



(RESEARCH ARTICLE)



Adsorption of heavy metals (lead and chromium) by *Calliandra surinamensis* chaff

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Abstract

Adsorption is one of the most effective ways of removing heavy metals such as Cu, Zn, Pb etc from media. This research work investigated the adsorption efficiency of *Calliandra surinamensis* pod chaff (Powder Puff flower) for the removal of heavy metals from their aqueous solutions using its chaff as the adsorbent. *C. surinamensis* chaff was activated using $ZnCl_2$ solution in water. The activated carbon obtained was characterized in terms of functional groups present in the carbon using FTIR which gave the infrared spectra of the activated carbon of the sample material. The impact of varying adsorption parameters such as pH of the solution, adsorbent dosage, particle size of the adsorbent and contact time on the amounts of lead and chromium removed from their solutions were evaluated to determine the maximum amounts of the metals adsorbed by changing the initial concentrations of the metals from 10 to 50 mg/l in solution. Analysis of variance was also done to determine the effects of varying process variables on the amounts of metals adsorbed. Increase in the process variables considered, pH, adsorbent dosage, particle size and contact time led to a corresponding increase in the amounts of lead and chromium adsorbed according to the order $10 < 20 < 30 < 40 < 50$ mg/l. However, chromium was more adsorbed than lead as pH, adsorbent dosage and contact time increased while lead was more adsorbed with increasing particle size. Analysis of variance showed a p-value > 0.05 for pH and > 0.10 for contact time showed that the variation in pH and contact time were not significant on the amounts of lead adsorbed by the adsorbent. While the p-value < 0.005 for pH and adsorbent dosage and < 0.05 for particle size showed that variations in adsorbent dosage and particle size are significant on the amounts of lead adsorbed. Also, for chromium, a p-value of 0.0145, 0.0001 and 0.0034 for pH, adsorbent dosage and particle size respectively showed that variations in pH, adsorbent dosage and particle size were significant on the amounts of chromium adsorbed by the adsorbent, while a p-value of 0.1679 for contact time showed that variation in contact time has no significant effect on the amount of chromium adsorbed by the adsorbent. In conclusion, *C. surinamensis* activated carbon is a promising and environmental friendly adsorbent for the treatment of aqueous solutions laden with heavy metals of lead and chromium.

Keywords: Adsorption; *Calliandra surinamensis*; Lead; Chromium; FTIR

1. Introduction

Heavy metals present in soils and water are a source of concern as they can cause effect to human health and the environment by ingestion through the food chain [1], [2]. Several water bodies are recipients of water contaminated with heavy metals from domestic, agricultural and industrial effluents [3],[4] Some documented deleterious effects of heavy metals such as lead includes disruption of the biosynthesis of haemoglobin and anaemia, rise in blood pressure and kidney failure, while Chromium can lead to airway obstruction, and lung, or sinus cancer [5]. In several towns and villages in Nigeria, people still depend on river water for both domestic (including drinking and cooking) and agricultural purposes [6]. The risks of which those persons are exposed to in the possible intake of heavy metals through consumption are enormous [7]. The treatment of water contaminated with heavy metals has been achieved with various methods such as Clay, ultrafiltration and agricultural waste materials [8],[9]. The removal of heavy metals from media

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containing them can be actualized by a process called adsorption. It is one of the preferred methods for the removal of heavy metals because it is economical and relatively effective.

Adsorption is the adhesion of atoms, ions or molecules from a gas, liquid or dissolved solid to a surface. This process creates a film of the *adsorbate* on the surface of the *adsorbent*. [5]

C. surinamensis is a genus of flowering plant that is widely distributed in the tropical and subtropical regions of southern Asia, Africa, Australia and America. It is a low branching evergreen tropical shrub that is named after *calliandria* which means 'beautify stamens' and *surinamensis* from the word Suriname, a country in Northern South America. The plant usually has complex branched multiple trunks and grows to a height of about 5 metres, although many sources suggest that it only attains a height of 3 metres. When Left unpruned it grows long thin branches that eventually droop down onto the ground [18]. *C. surinamensis* belongs to the plant family Fabaceae (leguminosae). It is an Angiosperm (flowering seed plants) that grows into shrubs. It is a perennial plant and its mode of nutrition like most plants is autotrophic [18]. Many plants find wide uses in adsorbing heavy metals from aqueous solutions, polluted as well as contaminated environments. *C. surinamensis* is one of such plants.

It has been used in folk medicine to treat various diseases and infections such as cough, wound healing and inflammation. [19]. One of the major uses of the plant *C. surinamensis* in the major areas it has been produced is for ornamental purposes [20]. In many areas, the *C. surinamensis* leaves have also been made use of as feeds for livestock by farmers. The flower pigment of *C. surinamensis* is used folklorically as a colourant and dye. [18]. The plant has been reported to possess antimicrobial, antioxidant activities and as antifungal agent [19],[20]. The Stem bark of *C. surinamensis* has been evaluated from which some chemical constituents have been isolated and characterized previously. They include: (β -Sitosterol, β -Sitosterol glucoside, β -amyryn, xanthone glycoside, and a flavanol glycoside). In a work reported by Falodun *et al.*, these chemical constituents were subjected to *in vitro* free radical scavenging and anti-cancer activities using DPPH free radical scavenging method and lung cancer cell lines. The flavanol and Xanthone glycosides revealed significant antioxidant and anti-cancer activities. The xanthone glycosides were also active on the cancer cell line used for the study [11]. In this study, effort was made to use the dry chaff of *C. surinamensis* to effect the removal of heavy metals from solution as a cheap and alternative method of water purification.

2. Material and methods

2.1. Preparation of *C. surinamensis* chaff sample

The moisture free *C. surinamensis* chaff were loaded into a mortar and cracked open using a pestle to expose the inner seed kernels. The seed kernels were then collected in polythene bags and kept for further usage.

2.2. Size reduction (filtration) of the *Calliandria surinamensis* chaff

The *C. surinamensis* chaff which were of non-uniform size was introduced into a mortar and then continuously crushed with a pestle into a powdery form. Thereafter, the chaff was sieved using a 250 μ m sieve filter to reduce the shafts to uniform size. The particles of uniform size were then washed thoroughly in order to remove the unwanted matter and impurities introduced during the crushing process and to obtain pure chaff.

2.3. Activation of *Calliandria surinamensis*

The carbon of *C. surinamensis* was modified chemically using ZnCl₂ solution prepared by dissolving zinc chloride(136.1 g) in de-ionized water in a 1000 cm³ volumetric flask and made up to the mark with distilled de-ionized water [13],[14],[15].

2.4. Characterization of the activated *Calliandria surinamensis* carbon

The activated carbon was characterized in terms of functional groups using Perkins Elmer Fourier Transform Infrared (FTIR).

2.5. Preparation of standard solutions of Pb²⁺ and Cr²⁺

Stock solutions of lead and chromium of 1000 mg/l from Pb(NO₃)₂ and potassium dichromate (K₂Cr₂O₇) salt solutions by dissolving 1.4675 g of the salts in 100 ml of deionized water which was then transferred into a 500ml Volumetric flask and made up to the mark with deionized water. Working solutions concentrations of 50, 100, 200, 250, 300, 400 and 500mg/l were prepared from the 1000mg/l of stock solution by serial dilution [8].

2.6. Preparation of reagents

1M H₃PO₄: 67.80 cm³ of phosphoric acid (specific gravity of 1.7 g/cm³) was measured into 1000 cm³ volumetric flask and made up to the mark with de-ionized water.

0.1M NaOH: sodium hydroxide (4.0 g) pellets was dissolved in de-ionized water into 1000 cm³ volumetric flask and made up to the mark with distilled de-ionized water.

0.1M ZnCl₂: zinc chloride (136.1 g) was dissolved in de-ionized water into 1000 cm³ volumetric flask and made up to the mark with distilled de-ionized water, [13],[14],[15].

2.7. Adsorption Experiments procedure

The experiments were carried out using the method of [9]. Exactly 50.0cm³ of lead working solution was measured into polythene bottles and adsorbent (0.5g) was transferred into these bottles. The mixtures in polythene bottles were agitated at the same time with a basic rotary mechanical shaker at 200 rpm for 3 hours and left undisturbed on the desk for 24 hrs to allow the system to equilibrate. After 24 hrs, the mixture was filtered through a whatman filter paper into another 100 ml polyethylene bottle. (Okieimen and Okieimen, 2001). The concentration of the residual or remaining lead ions in the filtrate after adsorption process was determined using AAS. The amount of the lead adsorbed was calculated using the equation;

$$q_e = \frac{v(C_o - C_e)}{m} \text{-----1}$$

q_e is the amount of adsorbate ion adsorbed in mg/g of the adsorbent, C_o is the initial concentration of the metal ion before the adsorption process, while C_e is the equilibrium concentration of the metal ion in the filtrate after adsorption process, m is the mass of the adsorbent and v is the volume of stock solution in ml.

Equation 2.7 gives the percentage of metal ion removal by the *C. surinamensis* adsorbent:

$$\% \text{ adsorbed} = \frac{(C_o - C_e)}{C_o} \times 100\% \text{-----2}$$

C_o is the initial concentration of the metal ion before the adsorption process while C_e is the equilibrium concentration of the metal ion in the filtrate after adsorption process

This same process was repeated using the chromium working solution.

2.8. Adsorption of metals with pH variation

Two grams (2g) of 0.4mm particle size of the processed samples were weighed into each separate 500ml conical flask, 50ml of the different lead and chromium solution (10, 20, 30, 40, 50mg/l) each was added. The pH of each metal solution was adjusted to pH 3, 4, 5, 6, and 7. The mixtures were agitated at 250 rpm with rotary mechanical shaker for 40min at ambient temperature. The mixtures were filtered and residual metal in the filtrate was determined with Atomic Absorbance Spectrometer.

2.9. Adsorption of metal with varying adsorbent dosage

Different grams of 0.5, 1.0, 1.5, 2.0, and 2.5 of 0.4 mm particle size of the processed sample was weighed into each separate 500 ml conical flask, 50ml of the different lead and chromium solution (10, 20, 30, 40, 50 mg/l) each was added. The pH of each metal solution was adjusted to pH 5. The mixtures were agitated at 250 rpm with rotary mechanical shaker for 40min at ambient temperature. The mixtures were filtered and residual metal in the filtrate was determined with Atomic Absorbance Spectrophotometer.

2.10. Adsorption of metals with variation in adsorbent particle size

Different particle sizes of 0.2, 0.4, 0.6, 0.8, and 1.0mm of the processed samples were weighed into each separate 500ml conical flask, 50ml of the different lead and chromium solutions (10, 20, 30, 40, 50mg/l) each was added. The pH of each metal solution was adjusted to pH 5. The mixtures were agitated at 250 rpm with rotary mechanical shaker for 40min at ambient temperature. The mixtures were filtered and residual metal in the filtrate was determined with Atomic Absorbance Spectrophotometer [19]

2.11. Adsorption of metals with variation in contact time

Two grams (2g) of 0.4mm particle size of the processed samples were weighed into five separate 500ml conical flask with each flask labeled 20, 40, 60, 80, and 100 minutes and each of the flask was prepared in quintuplicate. 50 ml of the different lead and chromium solution (10, 20, 30, 40, 50 mg/l) was added to each set of quintuplicate. The pH of each the solutions were adjusted to pH 5, each mixture was agitated at 250 rpm with rotary mechanical shaker for 40min at ambient temperature. The mixtures were filtered and residual metal in the filtrate was determined with Atomic Absorbance Spectrometer [19].

2.11.1. Atomic Absorption Spectrometry

The concentration of the residual or remaining metal ion(s) in the filtrates after adsorption process was determined using the Bulk Scientific Atomic Adsorption Spectrophotometer [11] was used for all the determination.

2.11.2. Adsorption experiment

Equilibrium adsorption experiments have been carried out to evaluate the adsorption capacity of the adsorbent. The batch equilibrium for the adsorption was performed in a set of Erlenmeyer flask (250 ml) where solutions of metal ions (50 ml) with different initial concentration (20-120 mg/l) were placed in these flasks. Equal mass (0.5 g) of particle size 325 μ m activated carbon was added to each metal solution. The pH was adjusted to 7 by adding either few drops of diluted hydrochloric acid or sodium hydroxide, operation temperature was ambient temperature (27 ± 2 °C). Thereafter, the different supernatants were filtered and the concentration remaining in the filtrates were determined using spectrophotometer.

The rates of adsorption of the various samples were determined from the uptake level of the selected heavy metals in their aqueous solution.

3. Results and discussion

3.1. Characterization of activated carbon (FTIR analysis)

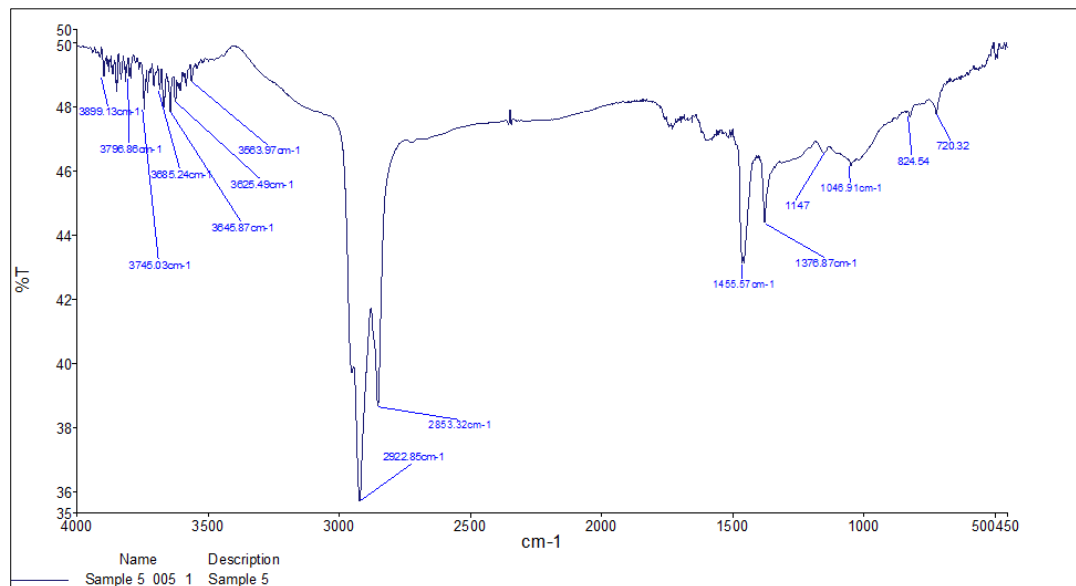


Figure 1 FTIR spectra of the Activated carbon from *C. surinamensis* chaff

Figure 3.1 gives the infrared spectra of the activated carbon (*C. surinamensis*). IR is a great method of identification of many different chemical functional groups such as ether, alkanol, alkane, alkanoate, alkene, alkyne, carboxylic, amide and amine are accountable for binding metallic ions to an adsorbent as presented [10]. In IR, the region 4000-1300 cm^{-1} is the functional group region which is the band to determine the functional group of an unknown compound, the region is also known as the group frequency because they tell us about the presence or absence of specific functional groups in a compound. The region 1300-400 cm^{-1} is the fingerprint region used to compare the spectra of one compound with another, which is known as the bending vibration mode. From the figure above, peak bands occur at 2922.85 cm^{-1}

where coupled vibration of CH₃/CH₂ group which represent Sp³ C–H stretching. At 2853.32 cm⁻¹, there is Sp² C–H stretching which shows the presence of an aromatic aldehyde. There is a peak band at 3563.97 cm⁻¹, which shows an intramolecular hydrogen bonded O–H stretching. There is C=C–H bending of cis di-substituted double bond at 700–720.32 cm⁻¹. At 720–750 cm⁻¹, there is an indication of mono-substituted benzene ring, while at 720–910 cm⁻¹, there is N–H bending. There is the presence of O–H bending at 824–940 cm⁻¹ while there is C–N stretching at 1046–1060 cm⁻¹. At 1147–1150 cm⁻¹, there S=O stretching while at 1220–1376 cm⁻¹, an O–C stretching was observed. An O–H band was seen at 1360–1376 cm⁻¹, CH₃ bending at 1376.87–1380 cm⁻¹, C–O–H in plane bend at 1400–1456 cm⁻¹ and CH₂ bending at 1450 – 1456.57 cm⁻¹. The presence of one or two strong bands below 900 cm⁻¹ indicate aromatic system. All of these bands occurred at the mid IR region of the spectrograph which is from 4000-400cm⁻¹.

3.2. Effect of changes in variables on adsorption of metals

The effects of adsorption process variables on the amounts of lead and chromium adsorbed by *C. surinamensis* activated carbon such as pH, adsorbent dosage, particle size and contact time were evaluated in this section for varying initial concentrations of the metals in solution.

3.2.1. Effect of pH on adsorption of the metals

Table 1 Effect of the variation of pH on the amounts of lead adsorbed by *C. surinamensis*

pH	Amount adsorbed, q (Pbmg/g)				
	10mg/l	20mg/l	30mg/l	40mg/l	50mg/l
3	3.62	8.40	11.73	16.29	17.23
4	3.23	7.28	9.82	13.71	16.06
5	2.06	4.77	3.73	2.37	2.23
6	1.40	3.18	5.38	9.14	11.45
7	1.88	3.34	6.05	9.67	14.84

Table 2 Effect of the variation of pH on the amounts of chromium adsorbed by *C. surinamensis*

Pp pH	Amount adsorbed, q (Crmg/g)				
	10mg/l	20mg/l	30mg/l	40mg/l	50mg/l
3	3.24	7.47	12.56	14.73	19.92
4	2.67	6.08	10.27	11.82	17.14
5	2.51	5.12	5.79	6.78	11.63
6	1.57	2.34	2.68	1.79	5.00
7	1.43	1.51	1.16	4.94	7.34

Tables 3.21 and 3.22 show the effect of the variation of pH on the amounts of lead and chromium adsorbed respectively by the activated *C. surinamensis* carbon as initial concentration of the metals in solution increases from 10 to 50 mg/l. adsorbent dosage, particle size and contact time were kept constant.

The amounts of lead adsorbed at initial concentrations of 10 and 20 mg/l of lead decreased as pH increased from 3 to 6, and only showed a slight increase as pH increased from 6 to 7. As pH increased from 3 to 5, the amount of lead adsorbed decreased for initial concentrations of 30, 40 and 50 mg/g of lead, and increased rapidly as pH increased from 5 to 7. The amounts of chromium adsorbed decreased rapidly for all initial concentrations (10 to 50 mg/l) of the metal in solution as pH increased from 3 to 6, while there was increase in the amounts of chromium adsorbed at initial concentrations of 40 and 50 mg/l as pH increased from 6 to 7, the amounts adsorbed showed further decrease at initial concentrations of 10, 20 and 30 mg/l. Adsorption of the metals shows evident decreases as pH increases from 3 to 5, and showed increase from pH of 5 to 7. The amounts of both lead and chromium adsorbed as shown in the Table 3.21

and 3.22 above increased according to the order 10<20<30<40<50 mg/l. Therefore, increase in the initial concentrations of the metals leads to increase in the amounts of the metals adsorbed in solution.

3.3. Effect of adsorbent dosage on adsorption of the metals

Table 3 Effect of the variation of adsorbent dosage on the amounts of lead adsorbed by *C. surinamensis*

Adsorbent dosage (g)	Amount adsorbed, q (Pbmg/g)				
	10mg/l	20mg/l	30mg/l	40mg/l	50mg/l
0.5	11.72	16.34	29.14	34.92	39.36
1.0	4.05	6.78	8.63	14.13	11.55
1.5	2.26	3.35	4.09	10.38	10.99
2.0	1.01	0.38	0.57	0.12	1.02
2.5	0.85	0.55	0.62	6.93	5.29

Table 4 Effect of the variation of adsorbent dosage on the amounts of chromium adsorbed by *C. surinamensis*

Adsorbent dosage (g)	Amount adsorbed, q (Crmg/g)				
	10mg/l	20mg/l	30mg/l	40mg/l	50mg/l
0.5	9.14	23.36	34.30	44.72	56.12
1.0	2.14	10.55	10.26	15.11	19.94
1.5	1.26	4.04	4.89	8.31	11.04
2.0	0.67	2.12	2.34	4.94	7.29
2.5	0.40	1.23	2.72	5.29	2.49

Table 3.31 and 3.32 shows the effect of adsorbent dosage vs amount of lead and chromium removed by *C. surinamensis* activated carbon when pH, contact time and particle size of adsorbent were kept constant. Initial concentration of the metals increased from 10 to 50 mg/l. The amount of the metals (Pb and Cr) adsorbed increased according to the order of initial concentrations 10<20<30<40<50 mg/l which indicate that increase in the initial concentrations of the metals led to increase in the amounts of the metals adsorbed in solution as adsorbent dosage increased. Chromium was adsorbed more than lead. Furthermore, as adsorbent dosage increased from 0.5 to 2.5 g, the amount of the metals adsorbed (Pb and Cr) decreased, except for initial concentrations 40 and 50 mg/l of lead which increased (amount adsorbed) as adsorbent dosage increased from 2.0 to 2.5 g (Table 3.31).

3.4. Effect of particle size on adsorption of the metals

Table 5 Effect of the variation of particle size on the amounts of lead adsorbed by *C. surinamensis*

Particle Size (mm)	Amount adsorbed, q (Pb mg/g)				
	10mg/l	20mg/l	30mg/l	40mg/l	50mg/l
0.2	4.62	8.06	7.64	3.43	4.24
0.4	3.22	2.29	2.07	3.53	5.47
0.6	4.23	6.33	5.74	9.17	13.05
0.8	3.93	7.90	9.65	12.06	14.23
1.0	4.88	8.45	12.61	13.73	18.14

Table 6 Effect of the variation of particle size on the amounts of Chromium adsorbed by *C. surinamensis*

Particle Size (mm)	Amount adsorbed, q - mgCr/g				
	10mg/l	20mg/l	30mg/l	40mg/l	50mg/l
0.2	4.72	6.12	2.51	5.78	6.40
0.4	2.09	0.84	0.72	0.44	4.77
0.6	4.45	1.84	3.63	3.68	7.34
0.8	4.59	5.13	7.52	8.73	11.23
1.0	4.78	7.24	9.79	12.06	20.39

Tables 3.41 and 3.42 shows the effect of particle size on the amount of lead and chromium removed by *C. surinamensis* activated carbon when pH, adsorbent dosage and contact time were kept constant. Initial concentration of the metals increased from 10 – 50 mg/l. As particle size increased from 0.2 to 1.0 mm, the amount of lead and chromium adsorbed by the activated carbon decreased initially for all initial concentrations at 0.2 to 0.4mm, followed by a further gradual increase in the amount of the metals adsorbed. More of lead were adsorbed than chromium as contact time increased. Again, the amounts of both metals adsorbed increased in the order of initial concentrations 10<20<30<40<50 mg/l.

Table 7 Effect of the variation of contact time on the amounts of lead adsorbed by *C. surinamensis*

Contact time (min)	Amount adsorbed, q (Pbmg/g)				
	10mg/l	20mg/l	30mg/l	40mg/l	50mg/l
20	2.29	6.07	7.07	12.62	3.18
40	0.72	0.77	1.51	1.39	2.90
60	1.84	2.13	3.23	7.18	9.63
80	2.58	2.67	3.34	9.98	12.34
100	3.06	5.74	6.01	10.12	14.53

Table 8 Effect of the variation of contact time on the amounts of lead adsorbed by *C. surinamensis*

Contact time (min)	Amount adsorbed, q(Pbmg/g)				
	10mg/l	20mg/l	30mg/l	40mg/l	50mg/l
20	3.39	7.07	8.72	8.17	5.08
40	0.98	5.39	2.62	2.34	4.90
60	2.72	0.54	4.54	9.08	12.61
80	3.60	3.17	6.28	10.84	14.83
100	4.24	7.40	8.37	12.81	15.19

There was an initial decrease in the amount of metals (Pb and Cr) adsorbed as contact time increased from 20 to 40 mins, and as contact time increased from 40 to 100 mins, the amount of metals (Pb and Cr) adsorbed increased gradually to a maximum level. More of chromium was adsorbed than lead as contact time increased. As previously observed, the amount of both metals adsorbed increased in the order of initial concentrations 10<20<30<40<50 mg/l.

3.5. Statistical analysis

One way analysis of variance (ANOVA) was employed to determine the effect of the variations in pH, adsorbent dosage, particle size and contact time on the amounts of both metals (lead and chromium) adsorbed by the adsorbent. The ANOVA (using Microsoft Excel 2013) was done at a probability of 5% (significant level of 0.05) which indicates that

there is 5% chance of random error in the experiments. A p-value of lesser than the set probability of 0.05 indicates that the factor is significant while a p-value greater than indicate that the factor is insignificant. For lead, a p-value of 0.0634 for pH (slightly insignificant) and 0.1237 for contact time shows that the variation in pH and contact time are not significant on the amounts of lead adsorbed by the adsorbent. While pH values of 0.0000 for adsorbent dosage and 0.011 for particle size shows that variations in adsorbent dosage and particle size are significant on the amounts of lead adsorbed. Also, for chromium, a p-value of 0.0145, 0.0001 and 0.0034 for pH, adsorbent dosage and particle size respectively showed that variations in pH, adsorbent dosage and particle size are significant on the amount of chromium adsorbed by the adsorbent, while a p-value of 0.1679 for contact time showed that variation in contact time has no significant effect on the amount of chromium adsorbed by the adsorbent.

4. Conclusion

In this work, *C. surinamensis* activated carbon was employed to remove lead and chromium from their aqueous solutions. The effects of varying the process variables considered showed that adsorption of the metals is maximum at a pH of 3, adsorbent dosage of 0.5 g, particle size of 1.0 mm and contact time of 100 minutes. Chromium was more adsorbed by the activated carbon than lead. The amount of the metals adsorbed increased in the order of initial concentrations 10<20<30<40<50 mg/l for all process variables considered. Hence, *C. surinamensis* activated carbon is a promising adsorbent for the removal of metals from aqueous solutions.

Compliance with ethical standards

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Disclosure of conflict of interest

The Authors declare no conflict of interest.

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