II), and Zn(II) ions at pH 3. Hg(II) showed the higher affinity to 4-VP grafted PET fibers and its uptake was not significantly affected by the presence of Ni(II) and Zn(II) ions in the solution. In the Hg(II)-Ni(II), Hg(II)-Zn(II), and Hg(II)-Ni(II)-Zn(II) systems, the adsorption selectivity exceeds 94% for Hg(II). Adsorption selectivity for Hg(II) at pH 3 was excellent in these binary and ternary systems. It can be applied to the quantitative and selective separation of Hg(II) ion in aqueous systems containing Ni(II) and Zn(II) ions.

Desorption studies

The study of desorption of Hg(II) ions was carried out and shown in Figure 13. The Hg(II) and adsorbed was easily desorbed by treating with 1M HNO₃ at room temperature. As shown in Figure 13, desorption ratio of metal ions increased over time and the desorption ratio of Hg(II) ions was 99%. Its



Figure 11 log *Q* versus to T^{-1} of Hg(II).



Figure 12 Selective adsorption of ions onto 4-VP grafted PET fibers: (a) Hg(II)-Ni(II); (b) Hg(II)-Zn(II); (c) Hg(II)-Ni(II)-Zn(II); pH: 3; ions concentration: 100 ppm; contact time: 120 min; temperature: 25°C; graft yield: 140%.



Figure 13 Desorption profile of Hg(II) ions adsorbed PET fibers: graft yield: 140%; ion concentration: 100 ppm; temperature: 25°C.

completion within 40 min indicates a fast desorption process.

CONCLUSIONS

In this work, adsorbent was prepared by grafting 4-VP monomer onto PET fiber. Effects of various parameters such as grafting yield, pH, adsorption time, initial ion concentration, and adsorption temperature on the adsorption amount of Hg(II) ions onto reactive fibers were investigated. The optimum pH for Hg(II) ion is 3. The adsorption amount of metal ions increased with the increase of grafting yield. The maximum adsorption capacity of Hg(II) is 137.18 mg g^{-1} . The adsorption kinetics is in better agreement with pseudofirst order kinetics and the adsorption data is good fit with Freundlich showing the heterogenous characteristics of the adsorption sites on 4-VP grafted PET fibers. It can be applied to the quantitative and selective separation of Hg(II) ion in aqueous systems containing Ni(II) and Zn(II) ions.

These results suggest that the 4-VP grafted PET fibers are an effective candidate as adsorbent for the removal of Hg(II) ions from wastewater.

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