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Rapid removal of heavy metal ions from aqueous solutions by low cost adsorbents

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Abstract: In the present investigation, different agricultural solid wastes namely: eggplant hull (EH), almond green hull (AGH), and walnut shell (WS), that are introduced as low cost adsorbents, were used for the removal of heavy metals (cobalt, strontium and mercury ions) from aqueous solutions. Activation process and/or chemical treatments using H_2O_2 and NH_3 were performed on these raw materials to increase their adsorption performances. The effectiveness of these adsorbents was studied in batch adsorption mode under a variety of experimental conditions such as: different chemical treatments, various amounts of adsorbents, initial metal-ion concentrations, pH of solutions, contact times, and solution temperatures. High metal adsorption efficiencies were achieved for all cases only in the first two to three minutes of adsorbents' contact time.

Maximum adsorption capacity of AGH sorbent for cobalt and EH for mercury were found to be 45.5 mg/g and 147.06 mg/g, respectively. The adsorption capacity of mercury for WS was also obtained as 151.5 and 100.9 mg/g for two different treated sorbents.

Keywords: environment; heavy metals; removal; low cost adsorbents.

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1 Introduction

Heavy ions are considered hazardous contaminants due to their toxicity, even at low concentration, and non-biodegradability. Heavy metal ions such as cobalt, mercury and strontium can be detected in the waste streams from mining operations, coal combustion and petrochemical complexes. There are various methods for removing heavy metals from aqueous streams such as: chemical precipitation (Mauchauffée and Meux, 2007), reverse osmosis (Mohsen-Nia et al., 2007), ion exchange (Verma et al., 2008), coagulation (El Samrani et al., 2008) and adsorption (Gupta et al., 1997, 2004, 2009; Mohan et al., 2000; Manohar et al., 2006; Gupta and Ali, 2006; Ali and Gupta, 2007; Wibowo et al., 2007; Zabihi et al., 2010).

The above mentioned ions are the priority pollutants listed by USEPA as they can easily pass the blood-brain barrier and affect the fetal brain. These heavy ions can be removed successfully from aqueous solutions by reverse osmosis, coagulation, and ion exchange. However, these methods are much less efficient for low concentrations, for which they can be expensive and even fail to reach the legal limits. In low concentration of these ions, it is appropriate to use adsorption methods.

Adsorption is as an effective and economic method for heavy metal wastewater treatment. Also, the adsorption process offers flexibility in the design and operation. In recent years, investigating low-cost adsorbents that have metal-binding capacities has been intensified. Materials that are locally available in large quantities such as natural materials and agricultural waste, can be used as low-cost adsorbents (Babel and Kurniawan, 2003; Gupta and Ali, 2004, 2006, 2008; Gupta et al., 1998, 1999, 2001, 2003, 2009; Gupta and Sharma, 2002, 2003; Gupta and Rastogi, 2008a, 2008b, 2008c, 2009; Gonçalves Rocha et al., 2009). Other materials which are usually used for water treatment and adsorption of pollutant are activated carbons. In this case, the design and operation of the process is convenient and can be handled easily, therefore the operational costs are comparatively low (Gupta et al., 1997; Mohan et al., 2000; Gupta et al., 2009; Wibowo et al., 2007; Zabihi et al., 2010).

In the present investigation, different agricultural solid wastes namely: eggplant hull (EH), almond green hull (AGH), and walnut shell (WS) were used. They are the most common agricultural products with large quantities in Iran. The object of the present work was to investigate the possibility of ions removal from water by low cost adsorbents prepared from the local agriculture solid wastes. Several prominent factors such as temperature, initial concentration, pH and also isotherm models were investigated in this study.

2 Materials and methods

2.1 Materials

All those mentioned agricultural solid wastes were collected from the local sources. WS which was used for the preparation of activated carbons, was dried in an oven for about one day at 120°C. Dried sample was powdered, sieved (below mesh No. 170), and impregnated with $ZnCl_2$ concentrated solution (136.28 g/mol, 98%) in a weight ratio of 1:0.5 (shell:$ZnCl_2$). The resulting black product was then dried in an oven at 120°C for about 5 h. Impregnated sample was placed on a ceramic boat, inserted in a tubular furnace, and then heated to the carbonisation temperature of 400°C under N_2 flow at the rate of 5°C/min. After carbonisation, the sample was cooled down in N_2 atmosphere. The carbonised sample was washed several times sequentially with hot and finally cold distilled water to remove any residual chemicals. Activated carbon product was then dried in an oven at 130°C. From the particle size distribution measurements the mean diameter was obtained about 0.088 mm.

The other adsorbents, i.e., AGH and EH, as agricultural residues, were washed with distilled water several times to remove water soluble impurities followed by drying in an oven at 180°C. Dried samples were ground and then the following two different chemical treatments were carried out to extract soluble organic compounds of the adsorbents and also to enhance chelating efficiencies:

- mixing with solution of hydrogen peroxide (2%vol) + ammonia (2%vol)
- mixing with solution of nitric acid (20%).

All the chemical and reagents used were of analytical reagent grade obtained from Merck Company.

2.2 Methods

All adsorption experiments were carried out using adsorbents with particle sizes less than 88 μm. Adsorbate containing solutions were prepared by dissolving necessary amount of heavy metal salt in the distilled water. Each solution was then diluted to obtain standard solutions. Batch adsorption studies were carried out with identified amount of sorbents and 50 mL of heavy metal solutions with a desired concentration in two conical flasks, simultaneously. The flasks containing adsorbent and adsorbate were agitated for predetermined time intervals on a mechanical shaker with 720 rpm. At the end of agitation, the suspensions were filtered by the aid of microporous filter paper. The amount of heavy metal ions in the final 25 mL volume was determined by atomic absorption spectrophotometer (Varian, spectra-110-220/880 Australia Pty., Ltd.) equipped with a Zeeman atomizer. The obtained results for two similar solutions were averaged and then reported as the experimental data.

3 Results and discussion

3.1 Sorbents characterisation

The characteristics of samples (WS activated carbons impregnated with $ZnCl_2$ and AGH treated with hydrogen peroxide (2%vol) + ammonia (2%vol)) were determined by nitrogen adsorption at −196°C with ASAP-1100, micromeritics. The specific surface area was calculated from the N_2 isotherms by using the BET equation. The textural characteristics of WS activated carbon and treated AGH are seen in Table 1. Also, the results of Bohem's method on estimation of surface functional groups of adsorbents are presented in Table 2.

Table 1 Textural characterisation of sorbents

Sample	*BET surface area (m^2/g)*	*Pore volume (cm^3/g)*	*Average pore size (nm)*
WS carbon	780	0.426	1.64
Treated AGH	186	0.056	1.92

Table 2 Surface functional groups of sorbents from the Bohem's method (mmol/g)

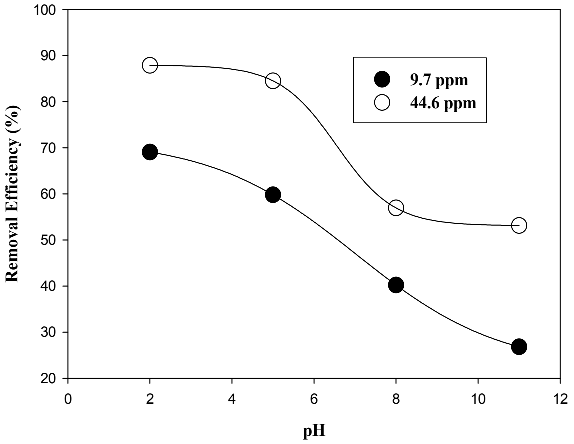
Sample	*Basic groups*	*Carboxylic groups*	*Lactonic groups*	*Phenolic groups*
Raw WS	0.52	0.45	0.49	0.39
WS carbon	0.42	0.54	0.48	0.35
Treated AGH	1.16	0.39	0.45	1.26

3.2 *Effect of pH*

In order to determine the effect of pH on Hg(II) removal by WS activated carbon, some experiments were conducted using two different initial concentrations of 9.7 and 44.6 mg/L. The effect of pH can be seen in Figure 1. The pH of each solution was adjusted by addition of HCl and/or NaOH. It is seen that the removal efficiency of Hg(II) decreases with increasing pH. Some authors indicated that surface charge is an important factor for the adsorption of metal ions (Huang, 1978) and it is very much dependent on the pH of the solution (Srivastava et al., 1989)

It is proposed that by increasing of OH^- concentration in the solution due to increase of pH, Hg(II) ions are extensively hydrolyzed and formed HgO (Gonçalves Rocha et al., 2009). This specie does not show electrostatic interaction with the active sites of the WS active carbon. Therefore, adsorption of mercury ions will be decreased.

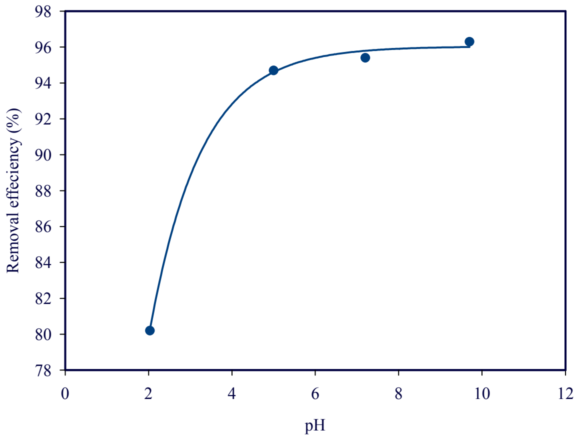
Figure 1 Effect of pH on the adsorption of Hg(II) by WS activated carbon (t = 15 min, T = 29°C)



As shown in Figure 1, the maximum adsorption is observed at pH = 2. It is seen that, for the dilute solution (9.7 ppm or mg/L) the removal efficiency was decreased with a mild slope, but for the concentrated one (44.6 ppm) it was constant in the initial stage (pH range of 2 to 5) and then sharply decreased. The removal efficiency reached a plateau at the pH range of 8 to 11. It can be concluded that at any pH, Hg(II) removal by activated carbon is very much greater for the concentrated solution than the dilute one. This is the result of stronger adsorption driving force in the concentrated solution. In general, the results indicated that the adsorption is highly pH dependent. Similar results have been also reported in other studies (Zhang et al., 2004).

On the other hand the effect of the solution pH on mercury ion adsorption for EH sorbent is illustrated in Figure 2 for initial metal concentrations of 52.6 mg/L. The results from the adsorption capacity studies show that the hydrogen ion concentration plays a substantial role in defining the mercury adsorption process. The adsorption percentage increases in the pH range from 2 to 5 and stays almost constant for pH higher than 5. The oxygen-containing functional groups on the EH surface are responsible for this metal adsorption. It is determined that various oxygen-containing groups with acidic character and different chemical properties (carboxylic, carboxylic in lactone-like binding structures, phenolic hydroxyl and carbonylic groups) are present on the EH surface. The mercury adsorption include formation of surface complexes with the participation of these functional groups on the surface of adsorbent. It is seems that at low pH there is a competition between H^+ and Hg^{++} ions adsorption on the sorbent surface, but in the case of WS activated carbon the tendency for H^+ ion is lower. This could be due to the different surface characteristics of these sorbents.

Figure 2 Effect of pH on the mercury ion removal by EH (C_0 = 52.6 mg/L, T = 25°C, Sorbent dose = 0.1 g, t = 5 min) (see online version for colours)



3.3 *Effect of temperature*

Figures 3 to 6 show the variation of heavy ions concentrations versus time at different adsorption temperatures. To remove Hg(II) ion from water by WS activated carbon, parameters with constant values in the solutions were: ion concentration of 44.6 mg/L, adsorbent dose of 0.05 g, and pH = 5. The experimental data in Figure 3 shows that solution concentration of Hg(II) ions reduces with the temperature increase, indicating an endothermic nature of the sorption processes, while the time required for reaching equilibrium remains practically unaffected. Also, the concentration of Hg(II) in solution decreases with time and attained equilibrium within 30 to 60 min. It is well recognised that the characteristic of the sorbent surface is a critical factor which affects sorption rate parameters. Also, diffusion resistance plays an important role in the overall transport of the ions. Improving the adsorption capacity with temperature suggests that active centres on the surface available for adsorption increase with the temperature. This could be attributed to the pore size variation and enhancing rate of intraparticle diffusion of solute since diffusion is an endothermic process (Zabihi et al., 2010). Similar results are also obtained for other adsorbents at different conditions. To support the above statement, pore volume distribution of AGH and WS carbon are given in Figure 7.

Figure 4 is depicted from the experimental data and shows increase in the adsorption rate of Hg(II) ion from water at different temperatures using EH. The experimental conditions for all cases were chosen to be the same with initial mercury concentration of 52.6 mg/L, sorbent dose of 0.1 g, and pH of 5. These results also confirm similar findings as above.

Figure 3 Effect of temperature on Hg(II) adsorption by WS activated carbon (see online version for colours)

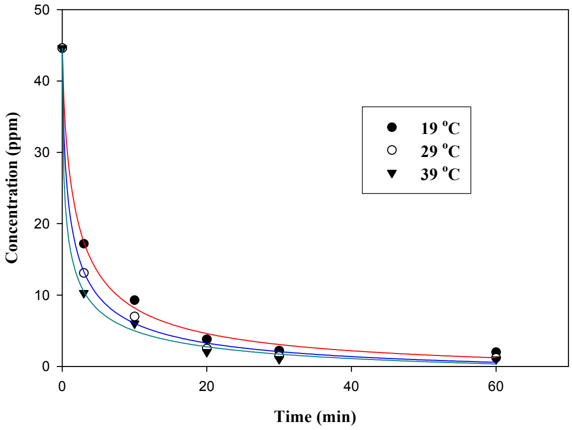


Figure 4 Effect of temperature on Hg(II) adsorption by EH (see online version for colours)

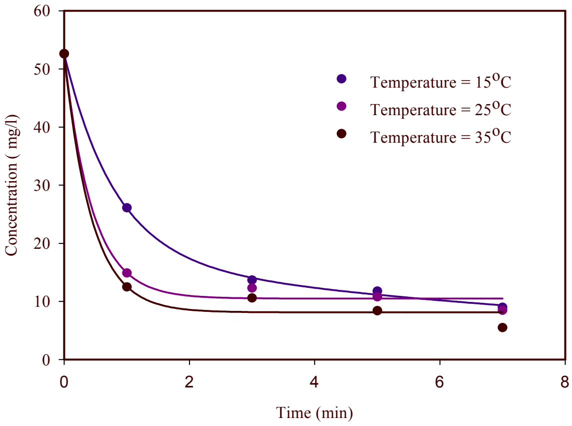


Figure 5 shows the effect of temperature (10, 25 and 45°C) on the adsorption of Sr(II) at different operating times for a fixed dose (0.3 g) of treated AGH. The initial concentration was kept constant at 45.5 mg/L. The experimental data indicate that Sr(II) ion adsorption increases rapidly during the early contact times and the process achieves almost 90% of its removal efficiency. The results also indicate that there is not a significant difference among the amounts of Sr(II) adsorbed at different temperatures especially after 3 min. This is another reason for the great tendency between Sr(II) and AGH and also independency of the adsorption process to the temperature.

Figure 5 Effect of temperature on Sr(II) adsorption by the treated AGH (see online version for colours)

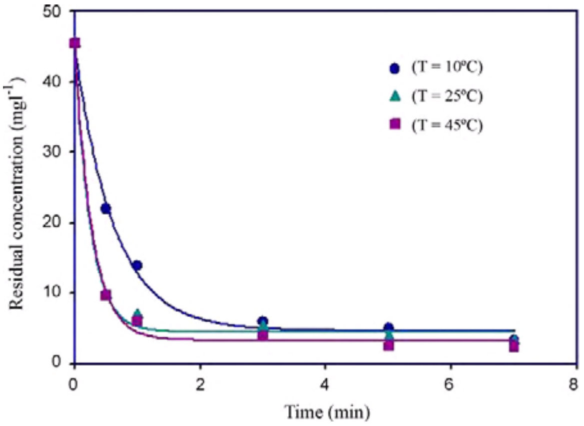


Figure 6 shows the effect of temperatures (15, 25 and 35°C) on adsorption of Co(II) at different times for a fixed sorbent dose of 0.25 g and particles with mesh No. 325. The initial concentration was 51.5 mg/L for all cases. The experimental data indicate that Co(II) ion adsorption was increased by increasing the time. This result is also similar to the above findings.

Figure 6 Effect of temperature on Co(II) adsorption by AGH (see online version for colours)

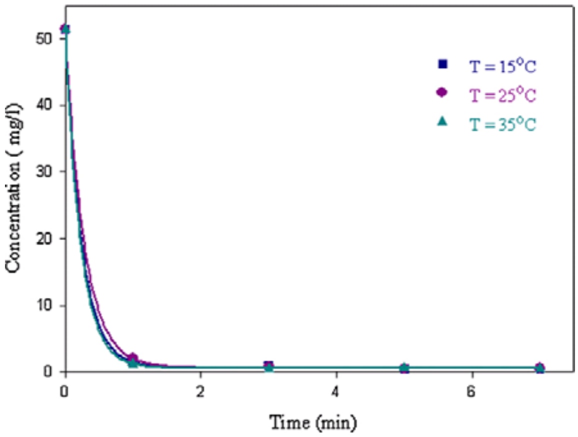
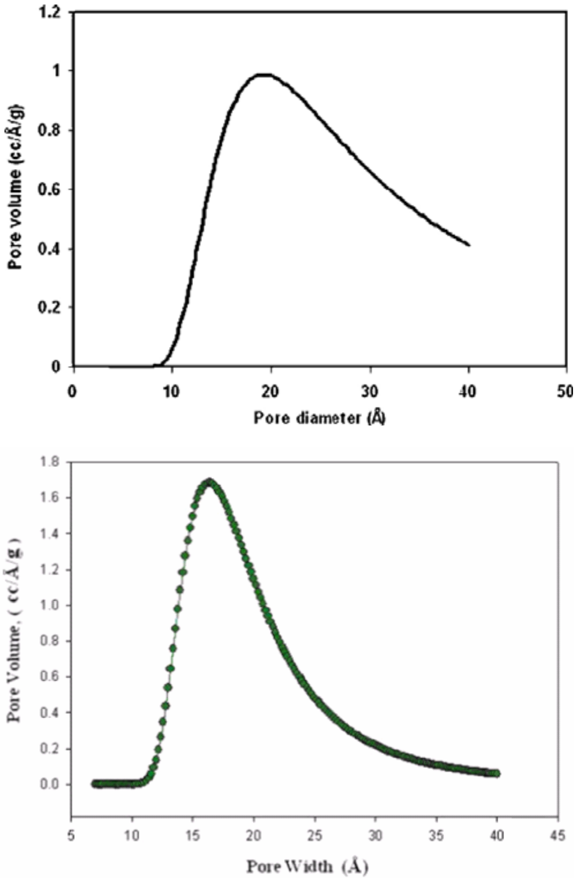


Figure 7 Pore volume distributions of AGH and WS activated carbon (see online version for colours)



3.4 *Effect of initial concentration and contact time*

Usually, in the heavy metal ions adsorption studies, it can be observed that the ions removal increase almost linearly with the enhancement of materials concentration. Many researchers have experienced similar results (Wan Ngah and Hanafiah, 2008; Babel and Kurniawan, 2003). In our investigation, the effect of initial heavy metal ions concentration was also studied using solutions with different initial concentrations, while keeping all other parameters constant. The results of two different types are shown in Figures 8 to 11. The first type is based on the adsorption capacity of adsorbents (mg/g) for WS activated carbon and EH, and the other one is based on the residual concentration in the final solutions (mg/L) for AGH.

Figure 8 shows the effect of initial Hg(II) concentration of 9.7, 20.8, 44.6 and 107mg/L on its removal efficiency using WS activated carbon at 29°C. The amount of Hg(II) ion adsorbed is increased in the initial stage (0 to 20 min range) and after that it has an enhancement to reach equilibrium in approximately 60 min time.

The removal of Hg(II) ion by EH as an adsorbent are studied with various concentrations of 11.2, 24.6, 52.6 and 105.6 mg/L at 25°C, while all other parameters were kept constant (Figure 9). Similar results were achieved in these experiments by increasing the initial concentrations.

Figure 8 Effect of initial concentration on Hg(II) adsorption using WS activated carbon (see online version for colours)

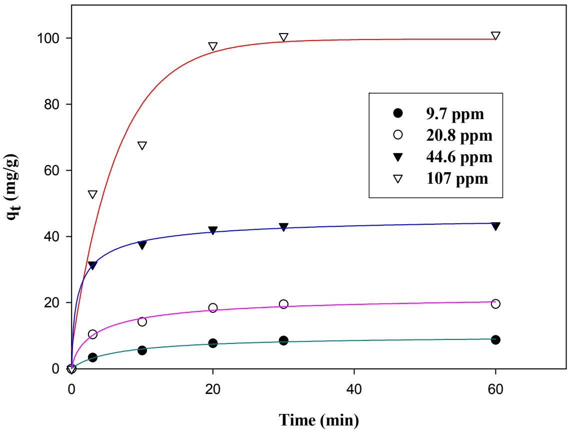
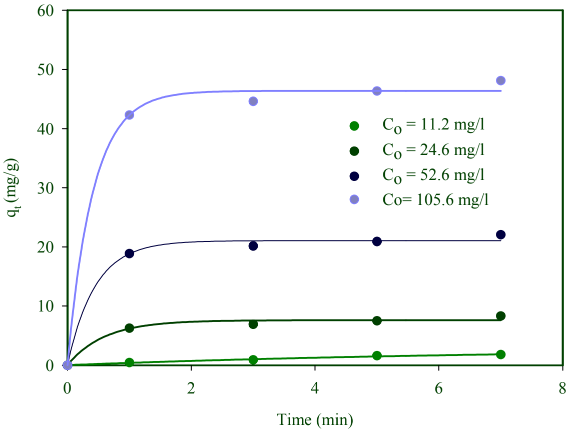


Figure 9 Effect of initial concentration on Hg(II) adsorption using EH (see online version for colours)



The adsorption of Sr(II) has been also investigated using two concentrations of 45 and 102 mg/L at different amounts of AGH (0.2, 0.3, and 0.4 g) and the results are shown in Figure 10.

The effect of initial concentration at different levels ranging from 51.5 to 110 mg/L and constant dose and size of AGH (0.25 g and mesh No. 325) can be seen in Figure 11. It is found that the removal efficiency of Co(II) was increased especially at the very short time of 1 min, but the absolute amount of Co(II) adsorbed per unit weight of AGH was decreased. Sorption efficiency and adsorbed amount per unit weight of Co(II) removal at 17.8, 51.5, and 110 mg/L concentrations and 1 min time are: 98.5% (3.50 mg/g), 97.22%

(10.01 mg/g), and 75.5% (16.60 mg/g), respectively. The equilibrium times are found to be the same for all different concentrations studied. Similar results are also reported by researchers for a variety of adsorbate-adsorbent systems (Manohar et al., 2006; Namsivayam and Ranganathan, 1995; Wibowo et al., 2007)

It is clear from all the above cases that the sorption amount of metal ions is enhanced by increasing the initial ion concentration. The amounts of metal ions adsorbed are also increased sharply with time in the initial stage and then have a gradual enhancement to reach equilibrium.

Figure 10 Effect of initial concentration on Sr(II) adsorption using the treated AGH (see online version for colours)

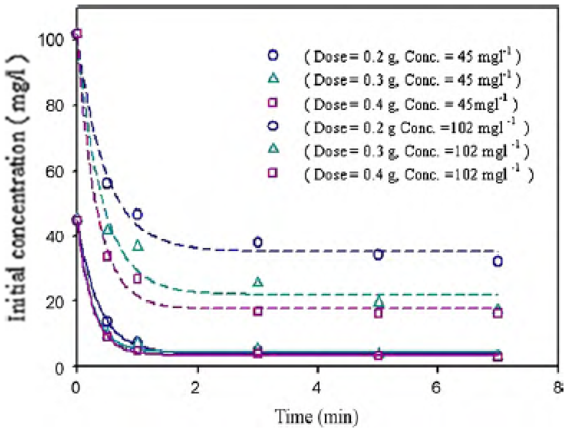
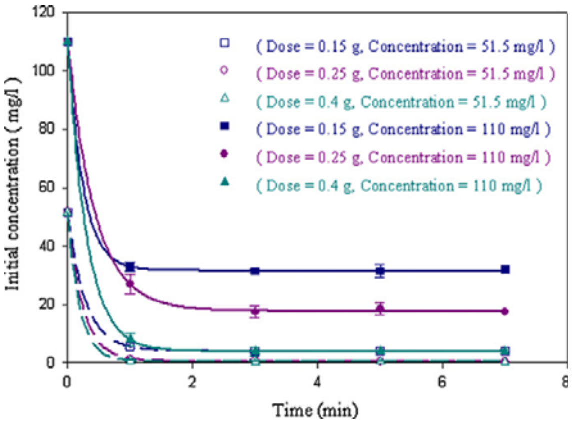


Figure 11 Effect of initial concentration on Co(II) adsorption using AGH (see online version for colours)



3.5 *Adsorption isotherms*

A study on the variation of initial ions concentration at a fixed amount of adsorbent per 50 mL solution was carried out at room temperature. The adsorption isotherms for metal ions sorption on different adsorbents were obtained for various concentrations, while keeping all other parameters constant. The isotherm data are correlated with Freundlich and Langmuir model for further investigation. Langmuir and Freundlich equations are commonly used for describing adsorption equilibrium for water and wastewater treatment applications. Freundlich model often gives a better fit particularly for adsorption from liquids and can be expressed as:

$$q_e = K_f C_e^{1/n} \tag{1}$$

where q_e (mg/g) is the amount of ion adsorbed at equilibrium, and K_f and n are Freundlich constants related to adsorption capacity and adsorption intensity. Usually, for a good adsorbent $1 < n < 10$. A smaller value of n indicates better adsorption and formation of relatively strong bond between adsorbate and adsorbent. Langmuir adsorption isotherm is given by the following equation:

$$\frac{C_e}{q_e} = \frac{1}{q_{m^b}} + \frac{1}{q_m} C_e \tag{2}$$

In this model, q_m(mg/g) is the amount of adsorption corresponding to complete monolayer coverage and b(L/mg) is the Langmuir constant related to the energy or net enthalpy of adsorption. When C_e/q_e is plotted against C_e, a straight line with the slope of $1/bq_m$ is obtained. The values of Freundlich and Langmuir constants obtained from the plots are given in Table 3 for comparison. It is seen that the Langmuir model fitted the results slightly better than the Freundlich model.

Table 3 Parameters of the isotherm models for adsorption of heavy metal ions from water

Freundlich model			*Langmuir model*			*Process*
R^2	*n*	*K_f(mg/g)*	*R^2*	*b(L/mg)*	*q_m(mg/g)*	
0.996	1.215	1.860	0.998	0.0091	151.51	Hg(II), WS carbon
0.999	1.136	1.160	0.989	0.0065	147.06	Hg(II), EH
0.992	1.950	3.590	0.994	0.0048	116.30	Sr(II), AGH
0.88	4.160	14.500	0.98	0.1700	45.50	Co(II), AGH

4 Conclusions

The present study revealed the feasibility of using new cheap adsorbents derived from the agriculture solid wastes for the removal of mercury, cobalt and strontium ions from aqueous solutions. The adsorption behaviour is well described by the pseudo-second-order kinetic and Langmuir isotherm models. The adsorption capacity of WS activated carbon, AGH and EH adsorbents are obtained as 151.51, 116.30, 45.5 and 147.06 mg/g for removing Hg(II), Sr(II), Co(II), and Hg(II) ions, respectively.

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