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## Evaluation on the Application of Immobilized Stereospermum kunthianum Stem-bark for the Removal of Pd<sup>2+</sup>, Cd<sup>2+</sup>, Cr<sup>2+</sup>, Fe<sup>2+</sup>, Zn<sup>2+</sup>, Mn<sup>2+</sup> in Aqueous Solution

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## Authors' contributions

This work was carried out in collaboration between all authors. All authors read and approved the final manuscript.

#### Article Information

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## ABSTRACT

This research studied the sorption capacity of *Stereospermum kunthianum* plant for the removal of heavy metal ions from waste water. The plant stem-bark was immobilized by entrapping or caging the bio-sorbent within a polymeric matrix of calcium- alginate to produce immobilized stem-bark (IMSB). The sorption efficiencies of heavy metals  $(Zn^{2+}, Pb^{2+}, Cd^{2+}, Mn^{2+}, Fe^{2+} and Cr^{2+})$  were investigated in aqueous solution using the IMSB and the residual metal ions in solution was determined using atomic absorption spectrophotometer (AAS). The effect of various parameters such as contact time, pH, ionic strength, initial metal ion concentration and temperature variation on the sorption of the above metal ions were investigated using batch experiments. The result obtained showed that the sorption efficiencies of  $Zn^{2+}$ ,  $Pb^{2+}$ ,  $Cr^{2+}$ ,  $Cd^{2+}$ ,  $Mn^{2+}$  and  $Fe^{2+}$  by IMSB were 91.60%, 97.85%, 78.46%, 85.08%, 65.20%, and 78.52% respectively. Result also shows that metal ion sorption increase in contact time; initial metal ion concentration and increase in

pH, and decreases with increase in temperature and ionic strength. In view of the above result, the abundant but presently wasted *Stereospermum kunthianum* plant can be used as a low-cost sorbent for the removal of heavy metal ions in waste water.

Keywords: Immobilization; Stereospermum kunthianum stem-bark; metal ions; sorption.

### 1. INTRODUCTION

Quality water is essential to human life and water of acceptable quality is essential for agriculture, industrial, domestic and commercial uses. All these activities are also responsible for polluting the water. Billions of gallons of waste from all these sources are thrown to freshwater bodies every day. The requirement for water is increasing while slowly all the water resources are becoming unfit for use due to improper waste disposal [1].

Environmental pollution is currently one of the most important issues facing humanity. Toxic heavy metals are considered one of the pollutants that have direct effect on man and animals [1]. The term heavy metal refers to any metallic chemical element that has a relatively high density and is toxic or poisonous at low concentrations. To a small extent they enter our bodies via food, drinking water and air. As trace elements, some heavy metals (e.g. copper, selenium, zinc) are essential to maintain the metabolism of the human body. However, at higher concentrations they can lead to poisoning. In recent years, management of pollution caused by heavy metals has been a major problem especially in developing countries. The introduction of heavy metals into the body of water and subsequently the consumption of it by human lead to hazardous effects such as blood and brain disorders (especially in young children), nephropathy, colic-like abdominal pains and miscarriage, as in the case of lead [2]. Cadmium and solutions of its compound are highly toxic, with cumulative effects causing irreversible brain, liver and kidney damage [3].

Unlike organic pollutants, heavy metals do not biodegrade and thus, pose a different kind of challenge for remediation [2]. To alleviate the problem of water pollution by heavy metals, various methods have been used to remove them from waste water such as chemical precipitation, coagulation, floatation, adsorption, ion exchange, reverse osmosis and electrodialysis [2]. The production of the sludge in the precipitation methods poses challenges in handling treatment and hand filling of the solid sludge. Ion exchange usually requires a high capital investment for the equipment as well as high operational cost. Electrolysis allows the removal of metal ions with the advantage that there is no need for additional chemicals and also there is no sludge generation, however, it is inefficient at a low metal concentration. Membrane processes such as reverse osmosis and electro-dialysis tend to suffer from the instability of the membranes in salty or acidic conditions and fouling by inorganic and organic substances present in waste water. Most of these techniques have some pre-treatment and additional treatments. In addition, some of them are less effective and require high cost.

This has encouraged research into discovering materials that are both efficient and cheap. In view of these, interest has recently risen in the investigation of some unconventional methods and low cost materials for sorption of heavy metal ions from waste water [4].

Abundant natural materials, particularly of cellulosic nature, have been suggested as potential bio-sorbents for heavy metals. Indeed, some biomass types are very effective in accumulating heavy metals. The economy of environmental remediation dictates that the biomass used for clean-up purposes should be abundantly available and must come from nature or even has to be a waste material. Konkoli leaves, moringa olefera bark, Seaweeds, pumpkin, among other kinds of biomass, have been tested for metal sorption with very encouraging results. Considering the number of biomass types, metals of interest and the number of experimental or process parameters, the task of prospecting for new and potentially feasible sorbents has a very wide scope.

Stereospermum kunthianum belongs to the family Bignoniaceae. It is a shrub or tree 3-15 m high. The plant is found in dry areas of deciduous forest, rocky outcrops, and termite mounds [5]. The plant is used in treating/purification of water by communities where the plant is commonly found. All the plant parts (stem-bark, root, leaves and seed) have medicinal values. The leaves are

palatable and are browsed by all ruminant animals; leaf infusion is used for treating wounds. Stem bark decoction is used to cure bronchitis and pneumonia [6]. Roots are used in treating venereal diseases and respiratory ailments. The pods are chewed with salt for coughs and are used in treatment of ulcers [7]. To the best of our knowledge, no report is presently available on industrial applications of *Stereospermum kunthianum* plant samples in water treatment which are presently wasting away in the wild.

The Immobilization of *Stereospermum kunthianum* plant stem bark involves the trapping of the biomass within the matrix of the polymer with sodium alginate and calcium chloride solutions to give a good bio-sorbent material [8]. However, bio sorbent has water retention capacity, high and fast sorption uptake [9]. Also because of immobilization, the bio- sorbent so obtained offer easy and conventional usage compare to free biomass, which is easily biodegradable [10].

In this experiment, we set out to evaluate the application of immobilized *Stereospermum kunthianum* plant (stem-bark) with sodium-alginate as a bio-sorbent for the removal of Lead, Cadmium, Chromium, Zinc, Iron, and Magnesium from aqueous solutions.

## 2. MATERIALS AND METHODS

#### 2.1 Materials

Sodium alginate, calcium chloride, sodium hydroxide, hydrochloric acid, lead nitrate, ferric chloride, cadmium (II) nitrate, chromium (III) nitrate, zinc nitrate. The chemicals were of analytical grade and were used as supplied. *Stereospermum kunthianum* Stem-bark was obtained from Waka creek in Biu, Borno state, Nigeria.

#### 2.2 Methods

#### 2.2.1 Preparation of Stereospermum kunthianum stem-bark sample

The stem bark, sample was freed from sand particles and dead dried tissues by carefully scraping with spatula and chopped to pieces, air dried for two weeks at  $30^{\circ}$ C, and milled into powder using pestle and mortar. This was then filtered through 100 µm sieve screen to obtain

fine powder. The pulverized sample was stored in polyethylene bag for further analysis.

#### 2.2.2 Dispersion of plant sample

The dispersion of the *Stereospermum kunthianum plant* (*stem-bark*) was done by weighing 4.00 g of the stem-bark powder and dissolved in  $100 \text{ cm}^3$  of water and the mixture was poured into a separating funnel and live to stand for 12 hours for separation into various fraction.

# 2.2.3 Preparation of sodium-alginate and calcium chloride solutions

Sodium alginate was prepared by weighing 4.00 g and making it up to 100 cm<sup>3</sup> mark with distilled water in a volumetric flask and left over night for complete dissolution to give 4% w/v solution. Calcium chloride (0.12 M) was prepared by weighing 26.28 g into 1 litre volumetric flask and making it up to the mark with distilled water [11].

#### 2.2.4 Synthesis of immobilized Stereospermum kunthianum stem-bark

25 ml viscous lavers of dissolved Stereospermum kunthianum plant stem-bark was mixed with 25 ml of 4% stock solution of Sodium alginate and stirred vigorously in 250 ml beaker. The mixture was then transferred into another beaker containing 30 ml of 0.12 M Calcium chloride solution. The reaction was kept for 1hour for complete precipitation. The precipitated blend solid of the sample was filtered and dried at room temperature for 24 hours. The dried solid was stored in polyethylene bag for further use [11].

#### 2.2.5 Preparation of synthetic waste water

The metal ions chosen for this study were  $Pb^{2+}$ ,  $Fe^{2+}$ ,  $Cr^{2+}$ ,  $Cd^{2+}$ ,  $Mn^{2+}$  and  $Zn^{2+}$ . The standards of these metal ion solutions were prepared from their anhydrate salts by dissolving 1.60, 2.90, 7.70, 2.29, 3.46 and 3.78 g of Lead nitrate, Ferric chloride, Chromium (III) nitrate, Cadmium (II) nitrate, Manganese nitrate and Zinc nitrate in distilled water respectively and made up to 1 litre in a Volumetric flask that gave the stock solution of 1000 ppm of the metal ions. Serial dilution of 200 ppm of each metal ion solution was prepared with distilled water. The synthetic waste water was kept for further use [12].

#### 2.2.6 Sorption experiment using immobilized <u>Stereospermum</u> kunthianum plant <u>samples</u>

Experiments were carried out in the batch mode for the measurement of sorption capacities. From 200 ppm of each metal ion solution, 50 ml was taken into a 250 ml conical flask and 0.2 g of the Stereospermum kunthianum stem-bark was added, corked with a rubber bung and shaken with a flask shaker for 2 hours at room temperature (30°C) at 150 rpm. The separation of the sorbents and solutions was carried out by filtration with whatman filter paper No 42 and the filtrates were stored in Sample cans for use. The residual metal ion Concentrations was Atomic determined using Absorption Spectrophotometer (AAS) Buck Scientific Model 210. (Air /Acetylene Flame, Integrated Model) Normal Parameters for Zn, Cd, Pb, Cr, Mn and Fe. The percentage sorption was calculated using the following equation

% Adsorption =  $[(Ci - Cf / Ci)] \times 100/1$ 

Where Ci = Initial metal ion Concentration and Cf = Equilibrium metal ion Concentration (mg/l) [13].

#### 2.2.7 Determination of the effect of contact time on sorption capacity of immobilized Stereospermum kunthianum plant stem-bark

To determine the kinetics of the membrane sorption, different sets of samples consisting of 0.2 g of the dried membrane and 50 ml of the metal ion solutions was prepared. The samples with solution were placed on flask shaker. As the samples were undergoing agitation, they were removed one after the other at a predetermined time interval ranging from 30 min-24 hrs. The solutions was filtered and analyzed for residual metal ion using AAS

#### 2.2.8 Determination of the effect of temperature on sorption capacity of immobilized Stereospermum kunthioanum plant stem bark

The Sorption Capacity of *Stereospermum Kunthianum* samples for the various metal ions were determined. Set of samples consisting of 0.2 g of the dried *Stereospermum Kunthianum* stem-bark and 50 ml of the metal ion solutions were inserted into 250 ml conical flasks, corked with rubber bungs and climbed on a flask shaker and shaken for 2 hrs. They were removed one after the other at different temperatures ranging from 30°C to 80°C. The solutions were filtered and analyzed for the residual metal ions using AAS.

#### 2.2.9 Determination of the effect of lonic strength on SORPTION capacity of immobilized Stereospermum kunthianum plant stem-bark

Useful information with regard to the effect of salt was obtained by measuring the sorption capacity of the membrane in various concentrations of NaCl solutions. Different concentrations (0.00 - 0.35) of NaCl was added to adjust the ionic strength of the metal ion solutions and the equilibrium concentration of the residual metal ions was determined using AAS.

#### 2.2.10 Determination of the effect of pH on sorption capacity of immobilized <u>Stereospermum kunthianum plant</u> stem-bark

The Sorption Capacity of the 0.2 g of Immobilized *Stereospermum kunthianum* stem bark sample with 50 ml of 200 ppm of various metal ion solutions at different pH values (1.0 to 6.0) were shaken for 2 hours using a flask shaker at 30°C. 1.0 M HCl and 1.0 M NaOH were used to adjust the pH of the solutions as the case may be. The residual metal ions were measured using Atomic Absorption Spectrophotometer AAS.

#### 2.2.11 Determination of the effect of initial metal ion concentration on sorption capacity of immobilized Stereospermum kunthianum Stembark

The Sorption Capacity of various samples consisting of 50 ml each of different metal ion concentrations ranging from 5 ppm to 100 ppm, each containing 0.2 g of the dried *Stereospermum Kunthianum* plant samples were prepared and shaken for 2 hours on a flask shaker at 30°C. The solutions were filtered and analyzed for residual metal ions using AAS.

## 3. RESULTS AND DISCUSSION

## 3.1 Sorption Capacity of Immobilized *Stereospermum kunthianum* Stembark

Fig. 1 shows the sorption capacity for different metal ions. The sorption capacity for  $Zn^{2+}$ ,  $Pb^{2+}$ ,

Cr<sup>2+</sup>, Cd<sup>2+</sup>, Mn<sup>2+</sup> and Fe<sup>2+</sup> using immobilized Stereospermum kunthianum bark were found to be 91.60%, 97.85%, 78.46%, 85.08%, 65.20%, and 78.52% respectively. This shows that the highest value of sorption capacity was recorded for  $Pb^{2+}$  and  $Cr^{2+}$ , while the lowest value was recorded for  $Cd^{2+}$ . The differences observed in the sorption capacity for the different metal ions can be explained in terms of difference in hydration free energy which is the energy released upon the attachment of ions to water molecules. The ability of metal ions to form covalent bonds with functional group present in the biomass and nature of the sorption sites as regard to porous nature of the bio-sorbent This is as a result of the biomass having negative charge functional groups such as hydroxyl, carbonyl and sulphate. When the biomass is immersed in water containing the metal ions, the positively charged metal ions combine with the negatively charged functional groups there by forming a covalent bond between the metal ions and the functional groups.

#### 3.2 Effect of Contact Time on Sorption Efficiency

The sorption of  $Zn^{2+}$ ,  $Pb^{2+}$ ,  $Cd^{2+}$ , $Mn^{2+}$ ,  $Fe^{2+}$  and  $Cr^{2+}$  by immobilized *Stereospermum kunthianum* stem-bark was studied as a function of time in order to find out the equilibrium time required for maximum sorption of metallic ions. Fig. 2 show the sorption of the metallic ions using

immobilized Stereospermum kunthianum stembark. It is observed that the concentration of metal ions adsorbed by the immobilized Stereospermum Kunthianum stem-bark increased with time. This is also due to the migration of higher fraction of the metal ions from the bulk solution through the sorbent boundary layer onto the active sites of the sorbent as time progresses [14]. This enhanced sorption of the metal ion with increase in agitation time. It may also be due to the decrease in boundary laver resistance to mass transfer in the bulk solution and an increase in kinetic energy of the hydrated metal ions. The removal of metal ions by Stereospermum kunthianum stem-bark could also be due to difference in sorption processes: ionexchange, adsorption, co-ordination, complexion as a result of the various types of ion binding groups such as hydroxyl, carboxylic, phenolic functional groups in Stereospermum Kunthianum Plant.

The initial faster rate may be due to the availability of the uncovered surface area of the sorbents, since the sorption kinetics depends on the surface area of the sorbents. The metal ions adsorption took place at the more reactive sites. As these sites were progressively filled, the more difficult the sorption becomes, as the sorption process tend to be more unfavourable. This is the general characteristics of sorption of metal ions [15].

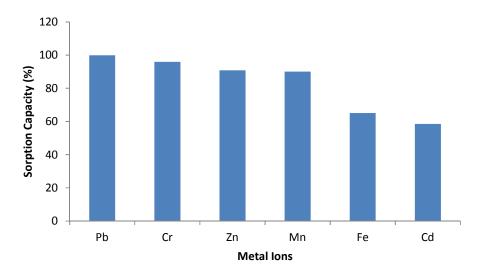


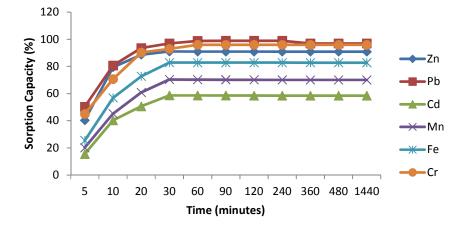
Fig. 1. Sorption Capacity of metal ions by Stereospermun kunthianum plant stem-bark stirring-time: 2 hrs, temperature: 30°C, initial metal concentration: 200 ppm

#### 3.3 Effect of Temperature on Sorption Efficiency

The effect of temperature on the sorption of Zn<sup>2+</sup> Pb<sup>2+</sup> Cd<sup>2+</sup>, Mn<sup>2+</sup>, Fe<sup>2+</sup> and Cr<sup>2+</sup> from solutions containing immobilized Stereospermum kunthianum stem-bark was studied. Fig. 3 show that the removal of the metals from aqueous Stereospermum solution bv immobilized kunthianum stem-bark is temperature dependent. Increase in temperature from 30°C to 80°C was found to result in a steady decrease in the removal efficiency of the sorbent for the metal ions. This is probably due to the effect of temperature on the interaction between the fibre surface and the metal ions in solution.

Increase in temperature probably weakens the bond formed between the metal ions and the sorption sites on the sorbent thereby resulting in a decrease in the amount of metal ions adsorbed on the sorbent. This implies that the decrease in the performance with increase in temperature may be physical sorption which is normally exothermic reaction, thus the extend of sorption generally increases with decreasing temperature [13].

Also increase in temperature result to the contraction of the plant fibres which causes the pores to be narrower and some suction sites to be hidden or inaccessible to the metal ions thereby decreasing the adsorption of the metal ions [16].



**Fig. 2. Effect of contact time on sorption capacity of ISKB** *Stirring-time: 2 hrs, Temperature: 30°C, initial metal concentration: 200 ppm* 

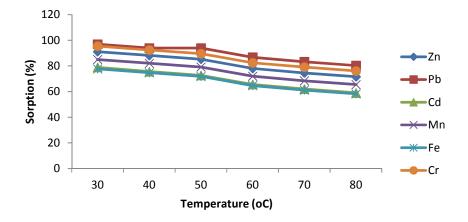


Fig. 3. Effect of temperature on sorption capacity of ISKP (Stem-Bark) Stirring-time: 2 hrs, initial metal concentration: 200 ppm

## 3.4 Effect of Ionic Strength on Sorption Efficiency

Fig. 4 represent the effect of the ionic strength on removal of heavy metal ions from aqueous immobilized solution by Stereospermum kunthianum stem-bark. Industrial waste water or natural water often contains ions such as Na<sup>+</sup> and Ca<sup>+</sup>, hence the influence of ionic strength on the sorption capacity of this immobilized plant stem-bark was investigated. It can be observed that the sorbent showed a decrease in sorption capacity with increase in ionic strength for all the metal ions studied. The decrease in sorption capacity with increasing ionic strength is primarily attributed to the reduction in movement of ions from lower to higher concentration between the sorbent and the external solution [17,10].

The differences in the movement of these ions from lower to higher concentration (osmotic pressure) between the sorbent and the external solution decrease as the ionic strength of external solution increases. Therefore, the sorption of metal ion decreases when the ionic strength of external solution increases [18].

From the above study, the coexistence of metal ions in the same solution caused the interference with the performance of immobilized *Stereospermum kunthianum* stem-bark sorbent. Therefore the pre-treatment of the effluent to remove the interfering ions will improve the efficiency of the sorbent [19].

#### 3.5 Effect of pH on Sorption Efficiency

The effect of pH on the adsorption of  $Zn^{2+} Pb^{2+}$ ,  $Cd^{2+}$ ,  $Mn^{2+}$ ,  $Fe^{2+}$  and  $Cr^{2++}$  from solutions containing immobilized *Stereospermum kunthianum* stem-bark was studied. Fig. 5; shows that the concentration of the metal ions increased gradually as the pH moves from lower to higher pH. This result of the effect of pH on the sorption of metal ions by immobilized *Stereospermum kunthianum* stem-bark shows that higher pH medium tend to support sorption more than lower pH medium.

pH is an important controlling parameter for sorption of metal ions because it affects the solubility of the metal ions, concentration of the counter ions on the functional group of the sorbent and the degree of ionization of the sorbate during reaction [20].

From the theory of acid-base equilibria in the pH range of 2.5 – 5.0, binding of heavy metals cations is determined primarily by the degree of dissociation of the weak acidic groups. Carboxylic acid groups (-COOH) normally found in biomass is one of the important functional groups for the uptake of metal ions by the sorbent [21]. The ionic state of a bio-sorbent can be used to explain the pH dependence of bio sorption. Low pH condition allows hydrogen and hydronium ions to compete with metal binding sites uptake. At higher pH values, there are lower numbers of competing hydrogen ions and more ligands are exposed with negatives resulting in greater metal ion sorption.

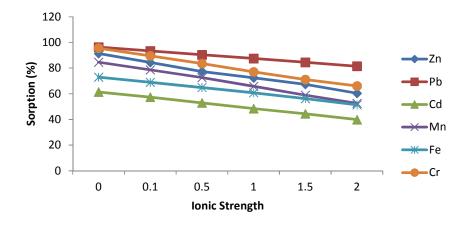


Fig. 4. Effect of lonic strength on sorption capacity of ISKP (Stem-Bark) Stirring-time: 2 hrs, temperature: 30°C, initial metal concentration: 200 ppm

### 3.6 Effect of Initial Metal ion Concentration on Sorption Efficiency

The result of metal ion sorption by immobilized *Stereospermum kunthianum* bark as a function of initial concentration is shown in Fig. 6. It can be observed that the removal capacity of the metal ions by the biomass increase with increase in the concentration of metal ions. A rapid sorption of the metal ions also occurred during the initial stages. This behaviour can be explained in terms of increase in flux, which is the driving force needed to overcome all mass transfer resistance

of metal ions between the solid and liquid phase and also the high collision probability between the metal ions and bio-sorbent surface [22].

The increase in sorption capacity can also be explained on the basis that, at lower concentration of metal ions, the number of the metal ions in the solution is less as compared to the available sites on the bio-sorbent. But at higher concentration, the available sites for the sorption become fewer and hence competition among metal ions for the few available sites, this then leads to increase in sorption capacity.

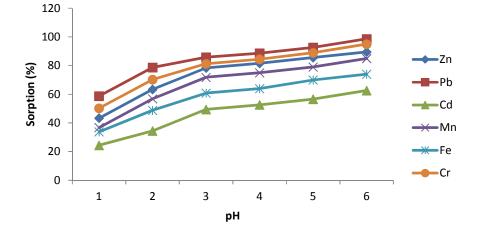


Fig. 5. Effect of pH on sorption capacity of Immobilized Stereospermum kunthianum stem-bark Stirring-time: 2 hrs, temperature: 30°C, initial metal concentration: 200 ppm

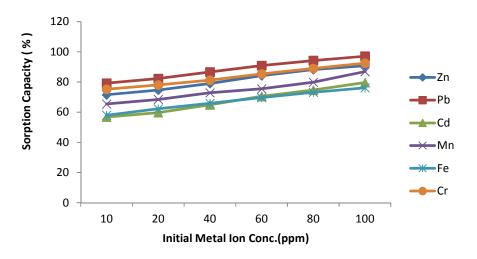


Fig. 6. Effect of initial metal ion concentration on sorption capacity of immobilized Stereospermum kunthianum stem-bark

Stirring-time: 2 hrs, temperature: 30°C, initial metal concentration: 200 ppm

## 4. CONCLUSIONS

This study aimed at investigating the sorption capacity of immobilized *Stereospermum kunthianum* stem-bark for  $Zn^{2+}$ ,  $Pb^{2+}$ ,  $Cd^{2+}$ ,  $Mn^{2+}$ ,  $Fe^{2+}$ , and  $Cr^{2+}$ , from aqueous solution and the effect of temperature variation on sorption and the effect of variation of pH on aqueous solutions.

The result obtained from this investigation shows that immobilized *Stereospermum kunthianum* stem-bark adsorbed all the metals above from aqueous solution and the concentration of the metal ions adsorbed increased with increase in contact time, increase in temperature and increase in pH for each metal.

The rate of sorption of metal ions in aqueous solution on the surface of immobilized *Stereospermum kunthianum* stem-bark is highest for Pb<sup>2+</sup>, Mn<sup>2+</sup>, Cr<sup>2+</sup>, Zn<sup>2+</sup> compare to Fe<sup>2+</sup> and Cd<sup>2+</sup> which was at its lowest.

It is therefore apparent to conclude that immobilized *Stereospermum kunthianum* stembark could serve as cheap readily available effective sorbent for the removal of the said metal ions from water as a way of treatment before discharging into the environment.

#### **COMPETING INTERESTS**

Authors have declared that no competing interests exist.

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