



Desorption of chromium (VI) and lead (II) ions and regeneration of the exhausted adsorbent

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Abstract

The desorption characteristics of previously adsorbed hexavalent chromium [Cr(VI)] and divalent lead [Pb(II)] ions on groundnut husk were tested by various desorption eluents such as tap water, de-ionized water, NaOH, HCl and H₂SO₄. Among them, HCl and H₂SO₄ were chosen as the best desorbing agents for Cr(VI) and Pb(II) ions, respectively, due to their high desorption efficiency. The desorption efficiency of HCl and H₂SO₄ for Cr(VI) and Pb(II) ions was about 76.1% and 82.1%, respectively, at a concentration of 0.1 M for both elution agents. The exhausted groundnut husk was regenerated up to five cycles, and the removal efficiency of Cr(VI) and Pb(II) ions on the recycled groundnut husk could be maintained at 53.5% and 54.6%, respectively, in the third cycle. The successive regeneration cycles resulted in the reduction of the desorption efficiency by 20.0% and 26.7% for Cr(VI) and Pb(II) ions, respectively, after the third cycle. The results show that groundnut husk could be recycled when used to remove Cr(VI) and Pb(II) ions from Cr(VI) and Pb(II)-polluted water and wastewaters.

Keywords Adsorbent · Biosorption · Desorption · Regeneration · Heavy metals

Introduction

Environmentalists and government agencies both locally and globally are worried about the health risks accompanying heavy metals poisoning in living organisms and underscore the need for continuous research intending to combat the problems of environmental pollution by these toxic metals. Industrial growth has led to a significant increase in naturally occurring metals' levels as experienced currently (Bayuo et al. 2019a, b; Mireles et al. 2012). Contamination of natural environment by heavy metals has to turn out to be a major issue worldwide, and Ghana is no exception because of their toxicity and potential health risks posed to biological organisms when consumed beyond the acceptable limits (Prabha and Udayashankara 2014).

It is a fact that agriculture and mining remain the major key contributors to chemical pollution in Ghana. Vegetable and crop farming in Ghanaian agriculture is prone to either misuse or overuse of fertilizers, pesticides and fungicides. Diseases and pests pose big challenges and problems in crop and vegetable production that usually compelled farmers to use chemical pesticides even if they have no technical know-how in application procedures. Furthermore, the rapid population growth along water sources in Ghana has necessitated proper conservation and efficient utilization of freshwater bodies due to heavy metals and other toxic chemicals. This is because there has been accelerated deterioration of water quality within water bodies due to increased domestic, municipal, industrial, mining and agricultural activities (Dadzie 2012). The sudden increase in illegal small-scale gold mining popularly called “galamsey” in the local communities, especially in Ghana, has led to concerns about the level of environmental pollution coming from these mining activities (Kpan et al. 2014). The emission of heavy metals into the environment from mining operations (Abas et al. 2014; Nriagu 1988) will pollute the surface and underground water sources as well as agricultural soil. When agricultural soils are polluted, these metals are taken up by plants and consequently accumulate in their tissues (Trüby 2003).

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Humans and animals that eat and graze on such contaminated plants and drink from polluted waters, as well as aquatic organisms that breed in heavy metal polluted waters, accumulate such toxic metals in their tissues and milk if lactating (Horsfall and Spiff 1999). These accumulated heavy metals tend to cause various diseases and disorders in living organisms (Tekere and Imamoglu 2005).

Chromium (VI) and lead (II) are being classified by the United States Environmental Protection Agency (USEPA) as potentially hazardous to most living species (Babu et al. 1987). Chromium (VI) is well known due to its health-related issues in humans including carcinogenic and mutagenic risks (Bayuo et al. 2019a, b; Igwe 2007), while neurological disorders in humans, particularly children and in a fetus, are due to the chronic consequences of lead (II) (Bayuo et al. 2019a, b; Lalhruiatluanga et al. 2010).

Therefore, it is necessary to remove metal-contaminated water and wastewaters before its discharge to the environment. However, the disposal of the used adsorbent is again an environmental problem and constitutes a disposal problem. Hence, there is also the need to desorb toxic ions on the surface of the used adsorbent to regenerate it for further use or to ensure safe disposal. Recently, the interest in desorption and re-utilization of adsorbents in metal recovery processes from water and wastewaters has deepened (Kwon and Jeon 2012). Desorption of ions can be performed to restore the adsorbent for further use (Bhuvaneshwari et al. 2012; Grover et al. 2012). After desorption, the metals can be recovered by extraction from the liquid phase so that the naturally low-cost adsorbents could be preserved. Biosorbent regeneration is the supreme fundamental method in assessing its commercial applicability, especially, in both batch and continuous processes, when expensive and selective sorbents are used (Lim and Aris 2014). During desorption procedures, ions accumulated on the biomaterial can be removed easily under appropriate operating procedures, and the adsorbent recycled several times to decrease material cost (Kahraman et al. 2008; Pan et al. 2009). Desorption can be performed by proton exchange using acids, chelating agents or exchange with other ions (Kwon and Jeon 2012). The choice of desorbing agents depends on the kind of adsorbent used and the metals adsorbed.

The removal of Cr(VI) and Pb(II) ions from wastewater has been widely studied using groundnut husk, and these studies have already shown that Cr(VI) and Pb(II) ions could be adsorbed by groundnut husk, which has economic merits compared to other adsorbents (Bayuo et al. 2019a, b). Unfortunately, however, there is little study on the recovery of the Cr(VI) and Pb(II) ions-laden adsorbents and regeneration of these adsorbents. The present study seeks to provide a greater understanding of the dynamic characteristics for desorption and regeneration of Cr(VI) and Pb(II) ions adsorbed onto groundnut husk using different desorbing agents.

Materials and methods

Adsorbent material and characterization

Raw groundnut husk powder was the adsorbent of interest in this study. The physicochemical composition of the groundnut husk was determined in our previous studies using Fourier transform infrared spectrometer (FT-IR) and scanning electron microscopy (SEM) (Bayuo et al. 2019a, b).

Adsorbate solutions

The chemicals and reagents employed for the experiments were of analytical grade and were supplied by the Technician of Applied Chemistry and Biochemistry Laboratory, C.K. Tedam University of Technology and Applied Sciences formerly, known as University for Development Studies, Navrongo Campus. Separate stock solutions (1000 mg/L) of Cr(VI) and Pb(II) were prepared by dissolving the appropriate amount of salts, 1.599 g of lead nitrate [Pb(NO₃)₂] and 2.829 g of potassium dichromate (K₂Cr₂O₇) in de-ionized water, and diluted to the 1000-mL mark with de-ionized water. Working solutions were prepared by diluting stock solutions with de-ionized water.

Desorption and regeneration studies

To carry out the desorption and regeneration studies, 2.0 g/L of the adsorbent of 0.063 mm particle size was introduced into Erlenmeyer flasks containing 25 mg/L of each metal ion [Cr(VI) and Pb(II)] in 100 mL. After equilibration for 60 min under room temperature, the adsorbent was recovered and the adsorption capacity determined. Residual metal ions on the used adsorbent surface were detached through washing for three consecutive times using distilled water and then dried in an oven at 105 °C to constant mass. Five different desorption agents, tap water, de-ionized water, 0.1 M NaOH, 0.1 M HCl and 0.1 M H₂SO₄, were used to desorb the metal ions from the adsorbent. Exactly, 40 mL of the desorption agents was kept in 100 mL Erlenmeyer flasks with the recovered adsorbent and agitated for 60 min with continuous agitation at a 120 rpm at a temperature of 30 ± 0.5 °C. The adsorbate–adsorbent mixtures were filtered after equilibration using Whatman 42 filter paper. The concentration of the Cr(VI) and Pb(II) ions in the filtrate was determined spectrophotometrically (UV–vis spectrophotometer) by diphenyl-carbazide (Bayuo et al. 2019a, b; Mahajan and Sud 2012) and dithizone (Bayuo et al. 2018; APHA 1989) methods, respectively.

Percentage desorption was calculated using the equation below (Katsou et al. 2011):

$$\text{Desorption efficiency (\%)} = \frac{C_{de}}{C_{ad}} \times 100 \quad (1)$$

where C_{ad} denotes the amount of metal ions adsorbed and C_{de} is the concentration of metal ions desorbed. The recovered adsorbent was dried at 105 °C to constant mass, and so the regenerated adsorbent was used in adsorption–desorption cycles to determine the reusability of the groundnut husk. The reusability of the groundnut husk was conducted in five successive adsorption–desorption cycles.

The amount of each metal desorbed from the groundnut husk into the solution per unit mass of adsorbent, Q_d (mg/g) at equilibrium is calculated by (Katsou et al. 2011):

$$Q_d = \frac{C_{de}}{m} \times V \quad (2)$$

where C_{de} (mg/l) is the liquid phase metal concentration detected in the desorbing solution at equilibrium conditions, m is mass of adsorbent and V (L) is the volume of the desorbing solution.

Results and discussion

Desorption of Cr(VI) and Pb(II) ions loaded on the groundnut husk

Desorption studies were conducted to select the optimum desorbing solution to be employed in successive regeneration cycles.

The desorption experiments shown in Fig. 1 indicate that the HCl and H₂SO₄ recovered about 76.1% and 56.0% of Cr(VI) adsorbed on the adsorbent surface, respectively. Conversely, in Fig. 2, H₂SO₄ and HCl were able to recover 82.1% and 74.2% of the Pb(II) adsorbed on the adsorbent surface, respectively. Tap water, distilled water and NaOH showed low desorption percentages of 8.4%, 24.7% and 34.4% for

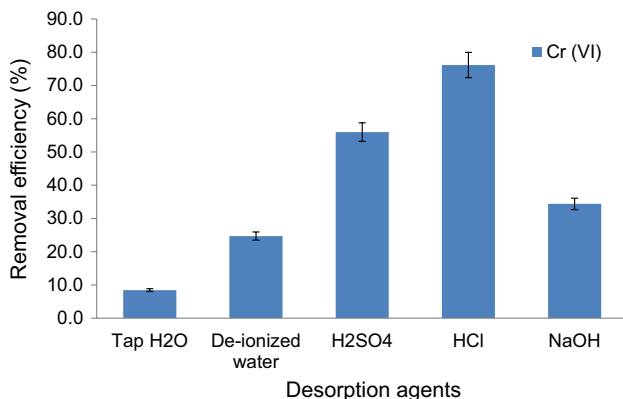


Fig. 1 Desorption of Cr(VI) ions adsorbed on groundnut husk

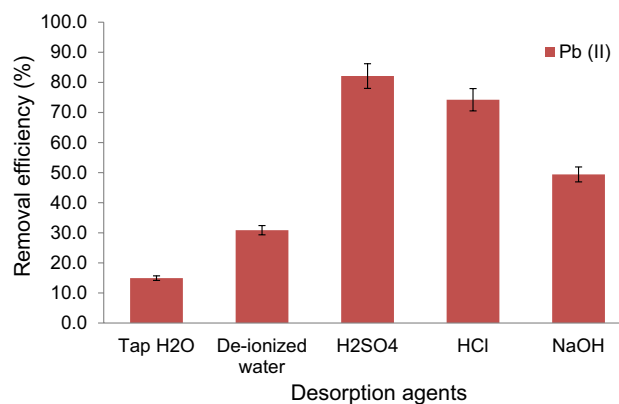


Fig. 2 Desorption of Pb(II) ions adsorbed on groundnut husk

Cr(VI) ion and 14.9%, 30.9% and 49.4% for Pb(II) ion, respectively. The study indicated that Cr(VI) and Pb(II) ions accumulated onto the groundnut husk could be desorbed and maximum percentage recovery of Cr(VI) and Pb(II) ions was 76.1% and 82.1% with 0.1 M HCl and 0.1 M H₂SO₄, respectively. Therefore, 0.1 M HCl and 0.1 M H₂SO₄ solutions were selected, respectively, as desorption reagents for removing Cr(VI) and Pb(II) ions from the groundnut husk surface.

The resultant desorption phenomenon observed in Figs. 1 and 2 for Cr(VI) and Pb(II) ions using HCl and H₂SO₄ as desorption agents, respectively, might be attributed to ion exchange type interaction rather than chemical sorption (Rotimi and Okeoghene 2014). Desorption is useful for the recovery and separation of metal ions in addition to the regeneration of the exhausted bisorbent (Rais et al. 2012, 2013). The hydroxyl and carboxylic groups in the groundnut husk made it amenable to easy desorption and regeneration with the acid solutions (Bayuo et al. 2019a, b; Johnson et al. 2008; Mireles et al. 2012).

The results of this study contradict the finding of Bhuvaneshwari et al. (2012) who studied on regeneration of chitosan after Cr(VI) sorption. In their study, 0.1 M H₂SO₄ was the best desorption agent and maximum desorption efficiency of Cr(VI) was found to be 88% under the following optimum conditions: agitation speed of 120 rpm, a contact time of 60 min and temperature of 40 °C. Furthermore, Wan et al. (2010) investigated the desorption of Pb(II) and Cu(II) ions from chitosan-coated sand in a batch system using tap water and diluted HCl solutions. The results signified that more metal ions could be recovered by the HCl with desorption efficiency of 97.91% and 99.22% for Pb(II) and Cu(II), respectively.

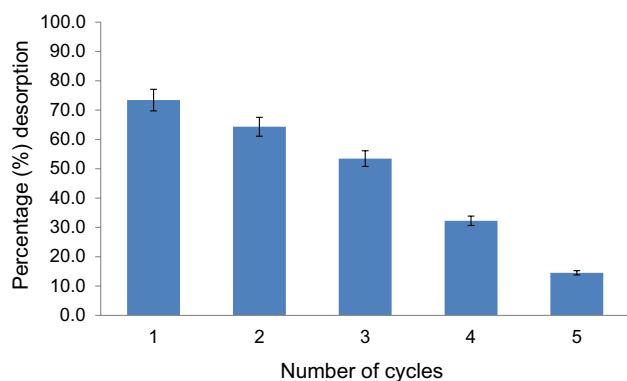


Fig. 3 Adsorption–desorption of Cr(VI) ions and reusability of adsorbent using HCl as desorbing agent

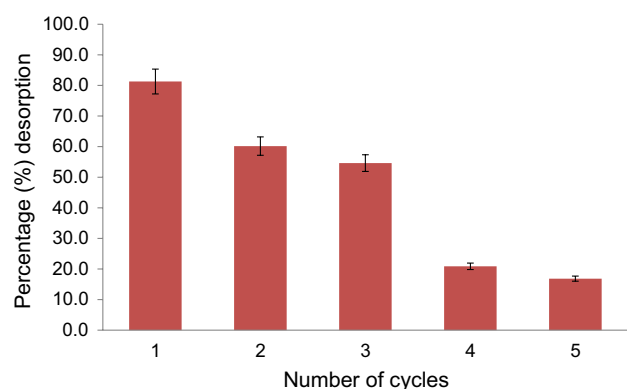


Fig. 4 Adsorption–desorption of Pb(II) ions and reusability of adsorbent using H₂SO₄ as desorbing agent

Regeneration and reusability of the groundnut husk

Several adsorption–desorption cycles were conducted for Cr(VI) and Pb(II) ions to determine the adsorption–desorption potential of the groundnut husk, once it had been regenerated several times. The reusability of the used adsorbent was investigated by performing five successive adsorption–desorption cycles of Cr(VI) and Pb(II) ions on the adsorbent using 0.1 M HCl and 0.1 M H₂SO₄ as respective desorption eluents. There was a gradual decrease in Cr(VI) and Pb(II) removal efficiencies with an increasing number of cycles as shown in Figs. 3 and 4, respectively. It was observed that cycles 1, 2 and 3 showed good retrieval of both metal ions accumulated on the adsorbent surface. For Cr(VI), the removal efficiencies of cycles 1, 2 and 3 were found to be 73.4%, 64.3% and 53.5%, respectively, as presented in Fig. 3. While in Fig. 4, the removal efficiencies of Pb(II) for cycles 1, 2 and 3 were found to be 81.3%, 60.2% and 54.6%, respectively. Hence, after the sequence of three cycles, the Cr(VI) and Pb(II) uptake capacity on the adsorbent was reduced from 73.4 to 53.5% and 81.3 to

54.6%, respectively. The regeneration experiments presented in Figs. 3 and 4 showed that the groundnut husk could be reused repeatedly in the adsorption and recovery of Cr(VI) and Pb(II) ions without significantly losing its adsorption properties.

Generally, regeneration is a key factor in improving the economy of the adsorption process and accessing the potential of the adsorbent for commercial applications. From the regeneration studies presented in Figs. 3 and 4, Cr(VI) and Pb(II) ions uptake capacity on the groundnut husk was reduced after a sequence of three cycles. After this, no significant desorption was observed which indicated that a considerable part of the metal ions adsorbed was due to irreversible sorption. Hence, chemisorption has an important influence on metal ions removal (Kulkarni and Kaware 2015). The loss in the uptake capacity of the groundnut husk for Cr(VI) and Pb(II) uptake during the first three cycles was found to be 19.96% and 26.65%, respectively. This could be attributed to the ignorable adsorbent load lost for the period of the adsorption–desorption cycles. The groundnut husk could be reutilized repeatedly in Cr(VI) and Pb(II) adsorption processes since the Cr(VI) and Pb(II) ions could be desorbed and adsorbent regenerated up to three cycles by 0.1 M HCl and 0.1 M H₂SO₄, respectively. This means that the adsorbent offered an economic benefit to be recycled repeatedly in Cr(VI) and Pb(II) adsorption process. It has been found that if the used adsorbent could be regenerated and used again at the end of the first cycle, then the adsorbent is said to be economical and could be used commercially (Rais et al. 2012).

The findings of this study could be compared to the findings of other studies on desorption and regeneration of exhausted biosorbents.

Gupta and Rastogi (2008) investigated sorption and desorption studies of chromium (VI) from the nonviable cyanobacterium *Nostoc muscorum* biomass. Sorption–desorption of chromium into inorganic solutions and distilled water were observed and the study indicated that the biosorbent could be regenerated using 0.1 M HNO₃ and EDTA with up to 80% recovery. The biosorbent was reused in five biosorption–desorption cycles without a significant loss in the biosorption capacity.

Katsou et al. (2011) studied the regeneration of natural zeolite polluted by lead (II) and zinc (II) in wastewater treatment systems. Several desorbing solutions such as HNO₃, NaCl, KCl and NH₄Cl were examined for the removal of Pb(II) and Zn(II) from zeolite, and the highest desorption efficiency was obtained for 3 M KCl and 1 M KCl, respectively. KCl was the most effective solution for the desorption process exhibiting very high desorption efficiencies (> 98.5%) for both metals.

Kwon and Jeon (2012) conducted a study on the desorption and regeneration characteristics for previously

adsorbed indium ions to phosphorylated sawdust. Various chemical reagents such as HCl, HNO₃, NaCl, ethylenediaminetetraacetic acid and nitrilotriacetic acid were tested on the desorption characteristics of indium ions adsorbed on phosphorylated sawdust. Among them, HCl was chosen as the best desorbing agent with a desorption efficiency of 97% at a concentration of 0.5 M.

A study was conducted by Hossain et al. (2012) on the adsorption and desorption of copper (II) ions onto garden grass. In their study, the desorption of adsorbed Cu(II) from exhausted garden grass was explored using eight types of desorption eluents including tap water, Milli-Q water, distilled water, 0.1 N H₂SO₄, 0.1 N HCl, 0.1 N HNO₃, 0.1 N NaOH and 0.1 N CH₃COOH. It was found that 0.1 N H₂SO₄ was a suitable eluent, which could be used up to five cycles of adsorption–desorption.

In most studies, it is found that the desorption of metal ions in acidic media appeared to be rapid and higher than in basic and neutral media (Samadder et al. 2014).

Conclusion

Batch adsorption–desorption studies were performed for the removal of chromium (VI) and lead (II) ions from aqueous solutions using groundnut husk. The selected adsorbent is viewed as a useful material while considering the economic aspects of wastewater treatment. The study indicated the suitability of the groundnut husk to be regenerated by removing chromium (VI) and lead (II) ions adsorbed on the adsorbent using HCl and H₂SO₄, respectively, as their best desorption eluents. The results showed that for most desorbing solutions lead (II) was more effectively desorbed than chromium (VI) with desorption efficiencies of 82.1% and 76.1%, respectively. Furthermore, the exhausted adsorbent was regenerated up to three cycles indicating its suitability to be utilized repeatedly in Cr(VI) and Pb(II) ions mitigation processes.

Compliance with ethical standards

Conflict of interest The authors declare that there is no conflict of interest regarding the publication of this paper.

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