Effective adsorption of lead and copper from aqueous solution by samaneasaman and banana stem

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Abstract. The sorption of metal ions with low-cost adsorbents plays an important role in sustainable development. In the present study, the efficacy of sugarcane bagasse, rain tree fruits (samaneasaman), banana stem and their mixtures, used as bio-sorbents, in the removal of Cu(II) and Pb(II) ions from aqueous solution is evaluated. Batch studies are conducted, and residual ions were measured using Inductively Coupled Plasma (ICP)-atomic spectrometer. Effect of pH, initial metal ion concentration, reaction time and adsorbent dosage are studied. The Pb(II) removal efficiency was observed to be 97.88%, 98.60% and 91.74% for rain tree fruits, banana stem and a mixture of adsorbents respectively. The highest Cu(II) ion removal was observed for sugarcane bagasse sorbent with an efficiency of 82.10% with a pH of 4.5 and a reaction time of 90 min. Finally, desorption studies were carried out to study the leaching potential of adsorbent, and it was found that the adsorbent is stable in water than the other leaching agents such as HCl, ammonium acetate, Sodium EDTA. Hence, these adsorbents can be effectively used for the removal of these heavy metals.

Keywords: low-cost adsorbents; heavy metal; lead; copper; banana stem; raintree fruit

1. Introduction

A more significant part of the world is facing freshwater shortage and expected to increase rapidly in coming decades as the population increases. Every year over 12 million people die due to water shortages, polluted water, and unsanitary living conditions (WHO 1997). In many developing countries ponds, lake and rivers are used for dumping untreated effluents and municipal sewage wastes, industrial poisons, and harmful chemicals. The industrial effluents, domestic waste, and agricultural runoff are the primary sources of heavy metal pollution in the environment. The pollutants which contain heavy metals (Cd, Cr, Cu, Hg, Pb, and Zn) can affect every human being, aquatic life, and the environment through food and water chain. Among all

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heavy metal contaminates, Pb and Cu are most common contaminants and the researchers are looking for removal of these heavy metals since they are very toxic to human health (Pehlivan *et al.* 2009 and Akar *et al.* 2009).

In the past decades, many researchers used chemical process for removing heavy metals from the pollutant water which was tedious and time-consuming (Pehlivan *et al.* 2009, Subhashini and Swamy 2013, Wuana *et al.* 2015, Yakout *et al.* 2016, Riahi *et al.* 2017, Wu *et al.* 2018,). Bouabidi *et al.* (2018) successfully utilized steel-making dust as a potential adsorbent for the removal of Pb(II) from an aqueous solution. Saleh *et al.* (2014) and Saleh and Al-Saadi (2015) used the activated waste rubber tire to produce carbonaceous material as an absorbent to remove methyl orange. Wuana *et al.*, 2015 prepared the activated carbon from Moringa oleifera for the removal of residual pharmaceutical. Nano-based adsorbents proved to be better than the normal adsorbent as these particles have a more surface area (Galhoum *et al.* 2016). Saleh (2015b) used the nanocomposites of silica incorporated with carbon nanotubes (silica/CNT), activated carbon (silica/AC) and multiwall carbon nanotubes (SiO_2-CNT) for efficient removal of Hg(II) ions from aqueous solutions. Ali *et al.* (2017) and Saleh *et al.* (2016) used the normal resin, activated resin, chelted resin and Hydrophobic cross-linked polyzwitterionic acid (HCPZA) resin as sorbent for the effective and complete removal of Cr(III), Co(II), organic contaminants, Hg(II), dye, chromium and Eriochrome black T (EBT) in various aqueous solutions and mixtures. Wuana *et al.* (2016) studied the phytoextraction for the removal of heavy metal such as Cd, Pb, Ni and Zn in the municipal waste dump. Hassan *et al.* (2017) could remove Hg(II) from wastewater effluent using Zinc oxide doped with Sulphur. AL-Hammadi *et al.* (2018) evaluated the removal of methylene blue dye using poly (trimesoyl chloride-melamine)-grafted palygorskite (PTMP) as an adsorbent in batch and column systems.

Various other researchers tried to find an alternative to the chemical process by utilizing the agricultural wastes which are easily available and cost-effective. Various materials like Turkish montmorillonite clay, coconut husk, coconut shell, pistachio shell, tea waste, sugarcane cellulose rubber wood fibre have been successfully used as adsorbents to remove heavy metals and acids from wastewater (Akar *et al.* 2009, Mahdavi *et al.* 2010, Dave *et al.* 2012, Parlayici-Karatas and Pehlivan 2012, Nandal *et al.* 2014, Zhong *et al.* 2014, Wang *et al.* 2017). Saleh *et al.* (2017) used polyethyleneimine modified activated carbon as a novel magnetic adsorbent for the removal of uranium from aqueous solution. Khan and Rao (2017) developed a high activity adsorbent of chemically modified Cucurbitamoschata for the removal of Cu(II) and Ni(II) from aqueous solution. Chemically modified porous cellulose adsorbent has the potential to chemisorb toxic metals like Cd(II), Cu(II) and Pb(II) from aqueous media (Barsbay *et al.* 2018). Thue *et al.* (2018) synthesized and characterized a novel organic-inorganic hybrid clay adsorbent for the removal of acid red 1 and acid green 25 from aqueous solutions. Green adsorbents were successfully prepared and applied for the removal of Cd, Cu, Fe, Pb and Zn from well water (Rashed *et al.* 2018). de Oliveira Ferreira *et al.* (2019) developed a modified activated carbon from coconut shell as a promising adsorbent for quinoline removal. Manganese dioxide-coated multiwall carbon nanotube (MnO_2/CNT) nanocomposite was synthesized and used for the removal lead in water with column studies by varying different flow rates (Saleh and Gupta 2012).

From the literature review, it is found that agricultural wastes can be effectively used as adsorbents to remove heavy metals from industrial wastewater. Results showed that the agricultural wastes as adsorbents are promising, cost-effective, feasible, easy to prepare and environmental friendly. However, some of the agricultural waste requires pre-treatment before using them as adsorbents (Wuana *et al.* 2015, Khan and Rao 2017, Saleh *et al.* 2017).

From the past study, it was found that the use of rain tree fruit and banana stem for removal of Cu(II) and Pb(II) ions from aqueous solutions has not been investigated. The present study aims to assess the capability of sugarcane bagasse, rain tree fruit and banana stem for the adsorptive removal of Cu(II) and Pb(II) ions from aqueous solutions and the effect of adsorption parameters were investigated. The adsorption parameters studied include pH, biosorbent dosage amount, contact time and heavy metal concentration.

2. Materials and methodology

2.1 Numerical simulation procedure

2.1.1 Raintree fruits (RT) – [SamaneaSaman, Fabaceae (legume family)]

The tree came to India from South America via Sri Lanka. It is a tropical tree and grows best in a moist area. It cannot withstand the cold climate. The mature fruit is blackish brown in colour, lumpy, oblong, 15 to 19 mm wide, 100 to 200 mm long and 6 mm thick. The pods can be straight or curved slightly. The fruit does not split but cracks irregularly. The pod is filled with sticky pulp which is sweet and edible. A typical tree and its fruit are shown in Fig. 1. The chemical composition is tabulated in Table 1.

2.1.2 Sugarcane bagasse (SB)

Sugarcane is tall genuine perennial grasses of the genus Saccharum. They are native to tropical regions of South Asia and Melanesia and grow well in warm temperature. They are mostly used for sugar production. It has stout, jointed and fibrous stalks which have high sucrose content. Fig. 2 shows a sugarcane tree and its bagasse. Bagasse is the dry fibrous pulp that remains after the juice is extracted from sugarcane. It is also used as biofuel and as building materials. The chemical composition is given in Table 1.

2.1.3 Banana pseudostem (BS)

The pseudostem is the piece of the plant that resembles a trunk. This 'false stem' is formed by the firmly pressed covering leaf sheaths. The pseudostem keeps on growing until the right stem emerges at the top. The leaves come from the pseudostem. The pseudostem is very strong even though it is fleshy and comprises of water. The chemical composition is tabulated in Table 1. A typical Banana pseudostem and its powder can be seen in Fig. 3.

Fig. 1 Raintree and its fruits

Fig. 2 Sugarcane and its bagasse

Fig. 3 Banana pseudostem and its powder

Table 1 Chemical composition of sorbents

Type	Percentage (%)		
	Rain tree fruit	Sugar cane bagasse	Banana pseudo stem
Dry Matter	95.98%	-	-
Total Ash	3.21%	-	-
Crude Protein	19.32%	-	-
Either Extract	3.4%	-	-
Crude Fibre	28.4%	-	-
Nitrogen Free Extract	54.33%	-	-
Neutral Detergent Fibre	39.72%	-	-
Acid Detergent Fibre	30.72%	-	-
Hemicellulose	9.72%	25%	-
Cellulose	17.48%	42%	-
Acid Detergent Lignin	8.89%	-	-
Calcium	0.24%	-	-
Phosphorus	0.13%	-	-
Lignin	-	20%	-
Glucose	-	-	71.76%
Xylose	-	-	11.20%
Arabinose	-	-	7.34%
Galactose	-	-	2.02%
Monnose	-	-	0.58%
Galacturonic acid	-	-	7.09%

Table 2 Variation of parameter values

Parameter	Values
Weight of sorbents (gm)	0.1, 0.3, 0.6
pH	4, 4.5, 5
Metal ion concentration (mg/L)	10, 20, 50
Contact time (minutes)	60, 90, 120

2.2 Preparation of sorbents

The selected sorbents are widely available in India. The collected samples (1 kg) were washed with tap water then brushed to remove any attached contaminants and then washed again with deionized water. The sample was dried at 100°C for 24 hours. The dried sample was then crushed and grinded using a mixer grinder machine. It was then sieved to 250 μm nylon sieve. To avoid contamination, the sorbents were stored in acid washed airtight polyethylene containers at room temperature. The sieved powder was directly applied without any physical and chemical treatment. However, in a few studies, the raw materials were pyrolysed and the adsorbent were prepared (Saleh *et al.* 2017, Saleh and Gupta 2012). Here, the stability of the natural adsorbent is studied, and the performance of the adsorption process was evaluated.

2.3 Preparation of stock solutions from metal salts

Lead Nitrate ($Pb(NO_3)_2$) and Copper Sulphate ($CuSO_4$) are used as the source to prepare lead and copper stock solution respectively. 1000 ppm stock lead and the copper solution was prepared by adding 1.615 gm of $Pb(NO_3)_2$ and 3.93 gm of $CuSO_4$ in 1 litre of distilled water respectively. From these, 10, 25 and 50 ppm stock solutions were prepared for testing. For example, 10 ppm lead stock solution was obtained by diluting 10 mL of 1000 ppm lead stock solution in 1000 mL volumetric flask with distilled water.

2.4 Variation of different parameters

The adsorption of $Cu2+$ metal ion was tested for different varying parameters. The tests were conducted by varying the weight of sorbents, pH, metal ion concentration and contact time. The variation in the parameters was selected from the literature (Al-Saadi *et al.* 2013, Gundogan *et al.* 2004, Ho and Mckay 1999, Saleh 2015a). Table 2 shows the values of different parameters considered in the study.

3. Calculating efficiency of metal removal

The following equation determined amount of material removed by sorbents through series of experiments

$$Removal_{\%} = \left[\frac{C_o - C_f}{C_o} \right] X100$$ (1)
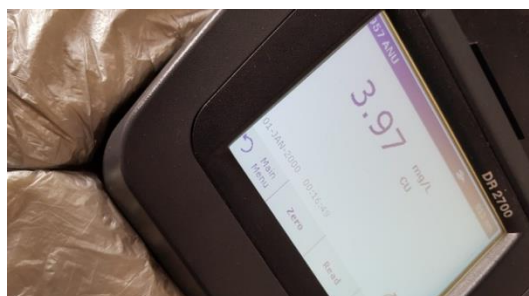
Fig. 4 Spectrophotometer

Fig. 5 ICP- atomic spectrometer

where C_o - is initial concentration and C_f - is the final concentration in ppm of metal ions after the filtration process.

For determining the Copper and lead concentration ICP-Atomic spectrophotometer was utilized. In a spectrophotometer, first, a calibration curve was made by testing the stock samples. Different concentrations of stock solution were used like 0, 10, 20, 50 and 100 ppm. Also, a blank sample was prepared with distilled water plus some sorbent (just to induce colour). Sorbents were filtered out before testing each sample. Now the calibration curve will provide a graph of absorbance v/s concentration. Also, with the help of the graph, the spectrometer will find the final concentration.

An ICP- Atomic spectrophotometer emits light from different sources and creates an emission spectrum which will later be absorbed by the detector and is then amplified to show an absorbance spectrum. The intensity of the spectrum will decide the concentration of metal ions in a coloured solution. An image of a spectrophotometer and ICP-Atomic spectrophotometer are shown in Fig. 4 and 5.

4. Results and discussions

4.1 Variation of pH

The surface charge of adsorbent as well as a chemical breakdown of adsorbate is significantly affected by pH and is considered as one of the critical parameter affecting the adsorption of metals from aqueous solution (Gundogan *et al.* 2004, Ho and Mckay 1999). The influence of pH was
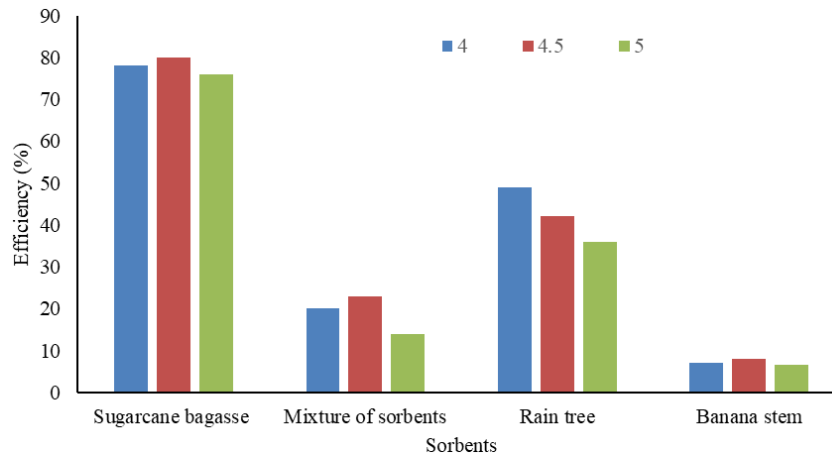
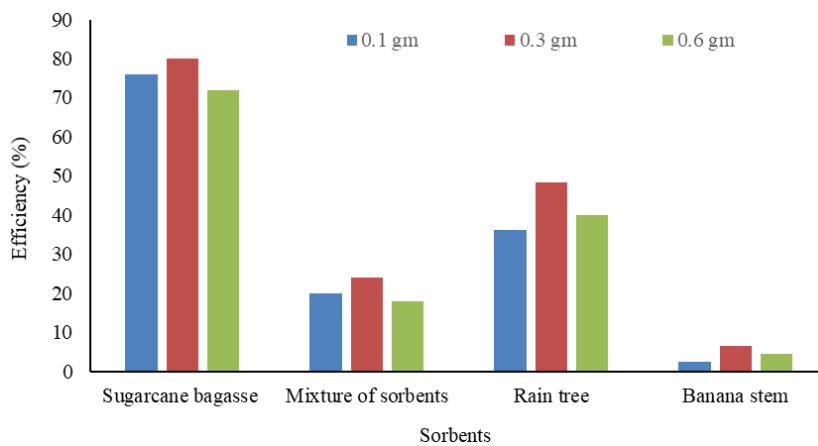Fig. 6 Effect of pH on the adsorption of Cu(II) ions

Fig. 7 Effect of the weight of sorbents on the adsorption of Cu(II) ions

studied over a range of values from 4 to 5 keeping initial concentration as 20 ppm, agitation time as 90 minutes and weight of sorbent 0.3 gm. The adsorption was low for all the adsorbents at a lower pH value of 4. As pH increases the adsorption of Cu2+ also increases. The increase may be attributed to the release of more and more H+ ions from the active sites and adsorption of Cu2+ ions. At pH values of 7 and above precipitation of hydroxide becomes predominant which must be avoided. For maximum adsorption, a pH value ranging between 4.5 and 5 may be utilized. A similar trend can also be observed from the studies by Al-Saadi *et al.* (2013). The optimum pH for Cu2+ ion adsorption was found out to be 4.5. SB showed the highest efficiency of 78% removal compared to the other adsorbents.

4.2 Variation of the weight of sorbents

The effect of variation of the weight of sorbents on adsorption was tested. The weight of sorbents was varied from 0.1 to 0.6 gm keeping ion concentration as 20 ppm, agitation time of 90 min and pH of 4.5. The efficiency of various sorbents in removing Cu2+ ions is shown in Fig. 7.
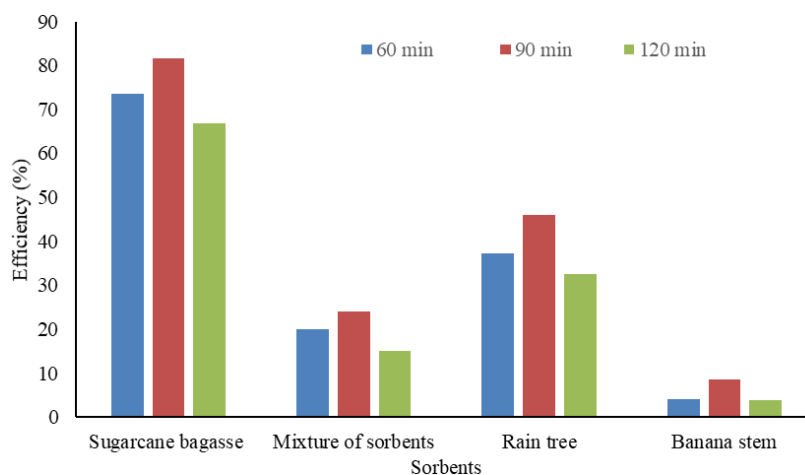
Fig. 8 Effect of contact time on the adsorption of Cu(II) ions

To the 50 mL standard solution, different weights of sorbents were added and mixed using a rotary shaker for 90 min at about 200 rpm. The mixture was then filtered using Whatman No. 1 filter paper. The experiments were conducted at room temperature. The filtrate and the solution were then analyzed in the spectrophotometer. Maximum removal of Cu^{2+} ions was observed for 0.3 gm for all the sorbents considered. SB showed maximum efficiency compared to other sorbents for all the weights of sorbents considered. Maximum efficiency of 82.1% was obtained for 0.3 gm SB sorbent.

4.3 Variation of contact time

The effect of contact time on adsorption of Cu^{2+} ions was investigated for an initial concentration of 20 ppm, pH of 4.5 and weight of sorbent 0.3 gm. The sorbent mixture was shaken for 60, 90 and 120 minutes. Initially, the removal of metal ions is high due to the ample availability of active sites. Increase in the contact time beyond 120 minutes did not show a considerable increase in the adsorption percentage since the systems had reached equilibrium. A similar trend was also observed by Saleh (2015). The percentage of removal of Cu^{2+} ions with various sorbents is shown in Fig. 8. The percentage of adsorption reduced from 81.63% to 66.89% as the contact time increased from 90 minutes to 120 minutes. At about 90 minutes after mixing, the maximum removal of Cu^{2+} was observed. Further, as the exterior sites exhausted, the uptake rate was controlled by the transport rate of adsorbate from the exterior to interior sites.

4.4 Variation of the concentration of metal ions

The effect of variation of concentration of metal ions on the adsorption process was tested keeping other parameters constant (Agitation time = 90 minutes, weight of sorbent = 0.3 gm, pH = 4.5). The efficiency of removal is shown in Fig. 9.

The standard solution of 10, 25 and 50 ppm concentration was prepared and tested as mentioned previously. The filtrate and the solution were further analyzed in a spectrophotometer. At low concentrations, the ratio of available surface to initial Cu^{2+} concentration was more
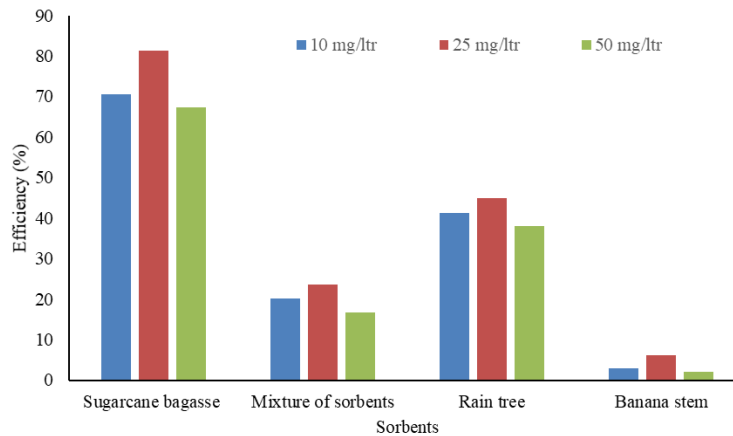
Fig. 9 Effect of the concentration of metal ions on the adsorption of Cu(II) ions

Table 3 Optimum results for copper ion

Parameters	Optimum results
Agitation time	90 minutes
pH	4.5
Weight of adsorbent	0.3 gm
Metal ion concentration	25ppm

Table 4 Efficiency of removal of hard metal by different sorbents

Parameters	Pb2+ ions (Removal %)	Cu2+ ions (Removal %)
Sugarcane Bagasse	73.00% (Wang *et al.* 2017)	82.10%
Rain Tree	97.88%	47.92%
Mixture	91.74%	22.80%
Banana Stem	98.60%	7.68%

significant than the ratio at high initial concentrations. The removal of metal ions is dependent on the initial concentration. It was further observed that the percentage of removal decreased with the increase in the initial concentration above 25 ppm. Maximum efficiency of 82.1% was observed for SB sorbent for 25 ppm.

4.5 Optimum conditions for the copper solution

The optimum values of parameters for Cu2+ ion removal are given in Table 3. The same values were further adopted for testing the removal of Pb2+ ions, and the efficiency is given in Table 4. It is clear from the table that SB is good at removal of both the metal ions with an efficiency of 73% and 82.1%. However, the highest removal of Pb2+ ions was observed with BS sorbents with an efficiency of 98.6%.

4.6 Desorption studies

Desorption of heavy metal plays a vital role in the stability of the adsorbent, and it also helps in
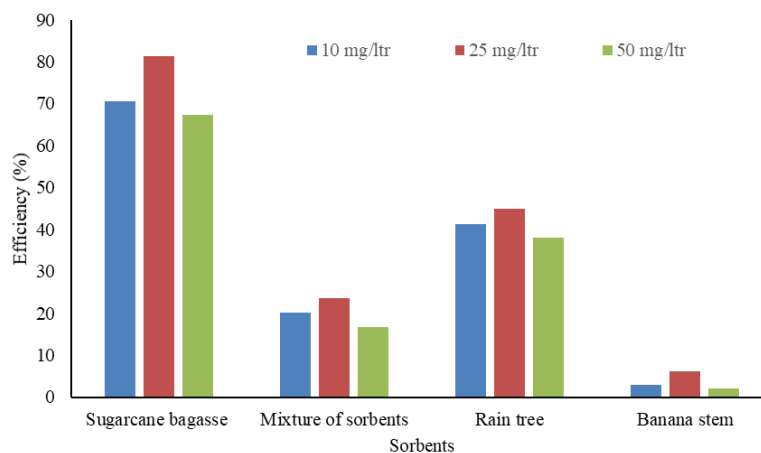
Fig. 9 Effect of the concentration of metal ions on the adsorption of Cu(II) ions
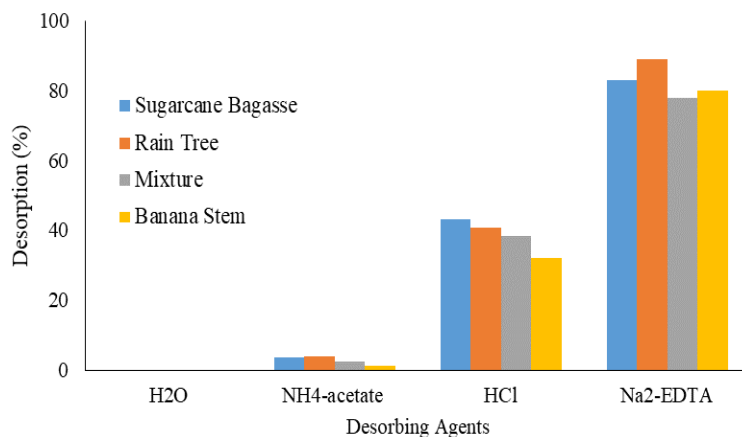

Fig. 10 Desorption studies on Pb(II) ions

the adsorbent regeneration studies. Here, four types of desorbing agents are preferred (sodium EDTA (0.2M), water, HCl (0.1N), ammonium acetate (0.2M)) based on the different reaction mechanism involved among the different adsorbent and adsorbents like acid-base reactions, complex formation, ions movement, etc. and more importantly in water. The results are shown in Fig. 10. It was observed that Sodium EDTA produced very less leaching of lead ions into the water system. However, significant leaching was observed for the other reagents. It means that the adsorption process was stable in water system than the other system. This may be due to the existence of a strong bond between the pollutant and adsorbent.

5. Conclusions

The adsorption potential was tested with agriculture waste material viz., sugarcane bagasse, banana stem, rain tree, and their mixtures. The effect of pH, ion concentration, sorbent concentration and contact time on removal of Cu2+ and Pb2+ metal ions were tested. The results obtained confirm that prepared SB sorbent can successfully remove both the ions from aqueous

solution. The optimum pH value for Cu2+ ion removal was 4.5. The contact time also affects the adsorption process, and it decreases with increase in contact time. The equilibrium contact time was 90 minutes after which the adsorption rate decreased. The adsorption process is stable in water system than the other desorbing agents. From the study, it is concluded that SB sorbent is a very promising sorbent for copper and BS and RT sorbent was effective in removing lead ions.

Acknowledgments

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