Comparison of the Sorption Capacity of Heavy Metals from Wastewater Using Immobilized, Grafted and Membrane *Stereospermum Kunthianum* Stem Bark

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Abstract- This study was conducted to explore the sorption capacity of three different industrial prepared products of *Stereospermum kunthianum* plant for the removal of some heavy metal ions from waste water.The plant stem-bark was first immobilized by entrapping the biosorbent with in a polymetric matrix of calcium alginate to produce immobilized stem-bark (IMSB). The stem-bark was also synthesised by reacting it with Acrylamide at 60^{o}c using Ceric Ammonium Nitrate as an initiator to obtaine grafted stem-bark (GSB) and the plant stem-bark was also mixed with glutaraldehyde to obtained Membrane stem-bark. The sorption efficiencies of some heavy metals (Pb, Mn, Zn, Cr, Fe and Cd) were investigated in aqueous solution using IMSB, GSB and MSB and the residual metal ions in solutions were determined using Atomic Absorption Spectrophotometer (AAS).The results obtained shows that the sorption efficiencies of Pb, Mn, Zn Cr, Fe and Cd by IMSB were 94.50%, 87.50%, 85.84%, 80.94%, 65.08% and 58.46% respectively. That of GSB were 97.85%, 90.00%, 91.60%, 95.89%, 78.52% and 65.20% respectively and sorption efficiency of MSB was 99.80%, 97.80%, 99.70%, 95.40%, and 97.70% for Pb, Mn, Zn, Cr, Fe and Cd respectively. MSB has maximum sorption capacity of metal ions in aqueous solution and occur in the trend MSB > GSB > IMSB. The result of this study present synthesized *Stereospermum kunthianum* stem-bark as potential bioremediation agent for treatment of water because it has the potential of removing heavy metals from water.

Index Terms- Stereospermum-kunthianum, Immobilization, Grafting, Membrane, Sorption and Heavy-metals.

I. Introduction

Environmental pollution is currently one of the most important issues facing humanity. It was increased exponentially in the past few years and reached alarming levels in terms of its effects on living creatures. Toxic heavy metals are considered one of the pollutants that have direct effect on man and animals. Industrial wastewater containing lead, copper, cadmium and chromium, etc for example can contaminate groundwater resources and thus lead to a serious groundwater pollution problem (Renge *et al*., 2012).

Water of high quality 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. The task of providing proper treatment facility for all polluting sources is difficult and also expensive, hence there is pressing demand for innovative technologies which are low cost, require low -maintenance and are energy efficient. The adsorption technique is economically favorable and technically easy to separate as the requirement of the control system is minimum. In this article, the technical feasibility of various low-cost adsorbents for heavy metal removal from contaminated water has been reviewed (Varsha *et al*., 2010).

Instead of using commercial activated carbon, researchers have worked on inexpensive materials, such as coconut shell, sawdust, mango leaves, chitosan. egg shell, and other adsorbents, which have high adsorption capacity and are locally available.

Stereospermum kunthianum plant

Stereospermum-kunthianum belongs to the family *Bignoniaceae*.The plant has vernacular names known by traditional herbalist and the communities where it is commonly found; *SanSami* (Hausa), *Ndengal-mbalu*, (Fulfulde), *Kengyar-tuma* (Babur-Bura), *Kera-thla* (Marghi), *Ayada* (Yoruba), *Alakiriti* (Ibo) and *Umana* (Tiv).

Stereospermum kunthianum is found in dry areas of deciduous forest, woodland, bush, rocky outcrops, termite mounds and margins of evergreen forests. The species is well spread all over the Sahel region and is often found near streams. Geographic distributions include Nigeria, Democratic Republic of Congo, Djibouti, Eritrea, Ethiopia, Kenya and Mozambique.

Biophysical limits Altitude **:** 500-2400 m, Mean annual temperature **:** Up to 40 ^{O}C, Mean annual rainfall **:** 450-900 mm. Soil type **:** Grows well on light silty and sandy soils. Reproductive **;** the bisexual flowers appear in the dry season before the new leaves, between February and March, and pods ripen between April and May (Aliyu Babayo, 2011).

Stereospermum kunthianum is a multipurpose plant of significant importance to local communities. According to traditional healer in Biu, Borno Nigeria (Bulama Fori, 2018), one of the major uses of the plant is in the treatment/purification of water by local communities where the plant is found. The plant is most useful when there is an outbreak of water born disease such as cholera. Apart from the above, the plant has numerous uses to the local communities where they are found. The plant leaves are used as feeds to animals (Cattles, Sheep, and goats). It has repellant property. The stem and roots of the plant if boiled and allowed to cool or if soaked in cold water over night could be used for many purposes according to Fori (2013).

The results of the heavy metals removal performance are compared to that of activated carbon and are presented in this study. It is evident that low-cost adsorbents have demonstrated outstanding removal capabilities for certain metal ions as compared to activated carbon. The adsorption process is being widely used by various researchers for the removal of heavy metals from waste streams despite its extensive use in the water and waste water treatment industries; activated carbon remains an expensive material. In recent years, the need for safe and economical methods for the elimination of heavy metals from contaminated waters has necessitated research interest towards the production of low cost alternatives to commercially available activated carbon. Therefore there is an urgent need that all possible sources of agro-based inexpensive adsorbents should be explored and their feasibility for the removal of heavy metals should be studied in detail.

Sources of heavy metals

The term heavy metal refers to any metallic chemical element that has a relatively high density and is toxic or poisonous at low concentrations. Examples of heavy metals include mercury (Hg), cadmium (Cd), arsenic (As), chromium (Cr). thallium (Tl). and lead (Pb). Heavy metals are natural components of the Earth's crust. They cannot be degraded or destroyed. To a small extent they enter our bodies via food, drinking water and air. As trace elements, some heavv 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. Heavy metal poisoning could result, for instance, from drinking-water contamination (e.g. lead pipes), high ambient air concentrations near emission sources, or intake via the food chain.

Heavy metals are dangerous because they tend to bioaccumulate. Bioaccumulation means an increase in the concentration of a chemical in a biological organism over time, compared to the chemical's concentration in the environment. Compounds accumulate in living things any time they are taken up and stored faster than they are broken down (metabolized) or excreted. Heavy metals can enter a water supply by industrial and consumer waste, or even from acidic rain breaking down soils and releasing heavy metals into / streams, lakes, rivers, and groundwater.

The effects of heavy metals

Many of the products, which we have used contain heavy metals in them. Heavy metals are in the foods we eat, water we drink, and the air we breathe. We need very little of only a few heavy metals including zinc, copper, cobalt, manganese, molybdenum, vanadium and strontium. These good heavy metals become toxic to us when the quantity is too high and it takes really very little to be too much. On top of that, we take in more than twenty heavy metals which are non-essential for our healthy functioning.

How do they get into the body? Heavy metals enter your body through drinking, eating, inhaling, and skin. Once in the body they do damage on the cellular level by causing dangerous free radicals production. The damage that they do is on the cellular level, and can cause cancer and many other diseases (Oremusova et al ., 2007).

II. MATERIALS AND METHODS

Sample Collection and Identification

A sample of the stem bark, of *Stereospermum Kunthianum* plant was collected in paper bags from creek Waka Biu, in Biu local government area, Borno State, Nigeria and was identified by Bulama Fori, Mbulamel Biu, Borno State, and confirmed by a plant Botanist Prof S.S Sanusi, Department of Biological Sciences, University of Maiduguri.

Sample Preparation

The stem bark, sample was freed from sand particles and dead dried tissues by carefully scraping with spatula. It was chopped to pieces, air dried for two weeks. The pulverized samples was stored in paper bags for further analysis (Osemeahon *et al*., 2007).

Dissolution of Plant Samples

The dissolution of the *stereospermum kunthianum* stem was done by weighing 4.00 g of the stem bark powder and dissolved in 100 cm^3 of water, then the mixture was poured into a separating funnel and allow to stand for 12 hrs to observe the possible separation into various fractions. (Osemeahon *et al*., 2007).

Preparation of Sodium Alginate and Calcium Chloride to Immobilized Sample

Sodium alginate was made by weighing 4.00 g and making it up to 100 cm^3 mark with distilled water in a volumetric flask and allow overnight for complete dissolution to give 4% w/w. Calcium chloride (0.12 M) was prepared by weighing 26.28 g into 1L volumetric flask and make up to the mark with distilled water (Wuyep *et al.*, 2007).

Synthesis of Immobilized Samples of *Stereospermum kunthianum*

25 cm^3 viscous layers of dissolved *Stereospermum kunthianum* stem bark sample were mixed with 25 cm^3 of 4 % stock solution of sodium alginate and stir vigorously for even mixing in 250 cm^3 beakers. The mixture was then transferred into another beaker containing 30 cm^3 of 0.2 M Calcium chloride solution. The reaction was allowed retention time of 1 hour for complete precipitation. The precipitate blend solid of the stem sample was filtered and dry at room temperature (30^0C) for 24 hrs. The dried solid was stored in polyethylene bag for further use (Wuyep, *et al.*, 2007).

Adsorption of Heavy Metals from Aqueous Solution Using Immobilized *Stereospermum-kunthianum* Plant

Preparation of synthetic waste water

The metal ions chosen for this study are Pb, Fe, Cr, Cd, Mn and Zn. The reason they are choosen is because of their toxicity. The standards of these metal ion solutions was prepared from their salts by dissolving 1.60 g, 3.54 g, 4.10 g, 2.10 g, 3.46 g and 2.90 g of Lead nitrate, Iron nitrate , Chromium (II) nitrate, Cadmium nitrate, Manganese nitrate and Zinc nitrate in deionised water respectively and made up to 1 litre in a Volumetric flask to 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 (Ogali *et al.,* 2008).

Immobilization of *Stereospermum kunthianum* Stem-barks

The immobilization of *Stereospermum kunthianum* stem-bark was achieved by entrapping or caging it within the polymeric matrix of Sodium alginate. It has been established that Sodium alginate consist of L-guluronic acid and D-manuronic acid units (Mary *et al*., 2005). The contacting of Ca^{2+} ions with gulunoric acid block forms an ionically cross-linked structure in aqueous environments. The cross linking of the polymer is due to binding of divalent cations (Ca^{2+}) to the carboxylic ($-COOH^-$) group of L-guluronic acid block (Mary *et al*., 2005). Divalent cations act as a cross-links and cause an ionic binding between G-blocks in polymer chains and forms three dimensional network (Naghan and Ageena, 2010). This network mobilizes *Stereospermum kunthianum* bark to produce a biosorbent.

Sorption experiment using immobilized Stereospermum kunthianum plant stem-bark

Experiments was carried out in the batch mode for the measurement of adsorption capacities. From 200 ppm of each metal ion solution, 50 cm^3 was taken into a 250 cm^3 conical flask and 0.2 g of the ***immobilized*** *Stereospermum kunthianum* plant samples was added and corked with a rubber bung and shaken with a flask shaker for 2 hrs at room temperature (30^{O}C) at 150 rpm. The separation of the adsorbents and solutions was carried out by filtration with whatman filter paper and the filtrates was stored in Sample cans for use. The residual metal ion Concentrations was determined using Atomic Absorption Spectrophotometer (AAS) The percentage adsorption was calculated using the following equation.

% Adsorption = [(Wx - Wy /Wx)] x 100

where Wx = Initial metal ion Concentration and Wy = Equilibrium metal ion Concentration (mg/l)

Synthesis of Grafted Copolymer

Stereospermum kunthianum polyacrylamide was synthesized by reacting *Stereospermum kunthianum* plant samples with acrylamide at 60^{o}C using Ceric ammonium nitrate (CAN) as an initiator (Osemeahon *et al.*, 2008). Aqueous *Stereospermum kunthianum* solution (2%) was prepared and stirred well for 1 hr with 0.105 mol of acrylamide at 60^{0}C. The initiator solution containing $2.5x10^{-3}$mol of CAN was added to the mixture and stirred well by using magnetic stirrer for another 5 hrs. The mass obtained at the end of this period was precipitated in excess acetone. This was then filtered, washed with a 7:3 ratio of water/ methanol mixture to remove the homopolymer formed. The solid mass was then dried in an electrically controlled oven at 40^{o}C and weighed using metter balance.

Sorption experiment using grafted stereospermum kunthianum stem-bark

Experiments were carried out in the batch mode for the measurement of sorption capacities. From the 200 ppm of each metal ion solution, 50 cm^3 was taken into a 250 cm^3 conical flask and 0.2 g of the **Grafted** *Stereospermum kunthianum* Stem-bark was added, corked with a rubber bung and shaken with a flask shaker for 2 hours at room temperature (30^{O}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 determined using Atomic Absorption Spectrophotometer (AAS) Buck Scientific Model 210. (Air/Acetylene Flame, Integrated Model). Normal Parameters for Cd, Pb and Cr. The percentage sorption was calculated using the following equation

% Adsorption = [(Wx - Wy /Wx)] x 100

where Wx = Initial metal ion Concentration and Wy = Equilibrium metal ion Concentration (mg/l)

Membrane Preparation

A 4% mass of stock solution of sodium alginate and *Stereospermum kunthianum* stem-bark was prepared in water mixed in the ratio of 80:20, (Osemeahon *et al*., 2008), (Toti *et al.*, 2002).

The stock solution with a volume of 100 cm^3 was taken in a beaker and mixed with 0.00175 mol of glutaraldehyde (GA). This mixture was stirred for 2 hrs at 25^{0}C and then poured uniformly on a plastic tray. The membrane was allowed to dry at room temperature (25^{O}C) for 12 hrs. The cast membrane was cross linked by immersing in a 1% HCl solution taken in an equimolar mixture of methanol and water for 24 hrs. It was washed thoroughly in water and allowed to dried.(Barminas *et al*., 2005).

Sorption capacity using membrane stereospermum kunthianum stem-bark

A concentration of 200 ppm of each of the metal ions were prepared with deionised water. From the 200 ppm concentration, 50 cm^3 of solution of metal ion was taken into a conical flask. 0.2 g of dried membrane was added and then shaken vigorously for 2 hrs by using flask shaker. The mixture was then filtered and the residual metal ion concentrations were determined using Atomic Absorption Spectrophotometer (AAS) (Barminas *et al*., 2005).

III. RESULTS AND DISCUSSION

Sorption Capacity of Immobilized *Stereospermum kunthianum* Stem-bark

The stem-bark was immobilized to obtained immobilized stem-bark (IMSB) and the sorption of heavy metal ions namely Pb^{2+} ,Mn^{2+}, Zn^{2+}, Cr^{2+}, Fe^{2+} and Cd^{2+} was determined in aqueous solution using IMSB and the residual metal ions in solution was determined using Atomic Absorption Spectrophotometer (AAS).

Figure 1 shows the sorption capacity for different metal ions. The sorption capacity for Zn^{2+}, Pb^{2+}, Cr^{2+}, Cd^{2+}, Mn^{2+} and Fe^{2+} using **immobilized** *Stereospermum kunthianum* Stem- 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+}, 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 is always a factor (Mahvi, 2008). 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.

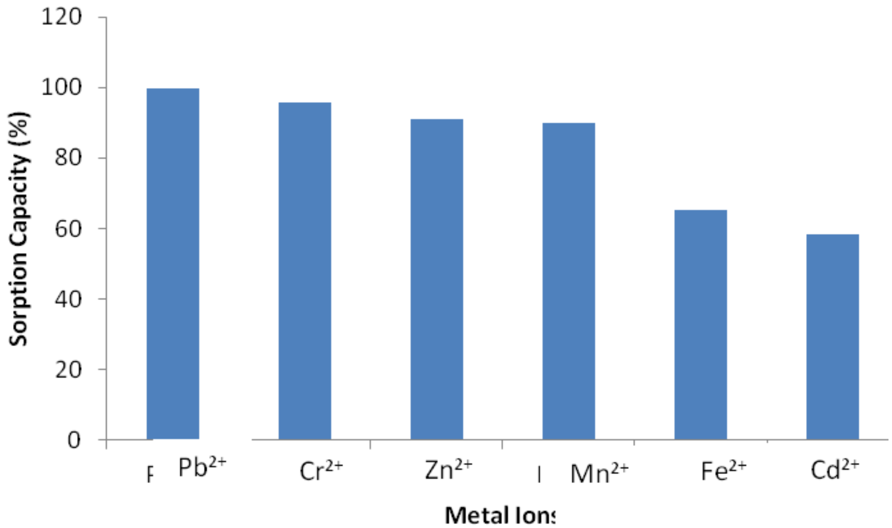

Figure 1: Sorption Capacity of Metal Ions by *Immobilized Stereospermum kunthianum* Stem- bark. (*Time: 2 hrs; Temperature: 30°C; Initial Metal Concentration: 200 ppm).*

Sorption Capacity of Grafted *Stereospermum kunthianum* Stem-bark

In order to improve the sorption capacity of the *Stereospermum kunthianum* stem-bark, it was grafted and the effect of grafting studied.

Effect of grafting on the sorption capacity of Stereospermum-kunthianum

Figure 2 indicate the equilibrium sorption for Pb^{2+}, Zn^{2+}, Cd^{2+}, Cr^{2+}, Fe^{2+} and Mn^{2+} by grafted *Stereospermum kunthianum* stem- bark (GSKB). It is observed that this grafted plant bark has the ability to remove significant quantity of the metal ions. The values 98.89 % , 97.84 %, 89.93 %, 80,94 %, 98.52 %, 90.20 % respectively were obtained as sorption capacity from the present study using grafted sample. This has more advantage over immobilized biomass by having higher values. The values obtained during metal sorption using immobilized biomass were 97.85%, 91.60%, 85.08%, 78.46%, 78.52% and 65.20% for Pb^{2+}, Zn^{2+}, Cd^{2+}, Cr^{2+}, Fe^{2+} and Mn^{2+} respectively. As can be seen from the results in Figure 2, grafted biomass has more efficiency over immobilized biomass.

By grafting the stem-bark, positive development has added value to the efficiency of the biomass. The sorption of the metal ions by the grafted biomass is higher than the immobilized biomass because of the added functional group. The Acrylamide that was used for the grafting has amide functional group and the nitrogen in the amide have three unpaired electrons and therefore has more negative charges that will attract and bind more metal ions.

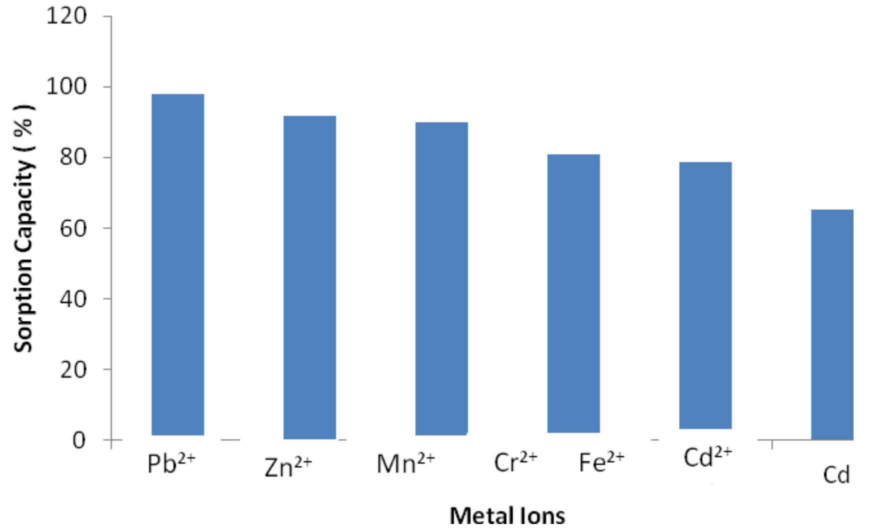

Figure 2: Sorption Capacity of Grafted *Stereospermum kunthianum* Stem-bark (Time*: 2hrs, temperature: 30°C, initial metal concentration: 200 ppm)*

Membrane Sorption Capacity

The stem-bark was also converted to Membrane to obtain membrane stem-bark (MSB). The conversion or preparation of the biomass into a membrane is to find out any possible advantage of the membrane over the grafted and immobilized plant stem-bark biomass.

Determination of Sorption Capacity of Membrane Stereospermum kunthianum Stem-bark

The sorption capacity for metal ions by membrane is presented in Figure 3. The result show that membrane can take up significant quantities of Pb^{2+}, Cr^{2+}, Fe^{2+} but relative lower amount of Zn^{2+}, Mn^{2+},and Cd^{2+}.

The sorption capacity of this membrane for the metal ions are in order Pb > Cr > Fe > Zn > Mn > Cd respectively. The values 99.80%, 99.70%, 98.40%, 97.80%, 97.80% and 97.70% respectively were obtained from the present studies and are higher than that of grafted which is 98.89%, 80.94%, 98.54%, 97.84%, 90.20% and 89.93% for Pb^{2+}, Cr^{2+}, Fe^{2+}, Zn^{2+}, Mn^{2+},and Cd^{2+} respectively.

The result shows that, membrane can take up more quantities of the metal ions than the grafted and immobilized biomass. The main reasons why membrane adsorbed more metal ions is because of the fact that, more or larger surface sorption sites were created, the binding sites and active surface area for attracting the metal ions were more.

The difference observed in the sorption capacity for the different metal ions can be explained in terms of the ability of the metal ions to form covalent bonds with ligands, the metal polymeric cations with in the membrane structure and the nature of the surface sites available (Cooper *et al*., 2000).

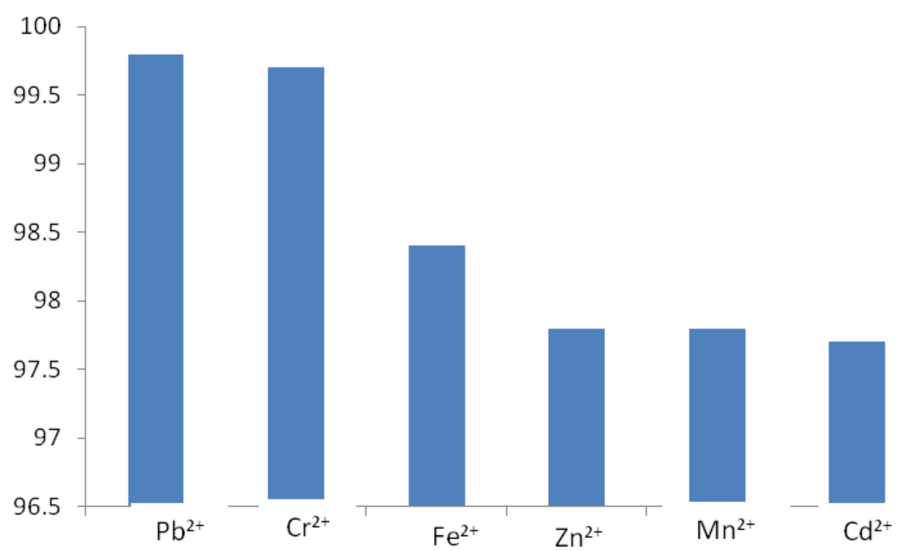

Figure 3: Sorption Capacity of Metals by Membrane *Stereospermum kunthianum* Stem-bark

(Time: 2 hrs, temperature: 30°C, initial metal concentration: 200 ppm)

IV. SUMMARY

The stem-bark was immobilized to obtained immobilized stem-bark (IMSB) and the sorption of heavy metal ions namely Pb^{2+} ,Mn^{2+}, Zn^{2+}, Cr^{2+}, Fe^{2+} and Cd^{2+} were determined in aqueous solution and the residual metal ions in solution were determined using Atomic Absorption Spectrophotometer (AAS). The sorption capacity for IMSB was 94.50%, 87.50%, 85.84%, 80.94%, 65.08% and 58.46% for Pb^{2+} ,Mn^{2+}, Zn^{2+}, Cr^{2+}, Fe^{2+} and Cd^{2+} respectively.

In order to improve the sorption capacity of the *Stereospermum kunthianum* stem-bark, it was grafted and the effect of grafting studied. The stem-bark was grafted to obtained grafted stem-bark (GSB), and was used for sorption of Pb^{2+}, Mn^{2+}, Zn^{2+}, Cr^{2+}, Fe^{2+} and Cd^{2+} in aqueous solution and the percentage sorption capacity obtained for the metal ions using GSB were 97.85%, 90.00%, 91.60%, 95.89%,78.52% and 65.20% respectively.

The stem-bark was also Membraned to obtain membrane stem-bark (MSB). The conversion or preparation of the biomass into a membrane was to find out any possible advantage of the membrane over the grafted and immobilized plant stem-bark biomass. The percentage sorption for the MSB was 99.80%, 97.80%, 97.80%, 99.70%, 95.40% and 97.70% for Pb^{2+}, Mn^{2+}, Zn^{2+}, Cr^{2+}, Fe^{2+} and Cd^{2+} respectively. MSB has maximum sorption capacity of metal ions in aqueous solution and occur in the trend MSB > GSB > IMSB.

V. CONCLUSION

The result of this study present *Stereospermum-kunthianum* as potential bioremediation agent for the treatment of wastewater because it has shown clearly that *Stereospermum kunthianum* Stem-bark has the potentials of removing heavy metals from water.

The result of this studies show that the quality of water for consumption for developing countries can be improved by first adding membrane stem-bark of *Stereospermum kunthianum* plant.

The use of Stem-bark of *Stereospermum kunthianum* plant is to be given attention for its effectiveness in waste water treatment. The technologies involved are economical, traditional, easy to implement and ideal for rural areas. The process being biological in nature does not generate any non-treatable wastes. These processes are easy to operate and require little or no maintenance.

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