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Removal of Mercury Ions from Simulated Wastewater Using Scapes of *Allium sativum* L. as Low-Cost Bio-Adsorbent

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Abstract

Mankind has witnessed various environmental issues with the onset of industrialization. Several industries, like paint, electroplating, leather, textiles, petroleum refining, dyes and pigments, etc., holds significant amounts of metals and heavy metals in their wastewater. Consequently, disturbance in ecosystems is quite visible in the form of water pollution. Conventional metals and heavy metal removal methods, such as reverse osmosis, chemical precipitation, ion exchange, electrodialysis, chemical oxidation, membrane separation, and so on, are energy intensive, expensive, and produce toxic by-products as secondary pollutants. Among the conventional methods, adsorption has been found to be cost-effective with high efficacy for removing heavy metals from wastewater. In the present study, finely powdered garlic scapes have been used as bio-adsorbents for mercury removal from simulated wastewater. Characterization of prepared bio-adsorbent was carried out by XRD, ATR-FTIR and FE-SEM, EDX. For the optimization of various parameters, batch studies were carried out using AAS (Atomic Adsorption Spectroscopy) by varying contact time, adsorbent dose, temperature and pH. Optimum results were obtained at 10 pH when the adsorbent dose was 50 mg for 45 minutes of contact time at 100ºC. The adsorption efficiency of the prepared biosorbent at optimum conditions was found to be 45–77%. Investigational data were best fitted into a pseudo-second order kinetic model (r^2=0.99993). Therefore, the prepared adsorbent has the potential to remove mercury ions from simulated wastewater and can be explored further at a pilot scale.

Keywords: *Adsorption, Garlic Scapes, Mercury*

1.0 Introduction

Through mining and minerals industries essential raw materials are supplied to various sectors and are vital for global economy. Heavy metals are toxic and are considered as hazardous pollutants at very minimal concentration. Toxic metals like chromium, lead, cadmium, arsenic, copper, and mercury are released into the environment, which causes damage to human health and ecosystems due to the rapid growth and development of modern industries. The US Environmental Protection Agency (EPA) has specified that contaminant level of mercury in drinking water is maximum about 2ppb (0.002 mg/L)[1]. In environment heavy metals are non-biodegradable. In human living tissues heavy metals gets accumulate and causes significant physiological disorders such as changes in blood composition and the central nervous system, as well as damage to vital organs. In wastewater (mining industry, industrial or natural) presence of heavy metals is one of the most serious worldwide environmental problems and a subject of great interest in environmental science, the treatment of

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which is a worldwide environmental concern[2-4]. Many methods like reverse osmosis, coagulation, chemical precipitation, solvent extraction and ion exchange were developed for heavy metals removal from wastewater, with disadvantageous features, such as expensive equipment, produces secondary pollutants and time-consuming process etc. Adsorption technology has gained more interest for heavy metal ions removal from wastewater[5,6]. Adsorption process has been proven to be an effective and simple separation process, main advantages are simplicity of design, low cost, and easy recovery of heavy metals[7,8]. Studies recently has been more focused on the development of efficient and cost-effective adsorbents. In recent years, there has been a surge of interest in cost-effective and waste-biomass-based adsorbents. The utmost striking rewards of bio-material use are cheap cost and easily available in huge quantities. To enhance adsorption efficiency and selectivity, adsorbents can be modified in number of ways. In the meantime, the biomaterials use makes the adsorption process more environmentally friendly. Several authors used various bio-adsorbents such as apple waste, soybean and cottonseed husk, rice husk, agricultural solid waste, peanut hull[9-13] etc. Scanty papers are available using scapes of *Allium sativum* L. as bio-adsorbent for heavy metal removal. Therefore, present study proposes usage of efficient bio-adsorbent prepared from the scapes of *Allium sativum* L. for the green remediation of mercury ions from the simulated wastewater.

2.0 Materials and Methods

2.1 Chemicals and Instruments

The AR Sigma grade chemicals were acquired from M/S. Merck India Ltd., and used for studies. By using double distilled water (DDW) all standard solutions were prepared. Before the use required glassware was soaked in 10% nitric acid (HNO_3) and then washed thoroughly with DDW. A metal ion solution was prepared using $HgCl_2$. Aimil Ltd.'s mechanical shaker (catalog no. AIM 054-1) was used for sieving. For weighing electrical balance of high-precision was used (make: Shimadzu, serial no. 0440E20487). A magnetic stirrer (make: REMI elektrotechnik Ltd. Serial no. ZDFY-15629), and a pH meter (make: Systronics, Serial no.11992) were used to maintain the pH of a solution.

2.2 Collection and Preparation of Bio-adsorbent

In clean brown paper bags wastes of garlic scapes (*Allium sativum* L.) were collected from the "M. S. Ramaiah University of Applied Science" canteen and local canteens in Bangalore. Garlic scapes were separated from garlic wastes and stirred for one hour at 300 rpm to remove mud particles. After washing thoroughly, they dried under sunlight for one week. Dried garlic scapes were ground and sieved through a 150-mesh mechanical shaker. Desiccator was used to remove moisture from powered garlic scapes. Finally stored in labelled airtight bottles prior to the experiments, as shown in Figure 1.

2.3 Characterisation

The shape, surface morphology, and elemental contents of the prepared bio-adsorbent were investigated by using different analysis techniques, i.e., Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR, Shimadzu), Field Emission Scanning Electron Microscopy with an Integrated Energy Dispersive X-ray system (FESEM-EDX, Hitachi S 3500), X-ray Diffractometer (XRD, Bruker), and batch studies were carried out using Atomic Adsorption Spectroscopy (AAS, Shimadzu).

2.4 Adsorption Studies

In 250-mL conical flask batch sorption experiments were conducted and shaken at a speed of 450–500 rpm using a magnetic stirrer. In 100 mL metal solutions (100 µ/L), the effect of prepared bio-adsorbent (10-70 mg) on Hg(II) sorption was evaluated. By varying contact time adsorbent dose, temperature and pH sorption studies were carried out. Whatman filter paper of grade 42 was used for



Figure 1. Preparation of bio-adsorbent.

filtration of solution, and the remaining concentration of the filtered solution was analysed by Atomic Absorption Spectroscopy (AAS). By using Origin 9.0 software, statistical analysis was carried out. The uptake capacity of prepared bio-adsorbent by garlic scapes for Hg(II) ions was evaluated by the following expression:

$$q = (C_0 - C_t)V / m \quad (1)$$

Where,

q (mg/g) is the amount of Hg (II) ions adsorbed onto the adsorbent

C_0 and C_t (mg/L) = represent the Hg (II) ion concentration before and after adsorption for t min.

V (L) is the volume of the solution and m (g) is the amount of the adsorbent dose.

3.0 Results and Discussion

3.1 Characterization of Garlic Scapes

3.1.1 Fourier Transforms Infrared Spectroscopy (FTIR)

The FT-IR spectrum of prepared bio-adsorbent from garlic scapes shows a broad and strong band at 3281.8 cm^{-1} which corresponds to N-H and O-H stretching vibrations. Peaks at 1500 and 1621.5 cm^{-1} attributes to N-H bending and C=C group stretching. Stretching vibration of the C-H bond peak was observed at 2923.1 cm^{-1}. The intense peak at 1019.9 cm^{-1} corresponds to the stretching of carboxylic acid[14-16] (Figure 2).

3.1.2 Scanning Electron Microscope (SEM)

Surface morphology of raw garlic scapes was examined by scanning electron microscopy equipped with an Energy Dispersive X-ray (EDX) as shown in Figures 3 and 4 indicates the fibrous and porous structure of the prepared bio-adsorbent, particle size was found to be 1.36µm. EDX results help provide supporting information regarding the composition of elements presents in raw garlic scapes[15-17].

3.1.3 X-Ray Diffractometer (XRD)

The XRD patterns of raw garlic scapes show a broad peak at an intensity of 550, within the range of 18-28° 2θ value[18]. From the graph (Figure 5), the crystallinity index was calculated, and the value obtained was 40.446%, which shows that prepared raw material is 40% crystalline and 60% amorphous in nature[17]. As prepared material is plant material, many elements are present in it, such as carbon, sulphur, phosphorus, nitrogen, etc. These were matched with standard JCPDS files for carbon, sulphur, and nitrogen

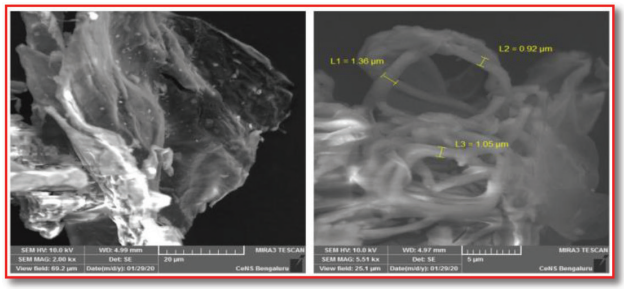


Figure 3. SEM images of raw garlic scapes.

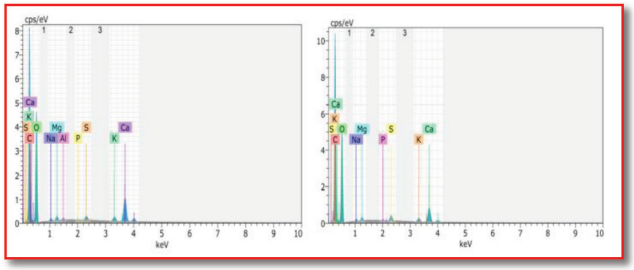


Figure 4. EDX results of raw garlic scapes captured at two different regions.

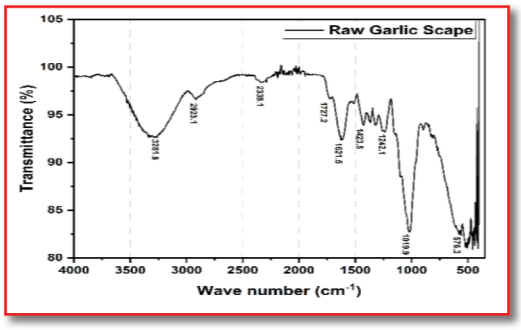


Figure 2. FT-IR spectra of raw garlic scapes.

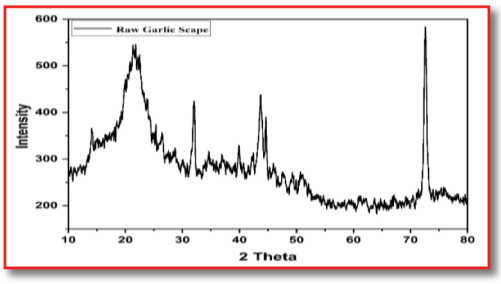


Figure 5. XRD graph of raw garlic scape.

(023-0455; 008-0247; 006-0675). The crystal size was found to be 107.6 nm by using Debye-Scherrer's equation[19].

3.1.4 Effect of Various Adsorption Parameters

Based on the following results the removal efficiencies of prepared adsorbent were investigated: Equation 1 calculates adsorption capacity Qe, while equation 2 calculates percentage removal of Hg(II)[20,21].

$$\%\,\mathrm{Re}\,moval = \frac{(C_o - C_e)}{C_o} \times 100 \qquad (2)$$

Where C_o = initial adsorbent concentration (mg/L), and C_e = final equilibrium adsorbate concentration (mg/L).

3.1.5 Effect of Adsorbent Dose and Contact Time

By keeping the metal ions constant, the adsorbent dose was varied, as shown in Figure 6, where the adsorption of Hg(II) increased by 45% with an increase in the adsorption dose and has been optimised at 50 mg. After this percentage of adsorption efficiency was decreased, an equilibrium was established between adsorbed mercury ions and unabsorbed mercury ions in the prepared solution. By retaining absorbent dose (50 mg) constant, contact time was varied. Equilibrium was attained in 45 minutes with 76% removal of Hg(II) ions[22].

3.1.6 Effect of pH and Temperature

In adsorption process the pH of the solution is an important variable and controlling parameter; it influences the degree of ionisation and speciation of the adsorbate during the process. By keeping the adsorbent dose (50 mg) and contact time (45 min) constant, pH was varied. Maximum adsorption occurred at pH 10, as shown in Figure 6, with 70% of the removal of Hg (II). The decrease in adsorption at low pH and high pH is due to the higher concentration of H^+ in the suspension and the formation of hydroxyl complexes. Temperature studies were carried out by keeping the adsorbent dose (50 mg), contact time (45 min), and pH (10) constant. The temperature was varied from 40 to 120 degrees Celsius (Figure 6). The adsorption process increases with increase in temperature, and 95% of maximum adsorption occurred at 100°C[19,21-23].

3.1.7 Adsorption Kinetics

The rate and mechanism of adsorption were determined by a kinetic model. Linear forms of various kinetic

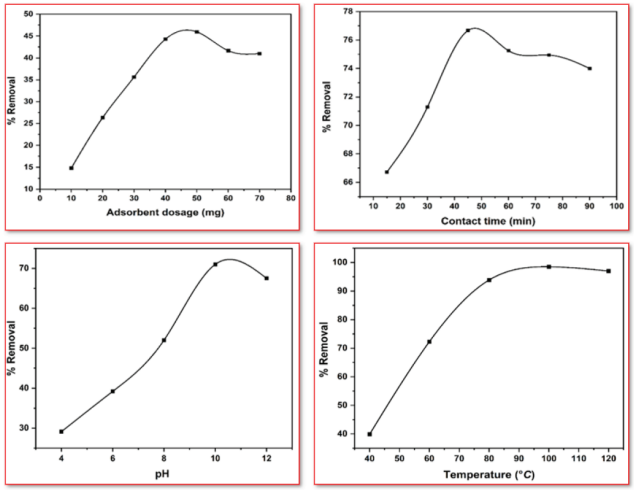


Figure 6. Effect of adsorbent dose, contact time, pH and temperature on the adsorption Hg(II) from aqueous solutions.

models, such as pseudo-first order and pseudo-second order, are as follows in equations 3 and 4 [24-26].

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \qquad (3)$$

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{q_e^2}{k_2} \qquad (4)$$

Where, qe and qt represent the amount of heavy metal adsorbed (mg/g) at equilibrium t, and k_1 (min^{-1}) and k_2 (g $mg^{-1}min^{-1}$) were pseudo-first and second order kinetic rate constants, respectively. The values of the kinetics parameters obtained from the respective liner plots were tabulated in Table 1 and shown in Figure 7. The experimental data was found to be best fitted in a pseudo-second order kinetic model with a determination co-efficient R^2 = 0.99993. The poor linearity of the Lagergren pseudo-first order kinetic plot observed for Hg(II) ions in the present study did not follow first order kinetics but obeyed to pseudo-second order kinetics[26,27].

3.1.8 Adsorption Isotherm

The equilibrium isotherms for Hg (II) ion adsorption on raw garlic scapes obtained at 100°C and pH 10 are presented in Figure 8. Langmuir and Freundlich models, respectively expressed by equations 5 and 6, were fitted to experimental data by linear regression.

$$\frac{C_e}{q_e} = \frac{1}{K_L q_{max}} + \frac{C_e}{q_{max}} \quad (5)$$

$$log\, q_e = log\, K_F + \frac{1}{n} log\, C_e \quad (6)$$

Where K_L and K_F are the Langmuir and Freundlich constant to the adsorption capacity, Q_{max} is maximum adsorbed amount and n is constant related to the intensity of adsorption which are presented in Table 2.

Determination co-efficients R^2 ≥0.97 shows quite good correlation between experimental and modelled data. Langmuir fittings (R^2 = 0.99937) presented lower uncertainties and lower standard errors for the regression with the respective parameter, which means they predict experimental data with a higher accuracy than the Freundlich model[18,28].

3.1.9 Thermodynamics

Thermodynamic parameters were studied to understand the nature of adsorption and the influence of reaction temperature on the removal of Hg(II) by raw garlic scapes shown in Figure 9. Estimated values of standard Gibb's free energy at various temperatures are shown in Table 3. At temperatures of 40, 60, 80, and 100°C, ΔG^0 was calculated to be -1111.05, -2613.98, -7954.59 and -12776.2 J/mol respectively. Negative values at all the reaction temperatures, suggest thermodynamically viable spontaneous nature of the metal cation adsorption on raw garlic scapes. ΔG^0 values were boosted as increase in reaction temperature which shows better possibility of adsorption at higher temperature. The positive value of enthalpy (ΔH^0= 61.6920) indicates the endothermic nature of the adsorption process at reaction temperature for Hg (II) ions. ΔH^0 values are greater than 20.9KJ/mol, which shows that the adsorption process mainly occurred due to ion-exchange or chemisorption mechanisms. A positive value of entropy (ΔS^0 = 197.67) indicates the

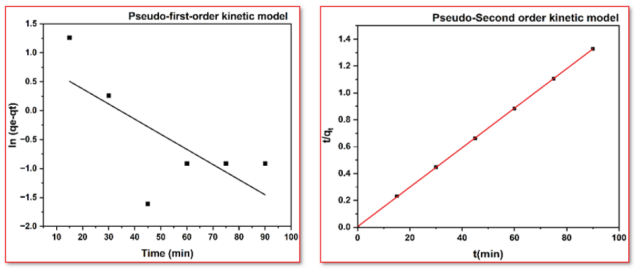


Figure 7. Pseudo-first order kinetic and Pseudo-second order kinetic model plot for adsorption of Hg(II) ions from aqueous solution using raw garlic scapes as adsorbent at pH 10, contact time 45min, 50mg adsorbent dose and at 100°C.

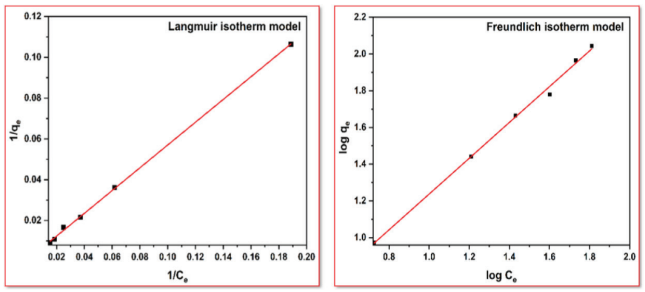


Figure 8. Adsorption Langmuir and Freundlich isotherm model graph.

Table 2. Adsorption equilibrium model

Langmuir isotherm model	Q_{max} (mg g^{-1})	854.7
	K_L (L mg^{-1})	0.002095
	R^2	**0.99937**
Freundlich isotherm model	n	0.97414
	K_F (mg g^{-1} (mg $L^{-1})^{-1/n}$)	1.8347
	R^2	**0.99604**

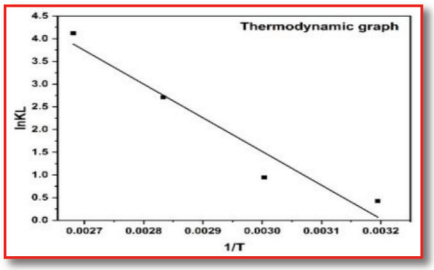


Figure 9. Thermodynamic plot for the sorption of Hg(II) by raw garlic scapes.

Table 1. Kinetic parameter for the adsorption of Hg(II) by raw garlic scapes.

Adsorbate	Adsorbent	Pseudo-first order			Pseudo-second order		
Hg (II)	Raw Garlic Scapes	K_1 (min^{-1})	q_e (mg g^{-1})	R^2	K_2 (g mg^{-1} min^{-1})	q_e (mg g^{-1})	R^2
		-0.00029	2.4565	0.3699	0.04293	68.11	0.99993

Table 3. Thermodynamic parameters value for the adsorption of Hg(II) by raw garlic scapes at different reaction temperatures (K)

Temperature (K)	313	333	353	373
ΔG° (KJ/mol)	-1.1110	-2.613.9	-7.9545	-12.776
ΔH° (KJ/mol)	61.6920			
ΔS° (KJ/mol K)	197.67			
R^2	0.91474			

increased randomness at the solid-solution interface during the fixation of adsorbed compounds on the active sites of the adsorbent[17,27,28].

4.0 Conclusion

The present work emphasized that low-cost raw garlic scapes could be effectively used as bio-adsorbents for removal of heavy metals from industries, mining industries etc. Raw garlic scapes act as reducing agents during batch adsorption for Hg(II) ions removal and equilibrium was reached at 45min of contact time, pH 10, 100°C and 50mg of adsorbent dose. Adsorption study proves that raw garlic scape is good adsorbent which was evident with adsorption data obeying Langmuir isotherm model (R^2-0.99937), pseudo-second order kinetic model (R^2-0.99993) and thermodynamic study (R^2-0.91474) revealed that adsorption process was endothermic and spontaneous in nature at high reaction temperature. Low-cost adsorbent performed well in removal of heavy metal (Hg), this can widely be adopted in industries which minimize cost efficiency and improves profitability. Use of low-cost adsorbents offers a lot of promising benefits for commercial purpose in future and also may contributes to sustainability of the surrounding environment. Also, it helps in reduction of waste generation. Application of raw garlic scapes could be alternative choice for the treatment of Hg(II) ions removal from industrial effluents and modification of raw garlic scape is under investigation. Adsorption of metals and heavy metals using this green bio-adsorbent will be a green remediation emerging as a sustainable solution for various kinds of industries that use metals and heavy metals, providing a greener ecosystem.

5.0 Acknowledgements

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