

Adsorption of mercury from aqueous solution using gum acacia-silica composite: kinetics, isotherms and thermodynamics studies

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ABSTRACT

In the present investigation, the remediation of mercury by using the gum acacia-silica composite as an adsorbent has been studied. Experiments revealed optimum parameters which were found to be pH 6, contact time 2 hours, Hg(II) concentration of 100 ppm, reflux temperature 30 °C and adsorbent dosage of 50 mg. The experimental data was subjected to modeling using the Langmuir and Freundlich isotherms. It was found that the data very well fitted to the Freundlich model. The pseudo second order kinetics confirms chemisorption with rate constant $3.1 \times 10^{-4} \text{ gm}^{-1}\text{min}^{-1}$. The calculated thermodynamic parameters (ΔG^0 , ΔS^0 , ΔH^0) revealed the exothermic and spontaneous nature of adsorption process at the solid-solution interface. The adsorbent could be recycled for six successive cycles with 31.5% loss in its efficiency. The adsorbent is found to be highly effective and economical for mercury remediation from water. Copyright © 2016 VBRI Press.

Keywords: Gum acacia; nanocomposite; mercury adsorption; tetraethyl orthosilicate (TEOS); sol-gel.

Introduction

The main way for mercury to enter into the environment is by the breakdown and dispersion of minerals into the soil and water. Elemental mercury converted into methyl mercury by microorganism present in soil and water, which accumulates in fish, mollusks, and other food sources, through which enters in humans [1]. Exposure of mercury causes large number of physical and psychological disorders in human beings [2]. The earlier study revealed that mercury enters the body of infants through breast-feeding [3].

Mercury contamination was found to be very high in the atmosphere as well as in living systems [4]. Hence remediation of mercury from water is very significant. Mercury remediation has been done in the past by using several techniques. Use of suitable adsorbent for the removal of metal ions is the most prominent and effective method among them. Activated carbon was found to be more expensive as adsorbent; hence there is a need for development of low cost adsorbent [5].

Polysaccharides are multifunctional which can potentially link to a wide variety of molecules [6-10]. Polysaccharide based materials have been widely used as biosensor [11], hydrogel [12,13], and also have potential applications in water retention, dyes removal and drug release [14,15]. Removal of toxic compounds using the polysaccharide based macromolecular adsorbents [16-21] has been the area of current research interest and the availability of voluminous literature reflects the importance of such adsorbents [22,23].

Composites of organic polymer and silica adsorbed mercury from aqueous medium due to porosity and high surface area [24, 25]. Polysaccharides are used as template in the sol-gel process [26-29] to generate silica. Mesoporous silica was found to be better adsorbent for remediation of various pollutants from aqueous solution [30-32]. Guar gum and grafted guar gum-silica composites have been proved to be better adsorbents for zinc [33-35] and cadmium [36]. Gum acacia is abundant and multifunctional, water miscible, it cannot be usefully exploited as adsorbent in aqueous systems; however, its hybridization with silica has resulted water insoluble porous material having high surface area and multifunctional [37]. In the current study, due to high toxicity of mercury contaminated water, our composite as adsorbent has been characterized for mercury remediation from water using batch adsorption technique at room temperature. Thermodynamics, kinetics and isothermal studies were carried out to fully understand the details of the process of adsorption.

Experimental

Materials

Gum acacia (CDH; LR) was used as a template. Tetraethyl orthosilicate (98% TEOS; Sigma Aldrich) was used as a silica precursor. NH_4OH (30% NH_3 ; Merck) was used as a catalyst in the sol-gel process. A mercury stock solution (1000 ppm) was prepared by using HgCl_2 (Merck). HNO_3 (Merck), HCl (Merck), NaOH (Merck) and buffer solutions

(Qualigen) were used. Rhodamine 6G (Merck) was used as a complexing agent and Gelatin (Merck) as a stabilizer. KI (Merck), potassium hydrogen phthalate ($C_8H_5O_4K^+$; Merck) and a pinch of sodium thiosulphate ($Na_2S_2O_3 \cdot 5H_2O$; Merck) were used for preparation of buffer.

Instrumentation

Schott AVS viscometer, Muffle Furnace, UV-visible Spectrophotometer, pH meter, FTIR spectrophotometer, scanning electron microscopy and EDAX-FEI Quanta 200 machine were used for instrumental analysis.

Preparation of adsorbent

General procedure for the preparation of gum acacia-silica composite material is shown in **Fig. 1**. Gum acacia (1.0 g) was dissolved in 10 mL of distilled water. Separately, 2.5 mL of TEOS was also dissolved in 2.5 mL of ethanol. A third solution incorporating 1.75 mL of 12N ammonium hydroxide was prepared separately. These solutions were immediately poured together rapidly into a reaction flask and kept under gentle stirring for 14 hours at room temperature to grow monodisperse SiO_2 particles within the biopolymer medium, which was subsequently washed several times with distilled water. The ensuing mixture was then subjected to slow evaporation at $40^\circ C$ for 4 hours and $60^\circ C$ until a dry material (H1) was obtained [34]. This material was subjected to calcination in a muffle furnace at $600^\circ C$ for 2 hours to obtain porous composite which was coded as H4 in our earlier work [37] and for convenience it is named as GH in this study.

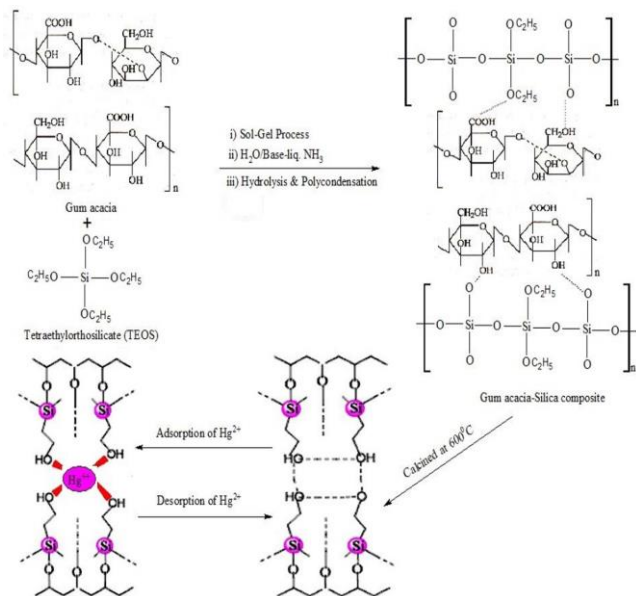


Fig. 1. Proposed mechanistic pathway for synthesis of gum acacia-silica nanocomposite and removal of Hg^{2+} from aqueous solution.

Preparation of the control silica

Pour 2.5 mL TEOS dissolved in 2.5 mL ethanol and 1.75 mL of 12 N ammonium hydroxide to 10 mL of distilled water. Then, the solution was stirred for 18 hours at room temperature. The obtained mixture was evaporated at $40^\circ C$ (3 hours), $60^\circ C$ (4 hours), $70^\circ C$ (2 hours) and $80^\circ C$ until dry material (CS) was obtained [34]. This material

was subjected to calcination in a Muffle furnace at $600^\circ C$ for 2 hours to obtain the control silica (CS_{600}).

Adsorption studies by batch method

Details of the procedure for adsorption studies by batch method has been given in our earlier work [16,25].

Desorption studies

Details of the procedure for desorption studies has been given in our earlier work [16,25].

Results and discussion

Our earlier work of characterizing of the gum acacia-silica composite [37], revealed that the material possess desirable characteristics to be an efficient adsorbent. The material (GH) was synthesized by polycondensation of tetraethyl orthosilicate in presence of gum acacia template, followed by controlled calcination (at $600^\circ C$ in air) of the hybrid gel. In a batch adsorption study, adsorbent GH (50 mg) effectively removed 113.63 mg/g $Hg(II)$ from aqueous solution in 2 hours at $30^\circ C$ from 100 ppm $Hg(II)$ at pH 6 and 90 rpm. The efficiency of adsorbent (GH) was found to be higher in comparison with all the adsorbents reported earlier for mercury remediation (**Table 1**). GH was found to adsorb 1.7 times more $Hg(II)$ than the uncalcined control silica (CS) synthesized in an identical manner, indicating the role of polysaccharide templating on the silica matrix. However, the adsorption by CS_{600} (control silica calcined at $600^\circ C$) was much lower than CS, this is explainable as silica xerogel shrinks on calcination due to condensation polymerization across the pores.

Table 1. Comparative adsorption capacity of $Hg(II)$ by different adsorbents.

Reported adsorption capacity of $Hg(II)$ by different adsorbents		
S. No.	Adsorbents	Q_{max} References
1.	2-mercaptobenzothiazole treated clay	2.81 [43]
2.	Silica aerogel	9.17 [44]
3.	Coconut coir pith grafted poly(hydroxyl EMA)	13.4 [45]
4.	Used tyre rubber	14.65 [46]
5.	Eucalyptus bark	33.11 [47]
6.	Sago waste carbon	55.6 [48]
7.	Biomass of Drepanocladus revolvens	94.4 [49]
8.	Fruit shell of Terminalia catappa	94.43 [50]
9.	Extracellular biopolymer poly(glutamic acid)	96.79 [51]
10.	Bicarbonate treated pea nut hull carbon	110 [52]
11.	Gum acacia-silica composite materials (GH)	113.63 Present study
12.	Control silica (CS)	64.39 Present study
13.	Control silica calcined at $600^\circ C$ (CS_{600})	31.67 Present study

Characterization of the gum acacia-silica composite

The adsorbent was characterized using FTIR, XRD, SEM, TEM, PL and BET as described elsewhere [37]. Brief description of FTIR and SEM are given here.

Infra-red spectroscopy

The shift of $-O-H$ stretching peak in GH to higher wave number (3448 cm^{-1}) is probably due to densification of gel

(Fig. 2). Detail investigation of effect of calcinations temperature over this composite as well as mercury loaded composite has already been discussed in our previously reported article [37].

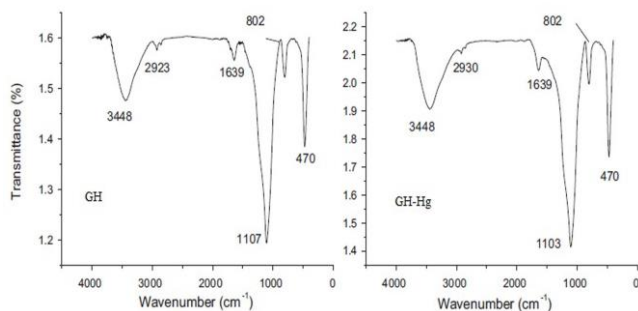


Fig. 2. FTIR spectra of composite calcined at 600°C (GH) and mercury loaded composite (GH-Hg).

Scanning electron microscopy

SEM and EDAX spectral details (Fig. 3) about composite GH and mercury loaded GH has been discussed in our earlier work [37].

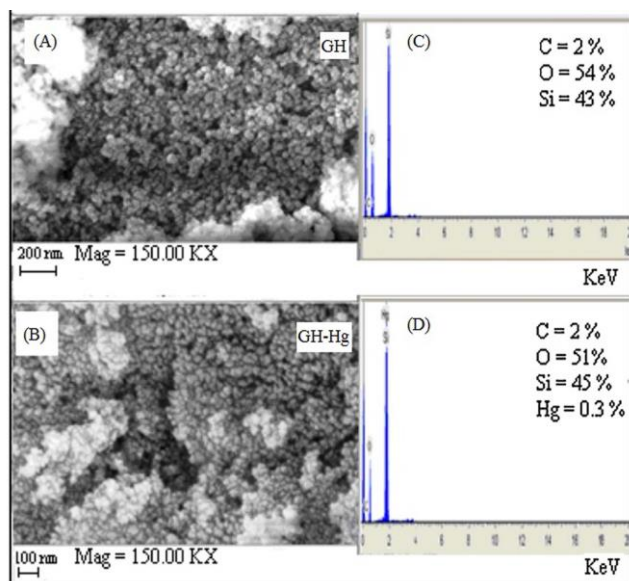


Fig. 3. SEM and EDAX picture of composite calcined at 600°C (GH) and mercury loaded composite (GH-Hg).

Evaluation of optimal conditions for mercury remediation

Influence of pH on mercury adsorption

Study on the influence of pH on mercury adsorption by the composite in the wide range of pH (Fig. 4A), reveals higher adsorption at pH 6. At 100 ppm initial mercury concentration, adsorption raised from 19.25% to 92.36% upon raising the pH up to 6 from 1. At pH 8.0 only 72.56% mercury was found to adsorb. A previously studied biopolymer, *i.e.*, poly (glutamic acid), with carboxylic acid functionality showed maximum adsorption at pH 6.0 [38].

Influence of the amount of adsorbent

The adsorption of mercury increased from 59.36% to 93.14% on raising the amount of adsorbent from 20 to 80

mg in 20 mL of 100 ppm mercury solution at 30°C for 2 hours (Fig. 4B). This increase in mercury adsorption by higher quantity of adsorbent is believed to be due to enhanced active binding sites availability for mercury binding. It was observed that 50 mg of adsorbent caused 92% of mercury remediation.

Influence of electrolytes

On raising the concentration of both NaCl and Na₂SO₄ from 0.01 M to 1.0 M, the amount of mercury removed by adsorbent decreased from 79.32 to 52.13 ppm and from 74.36 to 46.39 ppm, respectively (Fig. 4C). The reduced mercury removal is probably due to competitive adsorption of ions supplied due to the addition of electrolytes to occupy active binding sites. The Hg(II) adsorption was found to be less effective in Na₂SO₄ than in NaCl solution as a consequence to higher concentration of Na⁺ in the former solution.

Influence of temperature

The enhanced adsorption from 78.61% to 92.36% upon raising the temperature from 10°C to 30°C, confirms that the enthalpy change for the phenomenon of sorption is positive. However, at higher temperatures (>30°C), decreased adsorption indicates the initiation of desorption (Fig. 4D).

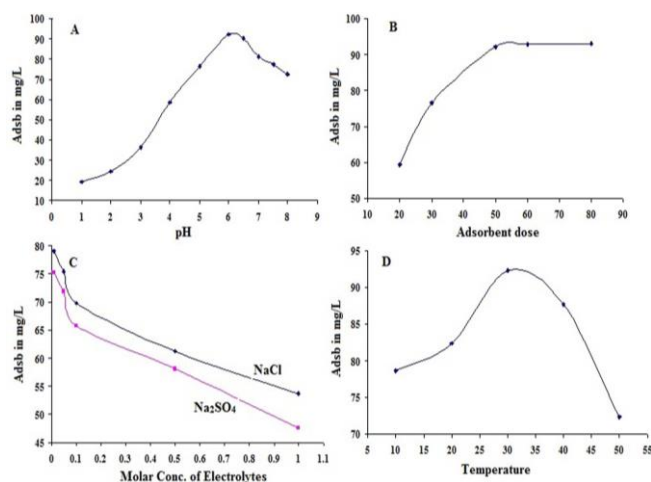


Fig. 4. Optimization of adsorption conditions (A) Adsorption at various pH (100 mg L⁻¹ Hg(II), 50 mg adsorbent dose, 2 hours contact time, 30°C temp and 90 rpm) (B) Adsorption at various adsorbent doses (100 mg L⁻¹ Hg(II), pH 6, 2 hours contact time, 30°C temp and 90 rpm) (C) Adsorption under the influence of different concentration of NaCl and Na₂SO₄, respectively (100 mg L⁻¹ Hg(II), 50 mg adsorbent dose, pH 6, 3 hours contact time, 30°C temp and 90 rpm) (D) Adsorption at various Temperature (50 mg adsorbent dose, pH 6, 2 hours contact time, 100 mg L⁻¹ Hg(II) solution and 90 rpm).

Kinetics of adsorption

The kinetics of mercury adsorption by gum acacia-silica composite indicated the effective binding of mercury to the adsorbent material in the initial period, followed by limited binding of mercury for a period of 2 hours until the attainment of equilibrium [39]. A little change was observed with respect to uptake capacity of composite for a period of 2.5 hours. Adsorption kinetics followed the pseudo second order [40], intra-particle diffusion model and pseudo first order (Fig. 5).

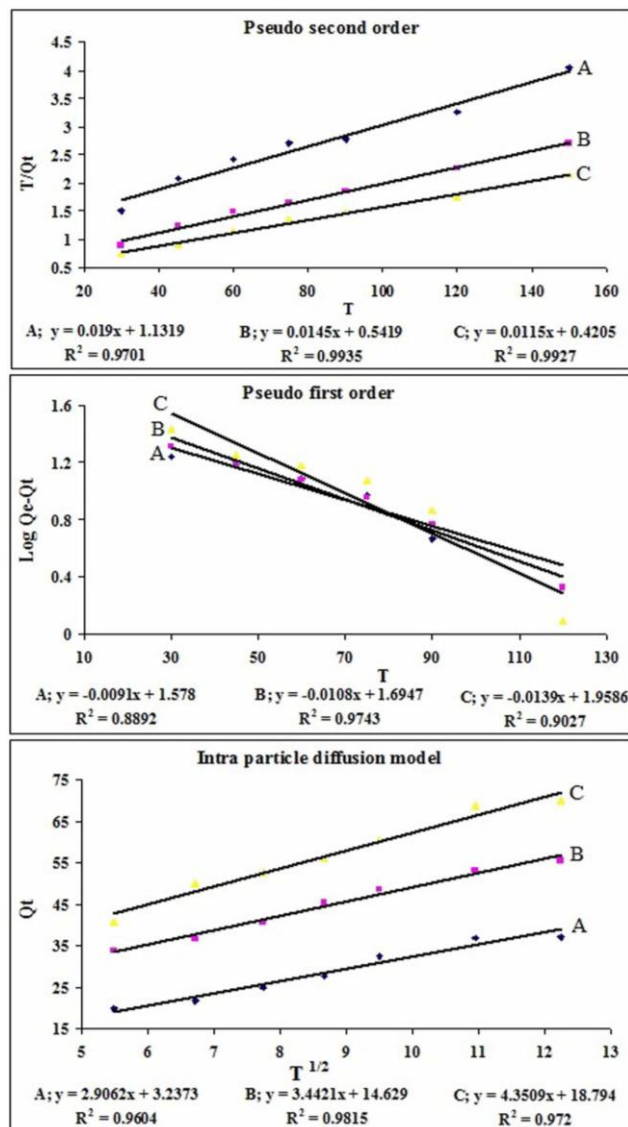


Fig. 5. Pseudo second order, Pseudo first order and Intra-particle diffusion model for the adsorption at 100 (A), 150 (B) and 200 (C) mg L⁻¹ initial Hg(II) concentrations.

Pseudo second order rate constant decreased for an initial increase in concentration from 100 to 200 ppm (Table 2A). The raise in solution temperature increased adsorption rate confirming endothermic nature of diffusion [41]. The computed data reveals that, the kinetics of adsorption is intra-particle diffusion controlled, in the initial period of time (up to 2 hours) and later it was followed by chemisorption.

Isothermal studies of adsorption

The isothermal studies were carried out at various concentrations of mercury (50 to 350 ppm) under optimum conditions of adsorption. The computed data fitted better to Freundlich model (Fig. 7) than to Langmuir model (Fig. 6). The estimated Q_0 value of 113.63 mg/g (Table 2B) can be attributed to the excellent performance of adsorbent for remediation of mercury. The Langmuir constant “b” was found to be directly proportional to temperature [42]. At 30°C for 100 ppm mercury, R_L value of 0.1374 is indicative of the adsorption as being favorable.

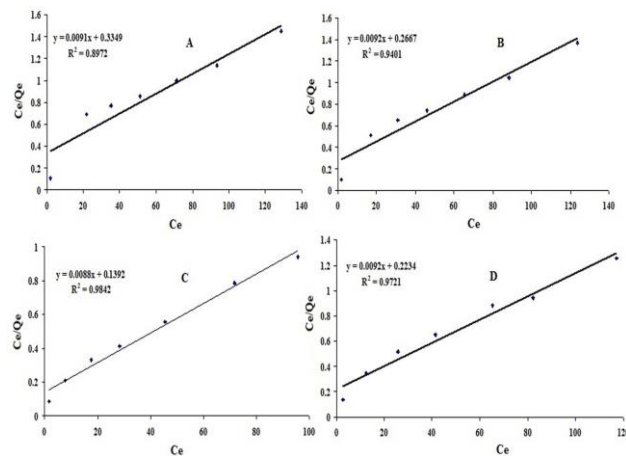


Fig. 6. Langmuir isotherms models at 10°C (A), 20°C (B), 30°C (C), and 40°C (D) temperatures for Hg(II) adsorption on gum acacia-silica composites using 50 mg adsorbent, pH 6 and rpm 90.

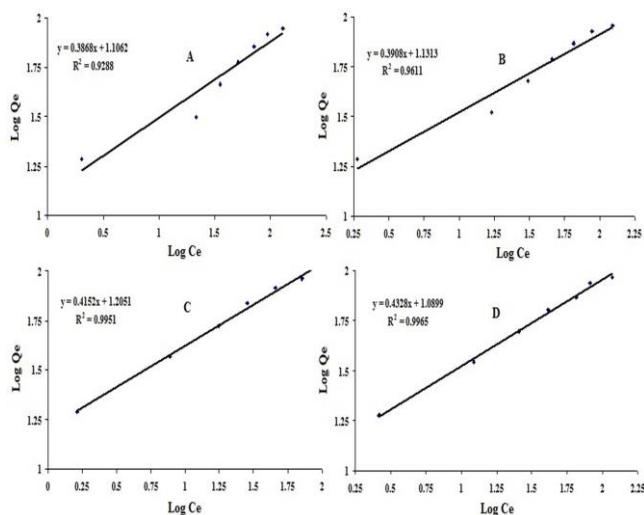


Fig. 7. Freundlich isotherms model at 10°C (A), 20°C (B), 30°C (C), and 40°C (D) temperatures for Hg(II) adsorption on gum acacia-silica composites using 50 mg adsorbent, pH 6 and rpm 90.

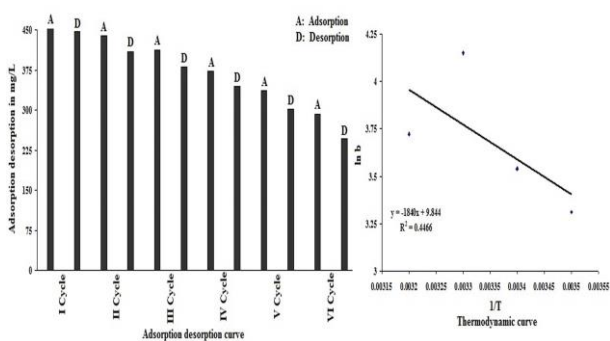


Fig. 8. Adsorption desorption cycle and thermodynamic simulation of the adsorption of Hg(II) by gum acacia-silica composites.

Reusability of the adsorbent

The reusability of the adsorbent after its repeated usage has been a crucial factor for economic compatibility. 92.35% of the Hg(II) was removed in the 1st cycle. The successive cycles were repeated six times at 500 ppm mercury solution, an adsorbent quantity of 250 mg, a pH of 6, 90 rpm and contact time of 2 hours, which resulted in to 96.7% stripping of Hg(II). The removal of mercury decreased

slightly up to sixth cycle (**Fig. 8**) confirming better efficiency of the adsorbent. In the last sixth cycle, 58.51% sorption was feasible. The experimental result reveals that the sorption ability of the adsorbent remained superior even after repeated adsorption–desorption cycles. The observed high efficiency of adsorbent material in removing Hg(II), would definitely open a great opportunity towards discovery of new adsorbent for the remediation of waste water with high metal contents.

Table 2. Kinetic models (A), Langmuir and Freundlich constant (B), Thermodynamic parameters (C) of Hg(II) adsorption by PVA-Silica composites.

(A) Pseudo second order, pseudo first order and intra particle diffusion kinetic model						
Hg(II) mg L ⁻¹	Pseudo second order R ²	K' (g mg ⁻¹ min ⁻¹)	Pseudo first order q _e (mg g ⁻¹)	R ²	K _d (min ⁻¹)	Intra particle diffusion K _{id} (mg g ⁻¹ min ^{0.5})
100	0.9701	3.12 × 10 ⁻⁴	52.63	0.8892	2.1 × 10 ⁻²	2.9062
150	0.9935	3.87 × 10 ⁻⁴	68.97	0.9743	2.5 × 10 ⁻²	3.4421
200	0.9927	3.12 × 10 ⁻⁴	86.95	0.9027	3.2 × 10 ⁻²	4.3509

(B) Langmuir and Freundlich adsorption isotherm						
Temperature (°K)	Langmuir Isotherm			Freundlich Isotherm		
	Q _{max} (mg g ⁻¹)	b (L mg ⁻¹)	R ²	n	K _f	R ²
283	109.89	27.10	0.8972	2.58	12.77	0.9288
293	108.69	34.49	0.9401	2.56	13.53	0.9611
303	113.63	63.22	0.9842	2.41	16.04	0.9951
313	108.69	41.18	0.9721	2.31	12.29	0.9965

(C) Thermodynamic Parameters			
Temperature (°K)	?G ⁰ (KJ mol ⁻¹)	?H ⁰ (KJ mol ⁻¹)	?S ⁰ (KJ mol ⁻¹)
283	-3.37	15.31	0.082
293	-3.72		
303	-4.51		
313	-4.21		

Adsorption thermodynamics

The change in entropy, ΔS and the change in enthalpy, ΔH were calculated by using the the plot of $\ln b$ against $1/T$ (**Fig. 8**). It has been observed that, on rising the temperature from 10°C to 30°C, the value of ΔG decreased from -3.37 kJ mol⁻¹ to -4.51 kJ mol⁻¹ (**Table 2C**). Thus, adsorption of Hg(II) onto the gum acacia-silica composite materials was found to increase at higher temperature. A positive value of enthalpy change (ΔH) confirms the endothermic nature of the adsorption process. Positive values of ΔS suggested good affinity of the metal ion toward the adsorbent and increased randomness at the solid-solution interface during the fixation of the metal ion to the active site of the adsorbent.

Conclusion

Gum acacia-silica composite was found to be better adsorbent in terms of simplicity in its synthesis, stability and photo luminescent nature, thus making it quite attractive for various industrial applications. Isotherm studies revealed unilayer adsorption. The positive ΔH^0 value confirms endothermic behavior of adsorption of mercury. Adsorption of mercury was found to follow pseudo second order kinetics. The adsorbent was found to be effective even after six successive adsorption-desorption cycles.

Acknowledgements

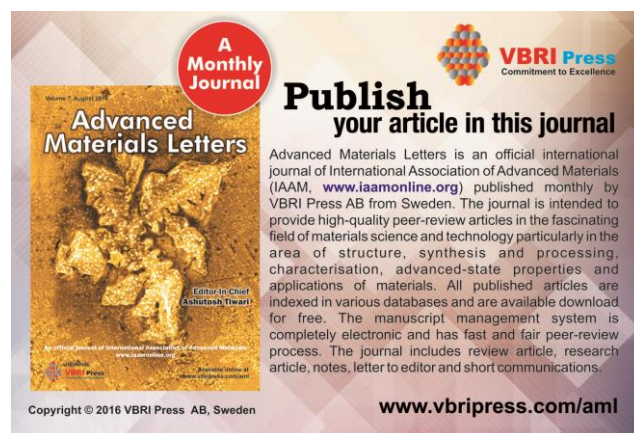
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