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Adsorption of Lead (ii), Chromium (vi) and Manganese (ii) metal ions from water using modified *Pennisetum purpureum* Schumach plant stalk

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ABSTRACT

Water pollution is a global problem affecting humanity. Availability of clean water is a fundamental prerequisite to public health safety and the survival of the human race as well as animals. However, pollution of river water by heavy metal ions deposition is a grave environmental problem. Presence of the heavy metals in the water, pose a serious health risk particularly to rural populations which rely majorly on the river water for domestic purposes. Several methods for elimination of the heavy metal pollutants from river waters have been previously employed. However, most of these methods are expensive and cumbersome, hence not sustainable. This research explored the potential of modified *Pennisetum purpureum* Schumach plant adsorbent in removal of Lead (II), Manganese (II) and Chromium (VI) ions from spiked water samples by adsorption. Surface analysis of the adsorbent using FTIR Photometer confirmed presence of functional groups such as $-NH_2$, C=O and O-H responsible for adsorption of heavy metal ions. Concentration of heavy metal ions in water samples was determined before and after adsorption process using Atomic Absorption Spectroscopy (AAS) and means concentration computed. Effect of changes in pH on adsorption was investigated by conducting adsorption at three different pH conditions of 5, 7 and 9. Modification of the adsorbent surface improved its adsorption capacity. For instance, at pH 5, adsorption of Cr^{+6} increased from 12% to 14.82% when modified adsorbent was used. The modified adsorbent achieved greater % adsorption in all the three metals. Increase in the pH of adsorption from 5 to 9 had a reducing effect on the percent adsorption. At pH of 5 the % adsorption of Pb^{+2} reduced from 90.26% to 73.75% at pH of 9. This reductions in percentage adsorption were recorded for Pb^{+2} and Mn^{+2} ions but not significant for Cr^{+6} ions. These results show the potential of *P. purpureum* plant for use as an adsorbent in purification/detoxification of river water which would present a cheaper and more readily available alternative to many current adsorbents and adsorption methods in use. However further research is needed to determine the optimal set of conditions as well as the most suitable modification treatment for maximum adsorption.

Keywords: *Pennisetum purpureum* Schumach, Adsorption, Functional groups, Atomic Absorption Spectroscopy.

INTRODUCTION

Water pollution is a global problem (Mateo-Sagasta *et al.*, 2017). Pollution is contamination of an ecosystem due to the introduction of harmful chemical compounds in the environment (Gunatilake, 2015). Although most of the earth's surface is covered with water, only 1% is available for human use, the rest is salty sea water. Where freshwater is available, it occurs in frozen form or is inaccessible to man for practical use (Zhongming *et al.*, 2018).

In rural Kenya, most people rely on use of river water for domestic purposes, such as drinking, cooking watering animals, brick making, watering crops and many others

(Munyao, 2017). River water pollution has great negative effects due to bio-accumulation and bio-amplification of persistent toxic heavy metals in human (Hussain *et al.*, 2019). Heavy metals are metallic elements with comparatively high atomic numbers (Kinuthia *et al.*, i2020). Heavy metal ions are reported as most detrimental pollutants due to their mobility in natural water ecosystems also due to their toxicity (Mangwandi, 2020).

Heavy metal toxicity results in lower energy levels in humans and affects blood composition, as well as damaging lungs, liver, kidney and other vital organs. Also, heavy metals, interferes with or reduces mental and central nervous functions. They are also believed to be key cancer-causing agents (Haseena *et al.*, 2017; Wasike *et al.*, 2019; Mangwandi, 2020). Therefore, it is very important to find a way of eliminating these metal ions from water.

Adsorption process is very simple, economical, and effective, therefore most preferred method for removal of toxic heavy metal contaminants in water (Jain *et al.*, 2016), other methods of removing heavy metal ions such as chemical precipitation, ion exchange, reverse osmosis etc. have several disadvantages which includes: High reagent requirements; Unpredictable metal ion removal; Generation of toxic sludge (Sulyman, 2017). Therefore, adsorption becomes preferred method of heavy metal ions removal.

Use of plant materials (bio-adsorbents), mainly low-cost agricultural wastes and by-products to remove heavy metals from contaminated water. These adsorbents are preferred due to their low cost and high adsorption efficiency which is accredited to presence of different active functional groups in them (Massimi *et al.*, 2018). Bio-adsorbents mainly consist of cellulose, lignin and hemicelluloses but other compounds such as starch, hydrocarbons, lipids, starch and simple proteins may also be present (Malik *et al.*, 2017). In addition, waste materials especially from agricultural operation are inexpensive and can be disposed of without expensive regeneration (Sulyman *et al.*, 2017). Plant materials contain active functional groups that have effective adsorption sites of heavy metal ions (Jain *et al.*, 2016).

This paper reports convenient and practical method that can be used to remove selected heavy metal ions from polluted river water using locally available materials. The research focuses on removal of heavy metal ions by adsorption using chemically modified plants stalks of *P. purpureum* from spiked water samples. *P. purpureum* is a type of grass commonly known as Napier grass. It grows in most parts of central Kenya as its used as common fodder crop for livestock. Traditionally, some farmers grow it in areas affected with soil diseases due to its soil cleansing properties. After adsorption of heavy metals, it should be disposed of safely.

METHODOLOGY

Chemicals and reagents

Analytical grade reagents were used in all preparations. These included concentrated nitric acid (HNO_3), Sodium hydroxide (NaOH), Hydrogen peroxide (H_2O_2), concentrated Hydrochloric acid (HCl), Potassium Hydroxide (KOH) and Potassium Bromide (KBr). Commercial stock solutions of Lead(II)nitrate [$Pb(NO_3)_2$], Manganese(II)sulphate, [$MnSO_4$] and Potassium dichromate(VI),[$K_2Cr_2O_7$] were used as purchased. Distilled water used to wash apparatus. Double distilled water to prepare spiked solutions.

Instrumentation

The mean concentration of selected heavy metal ions were determined using Atomic Absorption Spectroscopy (AA 6300 Shimadzu). The pH values of the analyte were determined using PHEP Hanna Instrument. Fourier Transform Infrared Spectrophotometer used for characterization of surface of the adsorbent was obtained from IRTracer-100 Shimadzu.

Preparation of adsorbent material and its chemical modification

Collection of *Pennisetum purpureum* stalks

Stalks from mature *P. purpureum* plants were collected (Plate 1). The grass of about four months from previous harvesting was collected from farms in the county of study. The stalks were then thoroughly washed in distilled water before being sun dried for four days. The dried stalks were then shred into pieces of about 1cm long after which about 1000 grams of the shred pieces were prepared, and soaked in boiling water for about 30 minutes to soften the adsorbent. After boiling the shredded pieces of *P. purpureum* were washed in running tap water then sun dried for about four days until they obtained a brittle texture. The dried brittle pieces were then ground into coarse powder using a laboratory mill. About half (500 grams) of the powder was stored in a clean stopped plastic container as unmodified Adsorbent.

The remaining *P. purpureum* powder (500 grams) was soaked in 0.5M KOH for 24 hours at room temperature (Sulyman *et al.*, 2017). At the end of the modification process, the adsorbent was filtered and rinsed with 0.5M HCl before washing with distilled water. It was left in distilled water for 2 to 3 hours during which the water was changed several times until the pH of filtrate remained

constant (pH 5) (Amer, 2015). The clean modified plant adsorbent was then sun dried and further dried in an oven. Drying was done for five hours at a temperature of 80^{0}C. Both the chemically modified and unmodified *P. purpureum* adsorbents were then weighed into 2 grams portions each and stored in separate labeled air-tight containers ready for adsorption studies.

Plate 1: *Pennisetum purpureum* (Napier grass) plant. (Source: Author)

Study of the surface of adsorbent material samples using FTIR

Surface analysis of the adsorbents to determine functional groups present was done using FTIR. About 2mg of modified sample of *P. purpureum* adsorbent was mixed with 200mg of KBr, dried and ground. The particles size was ground to less than 2 micrometers. The mixture was then squeezed to form transparent pellets ready to be analyzed using FTIR, (Ghiasvand, 2020; Sulyman *et al.*, 2017). The back ground spectrum was obtained by collecting an interferogram and its subsequent conversion to frequency data by inverse Fourier Transform.

Preparation of standard solutions and calibration

Commercial standard solutions Lead (II) nitrate, Manganese (II) sulphate and Potassium dichromate (VI) were used. Stock solution was diluted further by measuring 10 ml and placing it into a 100 ml volumetric flask topping up to the mark to make 100 ppm solution. Working standard solutions were prepared by serial dilution of 100 ppm stock solution by serial addition of distilled water. Concentration of 2,4,6,8 and 10 ppm for every metal ion was obtained. These solutions were maintained at pH value of 4 by addition of 0.5M Nitric acid or 0.5M Potassium hydroxide. Low pH value was maintained in order to ensure the metal ions do not precipitate. The solutions were used to calibrate the Atomic Absorption Spectrometer (AAS).

Adsorption process

This process was conducted in triplicate. Batch adsorption was carried out by putting 50ml water samples into clean 250 ml conical flasks. The water samples prepared earlier, whose concentration of heavy metal ions had already been determined. Then 2g of adsorbent was added into the conical flask. The mixture was shaken at 100 rpm for a time interval of 60 minutes. After which the suspension was separated by decantation. The solution was then separated further using vacuum filter machine fitted with Whatman filter paper no.42. The volume of the filtrate was determined and labeled waiting analysis of heavy metal ions concentration. The residual metal ion concentration was determined using AAS. The quantity of the heavy metal ions adsorbed was determined. Percentage adsorption was obtained by working out difference in mean concentration of each metal ion before and after adsorption multiplied by 100.

The results obtained were analyzed and compared with results of concentration of heavy metal ions obtained before adsorption. The effects of pH on adsorption process were initiated by adjusting pH of each sample solution to required value by adding 0.1M HNO_3 or 0.1M NaOH solutions. The pH ranges used were 5, 7 and 9. This research focused on comparative adsorption using plant adsorbent prepared from *P. purpureum* at various pH environments. The procedure was repeated four times using each 2g of adsorbent, at each pH environment and results of metal ion concentration in each case recorded. The average metal ion adsorbed was calculated at each pH environment.

Data analysis

The heavy metal ions present in water samples and their concentrations was determined prior adsorption. The average concentration was calculated. After adsorption, the filtrate was analyzed and the average concentration of heavy metal ion determined. The results obtained above were compared with mean concentrations obtained prior adsorption and analysis of variance determined using statistical analysis of variance (ANOVA). Optimum adsorption and other statistical analysis were done at 95% confidence interval was calculated using the SPSS statistical software version 25. Results with $p<0.05$ was considered statistically significant.

RESULTS AND DISCUSSION

FTIR Characterization of adsorbent material

Both the modified and unmodified adsorbents were chemically characterized for their chemical composition. FTIR confirmed that the hydroxyl, carbonyl, amine and double covalent bonds groups were the main groups present in the adsorbents. The carboxyl and amino functional groups provide the major adsorption sites for the lead ions binding. The analysis showed that NaOH modification leads to an increase in the number of adsorption sites

for metal uptake (Malik *et al.*, 2017). The unmodified adsorbent exhibited peaks at 3397 cm^{-1}, 3104 cm^{-1} and at 1641 cm^{-1} and i1403 cm^{-1} which were shifted to 3401 cm^{-1}, 3125 cm^{-1}, 1640 cm^{-1} and 1412 cm^{-1} respectively in the modified adsorbent (Figure 1).

The broad peak observed at 3397 – 3401 cm^{-1} in unmodified adsorbent is assigned to the stretching of hydroxyl (O–H) group as well the stretching of secondary amine (N-H) group (Al-Ghamdi and Khan, 2020). The O–H stretching vibrations occur within a broad range of frequencies indicating the presence of free hydroxyl groups and bonded O–H peaks of carboxylic acids (El-Naggar *et al.*, 2018). The peaks at 1641 and 1640 cm^{-1} are assigned to the carbonyl (C=O) stretching vibration of the ketone or due to the stretching vibrations of the double covalent bond (C=C) (Al-Ghamdi and Khan, 2020). The peak range 1403i– 1412 cm^{-1} is strongly associated with the presence of carbonate minerals (correlated to carbonyl group, C=O).

These functional group binds with heavy metal ions during adsorption. Amine groups are very effective in adsorption of Lead and Iron ions (El-Naggar *et al.*, 2018). At low pH values of 1-5, the functional groups are negatively polarized due to dissociation of H^+ ions attracting heavy metal ions (Massimi *et al.*, 2018). The binding of heavy metal ions present in water occurs by functional groups of adsorbent donating pair of electrons forming complex compounds (Al-Ghamdi and Khan, 2020). The interaction between heavy metal ions and functional groups on the adsorbent's surface results in adsorption of the metal ions from contaminated waters.

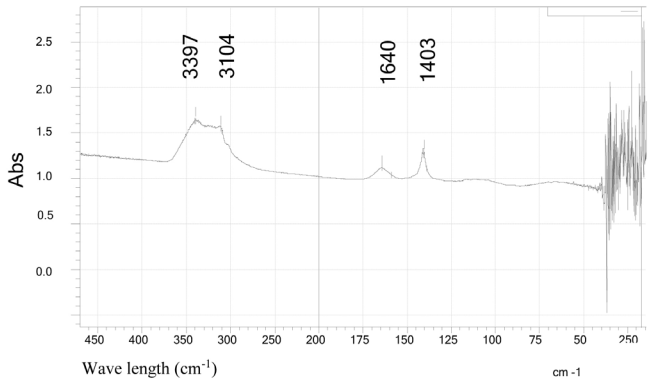

Figure 1: FTIR Spectrum for the unmodified adsorbent.

Modified adsorbent exhibited peaks at 3401, 3125, 1640 and 1412 cm^{-1} as shown in figure 2.

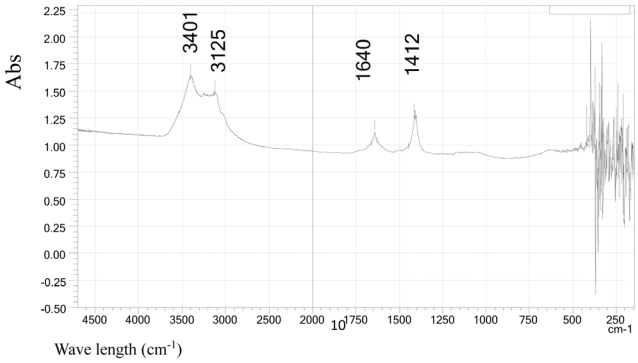

Figure 2: FTIR Spectrum for the modified adsorbent.

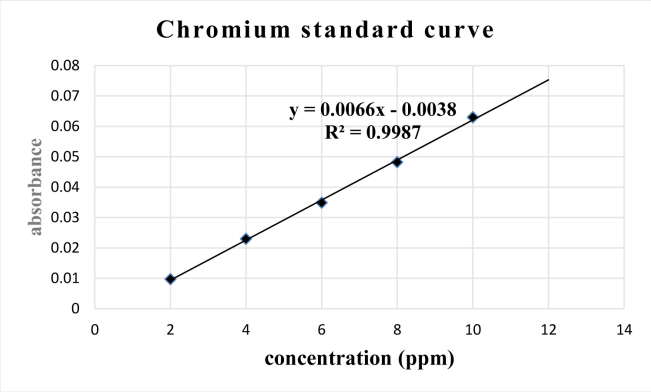

Figure 3: Standard curve for chromium analysis.

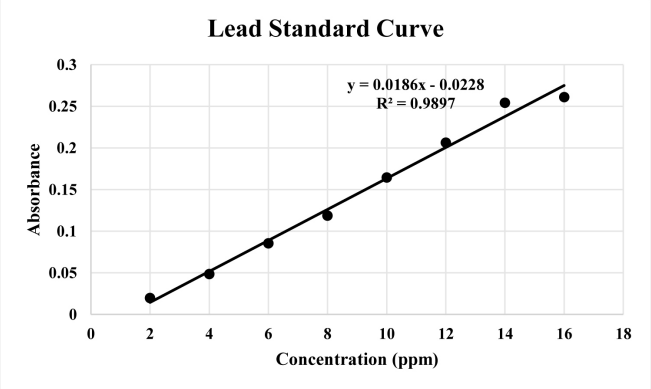

Figure 4: Standard curve for lead analysis.

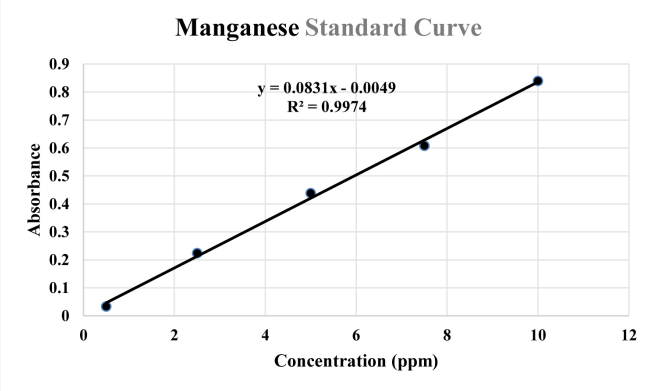

Figure 5: Standard curve for manganese analysis.

Atomic Absorption Spectrometer (AAS) validation results

The method and procedure were validated by use of standard spiked solutions of known concentrations. The calibration curve of Absorbance verses concentration of each heavy metal ion was used to determine concentration of Cr^{+6}, Pb^{+2} and Mn^{+2} ions in river water samples using AAS. Calibration curves for chromium as well as for lead and manganese analyses are presented in figures 3 to 5.

A linear plot of y =0.0066x – 0.0038 over the concentration range of 2.0-10.0 mg/l with a slope of 0.033 and a correlation coefficient of (R^2) = 0.9987 was obtained for chromium while linear plot of y =0.0186x – 0.0228 over the concentration range of 2.0-10.0 mg/l with a slope of 0.033 and a correlation coefficient of (R^2) = 0.9897 was obtained for lead and linear plot of y =0.0831x – 0.0049 over the concentration range of 2.0-10.0 mg/l with a slope of 0.033 and a correlation coefficient of (R^2) = 0.9974 was obtained for manganese.

High values of R^2 (tending to 1.00) obtained for the standard curves for each of the three elements indicate high accuracy of the results (the curve accurately represents the detector response). The intercept of 3 the curves were close to zero indicating minimum matrix interference and their slopes were above zero meaning that the instrument was sensitive. The curves were used to quantify the heavy metals in water samples after adsorption.

Batch adsorption of the selected heavy metals using unmodified and modified adsorbent

As shown in table 1, higher percentage adsorption was realized by use of modified adsorbent than unmodified adsorbent for all the three metal ions, chromium, lead and manganese. For both chromium and lead ion percent adsorption by modified adsorbent was significantly higher than that of unmodified ($p<0.05$) adsorbent for all the three different pH of adsorption. However, for manganese, although the modified adsorbent achieved higher adsorption than the unmodified adsorbent, the difference was not significant ($p>0.05$) for all the three different pH of adsorption.

Table 1: Comparison of adsorption of metal ions by unmodified and modified adsorbents under different pH conditions.

Metal	pH of adsorption		Mean % adsorption of spiked solution (100 ppm)		Statistics[1]		
			Unmodified adsorbent	Modified adsorbent	t	df	p-value
Chromium		PH 5	12.000±0.77[a]	14.818±0.32[a]	-3.38	4	0.028
		PH 7	9.639±0.33[b]	12.010±0.21[b]	-6.06	4	0.004
		PH 9	8.662±0.32[c]	9.670±0.12[c]	-2.98	4	0.041
	Statistic[2]	df	2	2			
		f-value	28.925	96.897			
		p-value	0.004	<0.001			
Lead		PH 5	68.354±0.27[a]	90.255±0.18[a]	-67.65	4	<0.001
		PH 7	60.811±0.95[b]	81.362±0.37[b]	-20.22	4	<0.001
		PH 9	54.905±1.17[c]	73.750±0.47[c]	-14.95	4	<0.001
	Statistic[2]	df	2	2			
		f-value	69.44	<0.001			
		p-value	0.001	0.006			
Manganese		pH 5	46.297±1.14[a]	53.102±3.52[a]	-1.84	4	0.140
		pH 7	39.885±0.93[b]	46.022±3.68[b]	-1.62	4	0.181
		pH 9	35.434±1.29[b]	40.912±6.73[c]	-1.38	4	0.238
	Statistic[2]	df	2	2			
		f-value	69.44	<0.001			
		p-value	0.001	0.006			

Note

- [1] – independent t-test
- [2] – repeated measures ANOVA
- values with superscripts of different letters in the same column are significantly different at $\alpha = 0.05$

Results for the adsorption experiments at different pH values are as shown in figures 6-8. The percentage adsorption of metal ions from spiked solutions increased from 9.67% to 14.8% for chromium, from 73.75 to 90.255% for lead, with a decrease in pH from 9 to 5 when modified adsorbent was used. Table 1 shows that a 2.0 increase in pH resulted in a significant reduction in the adsorption capacity of the adsorbent.

The findings of this study show that chemical modification of the adsorbent surface improved the adsorption capacity of the adsorbent. Higher percent adsorption was achieved for all the three analyte heavy metal ions from the river water samples. The percentage adsorption of metal ions from water samples after modification of adsorbents increased from 12 to 14.82% for chromium, 68.35 to 90.26% for Lead and 46.3 to 53.1% for manganese at solution pH of 5. At lower pH value of 2, modified dates had maximum percentage adsorption of 56.7%. The adsorption capacity reduced with increase in pH. This can be explained by increase of OH^{-1} ions which competes with active sites in bonding with metal ions. Also, there may be formation of precipitates of insoluble hydroxides with increase in pH (Mangwandi *et al.,* 2020). These finding are corroborated by those of other studies (Rajczykowski *et al.,* 2018; Schwantes *et al.*, 2016).

According to (Ngah and Hanafiah, 2008), chemical modification improved the adsorption capacity of adsorbents due to higher number of active binding sites after modification, better ion-exchange properties and formation of new functional groups that favours metal ions uptake. Besides more efficient sorption capacity, modification of biomass surface via chemicals also improves structural durability (Gupta *et al.,* 2015).

Adsorption isotherms

Adsorption isotherms exhibits the relationship between the amount of adsorbate removed from a liquid phase by a unit mass of adsorbent (Waweru, 2015). The R^2 values for Freundlich and Langmuir isotherms fit on the adsorption for lead (II) ions were 0.9921 and 0.997 respectively, while those for the adsorption of chromium ions were 0.994 and 0.998 for Freundlich and Langmuir isotherms respectively as shown in table 2.

The maximum adsorption capacity was 6.329 mg/g and 5.208 mg/g for Lead ions and Chromium ions respectively. R^2 values, the Langmuir adsorption isotherm had higher values of R^2 hence it fit better to the experimental data obtained than the Freundlich adsorption isotherm, for both Lead (II) ions and Chromium (VI) ions. This indicates that the adsorption of both metal ions on the *P. purpereum* adsorbent surface is monolayer (Waweru, 2015). The iR^2 values for Freundlich and Langmuir isotherms fit on the adsorption for manganese ions were 0.996 and 0.993 respectively.

The maximum adsorption capacity was 15.61 mg/g. R^2 values. Likewise, the Langmuir adsorption isotherm had higher values of R^2 hence it fit better to the experimental data obtained than the Freundlich adsorption isotherm, for manganese ions. Similar studies by (Mwangangi, 2015) on use of modified maize cobs in removal of lead and cadmium from water, the results obtained fitted to Langmuir and Freundlich adsorption isotherms. He found that adsorption of Pb^{+2} fitted better to Langmuir with R^2 value of 0.953 while Freundlich had a R^2 value of 0.052.

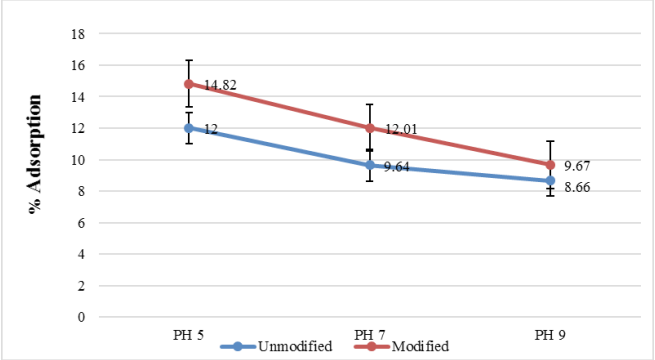

Figure 6: Effect of pH on the adsorption of chromium from spiked solutions by unmodified and modified adsorbents.

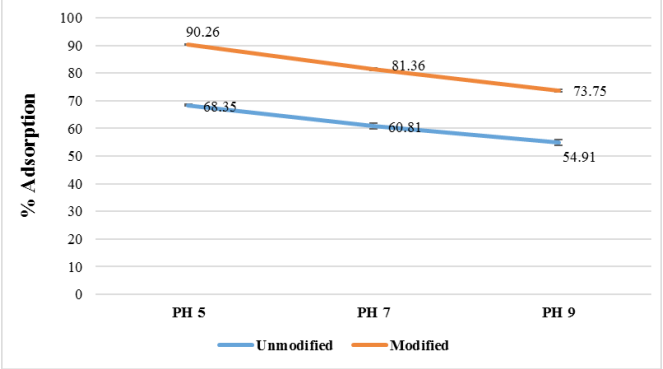

Figure 7: Effect of pH on the adsorption of lead ions from spiked solutions by unmodified and modified adsorbents.

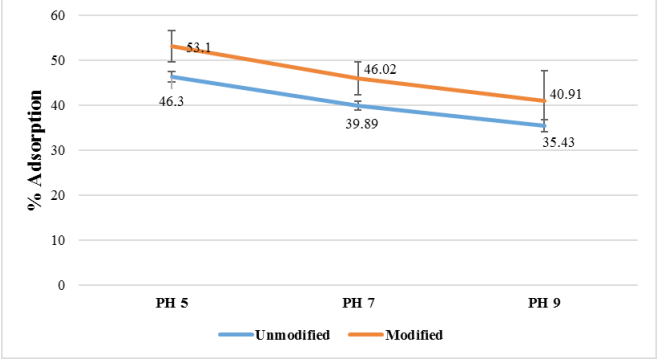

Figure 8: Effect of pH on the adsorption of manganese ions from spiked solutions by unmodified and modified adsorbents.

Table 2: Langmuir and Freundlich constants for removal of chromium, lead and manganese ions using modified *p. purpureum*.

Metal ion	Langmuir					Freundlich		
	Qmax	1/b	B	R^2	Kf	1/n	N	R^2
Chromium	5.208	-6.993	-0.143	0.998	23.988	-0.366	-2.732	0.994
Lead	6.329	-3.759	-0.266	0.997	18.072	-0.274	-3.650	0.9921
Manganese	15.61	1.338		0.988	2.59	0.902		0.953

Where Q_{Max} is adsorption capacity in mg/g
b- is Langmuir constant

CONCLUSIONS

The results obtained confirms the hypothesis that "the heavy metal ions can be removed effectively by adsorption using modified plant parts of *P. purpureum*. Chemical modification of the adsorbent surface improved the adsorption capacity of the adsorbent since higher percent adsorption was achieved by the modified adsorbent. The results also show that the pH of solution is a significant determinant of the adsorption capacity and efficiency of the adsorbent with lower pH values (more acidic environment) achieving greater adsorption.

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