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 marmelos* 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

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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 Numerous industries based on Aluminum are the potential sources of contamination². 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 ppm³. Al intake is hazardous to human³⁻⁶ as well as to aquatic life⁷⁻¹⁰. 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 $\text{Al}(\text{OH})_3$ 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 Carbon⁸, Granular activated carbon²¹, Amberlite IR-120H²¹, activated carbon- with oleic acid as surfactant²², Chitosan²³ and leaves, stems and their ashes of *Acacia Melanoxylon* and *Eichhornia Crassipes*²⁴, are investigated for Al-removal. Beach cast seaweed²⁵ 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

extraction conditions for the removal of Al^{3+} using the adsorbents derived from these materials.

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.

Butea monosperma tree

Aegle 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 *Aegle 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 oven 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 *Aegle 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 flask 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

magnitude of the targeted character but keeping constant the other parameters. The findings are depicted in Fig.-2 to 11 and Tables-1 and 2.

After establishing extraction conditions at optimum values, interference of two-fold excess of co-ions was investigated using simulated solutions. Regeneration as well as applications were also investigated. Results are noted in Fig.-12 and 13.

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.

Fig.-2: BMSP: Time Vs % Removal of Al^{3+} (Al^{3+} Conc.: 75 ppm; BMSP :0.150g/100 ml)

Fig.-3: AMSP: Time Vs Removal of Al^{3+} (Al^{3+} Conc. : 75 ppm; AMSP: 0.125 g/100 ml)

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 steady 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 Al^{3+} were removed from 100 ml of 75 ppm 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, Al^{3+} . So, the rate of adsorption of 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 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. pH_{zpc} was evaluated from the Fig.-5.

Fig.-4: pH Vs % Al(III) Removal
(Al^{3+} Conc. : 75 ppm; BMSP: 0.15 g/100 ml or
AMSP: 0.125 g/100 ml)

Fig.-5: pH_{zpc} Evaluation for BMSP and AMSP

It is interesting to note that substantial amounts of 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.

pH_{zpc} 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 pH_{zpc}, the dissociation is less probable; even protonation occurs at low pHs.

At low pH values, Al (III) exists as $\text{Al}[(\text{H}_2\text{O})_6]^{3+}$. With raise in pH, $\text{Al}(\text{OH})^{2+}$ and $\text{Al}(\text{OH})_2^+$ are increasingly formed³³. In neutral conditions amorphous $\text{Al}(\text{OH})_3$ is nucleated. In basic conditions, the aggregates dissolve to form $\text{Al}(\text{OH})_4^-$. Between pH: 6 to 8, hydrated $\text{Al}(\text{OH})_3$ nucleates are not precipitated as their formation from $\text{Al}(\text{OH})_2^+$ is inhibited.³³

The good affinity of Al^{3+} at low pHs, indicates binding between Al-species and functional groups of sorbents: -OH/COOH, which are not dissociated as is reflected from pH_{zpc} 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 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 BMSP 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}

Thermodynamic parameters: free energy: ΔG ; change in enthalpy, ΔH and change in entropy ΔS , 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 ΔH indicate the endothermic process of sorption. R^2 values confirm the same. Positive ΔS values signify the disorderliness at a solid-liquid interface with a raise in temperature. Negative ΔG 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 isotherms.^{40,41} Linear forms of Freundlich Isotherm: $\log(q_e) = \log k_f + (\frac{1}{n}) \log C_e$ 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

It can be inferred from the plots that the Correlation coefficient, R^2 , values are more for Langmuir isotherms: 0.9985 for BMSP and 0.9938 for AMSP than Freundlich isotherms: 0.8868 for BMSP and 0.8605 for AMSP. So Langmuir isotherms 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

	ΔH , kJ/mol	ΔS , J/mol/K	ΔG , kJ/mol			R^2
Temp. (K)			303	313	323	
BMSP	59.19	206.9	-3.713	-4.859	-7.875	0.974
AMSP	12.10	41.90	-0.606	-1.027	-1.448	0.958

Kinetics of Adsorption Process

Kinetics of adsorption was analysed by various standard models. The equations used are: pseudo first-order: $\log(q_e - q_t) = \log q_e - k_1 t / 2.303$ ^{41,42}; pseudo second-order: $t/q_t = 1/k_2 q_e^2 - (1/q_e) t$ ^{42,43}; Weber and Morris intraparticle diffusion: $q_t = k_{ip} t^{1/2} + c$ ^{39,44}; Bangham's pore diffusion: $\log[\log(C_i/C_i - q_{tm})] = \log(k_o/2.303V) + \alpha \log(t)$ ^{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

Table-2: Kinetics of Adsorption: Various Characteristics

S. No.	Sorbent	Slope	Intercept	R ²
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, 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

Fig.-12b: Interference of Co-Cations

Fig.-13: Regeneration and Reuse

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.

Table-3: Adsorption Capacities Comparison

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

2	Modified activated carbon as a sorbent and oleic acid as surfactant	7	1.35	100% removal	22
3	Chitosan	4	5–40	49.0 mg/g	23
4	<i>Acacia Melanoxylon</i> and <i>Eichhornia Crassipes</i>	6	50	20 mg/g (stems) and 25 mg/g (stems)	24
5	<i>Moryngea Millingtonia</i> Leaves and their ash	6	50	16.7 mg/g for leaves 20.0mg/g for ashes	28
	<i>Cygium Arjunum:</i> Barks and their ash	6	50	14.8 for barks and 20.5 for their ash	
6	<i>Ficus Racemosa</i> Plants	6	50	25.0 mg/g	26
7	<i>Cassia Occidentalis</i>	6-8	50	26.0 mg/g	27
8	Refused beach cast seaweed	4	10 to 100	22.5 mg g ⁻¹	25
9	Granular activated carbon and Amberlite IR-120H	5	100	99.2 and 99.55%	21
9	BMSP	3-9 Maximus at:8	75	100% Removal 50 mg/g	PRESENT WOK
10	AMSP	3-9 Maximus at:8	75	100% Removal 62.0 mg/g	

C_i = initial concentration of Chromium (VI)

Applications

BMSP and AMSP effectiveness as adsorbents in treating Al³⁺ contaminated real water samples were investigated. Samples from Al-based industries and polluted lake waters were collected. The amounts of Al³⁺ present were estimated. In the case of lake waters, only traces of Al³⁺ was present. Hence, known amounts of Al³⁺ 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 Al³⁺. The findings were enlisted in Table-4.

Table-4: Applications: Al³⁺ Removal
(pH:8, Sorbent Dosage: 0.125 g/100 ml for AMSP and 0.150 g/100 ml for BMSP; Time:90 min, rpm:300, Temperature: 303 K)

Samples	C _i *	C _e *	% 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%

C_i* and C_e* 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 Al³⁺.

CONCLUSION

- Sorbents derived from stems of *Burea monosperma* (BMSP) and *Angle marmelos* (AMSP) are found to be very effective in the removal of Al³⁺ 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 Al³⁺ from 100 ml of 75 ppm solution of Al³⁺ after an equilibration time of 90 minutes at room temp:303 K and 300 rpm.

- 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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