Removal of Lead Ion from Industrial Effluent Using Plantain (*Musa paradisiaca*) Wastes

O. T. Okareh[1*] and A. T. Adeolu[1]

[1]*Department of Environmental Health Sciences, Faculty of Public Health, University of Ibadan, Ibadan, Oyo State, Nigeria.*

Authors' contributions

This work was carried out in collaboration between authors OTO and ATA. Author OTO designed the study, performed the statistical analysis, wrote the protocol, and wrote the draft of the manuscript and managed literature searches while author ATA managed the analyses of the study and literature searches. The two authors read and approved the final manuscript.

Original Research Article

ABSTRACT

Introduction: Industrial effluent often contains heavy metals which bio-accumulate in biological systems and persist in the environment, thereby constituting public health problems. Plantain (*Musa paradisiaca*) wastes, which are easily available, could be used to produce resource materials such as activated carbon that are of economic importance.
Aims: This study assessed the use of plantain wastes for the removal of lead in effluent from battery recycling plant.
Methodology: Plantain wastes were collected from a plantation, sun-dried and ground. These were then carbonized and activated using industrial oven at $400^{o}C$. An Acid-Lead battery recycling plant in Ogunpa, Ibadan, Nigeria was purposively selected. Samples of effluent from the point of discharge into Ogunpa River (100 m from the residential area) were subjected to physico-chemical (pH, conductivity, Total Suspended Solid (TSS), and Lead (Pb)) analyses, using American Public Health Association methods. Batch experiment was adopted in determining the adsorption isotherms of the adsorbents, using Association of Official Analytical Chemist method at varied effects of pH (2 to 12) and adsorbent doses (0.1 to 2.0 g) with treatments of Plantain Peel Activated Carbon (PAC), Plantain Leaf Activated Carbon (LAC), Plantain Bract Activated Carbon (BAC),

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Corresponding author: E-mail: dapsy2001@yahoo.co.uk;

Plantain Stalk Activated Carbon (SAC) while Commercial Activated Carbon (CAC) served as control. Initial and final concentrations of Pb were determined by Atomic Absorption Spectroscopy.
Results: Means of pH, conductivity, TSS, and Pb of the effluent sample were: 2.0±0.2, 2164.7±0.6 µs/cm, 2001.7±25.2 mg/l, 31.3±0.0 mg/l. The highest quantities (94.97%) of Pb were removed at pH^{10}. Optimum dosage of Pb occurred at 1.5 g.
Conclusion: Carbonized and activated plantain waste used as adsorbents had potentials for effectively removing lead from effluent generated from battery recycling plant. Treatment of effluent with plantain wastes should be encouraged in battery recycling plant for improved public health and safety status, and to enhance effective waste management.

1. INTRODUCTION

Over two-thirds of earth's surface is covered by water; less than a third is taken up by land. When earth's population was much smaller, no one believed pollution would ever present a serious problem. As earth's population continues to grow, people are putting ever-increasing pressure on the planet's water resources [1,2,3]. As industrialization has spread around the globe, so the problem of water pollution has spread with it. It was once popularly believed that the oceans were far too big to pollute. Today, with over 8 billion people on the planet, it has become apparent that there are limits and water pollution is one of the signs that humans have exceeded those limits. Water pollution has always been a major problem to the environment.

Water pollution is the process of contamination of streams, lakes, underground water, bays or oceans by substances harmful to living things. Water pollutants may occur naturally or through anthropogenic means. A variety of industries are responsible for the discharge of heavy metals into the environment through their waste water [4]. Some of these water pollutants, specifically heavy metals, are of serious health implications because of their persistence and bio-accumulation potential in the environment. For example, Lead is a metal ion toxic to the human biosystem, and is among the common global pollutants arising from increasing industrialization. The assimilation of relatively small amounts of lead over a long period of time in the human body can lead to the malfunctioning of the organs and chronic toxicity [5].

Based on the increasing rate at which heavy metals are being generated and discharged into water bodies through various industrial applications, as well as the known deleterious effects of these heavy metals, there is a need to proffer a cost-effective solution to heavy metals removal in polluted water. Over the years, a wide range of techniques for removing heavy metals from polluted water have been developed, some of which are precipitation, ion exchange, adsorption, electro-dialysis and filtration. Studies on the treatment of effluent bearing heavy metal have revealed adsorption to be a highly effective technique for the removal of heavy metal from waste stream and activated has been widely used as an adsorbent [6].

Despite its extensive use in the water and waste water treatment industries, activated carbon remains an expensive material. Therefore, the quest for cost effectiveness has formed the basis for the use of low cost agricultural wastes, which are produced in excessively large quantities, in heavy metals adsorption from effluent. Therefore, there is an urgent need to explore some agro-based inexpensive adsorbents and their feasibility for the removal of heavy metals. Plantain wastes, which are easily available and often constitutes nuisance to the environment could be used to produce resource materials such as activated carbon commonly used as filter in water treatment systems. Therefore, this study assessed the use of plantain wastes in the removal of lead ion in effluent from battery recycling plants.

2. MATERIALS AND METHODS

2.1 Plantain Wastes

Ripe plantain and fruit stalk were collected in market within Ibadan in Oyo State while plantain bract and leaves were collected from plantation in Osun State, Nigeria. The peel and fruit stalk were removed, washed with distilled water and sun dried for 168 hours and then oven dried at 45ºC to constant weight. The samples were ground, passed through a 0.14 mm mesh size and, stored in polythene container for analysis. Four types of adsorbents, Plantain Peel

Activated Carbon (PAC), Plantain Leaf Activated Carbon (LAC), Plantain Bract Activated Carbon (BAC), Plantain Stalk Activated Carbon (SAC) and Commercial Activated Carbon (CAC) which served as control, were used as the media (adsorbent) to remove lead ion in wastewater.

2.2 Effluent Collection

Effluent was collected from the point of discharge into Ogunpa River into a 5-litre plastic bottle from Acid-Lead battery recycling plant, Ogunpa, Ibadan North-West Local Government, Oyo State. Containers used for sample collection were pre-treated by washing with dilute hydrochloric acid and later rinsed with distilled water. The containers were later dried in an oven for one hour at 105ºC and allowed to cool to ambient temperature. At the collection point, containers were rinsed with samples 3 times and then filled with sample, corked tightly, and taken to the laboratory for treatment and analysis. The method of analysis was consistent with the standard methods [7].

The pH of the sample was taken at the site and other parameters were measured in the laboratory. Samples were stored at below 4ºC to avoid any change in physico-chemical characteristics. An atomic absorption spectrophotometer was used: model Phillip PU 9100 × with a hollow cathode lamp and a fuel rich flame (air acetylene). Each sample was aspirated and the mean signal response recorded at the metal ion's wavelength. This was used to compute the concentrations of metal ions absorbed by the adsorbents.

2.3 Carbonization and Activation of Plantain Wastes

The samples were carbonized and activated by the two steps method. A quantity (100 g) of each raw ground plantain waste samples was prepared by drying at 400ºC for 1hr under a closed system in a porcelain crucible and then cooled to room temperature. The charcoal was subjected to H_3PO_4 activation. The charcoal was agitated in H_3PO_4 after the agitation; the pre-carbonized charcoal slurry was left overnight at room temperature and, then, dried at 110ºC for 24 hours. The samples were activated in a closed system. Consequently, the samples were heated to optimize temperatures of 400ºC and maintained at a constant temperature for 1hour before cooling. After cooling down, the activated charcoal was washed successively several times with distilled water to remove the excess activating agent and impurity. The prepared activated carbons were then subjected to various analyses.

2.3.1 Commercial activated carbon

The commercial carbon, Calgon carbon (F-300), was obtained from Calgon Carbon Inc., Pittsburgh, PA, USA.

2.4 Effect of Operating Conditions

2.4.1 Effect of pH

Volume of 50 cm^3 of effluents was measured into each 250 ml conical flask at adjusted pH of 2, 4, 6, 8, 10 and 12. The desired pH was maintained using concentrated NaOH to adjust the pH. Activated carbon (1.0 g) was added into each flask and agitated intermittently for the desired time periods. The mixture was shaken thoroughly at 200 rpm with an electric shaker for 90 minutes to attain equilibrium. The suspension was filtered using Whatman No. 1 filter paper to remove suspended adsorbent. Initial and final concentrations of lead ions were analyzed for the lead ions using an Atomic Absorption Spectrophotometer, AAS (Computer-Aided Scalar Series, Model 969) while the amount adsorbed was calculated by difference.

2.4.2 Effect of contact time

The procedure was repeated for optimum contact time in which 50 ml of effluent was measured into each 250 cm^3 conical flask at optimum pH (obtained from experiment above). Activated carbon (1.0 g) was added into each flask and agitated intermittently for the desired time periods. The mixture was shaken thoroughly at 200 rpm with an electric shaker for 30, 60, 90, 120 and 150 minutes to attain equilibrium. The suspension was filtered using Whatman No. 1 filter paper to remove suspended adsorbent. Initial and final concentrations of lead ions were analyzed for the lead ions using an AAS, while the amount adsorbed was calculated by difference.

2.4.3 Effect of adsorbent doses

The procedure was repeated for optimum adsorbent dose in which 50 ml of effluent was measured into each 250 cm^3 conical flask at optimum pH. A known amount of activated carbon 0.1, 0.5, 1.0, 1.5 and 2.0 g was added

into each flask and agitated intermittently for the period of 90 minutes. The mixture was shaken thoroughly at 200 rpm with an electric shaker for optimum contact time to attain equilibrium. The suspension was filtered through Whatman No. 1 filter paper to remove any suspended adsorbent. Initial and final concentrations of lead ions were determined by AAS, while the amount adsorbed was calculated by difference.

2.4.4 Effect of initial metal ion concentration

The stock solution of 1000 mg/L of the standardized Pb^{2+} was prepared from the chloride using effluent sample. The solution was adjusted to pH 6.0±0.2 with 0.1 M HCl. Batch sorption experiments were performed in which 50 ml of effluent was measured into each 250 ml conical flask and 1.0 g of the adsorbent was added into each flask and agitated intermittently for the desired time periods. The mixture was shaken thoroughly at 200 rpm with an electric shaker for 90 minutes to attain equilibrium. The suspension was filtered through Whatman No. 1 filter paper to remove any suspended adsorbent. Initial and final concentrations of lead ions were determined by AAS, while the amount adsorbed was calculated by difference.

A summary index was used to determine the mean values of results obtained from various parameters. One-way ANOVA was used to test for significant differences in characteristics of the prepared plantain wastes. The results were investigated by using the least significant difference at a 95% confidence level using SPSS 16.

2.5 Adsorption Isotherms

A series of batch experiments was carried out to determine the adsorption isotherms of lead ion on the adsorbents [6].

The amount of metal ion adsorbed (Q_e) during the series of batch investigations was determined using a mass balance equation:

$$Q_e = \frac{(C_v - C_f) x V}{M}$$

Where Q_e is the metal uptake (mg/g); C_v and C_f are the initial and final metal equilibrium concentration in the effluent sample (mg/l) respectively, M is the mass of the adsorbent (g) and V is the volume of the effluent sample (l).

The definition of removal efficiency is as follows:

Removal efficiency (%) = $\frac{C_v - C_f}{C_v} x 100$ [8]

Where C_v and C_f are the initial and final metal equilibrium concentration in the effluent sample (mg/l) respectively.

3. RESULTS AND DISCUSSION

3.1 Physico-chemical Characteristic of the Effluent

Table 1 shows the physico-chemical analysis of the effluent sample, showing the level of lead contamination against the limits.

Table 1. Physico-chemical characteristic of the effluent

Parameters	Test value	NESREA standards
pH	2.0±0.15	6-9
Temperature (ºC)	30.0±1.5	<40
TSS (mg/L)	2001.7±25.2	28
Conductivity (μScm^{-3})	2164.7±0.6	1000
Lead (Pb) (mg/L)	31.3±0.0	0.01

All data were mean ± standard deviation of triplicate determinations

High or low pH values in a river have been reported to affect aquatic life and alter toxicity of other pollutants in one form or the other [9]. The mean pH value of the effluent indicated that the effluent was highly acidic than pH 6-9 of NESREA recommended limits for battery factory effluents (Table 1). Low pH value impaired recreational uses of water and affect aquatic life. Since pH is a function of hydrogen ion concentration (i.e pH = -log [H^+]), the high acidity was probably due to the high concentration of hydrogen ion [H^+] in the effluent, sourced mainly by sulphuric acid-one of the major raw materials in lead acid battery manufacture.

Temperature is basically important for its effect on other properties of wastewater. The mean temperature value of the effluent is within the range of NESREA limit of 30ºC. The averaged Total Suspended Solids (TSS) value of the effluent is very high. Conductivity of water is a measure of the total concentration of ionic species or salt content. The mean conductivity value of the effluent is very high. The high conductivity value (i.e high salt concentration) in the effluents can increase the salinity of the receiving river, which may result in adverse

ecological effects on the aquatic biota and such high salt concentrations hold potential health hazard [10].

Lead is a suspected pollutant in a battery recycling effluent because it is a major raw material in the manufacture of lead acid accumulated batteries. Lead at very low concentration is toxic and hazardous to most forms of life. The chronic effect of Pb on man includes neurological disorders, especially in the foetus and in children. This can lead to behavioural changes and impaired performance in IQ tests [11,12]. The Pb level was exceeded in the effluent being studied; the direct use of water from this receiving river (Ogunpa) for domestic purposes without treatment could be detrimental to young children in the vicinity of the catchment (Table 1). The receiving river also would not be suitable for maintenance of the aquatic ecosystem.

3.2 Characteristics of the Adsorbents

The bulk density is important physical parameter because it determines the mass of carbon that can be contained in a filter of given solids capacity and the amount of treated liquid that can be retained by the filter cake [13]. Activated carbon with high bulk density has ability to filter more liquor volume before available cake space is filled. It equally has a low amount of ash (Table 2). Ash content reduced the overall activity of activated carbon and the efficiency of reactivation. The lower the ash value therefore the better the activated carbon for use as adsorbent.

3.3 Surface Chemistry of the Adsorbents

Iodine number is a fundamental parameter used to characterize activated carbon performance by measuring surface area. It is a measure of the micro-pores content of the activated carbon and is obtained by the adsorption of iodine from solution by the activated carbon sample. The micro-pores are responsible for the large surface area of activated carbon particles and are created during the activation process. The surface areas of the adsorbents are higher when compared to commercial activated carbon (Table 2). The surface titration method stipulates that only strongly acidic carboxylic groups are neutralized by sodium bicarbonates (Na_2HCO_3), whereas those neutralized by sodium carbonate (Na_2CO_3) are thought to be lactonic and carboxylic group. The weakly acidic phenolic groups only react with strong alkali, sodium hydroxide (NaOH). Neutralization with hydrochloric acid (HCl) characterizes the amount of surface basic group's pyrones and chromenes that are present in the activated carbon [14,15]. The values of surface chemistry of the adsorbents are almost the same when compared to commercial activated carbon (Table 2).

3.4 Operating Conditions

These are conditions necessary for the removal of heavy metals from the effluent. They include pH, contact time, adsorbent dose, and initial ion metals concentration. The effect of these conditions on the removal of heavy metals was studied and recorded.

Table 2. Characteristic of the prepared activated carbons

Parameter	BAC	LAC	PAC	SAC	CAC
Ash (%)	5.0±0.01[b*]	5.0±0.01[a*]	5.2±0.01[c*]	6.2±0.01[e*]	5.4±0.01[d]
Bulk density (kg/m^3)	0.8±0.00[c*]	0.7±0.001[a]	0.8±0.01[d*]	0.8±0.01[b*]	0.6±0.01[a]
Surface Area (m^2/g)	486.1±0.01[b*]	524.7±0.01[c*]	625.6±0.01[e*]	530.7±0.01[d*]	200.4±0.01[a]
Carboxylic	0.1±0.01[a*]	0.1±0.01[b]	0.1±0.01[a*]	0.1±0.01[b]	0.1±0.01[b]
Phenolic	0.2±0.01[b*]	0.3±0.01[c]	0.1±0.0[a*]	0.3±0.01[c]	0.3±0.01[c]
Lactones	0.6±0.01[c]	0.5±0.01[a*]	0.6±0.01[b]	0.6±0.01[d*]	0.6±0.01[b]
Acidic	0.9±0.01[b*]	0.9±0.01[b]	0.8±0.01[a*]	10.0±0.01[c]	10.0±0.01[c]
Basic	0.5±0.01[a*]	0.6±0.01[c*]	0.6±0.01[d*]	0.6±0.01[c*]	0.5±0.01[b]

All data were mean ± standard deviation of triplicate determinations
[a,b,c,d,e] *are means from least to the highest. Means of the same row with the same alphabet(s) are not significantly different, Means within the same row with the different alphabet(s) are significantly different at P<0.05*
* = The mean difference is significant at the 0.05 level

3.4.1 Effect of pH

The pH of the aqueous solution is an important controlling parameter in the adsorption process and thus the role of H^+ concentration was examined from samples at different pH covering a range of 2-12. Fig. 1a shows that percentage removal of Pb (II) increased at a steady rate as pH increased up to 10, attaining a maximum value of around 95.58%. Also, the adsorption capacity (Q_e) of Pb (II) increased at a steady rate as pH increased up to 10, attaining a maximum value of 1.98 (Fig. 1b).

It should be noted that after pH 10, there was a decrease in the adsorption, which may be due to the formation of soluble hydroxyl complexes. At low pH values, the surface of the adsorbent would be closely associated with hydroxonium ions (H_3O^-) by repulsive forces, to the surface functional groups, consequently decreasing the percentage removal of metal [16]. As the solution pH increases, the onset of the metal hydrolysis, the precipitation began and the onset of adsorption therefore occurs before the beginning of hydrolysis [8]. When the pH of the adsorbing medium was increased from 2 to 10, there was a corresponding increase in deprotonation of the adsorbent surface, leading to a decrease in H^+ ion on the adsorbent surface. This creates more negative charges on the adsorbent surface, which favours adsorption of positively charge species and the positive sites on the adsorbent surface [5].

The hydrolysis of cations by the replacement of metal ligands in the inner coordination sphere with the hydroxyl groups; this replacement occurs after the removal of the outer hydration sphere of metal cations. In addition, increasing pH decreases the concentration of H^+, therefore reducing the competition between metal ions and protons for adsorption sites on the particle surface. Another factor that contributes to enhancing metal ion adsorption is the increasing pH which encourages metal ion precipitation from the solution in the form of hydroxides. The adsorption of the metal ions into plantain waste adsorbent was largely influenced by pH.

3.4.2 Effect of contact time

The result showed that for all adsorbents the removal rate was rapid within the first 30 minutes, sharply increased for 60 minutes and gradually increased between 90 and 150 minutes for Pb (II). The Fig. 2a shows that percentage removal of Pb (II) increased at a steady rate as contact time increased up to about 150 minutes, attaining a maximum value of around 95.78%. The adsorption capacity (q_e) of Pb (II) increased at a steady rate as contact time increased up to 150 minutes, attaining a maximum value of 1.50 (Fig. 2b).

The initial faster rate was due to the availability of the uncovered surface area of the adsorbents, since the adsorption kinetics depends on the surface area of the adsorbents. The lead adsorption takes place at the more reactive sites. As these sites are progressively filled the more difficult the sorption becomes, as the sorption process tends to be more unfavourable [7].

3.4.3 Effect of adsorbent dose

The values were generated by varying the adsorbent doses (0.1 to 2.0 g) at room temperature with different adsorbents. The result suggested that after a certain dose of adsorbent the maximum adsorption sets in and hence the amount of ions bound to the adsorbent and the amount of free ions remain constant even with further addition of the dose of adsorbent.

The Fig. 3a above showed that percentage removal of Pb (II) increased at a steady rate as adsorbent dose increased up to about 1.5 g for Pb (II), attaining a maximum value of 96.80%. The adsorption capacity (q_e) of Pb (II) decreased at a steady rate as adsorbent dose increased up to 1.5 g, attaining a minimum value of 0.60 (Fig. 3b). The increased percentage adsorption by adsorbent was as a result of increased surface area and increased adsorption site occasioned by increased adsorbent dose [17]. The observed decrease in adsorption capacity may be due to decrease in liquid–solid ratio. The change in the solid-liquid ratio may have directly resulted in this trend since amount adsorbed, qe, has an inverse proportionality function to weight of biosorbent, but a direct proportionality function to percentage adsorbed [18].

3.4.4 Effect of initial metal ion concentration

The result showed that percentage removal of Pb (II) increased at a steady rate as initial concentration increased up to 500 mg/L, attaining a maximum value of 99.21%. The adsorption capacity (q_e) of Pb (II) increased at a steady rate as initial concentration increased up to 500 mg/L, attaining a maximum value of 24.80. Details are shown Fig. 4a and 4b.

With increasing metal ion concentration, there is increase in the amount of metal ion adsorbed due to increasing driving force of the metal ions towards the active sites on the defatted plantain waste adsorbents. This indicates a decrease in the active sites on the sorbents as more metal ions are adsorbed [19].
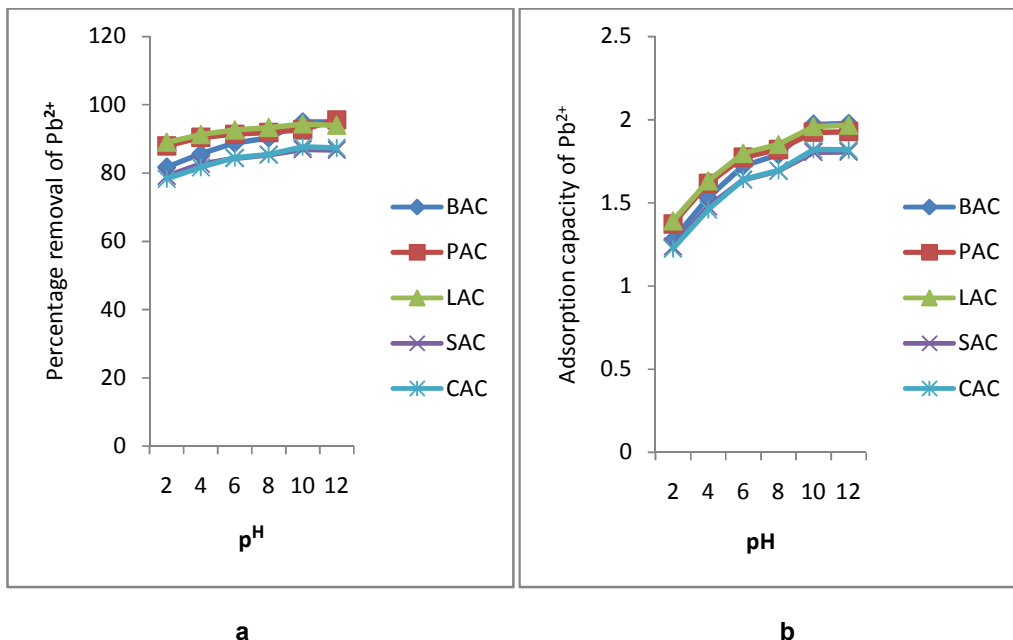

a

b

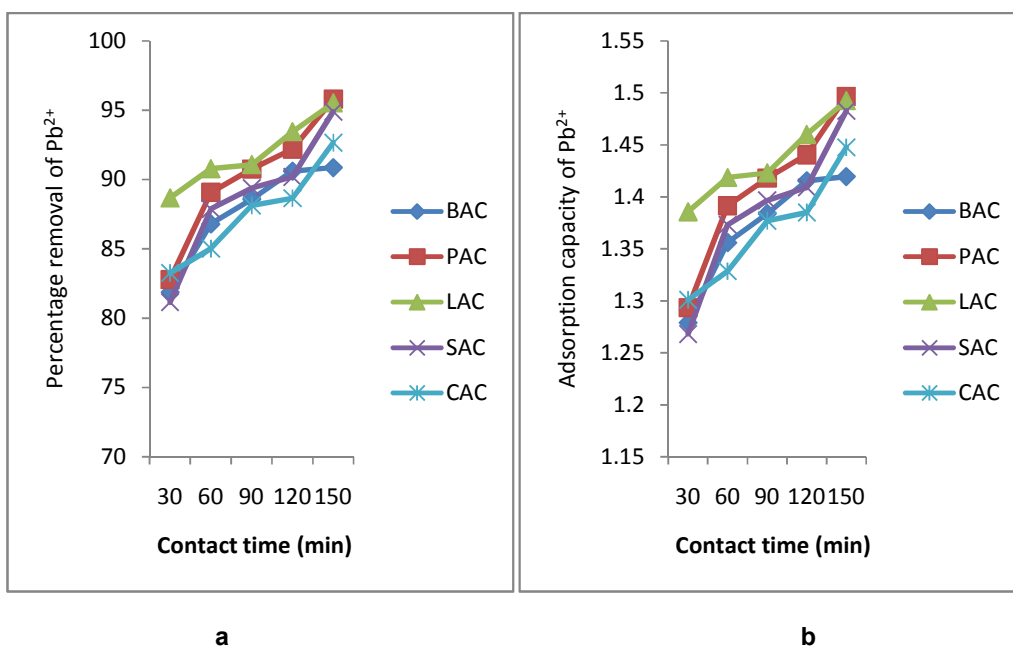**Fig. 1a & b. Effect of pH on the percentage removal and adsorption capacity**

a

b

Fig. 2a & b. Effect of contact time on the percentage removal and adsorption capacity
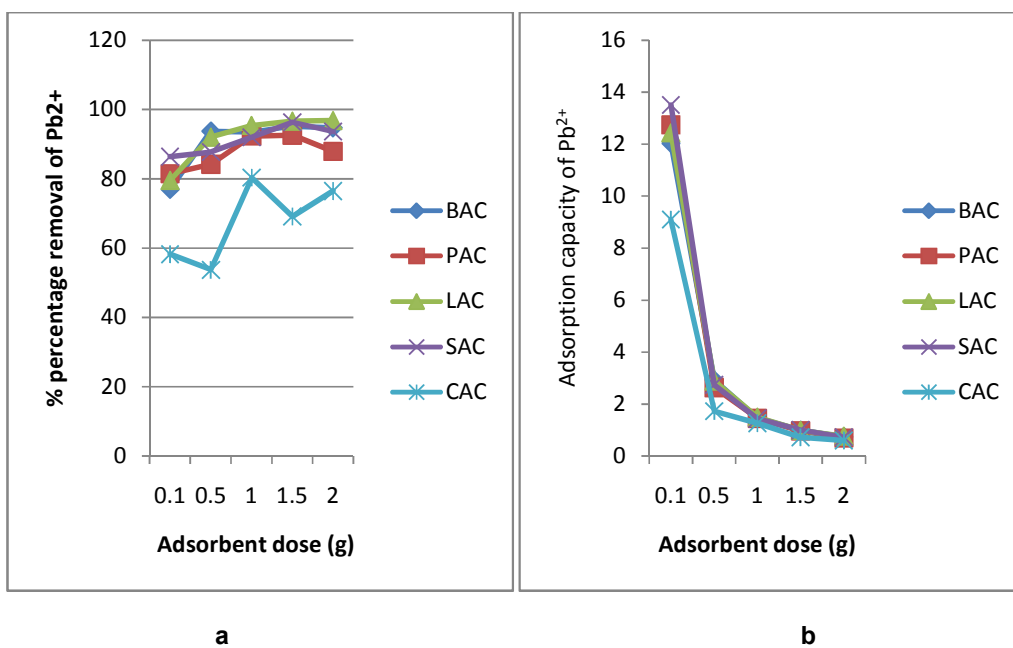
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Fig. 3a & b. Effect of adsorbent dose on the percentage removal and adsorption capacity
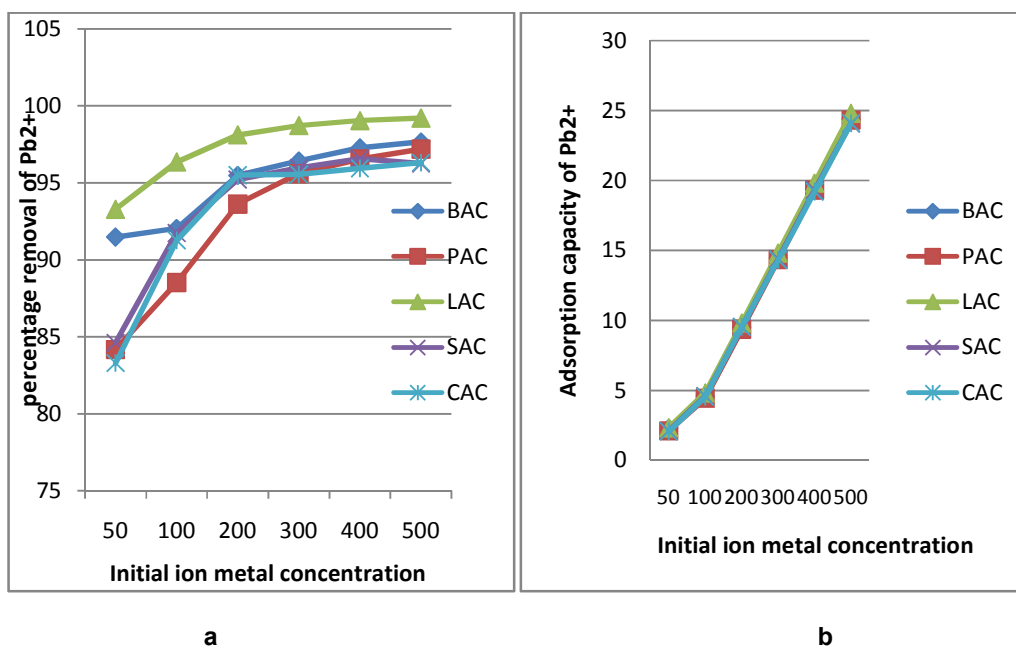

Fig. 4a & 4b. Effect of initial ion concentration on the percentage removal and adsorption capacity

4. CONCLUSION

The study confirmed that the discharge of untreated effluent from battery recycling plant, containing heavy metals and hazardous materials such as lead, over a long period of time into the receiving water body is a potential source of contamination. The study revealed that the background level of lead in the effluent was 31.25 mg/l, which was above the recommended limits of NESREA. Plantain wastes have been successfully used to produce high quality

activated carbon because of its inherent high densities and carbon content. Experiments conducted on the adsorption of Pb^{2+} by BAC, PAC, LAC and SAC showed higher quantity of lead adsorbed when compared with the CAC. The result showed that plantain waste activated carbon can compete favourably with CAC.

From the analysis of the result, it is evident that the plantain wastes, processed into activated carbon have great potential for the uptake of lead from the industrial effluent. It has also been observed that the sorption capacity of activated carbon made from plantain wastes depends on conditions such as pH, initial metal ion concentration, adsorbent dose and contact time. Plantain wastes, which constitute nuisance to the environment, could be used to produce resource materials (activated carbon), which can be used to remove heavy metals from the industrial effluent that are of public health concern. Converting these wastes into activated carbon will greatly help to reduce its menace in the environment and enhance effective waste management.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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