REMOVAL OF ALUMINUM (III) FROM POLLUTED WATER USING NEW EFFECTIVE BIO-SORBENTS DERIVED FROM STEMS OF *Burea monosperma* AND *Angle marmelo* PLANTS

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ABSTRACT

Highly effective sorbents are prepared from *Burea monosperma* stem powder (BMSP) and *Angle marmelo* stem powder (AMSP) for the removal of Al$^{3+}$ from water. Extraction conditions are optimized. Adsorption capacities are as high as: 50.0 mg/g for BMSP and 62.0 mg/g for AMSP. Good extractions are observed in a wide range of pH: 3 to 9 with a maximum at pH:8, emphasizing the successful utility of both the adsorbents even in low acidic conditions. Common co-ions interfere marginally. Thermodynamic parameters reveal the spontaneity of sorption of Al$^{3+}$ onto the sorbents. Analysis of adsorption isotherms confirms Langmuir model of adsorption. Of the various kinetic models applied pseudo-second-order explains well the kinetics of Al$^{3+}$ sorption. Both the spent sorbents can be regenerated and reused for three cycles with little loss of sorption capacities. BMSP and AMSP are successfully applied to remove completely Al$^{3+}$ from the samples of industrial effluents as well as polluted lakes.

Keywords: Al-pollution Control, Bio-sorbents, *Burea monosperma*, *Angle marmelo*, Thermodynamics, Kinetics, Isotherms, Applications

INTRODUCTION

Aluminum is next to iron in human utility and it is abundantly available in the crust of earth$^{1,2}$. In acidic environments, Al$^{3+}$-ions leach into water bodies.$^{1,3}$ Numerous industries based on Aluminum are the potential sources of contamination.$^{2}$ Ill-treated or un-treated sewages from these industries enter into water bodies.$^{1-3}$ As Al$^{3+}$-salts are non-degradable, the Al-concentration increases in these water sources crossing the EPA maximum allowed limit of 0.2 ppm$^{3}$. Al intake is hazardous to human$^{3-6}$ as well as to aquatic life$^{7-10}$. Anemia, orthopedic ailments, neurological problems, brain hemorrhage etc., are some of the many problems caused by Al-intake.$^{3-6}$ Hence removal Al$^{3+}$ from contaminated water is one of the main aspects of water pollution control research. Earlier methods used are precipitating the Al$^{3+}$ as Al (OH)$_3$ by increasing solution pHs. These Precipitation methods are not satisfactory when Al$^{3+}$-concentration is low. Reverse osmosis, electro dialysis and ion-exchange methods of purification, though good, are non-economical and they are not adaptable to treat large amounts of water/sewage.$^{11-17}$ Methods based on adsorption are attracting researchers as these procedures are simple and effective.$^{18-20}$ When adsorbents are derived from plant materials, the adsorption process is becoming more interesting in view of their low cost, renewable plant sources and abundance.$^{18-20}$ Active Carbon$^8$, Granular activated carbon$^{21}$, Amberlite IR-120H$^{21}$, activated carbon- with oleic acid as surfactant$^{22}$, Chitosan$^3$ and leaves, stems and their ashes of *Acacia Melanoxylon* and *Eichhornia Crassipes$^{24}$, are investigated for Al-removal. Beach cast seaweed$^{22}$ and plant materials of *Ficus Racemosa$^{26}$, Cassia Occidentalis$^{27}$, Moryngea Millingtonia$^{28}$ and *Cygium Arjunum$^{28}$ are investigated as sorbents. Water hyacinth sorption ability for Al$^{3+}$ was investigated by growing it in wetlands.$^{29}$ We tried to identify plant materials having Al$^{3+}$-affinity. Stems of *Butea Monosperma* and *Angle marmelos*, are showing Al$^{3+}$-sorption character. The present research work is related to optimize the
EXPERIMENTAL

Material and Methods
Chemicals
Analytical grade chemicals were used. For the preparation of simulated as well as reagents, double distilled water was used. 500 ppm Al\(^{3+}\) stock solution was prepared. As per the requirement, it was diluted. Eriochrome cyanine R solution, buffer solutions, 5 vol Hydrogen peroxide solution were prepared as described in the literature.\(^{30}\)

Adsorbents
_Butea monosperma_ is a dry season quickly growing tree. It belongs to _Butea_ species of the plant kingdom. It grows well in tropical and sub-tropical areas to a height of 15 m. It is a sacred tree for Hindus and its wood pieces are used for fire rituals.

_Angle marmelos_ tree

Fig.-1: Plants showing Strong Adsorption for Al\(^{3+}\) Ions

_Aegle marmelos_ belongs to _Rutaceae_ family and its trivial name is bael /bhel. It is widely grown in India. It is a sacred tree for Hindus.

The stems of _Butea monosperma_ tree and _Angle marmelos_ tree were cut into pieces. They were washed with distilled water and dried in Sunlight for one week. Then they were dried at 105 °C in hot air over for one hour. Then the pieces were crushed to a fine powder and passed through a sieve having pores less than 75 microns in size. Powders were stored in dry bottles. These were investigated as adsorbents. _Butea monosperma_ stem powder was named as: BMSP while _Angle marmelos_ stem powder was named as: AMSP. This terminology is used throughout this manuscript.

Adsorption Experiment
Batch modes of extraction were employed in the Al-extraction investigations.\(^ {31,32}\) Synthetic simulated solutions having known Al\(^{3+}\) concentrations were prepared and were subjected to investigations. The general procedure employed was as detailed hereunder.

General Procedure
100 ml of simulated Al\(^{3+}\)-solutions were taken into stoppered 250 ml bottles. Then known amounts of BMSP or AMSP were added. pHs of solutions were adjusted to desired values with the dropwise addition of diluted solutions of acid or base. Then the bottles were agitated at 300 rpm in Shakers for the required periods. Then the contents in the flash were filtered. The residual amounts of Al\(^{3+}\) in the filtrate were determined using “Eriochrome cyanine R” method as described in Vogel.\(^ {30}\) The effect of each extraction parameter on Al\(^{3+}\)-sorption was investigated by gradually changing the...
RESULTS AND DISCUSSION

BMSP and AMSP were evaluated as adsorbents for the extraction of Al$^{3+}$ ions from water. The investigations were performed to optimize different extraction conditions to develop maximum affinity between Al$^{3+}$ and the sorbents. The findings are as follows.

Equilibration Time

The minimum equilibration time needed for the maximum extraction of Al$^{3+}$, is assessed. For this, known amounts of BMSP/AMSP were added to 50 ppm Al$^{3+}$ solution. Then the pH was adjusted to 8 using dil HCl or NaOH. Then the solutions were shaken at 300 rpm for different time intervals. Then the solution was filtered and the filtrate was analyzed for residual Al$^{3+}$. The findings are plotted in Fig.-2 and 3.

With the rise in time, % removal of Al$^{3+}$is increased for both the adsorbents at all initial pHs. Initially, the rate of adsorption is more and it decreases with time. After a certain time, almost a study state is reached with a further marginal increase in adsorption. With 15 minutes of equilibration and at pH: 8, 64.0% and
70.0% of \( \text{Al}^{3+} \) were removed from 100 ml of 75 ppm \( \text{Al}^{3+} \) solution with BMSP and AMSP respectively as adsorbents. A time of 90 min has been taken to achieve 98.0% removal with BMSP and 100.0% with AMSP. Further, as is seen from the curves, the equilibration time needed to reach the study state also depends upon the initial pH. The equilibration time decreases with a rise in pH.

For a fixed amount of adsorbent, the active sites are limited. Initially, the number of active sites available is more and so, the rate of adsorption is more. With the lapse of time, the active sites are used up or engaged with adsorbate, \( \text{Al}^{3+} \). So, the rate of adsorption of \( \text{Al}^{3+} \) falls. A study state is reached when all the active sites are practically used up.

**Effect of pH**

The initial pH effect on the extraction of \( \text{Al}^{3+} \) was investigated by varying the pH from 2 to 10 while maintaining all other extraction conditions at constant levels. The results are presented in Fig.-4. pHzpc was evaluated from the Fig.-5.

It is interesting to note that substantial amounts of \( \text{Al}^{3+} \) are removed in a wide range of pHs: 3 to 9. With BMSP, 76.0% at pH:3 to 98.0% at 8 and with AMSP 95% at pH: 3 to 100.0% at pH:8. The maximum extraction is noted at pH: 8. Above this pH, the sorption is less favoured and so % removal falls.

pHzpc values for BMSP and AMSP are 6.3 and 7.3 respectively. Above these values, the surface of the said sorbents acquires a negative charge. It is due to the dissociation of functional groups. Below this pHzpc, the dissociation is less probable; even protonation occurs at low pHs.

At low pH values, \( \text{Al} \) (III) exists as \( \text{Al}[(\text{H}_2\text{O})_6]^{3+} \). With raise in pH, \( \text{Al(OH)}^{2+} \) and \( \text{Al(OH)}^{3+} \) are increasingly formed. In neutral conditions amorphous \( \text{Al(OH)}_3 \) is nucleated. In basic conditions, the aggregates dissolve to form \( \text{Al(OH)}^+ \). Between pH: 6 to 8, hydrated \( \text{Al(OH)}_3 \) nucleates are not precipitated as their formation from \( \text{Al(OH)}_2^+ \) is inhibited. The good affinity of \( \text{Al}^{3+} \) at low pHs, indicates binding between \( \text{Al} \)-species and functional groups of sorbents: \(-\text{OH}/\text{COOH}\), which are not dissociated as is reflected from pHzpc values. Due to negative charges on the sorbent surface and Al (III) species, they get repelled. Hence fall of adsorption at high pHs.

**AMSP/ BMSP Concentrations**

The minimum AMSP/ BMSP Concentration required for \( \text{Al}^{3+} \)removal was investigated. By changing the AMSP/BMSP concentration but maintain the other extraction conditions at constant levels, % removal of
Al^{3+} was assessed. The results are depicted in Fig.-6. 1.25 g/L for AMSP and 1.5g/ L for BMSP are minimum needed for removing completely Al^{3+} from a solution of conc. 75 ppm.

As is evident from the Fig that initially, % removal of Al^{3+} is linearly increased with the increase in conc. of AMSP/BMSP. But after a certain concentration, % removal is reduced and linearity is lost. Further after 1.25g/l for AMSP and 1.50 g/l for BMSP, study states are reached.

Fig.-6: Sorbent Dosage Vs % Removal Al^{3+}(Al^{3+}: 75 ppm; pH:8; Time: 90 min)

**Initial Concentration of Al^{3+}**
The effect of initial conc. of Al^{3+} on the adsorption capacity of AMSP and BMSP was investigated. For this 100 ml of different concentrations: 25 ppm to 200 ppm of Al^{3+} were equilibrated with 0.125 g/100ml of AMSP or 0.150 g/100 ml of BNSP at pH: 8 for 90 minutes. The solutions were filtered. Residual Al^{3+} was estimated. The results are plotted in Fig.-7 and 8.

Fig.- 7: Initial Concentration Vs % Removal
Fig.- 8: Adsorption Capacity Vs Initial Concentration

With the increase in the concentration of Al^{3+}, more Al^{3+} ions are drifted towards the surface of the adsorbents due to diffusion and so, adsorption capacity (in taking capacity), is increased. But for a fixed amount of adsorbent, the active sites are limited and the needed number of sites proportional to the increase in concentration, are not available. Hence, a decrease in % removal.
Temperature Effect

The effect of temperature on the adsorption abilities of BMSP and AMSP towards Al$^{3+}$ was investigated at temperatures: 303, 313 and 323 K. The results were depicted by plotting as ln $K_d$ Vs 1/T, Fig.-9a and b$^{34,35}$

![Fig.-9a: 1/T vs ln $K_d$](image)

![Fig.-9b: 1/T vs ln $K_d$](image)

Thermodynamic parameters: free energy: $\Delta G$, change in enthalpy, $\Delta H$ and change in entropy $\Delta S$, were evaluated employing the equations: $\Delta G = -RT \ln K_d$ ; $\ln K_d = \Delta S/R - \Delta H/RT$ ; $K_d = q_e/C_e$ & $\Delta G = \Delta H - T\Delta S$, where $K_d$ = distribution coefficient, $q_e$ - amount of adsorbed Al$^{3+}$, $C_e$ = Equilibrium conc. of Al$^{3+}$, T = Temp. in K, R = gas constant.$^{36,37}$The values are tabulated in Table-1.

The positive values of $\Delta H$ indicate the endothermic process of sorption. $R^2$ values confirm the same. Positive $\Delta S$ values signify the disorderliness at a solid-liquid interface with a raise in temperature. Negative $\Delta G$ values indicate that the adsorption is spontaneous.

Adsorption Isotherms

The sorption nature of BMSP and AMSP towards Al$^{3+}$ was analyzed using Freundlich$^{38,39}$ and Langmuir isothersms.$^{40,41}$ Linear forms of Freundlich Isotherm : $\log (q_e) = \log k_f + \left(\frac{1}{n}\right) \log C_e$ and Langmuir Isotherm: $C_e/q_e = (a_l/k_l)C_e + 1/k_l$ were used. The relating plots are depicted in Fig.-10 and 11.

![Fig.-10: Freundlich Isotherms](image)

![Fig.-11: Langmuir Isotherms](image)

It can be inferred from the plots that the Correlation coefficient, $R^2$, values are more for Langmuir isothersms: 0.9985 for BMSP and 0.9938 for AMSP than Freundlich isothersms: 0.8868 for BMSP and 0.8605 for AMSP. So Langmuir isothersms describe well the adsorption on both the adsorbents. This indicates homogenous surfaces of BMSP and AMSP and also monolayer adsorption.

![Table-1: Thermodynamic Parameters: for Al$^{3+}$ Ion Adsorption onto BMSP & AMSP](image)
Kinetics of Adsorption Process

Kinetics of adsorption was analysed by various standard models. The equations used are: pseudo first-order: \( \log (q_e - q_t) = \log q_e - k_1 t/2.303 \); pseudo second-order: \( t/q_t = 1/k_2 q_e^2 - 1/q_e \) t \( ^{42,43} \); Weber and Morris intraparticle diffusion: \( q_t = k_{\text{ip}} t^{1/2} + c \); \( ^{39,44} \); Bangham’s pore diffusion: \( \log [\log (C_i/C_i - q_t)] = \log (k_\alpha/2.303V) + \alpha \log(t) \); \( ^{36,39,45} \); Elovich equation: \( q_t = 1/\beta \ln(\alpha \beta) + 1/\beta \ln(t) \). \( ^{36,37,46,47} \) The pertaining values are presented in Table-2.

![Fig.-11: Langmuir Isotherms](image)

Figure-11: Langmuir Isotherms

Table-2: Kinetics of Adsorption: Various Characteristics

| S. No. | Sorbent | Slope | Intercept | R² |
|--------|---------|-------|-----------|----|
| 1      | Pseudo First-order |       |           |    |
| BMSP   | -0.0078 | 0.7053 | 0.9788    |    |
| AMSP   | -0.0102 | 0.7822 | 0.9808    |    |
| 2      | Pseudo Second-order |       |           |    |
| BMSP   | 0.0664  | 0.6734 | 0.9900    |    |
| AMSP   | 0.0389  | 0.2060 | 0.9925    |    |
| 3      | Weber-Mories Intra Particle Diffusion Model |       |           |    |
| BMSP   | 0.3552  | 10.760 | 0.8668    |    |
| AMSP   | 0.3243  | 15.56  | 0.8456    |    |
| 4      | Bangham’s pore Diffusion |       |           |    |
| BMSP   | 0.7001  | -1.3112 | 0.9301    |    |
| AMSP   | 0.6224  | -1.0788 | 0.9623    |    |
| 5      | Elovich Model |       |           |    |
| BMSP   | 1.9760  | 6.3454 | 0.9701    |    |
| AMSP   | 1.4320  | 12.90  | 0.9569    |    |

Among the five models studied, Pseud Pseudo second-order has higher R² values for both the adsorbents: BMSP and AMSP. Hence, Pseudo second-order describes well the kinetics process.

Interfering Ions

Possible interference caused by commonly co-existing cations and anions in water is one of the aspects of the investigation. So in the present study synthetic simulated solutions of 75 ppm of Al³⁺ having two-fold excess of targeted co-ions were prepared. They were subjected to extraction at the optimum condition of pH: 8, time of equilibration: 90 min, sorbent dosage of: 0.125 g/100 ml for AMSP and 0.150 g/100 ml for BMSP, 300 rpm and at 303 K. The results are noted in Fig.-12. It may be seen that anions (except fluoride) and cations have marginally interfered with the extraction procedures developed in this work with AMSP and BMSP.

Regeneration of Spent AMSP AND BMSP

To make the process cost-effective, the spent adsorbents were tried to be regenerated for subsequent use. Various eluents were investigated for this purpose. 0.1 N NaOH solution was found to be effective. The
loss of adsorption ability for the removal Al$^{3+}$ with each successive regeneration was investigated as per the method developed. The results are furnished in Fig.-13.

It may be noted that until three cycles of regenerations, AMSP, as well as BMSP, retained their sorption ability with marginal loss.

![Fig.-12a: Interference of Co-anions](image1)

![Fig.-12b: Interference of Co-Cations](image2)

![Fig.-13: Regeneration and Reuse](image3)

**Comparison with Previous Works**

The merits of BMSP and AMSP are compared with the existing sorbents. A comparative statement is presented in Table-3.

It can be seen from the Table that BMSP and AMSP have more adsorption capacity than many of the reported adsorbents. Further, these are effective in a wide pH range: 3 to 9 and are applicable even in fair acidic solutions besides neutral and faintly basic solutions.

| S. No. | Adsorbent      | pH  | C$_i$ Of Cr(VI) (in ppm) | Adsorption Capacity (% Removal) | References |
|--------|----------------|-----|--------------------------|-------------------------------|------------|
| 1      | Activated carbon | 4-8 | 3                        | 10 mg/g                        | 3          |
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Applications

BMSP and AMSP effectiveness as adsorbents in treating Al\(^{3+}\) contaminated real water samples were investigated. Samples from Al-based industries and polluted lake waters were collected. The amounts of Al\(^{3+}\) present were estimated. In the case of lake waters, only traces of Al\(^{3+}\) was present. Hence, known amounts of Al\(^{3+}\) were added to them. Then the samples were treated with BMSP (0.150 g/100 ml) and AMSP (0.125 g/100 ml) at pH:8 for 90 minutes at 303 K and 300 rpm. The solutions were filtered and the filtrates were estimated for Al\(^{3+}\). The findings were enlisted in Table-4.

Table-4: Applications: Al\(^{3+}\) Removal (pH:8, Sorbent Dosage: 0.150 g/100 ml for BMSP or 0.125 g/100 ml for AMSP; Time:90 min, rpm:300 , Temperature: 303 K)

| Samples                | Ci\(^*\) | Ce\(^*\) | % Removal |
|------------------------|----------|----------|-----------|
| Al-based Industries    |          |          |           |
| 1                      | 9.5 ppm  | Zero     | 100%      |
| 2                      | 12.6 ppm | Zero     | 100%      |
| 3                      | 18.0 ppm | Zero     | 100%      |
| Lake Samples -fed with known Amounts of Al\(^{3+}\) |          |          |           |
| 1                      | 15.0 ppm | Zero     | 100%      |
| 2                      | 20.0 ppm | Zero     | 100%      |
| 3                      | 30.0 ppm | Zero     | 100%      |

Ci\(^*\) and Ce\(^*\) are the initial and final concentration of Cr(VI); The values are average of 3 samples: Standard Deviation: 0.05%

It can be inferred from the data that BMSP and AMSP are very effective in the removal of Al\(^{3+}\).

CONCLUSION

- Sorbents derived from stems of Burea monosperma (BMSP) and Angle marmelos (AMSP) are found to be very effective in the removal of Al\(^{3+}\) from water.
- Extraction conditions are optimized. At pH:8, both the adsorbents (0.150 g/100 ml of BMSP or 0.125 g/100 ml of AMSP), remove completely Al\(^{3+}\) from 100 ml of 75 ppm solution of Al\(^{3+}\) after an equilibration time of 90 minutes at room temp:303 K and 300 rpm.
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- Adsorption capacities are as high as: 50.0 mg/g for BMSP and 62.0 mg/g for AMSP.
- Good extractions are observed in a wide range of pH:3 to 9 with a maximum at pH: 8 for both the sorbents. This facilitates the applications of these sorbents even for acidic waters beside their applications for neutral and slightly basic solutions.
- Interference studies reveal the common co-ions interfere, is marginal.
- Thermodynamic parameters are evaluated and they emphasize the spontaneity of sorption of Al\(^{3+}\) onto BMSP and AMSP.
- The application of adsorption isotherms supports Langmuir's mode of adsorption.
- Of the various kinetic models applied for adsorption, pseudo-second-order fits well.
- Spent BMSP, as well as AMSP, can be regenerated and reused until three cycles with marginal loss of sorption capacities.
- BMSP and AMSP are successfully applied to remove completely Al\(^{3+}\) from the samples of industrial effluents as well as polluted lakes.

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