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Nasir Khalid[a]; Shujaat Ahmad[a]; Seema Naseer Kiani[b]; Jamil Ahmed[b]
[a] NUCLEAR CHEMISTRY DIVISION, PAKISTAN INSTITUTE OF NUCLEAR SCIENCE AND TECHNOLOGY, ISLAMABAD, PAKISTAN [b] DEPARTMENT OF CHEMISTRY, QUAID-I-AZAM UNIVERSITY, ISLAMABAD, PAKISTAN

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Removal of Mercury from Aqueous Solutions by Adsorption to Rice Husks

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NASIR KHALID and SHUJAAT AHMAD*
NUCLEAR CHEMISTRY DIVISION
PAKISTAN INSTITUTE OF NUCLEAR SCIENCE AND TECHNOLOGY
P.O. NILORE, ISLAMABAD, PAKISTAN

SEEMA NASEER KIANI and JAMIL AHMED
DEPARTMENT OF CHEMISTRY
QUAID-I-AZAM UNIVERSITY
ISLAMABAD, PAKISTAN

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ABSTRACT

The adsorption of mercury ions from aqueous solutions on rice husk has been investigated as a function of appropriate electrolyte, contact time, concentrations of adsorbent and adsorbate, and temperature. The radiotracer technique was used to determine the distribution of mercury. Maximum adsorption was observed at 0.01 mol·dm^{-3} acid solutions (HNO_3, HCl, H_2SO_4, and $HClO_4$) using 1.0 g of adsorbent for 1.30×10^{-3} mol·dm^{-3} mercury concentration in 5 minutes of equilibration time. Studies show that the adsorption decreases with an increase in the concentrations of all the acids. The adsorption data follow the Freundlich isotherm over the range of 1.3×10^{-4} to 2.6×10^{-3} mol·dm^{-3} mercury concentration. The characteristic Freundlich constants, i.e., $1/n = 0.89 \pm 0.05$ and $A = 208 \pm 2.1$ m·mol·g^{-1}, have been computed for the sorption system. The uptake of mercury increases with a rise in temperature. Thermodynamic parameters, i.e., $\Delta G°$, $\Delta S°$, and $\Delta H°$, have also been calculated for the system. The sorption process was found to be endothermic. Application of the method to the effluents of medium-sized industries showed that 9.9 kg of rice husk was sufficient for their decontamination.

INTRODUCTION

Mercury has long been identified as a potential health and environmental hazardous metal. Prolonged exposure to it produces nervous disorders: mus-

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cle tremors, depression, memory loss, paralysis insanity, and myocardial necrosis, whereas large doses may damage the liver and kidney (1, 2). Major sources of mercury contamination are industries producing chlorine and caustic soda, vinyl chloride, plastics, acetaldehyde, electrical equipment, fungicides, cement, paints, pulp and paper, etc. Significant amounts of mercury in sewage sludge, household bleaching solution, and coal and oil have been reported (3). Nowadays mercury is also used as a catalyst in the production of polyurethane foams and may be hazardous if not properly controlled. Hence, its removal from such effluents is desirable for the sake of a clean environment.

Many processes are utilized to clean industrial wastewater from mercury. The most commonly used procedures include precipitation (4, 5), ion exchange (6, 7), solvent extraction (8), and adsorption. Among these, the adsorption process has been used extensively for the removal of heavy metals from polluted wastewaters. The process is useful and popular due to its low maintenance cost, high efficiency, and ease of operation. The adsorption of mercury on MnO_2 (9), TiO_2 (10), zirconium oxide (3, 11), soil (12), silica gel (13, 14), activated carbon (15), and titanates of alkali and alkaline earth metals (16, 17) has been studied.

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Beside these, such unconventional materials as tree barks, hair, wool, peanut and onion skin, etc. have also been used for the adsorption of mercury (18–22). The present work deals with a series of experiments to assess the utility of rice husk as an adsorbent for the removal of mercury from aqueous solutions. As Pakistan is a major producer of rice (4.144 million tons annually), and husk accounts for about 20–23% of the whole rice, the selected material is abundantly available at no cost as waste material even after its wide consumption as a fuel in mills, thus posing a disposal problem for mill owners. It contains 15–22% SiO_2 in a hydrated amorphous form like silica gel (23), which is a good adsorbent. The evaluation of this naturally occurring cheaper material as an adsorbent for the decontamination of industrial effluents is a continuation of our earlier studies (24, 25).

EXPERIMENTAL PROCEDURE

Preparation of Radiotracer

The radiotracer of mercury used in the present study was prepared by irradiating a known weight of spec-pure mercury oxide (Johnson & Mathey) in a 10 MW swimming-pool-type reactor (PARR-1) of this institute for a appropriate time at a neutron flux of 4.5×10^{13} $cm^{-2}\cdot s^{-1}$. After a suitable cooling time, the target was dissolved in concentrated distilled nitric acid and diluted to a suitable volume with water.

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Reagents

The husk of basmati rice was obtained from a rice mill. The husk was thoroughly washed with water to remove all dirt and was oven-dried at 80°C to constant weight. The dried husk was stored in a precleaned airtight container and was used as such without any physical or chemical pretreatment.

Buffer solutions of pH 1 to 10, with an ionic strength of 0.1 M, were prepared by using appropriate aliquots of solutions of KCl and HCl (pH 1–3), CH_3COONa and CH_3COOH (pH 4–6), NH_4Cl and NH_4OH (pH 6–7.5), and H_3BO_3, NaCl, and NaOH (pH 8–10). All pH measurements were carried out with a digital pH meter-605 from Metrohm. All the reagents used were of Analytical Grade and were used as such. Glassware was cleaned by overnight soaking in nitric acid (1:1) followed by multiple rinsing with water. Distilled and deionized water was used throughout.

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Sorption Measurements

Adsorption of mercury was carried out by a batch technique at room temperature (296 ± 1 K) except where otherwise specified. A known amount of rice husk was taken in a 50-cm^3 culture tube with a screwed polyethylene cap and soaked in the acid or buffer in which the adsorption was to be carried out. After 5 minutes the excess solution was drained out and 10 cm^3 of standard acid solution or buffer solution of known pH was added along with a fixed amount of stock radiotracer solution. The contents were equilibrated on a mechanical shaker for 10 minutes, centrifuged at 5000 rpm for 3 minutes for phase separation, and the supernatant solution was withdrawn. The radioactivities of the liquid phase were determined before and after equilibration with a NaI well-type scintillation counter (Camberra) coupled with a counter-scaler (Nuclear Chicago). A volume of 1.0 cm^3 was usually used to measure the activity.

TREATMENT OF DATA

Adsorption from Solution

The percentage sorption of metal ion "M" from the solution was calculated using the equation

$$\%\ \text{Sorption} = \frac{A_i - A_f}{A_i} \times 100 \tag{1}$$

where A_i = initial radioactivity of the solution
A_f = radioactivity of solution after equilibrium

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The distribution coefficient (K_d) was calculated from

$$K_d = A_i - \frac{A_f}{A_f} \times \frac{V}{m} \qquad (2)$$

where V = volume of adsorbate (cm^3)
m = amount of adsorbent (g)

Calculation of Thermodynamical Parameters

The values of $\Delta H°$ and $\Delta S°$ were calculated from the slope and intercept of the linear van't Hoff plot, respectively, using the relation

$$\ln K_d = \frac{\Delta S°}{R} - \frac{\Delta H°}{RT} \qquad (3)$$

where K_d = distribution coefficient (cm^3/g)
$\Delta S°$ = entropy change for the process
$\Delta H°$ = enthalpy change for the process
R = gas constant
T = absolute temperature

The change in standard free energy ($\Delta G°$) and entropy ($\Delta S°$) for the specific adsorption was calculated using the following equations:

$$\Delta G° = -RT \ln K_d \qquad (4)$$

$$\Delta G° = \Delta H° - T\Delta S° \qquad (5)$$

where the symbols have their usual significance.

RESULTS AND DISCUSSIONS

The adsorption of mercury in aqueous solutions on rice husk was examined by optimizing various parameters such as variable concentrations of mineral acids, pH, equilibration time, and the amount of adsorbent and adsorbate using the radiotracer technique. The criterion for optimization was the selection of parameters where maximum adsorption occurred. The effect of temperature on the adsorption of mercury was also checked. All the reported results are the average of at least triplicate measurements. The expected error was within ±3.0%.

The metal contents of 10 samples of basmati rice husk were determined by employing neutron activation analysis and atomic absorption spectrophotometry. The results have been reported elsewhere (24).

Effect of Acid Concentration

The chemical treatment of the metal oxides resulted in certain changes in surface properties. Therefore, the adsorption behavior of mercury (1.3×10^{-3}

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mol·L^{-1}) was checked in mineral acid solutions (HNO_3, H_2SO_4, HCl, and $HClO_4$) with concentrations ranging from 0.01 to 3.0 M. The results shown in Figs. 1a and 1b show a decrease in the adsorption of mercury with an increase in acid concentration from 0.01 to 3.0 M. This is explicable on the basis of competition between the excess of H^+ ions in the medium and the positively charged hydrolyzed species present in the solution. A higher acid concentration appears to suppress hydrolysis of the element in the hydrolyzed species, resulting in lower adsorption. The adsorption trends in the presence of all the acids was similar. It was also observed that maximum adsorption occurred in HNO_3 (0.01 M) compared to the other acids. Therefore, this concentration of HNO_3 was used for all the subsequent experiments.

The adsorption of metal ions on a hydrous oxide surface can probably be explained on the basis of the surface complex formation model. In this model the hydrolysis of silicon dioxide produces the hydrous oxide surface group —SiOH, i.e., the silanol group. This results in the formation of a weakly acidic silica surface with $K_a = 10^{-6}$ to 10^{-8} (26). The cation-exchange reaction can occur in aqueous solutions as follows:

$$m(\text{—SiOH}) \quad \leftrightarrow \quad m(\text{—SiO}^-) + m\text{H}^+ \tag{6}$$

$$\text{M}^{n+} + m(\text{—SiO}^-) \quad \leftrightarrow \quad \text{M(OSi—)}_m^{n-m} \tag{7}$$

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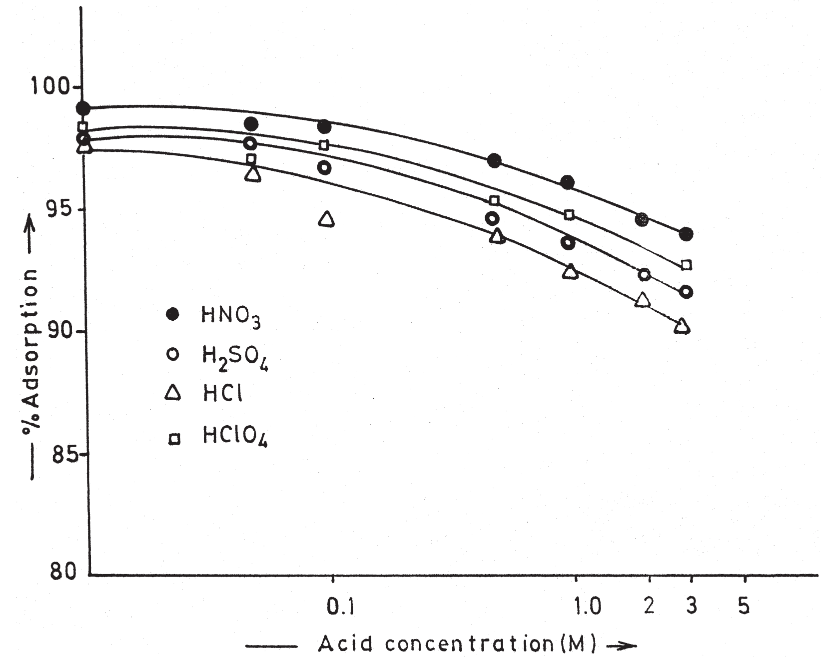

FIG. 1a Variation of percent adsorption of mercury on rice husk as a function of acid concentration.

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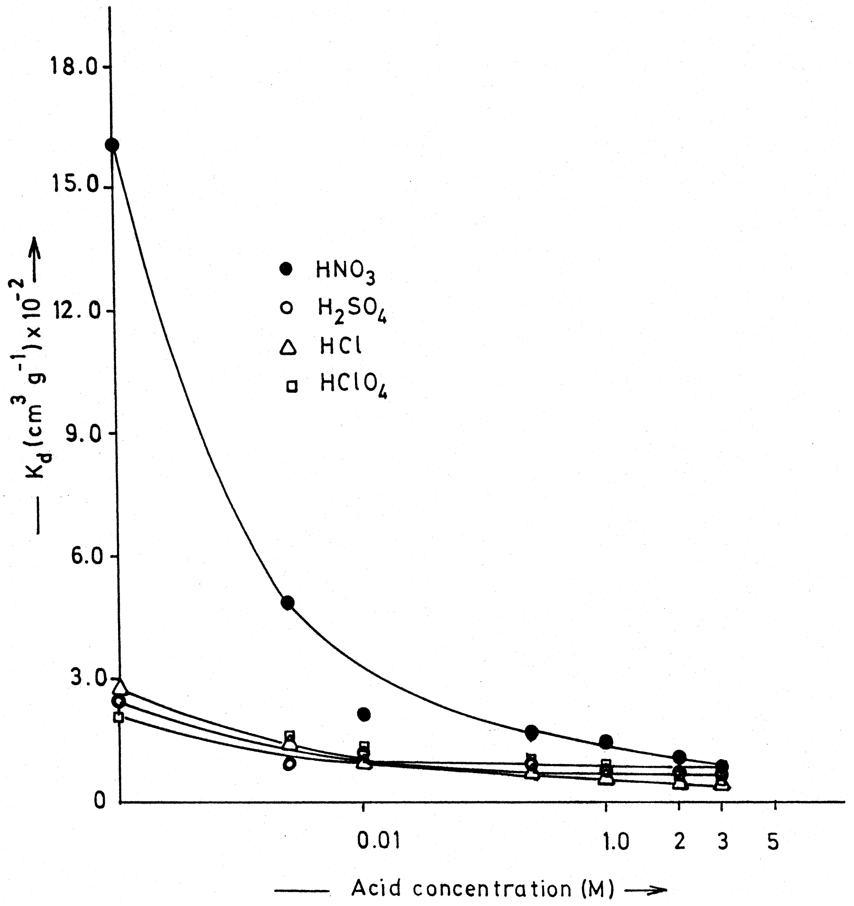

FIG. 1b Variation of K_d for the adsorption of mercury on rice husk as a function of acid concentration.

The overall reaction can be represented as

$$M^{n+} + m(\text{—SiOH}) \leftrightarrow M(\text{OSi—})_m^{n-m} + mH^+ \tag{8}$$

where M^{n+} = metal ion with n^+ charge
—SiOH = silanol group on SiO_2 surface
mH^+ = number of protons released

Reaction (8) is reversible in acid solution. In the present study the decrease in the adsorption of mercury at higher acid concentrations can be explained on the basis of the competitive reaction of protons and metal ions. The maximum adsorption of metal at 0.01 M acid concentration is in accordance with the concept of zero point charge (ZPC) which lies in the 1.8–2.2 pH range for SiO_2 (27). This indicates that the adsorption of metal occurs on the surface of SiO_2 present in rice husk.

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Effect of pH

The removal of mercury from wastewaters by adsorption is highly dependent on the pH of the solution which affects the surface charge of the adsorbent, the degree of ionization, and the speciation of the adsorbate species. The adsorption behavior of mercury ions was studied in aqueous solutions of different pH values (1 to 10) using 1.0 g of rice husk and a fixed amount of mercury (1.30×10^{-3} mol L^{-1}). The results obtained are presented in Fig. 2. It was observed that the maximum adsorption of mercury on rice husk occurred at pH 6, which gradually decreased with a further increase in pH. These results are similar to the reported values of pH for the maximum adsorption of mercury on onion skin (22). Friedman et al. (20) reported that maximum adsorption of Hg^{2+} ions takes place on wool at pH 2 and 9.

The adsorption at near-neutral pH could be attributed to the cellulose part of the rice husk, where site-binding adsorption might be occurring. This could probably be due to the surface complexation phenomenon which is facilitated by dissociation of the acidic functional groups (—COOH, — SH, etc.) present in the rice husk, which is essentially dependent upon their pK_a values. The pK_a values of some organic constituents of rice husk are given in Table 1. The removal of mercury could, therefore, probably be due to the mixed effect of "ion exchange" and "surface complexation" phenomena on the surface of rice husk. The decrease in adsorption of mercury noted at alkaline pH is probably due to the formation of hydroxide.

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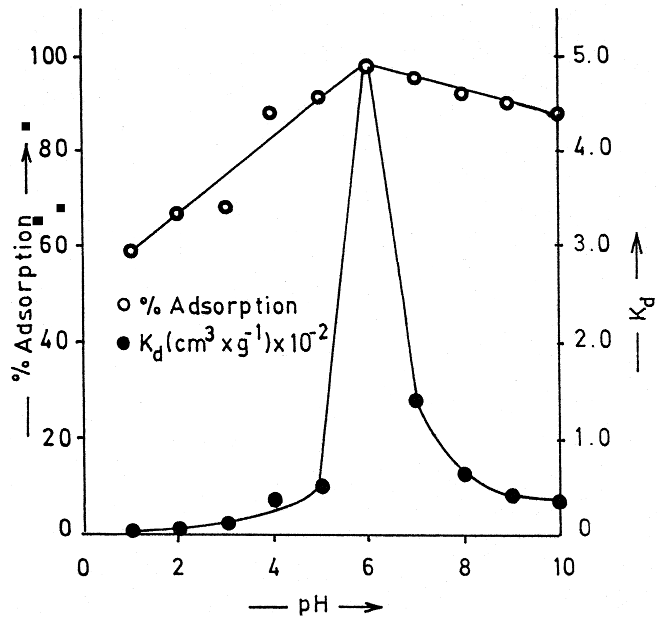

FIG. 2 Effect of pH on the adsorption of mercury on rice husk.

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TABLE 1
pK_a Values of Some Components of Rice Husk (28)

Component	Formula	pK_a
Aspartic acid	$C_4H_7NO_4$	1.99, 3.9, 9.9
L-Glutamic acid	$C_5H_9NO_4$	2.13, 4.3
Cysteine	$C_3H_7NO_2S$	1.9, 8.4, 10.7
Histidine	$C_6H_9N_3O_2$	1.8, 6.04, 9.3
Lysine	$C_6H_{14}N_2O_2$	2.2, 9.06, 10.5
Tyrosine	$C_9H_{11}NO_3$	2.2, 9.21, 10.5
Arginine	$C_6H_{14}N_4O_2$	1.8, 8.99, 12.5

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Effect of Shaking Time

The variation of percent adsorption of mercury with shaking time was also studied using 5.0 cm^3 of 1.3×10^{-3} mol·L^{-1} of mercury solution in 0.01 M HNO_3 with 1.0 g of rice husk. The shaking time was varied from 0.5 to 25 minutes, and the results are shown in Fig. 3, which is a plot of percent adsorption of mercury versus shaking time. It is seen that the percentage adsorption increases with an increase in shaking time. Maximum adsorption was observed at 5 minutes time, beyond which there is no further increase in the adsorption. Therefore, 5 minutes shaking time was considered to be sufficient

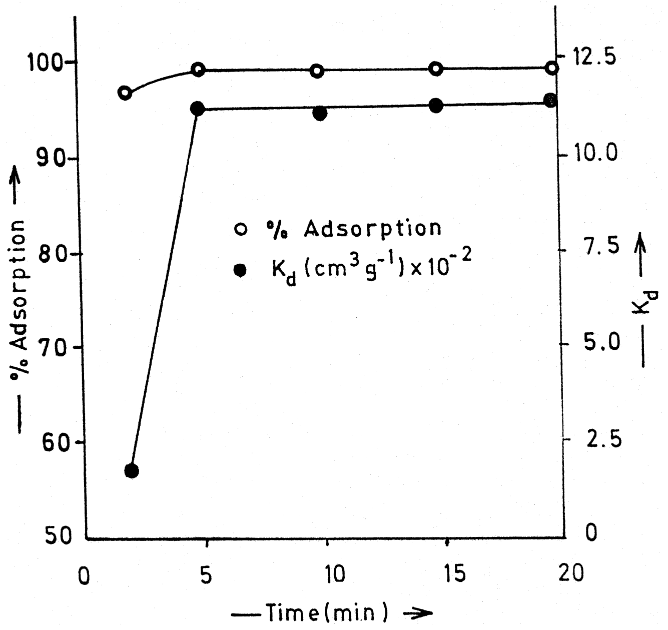

FIG. 3 Influence of equilibration time on the adsorption of mercury on rice husk.

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for the adsorption of mercury on rice husk and was used for all subsequent experiments.

Effect of Amount of Adsorbent

The amount of adsorbent also affects the efficiency of the adsorption, and this parameter was also optimized by shaking 1.3×10^{-3} mol·L^{-1} of mercury solution using the optimized parameters. The amount of rice husk was varied from 0.2 to 2.0 g and the results are presented in Fig. 4 which depicts that just 0.6 g of rice husk is sufficient for the quantitative removal of mercury from the aqueous solution used . This amount of rice husk was used for further investigations.

Effect of Concentration of Adsorbate

The effect of mercury concentration on the adsorption process was studied under the optimized conditions of shaking time, pH, and the amount of adsorbent. The concentration of mercury was varied from 1.3×10^{-4} to 6.5×10^{-3} mol·L^{-1}. The results, given in Fig. 5, show that the adsorption of mercury was almost constant up to 2.6×10^{-3} mol·L^{-1} of mercury. Beyond this concentration the adsorption gradually decreased with an increase in the concentration of mercury. This can be explained in terms of the relatively lesser number of active sites at higher concentrations of mercury. The loading capacity of rice husk was calculated and found to be 10.2 g mercury per kg of rice husk.

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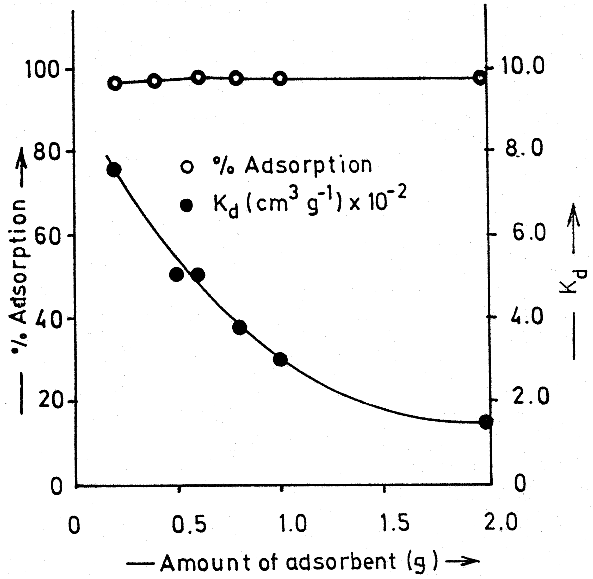

FIG. 4 Effect of amount of adsorbent on the adsorption of mercury.

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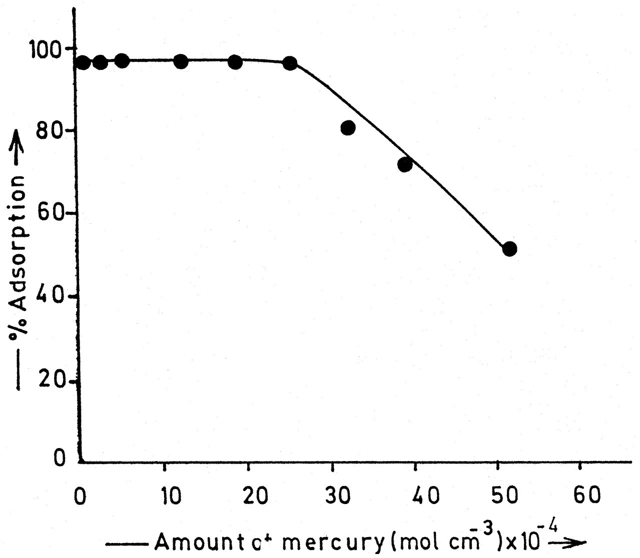

FIG. 5 Effect of amount of adsorbate on the adsorption of mercury on rice husk.

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The adsorption data of mercury were further analyzed by fitting to Langmuir and Freundlich adsorption isotherms. The data do not fit to the Langmuir equation. However, the Freundlich adsorption isotherm was capable of describing the data over the concentration range of 1.3×10^{-3} to 2.6×10^{-3} mol·L^{-1}. The Freundlich isotherm was tested in the following linearized form:

$$\log C_{\mathrm{ads}} = \log A + 1/n \log C_{\mathrm{eq}}$$

where C_{ads} = amount of mercury adsorbed at equilibrium (mol·g^{-1})
C_{eq} = equilibrium concentration of mercury in solution (mol·L^{-1})
A and $1/n$ = Freundlich constants

A plot of log C_{ads} versus log C_{eq} yields a straight line. This linear plot (Fig. 6) supports the applicability of the Freundlich adsorption isotherm in the present study. The values of Freundlich constants $1/n$ and A, which are rough measures of the adsorption intensity and adsorption capacity of the adsorbent, respectively, have been determined from the slope and the intercept of the straight line in Fig. 6 using a least-squares fit program and were found to be 0.89 ± 0.05 and 208.4 ± 2.1 m mol·g^{-1}, respectively. The higher fractional value of $1/n$ ($0 < 1/n < 1$) signifies that the surface of the adsorbent is heterogeneous in nature (29). The higher values of A further confirms the higher affinity of mercury ions toward the rice husk surface. The applicability of the Freundlich isotherm has also been reported for the adsorption of Hg^{2+} ions on the surfaces of wool (20) and metal oxides (3, 9, 11).

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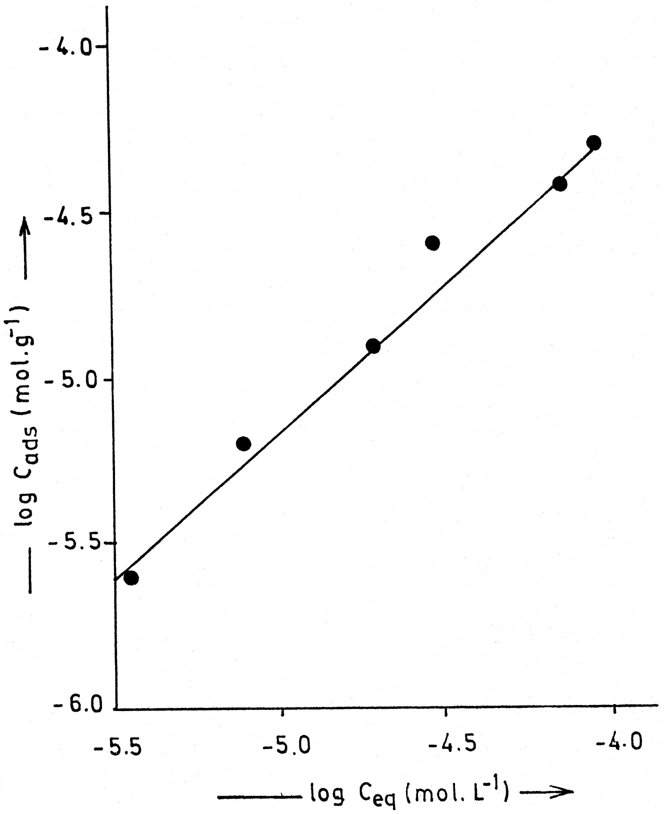

FIG. 6 Freundlich plot for the adsorption of mercury on rice husk.

Effect of Temperature

The effect of temperature on the adsorption of mercury ions (6.5×10^{-4} mol·L^{-1}) on rice husk was also checked using the optimized conditions. The temperature was varied from 283 to 323 K. The amounts of mercury ion adsorbed at equilibrium at various temperatures are shown in Table 2, which reveals that the K_d value increases with a rise in temperature, indicating better adsorption at a higher temperature. The increase in the amount of mercury adsorbed at equilibrium with an increase in temperature may be either to acceleration of some originally slow adsorption step(s) or to the creation of some new active sites on the adsorbent surface. Similar results have been reported for the adsorption of mercury on zirconium oxide (3).

The amounts of mercury adsorbed at equilibrium at different temperatures have been utilized to evaluate the change in standard enthalpy $\Delta H°$. A graph was plotted of ln K_d vs $1/T$ and yielded a straight line (Fig. 7), the slope of which was equal to $-\Delta H°/R$, thus allowing $\Delta H°$ to be calculated. The estimated $\Delta H°$ value for the present system is 12.09 kJ·mol^{-1} (Table 3). The positive value of $\Delta H°$ indicates that the process is endothermic in nature. The val-

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TABLE 2
Adsorption Studies of Mercury Ions on Rice Husk as a Function of Temperature

Rice husk	500 mg
Shaking time	5 minutes
Volume equilibrated	5 cm^3
Concentration of HNO_3	0.01 M
Concentration of mercury	6.50×10^{-4} M

Temperature (K)	1/*T* (K^{-1})	Concentration adsorbed per gram of adsorbent ($mol \cdot g^{-1}$)	Concentration in bulk ($mol \cdot L^{-1}$)	K_d ($cm^{-3} \cdot g^{-1}$)	Ln K_d
283	3.533×10^{-3}	6.264×10^{-6}	2.36×10^{-5}	265.42	5.58
293	3.413×10^{-3}	6.277×10^{-6}	2.23×10^{-5}	281.48	5.64
303	3.300×10^{-3}	6.328×10^{-6}	1.72×10^{-5}	367.91	5.91
313	3.195×10^{-3}	6.407×10^{-6}	9.26×10^{-6}	391.94	6.54
323	3.096×10^{-3}	6.368×10^{-6}	1.32×10^{-5}	482.42	6.18

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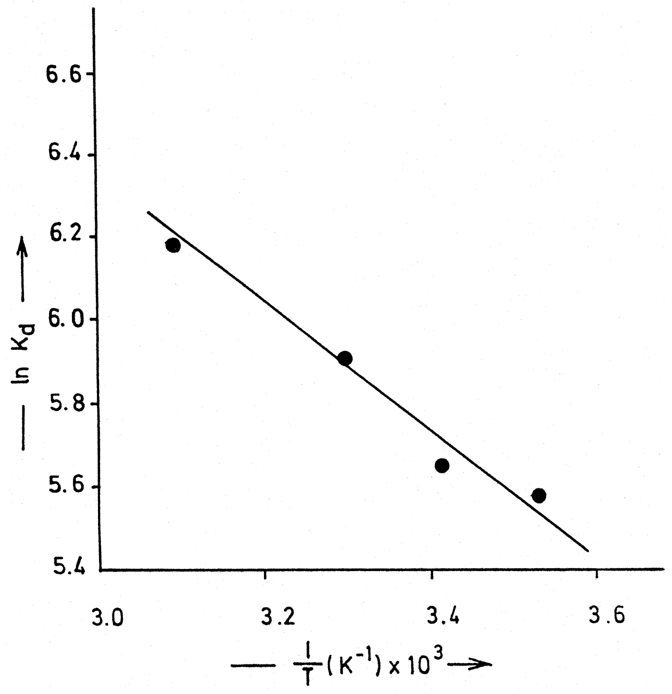

FIG. 7 van't Hoff plot of the adsorption of mercury on rice husk; ln K_d vs 1/*T*.

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TABLE 3
Thermodynamic Parameters for Adsorption of Mercury Ions on Rice Husk

Temperature (K)	$\Delta G°$ ($kJ \cdot mol^{-1}$)	$\Delta H°$ ($kJ \cdot mol^{-1}$)	$\Delta S°$ ($J \cdot deg^{-1} \cdot mol^{-1}$)
283	−13.129	12.096	89.134
293	−13.739		88.174
303	−14.889		89.059
313	−17.019		93.019
323	−16.596		88.830

ues of the free energy of specific adsorption, $\Delta G°$, were calculated using Eqs. (4) and (5) and are in good agreement with each other (see in Table 3).

The negative values of $\Delta G°$ indicate that the adsorption of mercury on rice husk is a spontaneous process. The positive values of $\Delta S°$ suggest the increased randomness at the solid–solution interface during the adsorption of mercury. The adsorbed solvent molecules, which are displaced by the adsorbate species, gain more translational entropy than is lost by the adsorbate ions, thus allowing for the prevalence of randomness in the system.

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TABLE 4
Chemical Analysis of Effluents
of Caustic Soda Industries

Ions	Concentration in $mg \cdot L^{-1}$ I_1	I_2
Na^+	248.0	198.0
K^+	11.2	8.6
Ca^{2+}	51.6	83.2
Mg^{2+}	10.1	16.4
Cl^-	96.3	125.4
SO_4^{2-}	48.0	28.3
HCO_3^-	75.0	80.0
Cu^{2+}	0.1	1.2
Pb^{2+}	0.1	0.07
Cd^{2+}	0.01	0.01
Zn^{2+}	0.3	0.4
Fe^{2+}	0.15	0.1
Hg^{2+}	5.8	10.1
Hg^{2+a}	0.01	0.01

[a] Concentration after adsorption on rice husk.

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Removal of Mercury from Industrial Effluents

Analysis of effluents of medium-sized caustic soda industries (I_1 and I_2) is given in Table 4. The samples were collected from the presetting tanks before their disposal. The amount of discharge from such industries is approximated to be 10,000 L/day, which contains up to 10 mg of mercury per liter. The recommended permissible levels of the National Environmental Quality Standards (NEQS) of Pakistan is 0.01 mg of mercury per liter. Hence, there is a need to remove about 10 $\text{mg}\cdot\text{L}^{-1}$ of mercury to meet the requirement of the NEQS. The data in Table 4 show that by using the optimized conditions for the present system, 9.9 kg of rice husk is sufficient to decontaminate mercury from such effluents.

CONCLUSION

The rate of adsorption of mercury ions from aqueous solutions on rice husk is reasonably fast, requiring a contact time of 5 minutes. The reaction was found to be endothermic and occurs in the presence of a variety of anions, with maximum adsorption noted in the presence of 0.01 M HNO_3. The loading capacity of rice husk for mercury under the optimal conditions was found to be 10.2 g mercury per kg of rice husk. Based upon these data, it is concluded that abundantly available rice husk has tremendous potential as a cheap and effective adsorbent for mercury from industrial effluents and can be effectively utilized for waste management. Recycling can be achieved by heating the loaded husk.

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