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## Removal of aluminium (III) from waste waters using bio-sorbents pertaining to *Withania somnifera* plant

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### ABSTRACT

Removal of Aluminum (III) from waste waters using bio-adsorbents derived from plant materials of *Withania somnifera* has been investigated by varying various physicochemical parameters such as pH, time of equilibration, sorbent dosage, agitation time, initial concentration of Aluminum ions, temperature and presence of foreign ions using simulated waters and by adopting batch methods of extraction. Optimum conditions for the maximum extraction of Al (III) ions have been investigated. The adsorption process is analyzed with Freundlich, Langmuir, Temkin and Dubinin-Radushkevich (D-R) isotherm models and found that the Langmuir isotherm model better describes the adsorption process emphasizing the mono-layer formation of the Al (III) ions on the adsorbent and further, the mean free energy (E) and heats of sorption (B) of Temkin isotherm and Dubinin-Radushkevich isotherms indicate that the adsorption is 'physisorption' in nature. Kinetics of adsorption is quantified using pseudo first-order, pseudo second-order, Weber and Morris intraparticle diffusion, Bangham's pore diffusion and Elovich equations and found that the adsorption process has good correlation coefficient values with pseudo-second-order model. The endothermic nature of the adsorption is found on the analysis of the thermodynamic parameters,  $\Delta H$ ,  $\Delta S$  and  $\Delta G$ . Interference of common co-ions has been studied. The methodologies developed are successfully applied to industrial sewages and polluted natural waters.

**Key words:** Aluminum (III), pollution control, bio-sorbents, kinetics, adsorption isotherms, *Withania somnifera*

### INTRODUCTION

Exploring the adsorption abilities of bio-sorbents derived from plant materials towards different pollutants in the removal of the latter from waste or polluted waters is being increasingly investigated throughout the Globe. Our research group has been investigating on these bio-methods of pollution and successfully developed methods for the removal of Chromium (VI) (1-4), Zinc (5,6), Aluminium (III) (7-10), Fluoride (11-17), Nitrite (18,19), Nitrates (20), Ammonia(21-23), Phosphate (24,25) and Dyes (26-29). Further, bio-remediation methods are developed for the control of metal ions using oxidation ponds and different bio-masses (30). In the present work, bio-sorbents derived from plant material of *Withania somnifera* plant have been investigated for their effectiveness in the removal of toxic Aluminium (III) ions from polluted waters.

The presence of Aluminum (III) in water-bodies is harmful to aquatic life and it a neurotoxin (31) and its consumption by human beings, results in serious disorders such as kidney disorders, *Parkinson and Alzheimer's disease* (32), *mellowing of bones* (33), *affecting the eye sight, pulmonary fibrosis and microcytic anemia* (34). Hence, as per WHO and US drinking waters standards, the maximum permissible limit of Aluminium (III) is: 0.2 ppm. Inadequately treated effluents from industries pertaining to food and beverages, dyeing, textile and drugs and residual aluminum in treated municipal waters, are the main source of contamination. Further, nearly 7% of Aluminium present in the earth's crust is leached into the nearby water bodies under acidic conditions and it becomes detrimental to the biota, micro-organisms and human beings.

The methods based on aeration/stripping, sedimentation and filtration are ineffective at micro levels and methods based on ion-exchange, reverse osmosis Electro dialysis are expensive (35-38). The non-conventional methods based on bio-adsorbents are being used in removing Aluminium ions from polluted waters. Algal biomass (39), powdered marble wastes (40), combination of active carbon as adsorbent and oleic acid as surfactant (41), water hyacinth (42), Chitosan (43), rice husk char and activated rice husk char (35,44) have been investigated for the removal of Aluminium ions from waste waters. Bio-adsorbents derived from *Acacia Melanoxylon* and *Eichhornia Crassipes* Plants (7), *Moryngea Millingtonia* and *Cygium Arjunum Plant* (9), *Achiranthus aspera* and *Cassia Occidentalis* (10), have been successfully used to remove Aluminium (III) from polluted waters.

In the present work, bio-adsorbent derived from leaves and stems of *Withania somnifera* plant have been investigated by varying various physicochemical parameters to optimize the conditions of extraction for the maximum removal of Aluminium ions from polluted waters.

## MATERIALS AND METHODS

Bi-adsorbent derived from the leaves and stems of *Withania somnifera* plant (Figure 1) are found to have affinity for Aluminium (III) ions. *Withania somnifera* (ashwagandha) is an herb in the Solanaceae family and it grows in wet lands. It has many medicinal values and this plants parts are used in the preparation of some Ayurvedic medicines in the cure of tumors, tubercular glands, carbuncles, and ulcers.



Figure 1: *Withania somnifera* plant

Adsorbents are prepared from the leaves and stems of *Withania somnifera* plant and are used in the extraction of Aluminium ions from polluted waters using batch methods of extraction (47-49).

**Preparation of the Bi-adsorbents:** The leaves and stems of *Withania somnifera* plant were cut, washed with tap water followed by distilled water and then sun dried. The dried materials were powdered to a fine mesh of size: < than 75 microns and activated at 105<sup>o</sup> C in an oven and then employed in this study. Further, these leaves and stems were burnt to ashes and these ashes were also used in this work.

**Extraction Method:** Weighted quantities of adsorbents were taken in to previously washed 1 lit/500 ml stopper bottles containing 500ml/250 ml of Aluminum Potassium Sulphate solution of predetermined concentrations. The various initial pH values of the suspensions were adjusted with dil HCl or dil NaOH solution using pH meter. The samples were shaken vigorously in mechanical shakers and were allowed to be in equilibrium for the desired time. After the equilibration period, an aliquot of the sample was taken for Aluminum determination. Aluminum (III) was determined spectrophotometrically by using "Eriochrome cyanine R" method (50). In this work all chemical used were of analytical grade and double distilled water was used for the preparation of solution.

The sorption nature of the bio-adsorbents were studied by varying physicochemical parameters such as pH, time of equilibration, initial concentration of the adsorbate, adsorbent dosage and temperature. The results were presented in the Graph No. 1 to 13 and Table 1-3.

**Effect of Co-ions:** The presence of tenfold excess of common co-ions on the % of extraction of the Aluminium ion was studied at other optimum conditions of extraction as inferred from the Graph No. 1-3 and the results were presented in the Table: 4.

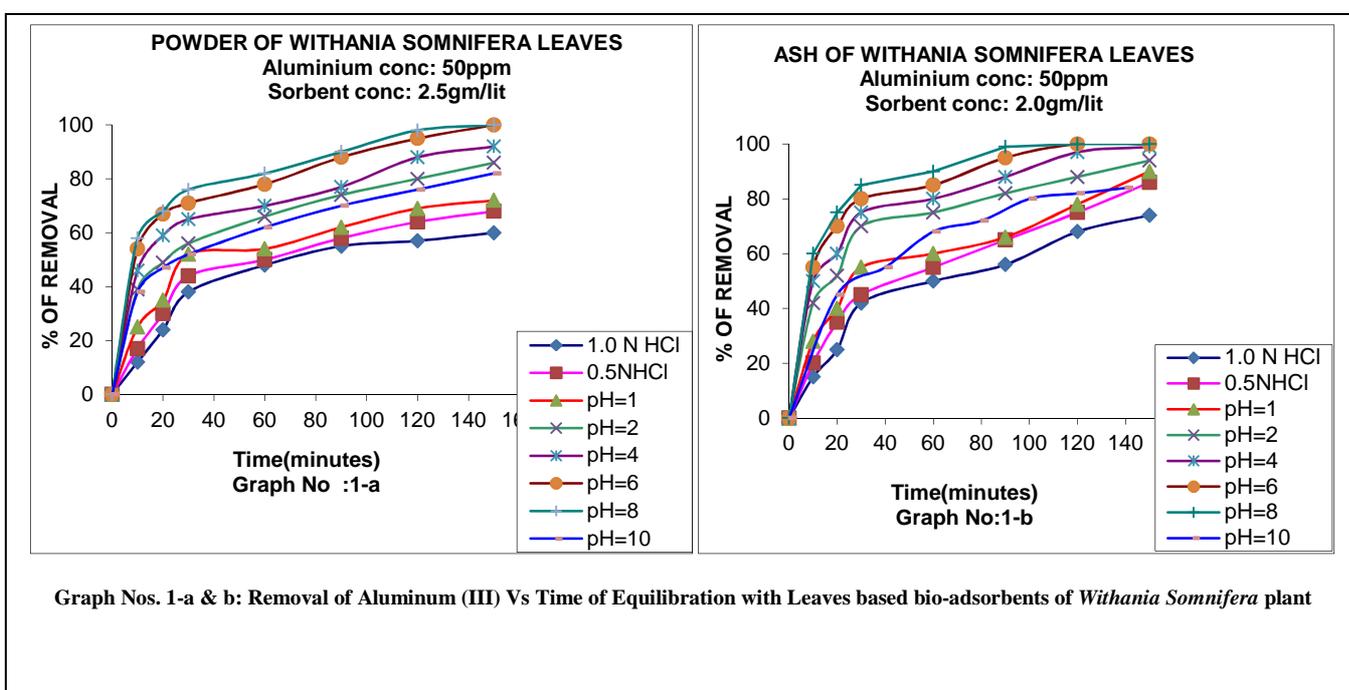
**E: APPLICATIONS:** The procedures developed for the extraction of Aluminium ions using synthetic simulated solutions, were applied to samples collected from the effluents of Aluminum based industries and the results were presented in the Table 5.

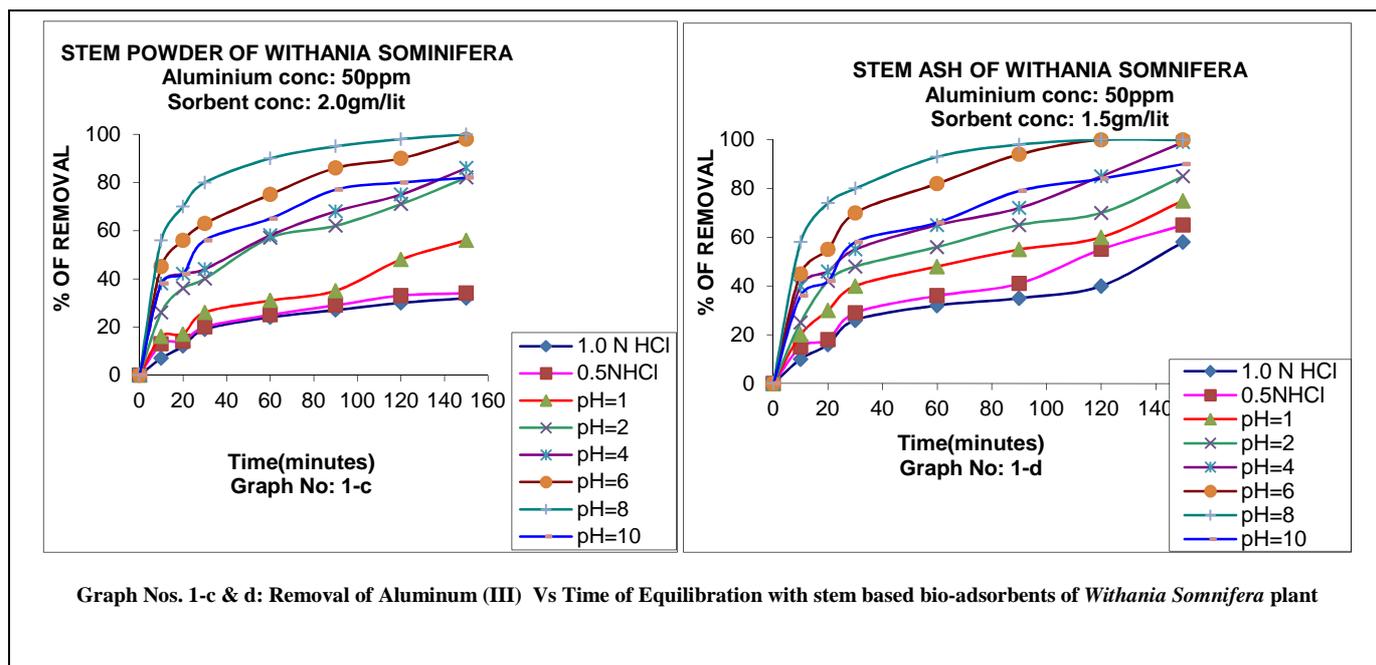
### RESULTS AND DISCUSSION

It is observed that the % of extraction is affected by pH, time of equilibration, sorbent dosage, temp., and initial concentration of the adsorbate.

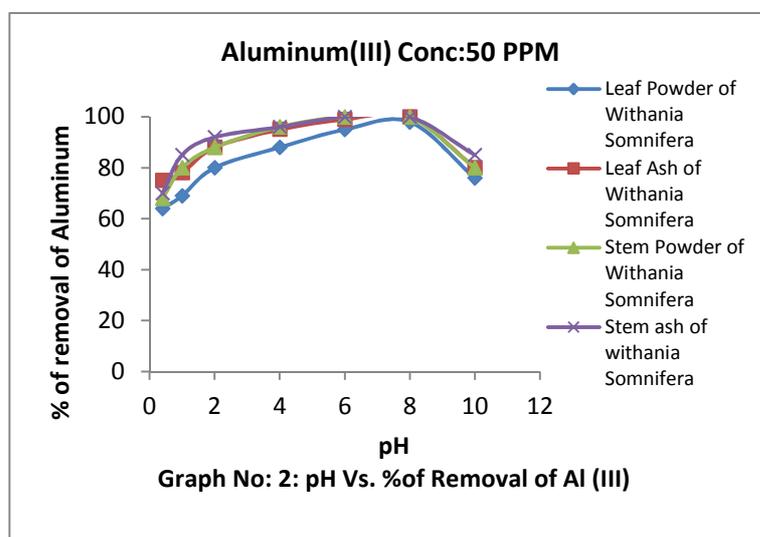
#### A: Effect of Time of Equilibration:

With the increase in time of equilibration, % removal of Aluminium ions also increases and after certain time, no more adsorption is found (vide Graph Nos. A: 1-a to 1-d). For instance, at other optimum conditions of extraction, % removal is found to be: 58% at 10 minutes, 68 % at 20 minutes, 76% at 30 minutes, 82% at 60 minutes, 90% at 90 minutes, 98% at 120 minutes and 100% at 150 minutes or above with the bio-adsorbents pertaining to leaves powders (vide Graph No.1-a). The optimum time for agitation is found to be 150 minutes for bio-adsorbent pertaining to the leaves and stems at optimum pH: 8 while it is 120 minutes with their ashes. (vide Graph No: 1)

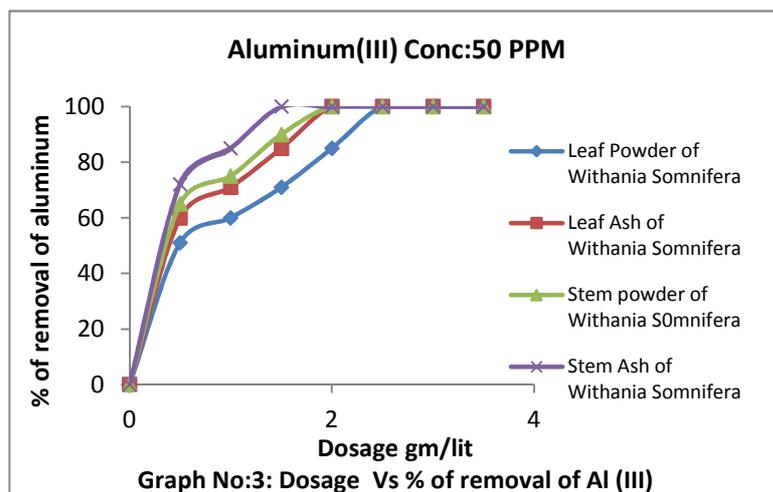




**B: Effect of pH:** The optimum pH is found to be between 6 to 8 (Vide Graph: 2). 100% of removal of Aluminium is observed with all the bio-adsorbents developed in this work when all other conditions of extractions are optimized namely: equilibration time : 150 minutes and sorption dosage: 2.5gm/lit for leaves powders; equilibration time of 120 minutes and sorption dosage: 2.0 gm/lit for .leaves ashes; equilibration time : 150minutes and sorbent dosage:2.0gm/lit for stems powder and equilibration time of 120 minutes and sorbent dosage :1.5gm/lit for stem ashes.



**C: Sorbent Dosage:** The optimum sorbent dosage is found to be 2.5 gram/lit for the leaves bio-adsorbent and 2.0 gm/lit with its ashes. With stems bio-adsorbent, the dosage is found to be: 2.0gm/lit and 1.5 gm/lit. with their ashes (vide Graph No : 3:)



#### D: Effect of Temperature:

The influence of temperature on the extraction of Aluminum was investigated at 303, 313 and 323K with simulated 5.0 mg/l Aluminum solutions and at other optimum conditions of extractions. The observations are presented as  $\ln K_d$  Vs  $1/T$  as shown in Graph No.4-a to 4-d. Thermodynamic parameters free energy ( $\Delta G$ ) (kJ/mole), change in enthalpy ( $\Delta H$ ) (kJ/mole) and change in entropy ( $\Delta S$ ) (J/K/mole) were determined at different temperatures by using the equations [51-53],  $\Delta G = -RT \ln K_d$ ;  $\ln K_d = \Delta S/R - \Delta H/RT$ ;  $K_d = q_e/C_e$  and  $\Delta G = \Delta H - T\Delta S$ , where  $K_d$  is the distribution coefficient for the adsorption,  $q_e$  is the amount of Aluminum ion adsorbed on the adsorbent per liter of solution at equilibrium,  $C_e$  is the equilibrium concentration of Aluminum(III) ion solution,  $T$  is the absolute temperature in Kelvin,  $R$  is the gas constant. The values of  $\Delta H$  and  $\Delta S$  were obtained from the slope and intercept of a plot between  $\ln K_d$  and  $1/T$  and  $\Delta G$  values were obtained from the equation  $\Delta G = \Delta H - T\Delta S$  and tabulated (vide Table 1) [54, 55].

**Table 1: Thermodynamic parameters of Aluminum (III) ion adsorption on Bio-adsorbents**

Parameter	$\Delta H(\text{kJ/mol})$	$\Delta S(\text{J/mol/K})$	$\Delta G (\text{kJ/mol})$			$R^2$
			303	313	323	
Leaf Powder	11.88	40.273	-0.3227	-0.7251	-1.1281	0.9888
Leaf ash	12.22	42.509	-0.65922	-1.0843	-1.5094	0.9771
Stem Powder	45.169	158.04	-2.7171	-4.2975	-5.8779	0.9772
Stem ash	67.459	234.122	-3.480	-5.8211	-8.1624	0.9805

As the temperature increases from 303 to 323K (30 to 50°C), the % extraction Aluminum(III) ion increases from 71.5 to 75.5% in the case leaves bio-sorbent; 80.0 to 86.5% with ashes of leaves; 74.5 to 79.5% with stems bio-sorbent; 83.0 to 88.5% with stems ash. With the increase in temperature, the diffusion of  $\text{Al}^{3+}$  into the surface layers of the adsorbent is more facilitated due to the increase in K.E. of the ions and decrease in the density of outer surface of the adsorbent.

From the Table 1, it can be inferred that the adsorption process is:

- endothermic in nature as the  $R^2$  is near to "one" and  $\Delta H$  is positive
- physisorption as the  $\Delta H$  is positive [56]
- increased disorder and randomness at the solid solution interface as the  $\Delta S$  is positive [57].
- spontaneous nature as  $\Delta G$  is negative [58].

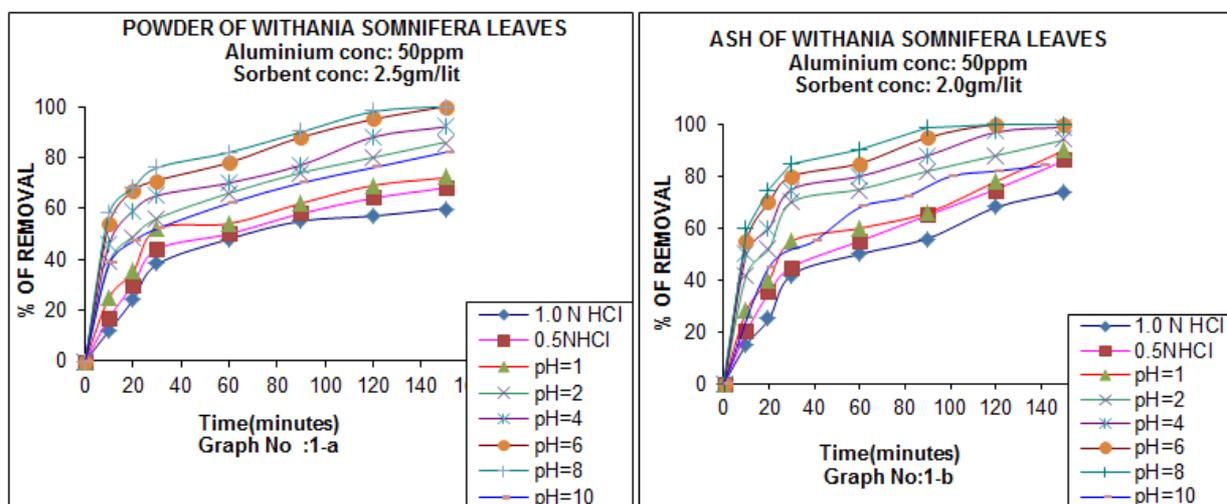
#### E: Adsorption Isotherms:

Freundlich [59], Langmuir [60], Temkin [61] and Dubinin-Radushkevich [62] isotherms are used to evaluate the nature of adsorption.

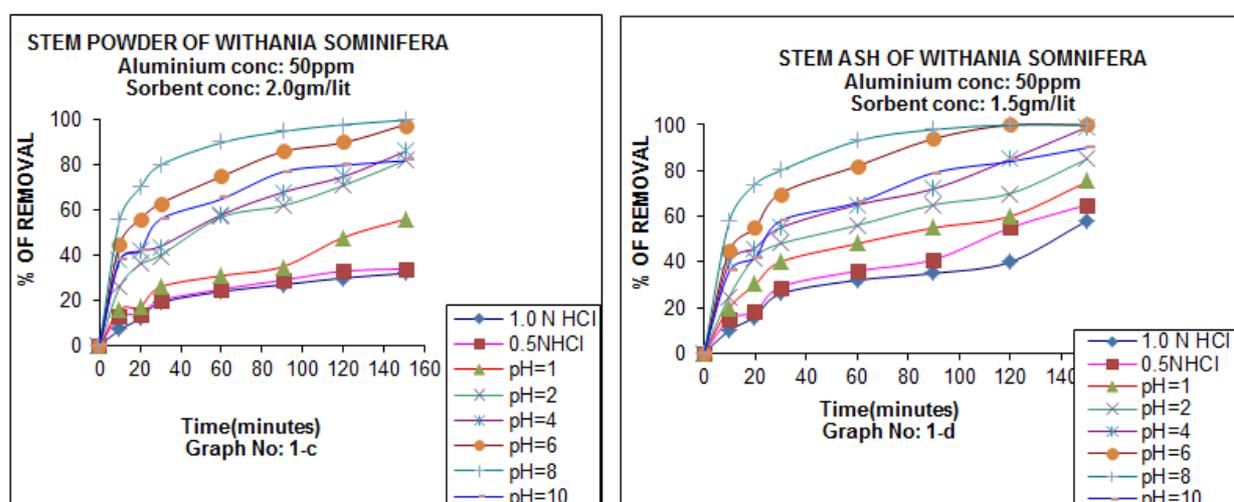
Linear form of Freundlich equation is  $\log(q_e) = \log k_f + \left(\frac{1}{n}\right) \log C_e$  and Langmuir equation is  $C_e/q_e = (a_L/k_L)C_e + 1/k_L$ . Significant feature of the Langmuir isotherm model is the dimensionless separation factor,  $R_L = 1/(1 + a_L C_i)$ . According to Hall et al [63], the nature of the adsorption process is unfavorable if  $R_L > 1$ , linear if  $R_L = 1$ , favourable if:  $0 < R_L < 1$  and irreversible if:  $R_L = 0$ .

The linear results are presented in Graph. No :5-a to d & 6-a to d and isothermal constants along with the correlation coefficient values are presented in Table 2-A.

The high correlation coefficient ( $R^2=0.9985, 0.9938, 0.9689$  and  $0.9983$ ) values than Freundlich isotherm and dimensionless separation factor ( $R_L= 0.01594, 0.0142, 0.0212$  and  $0.01174$ ) values in the range of 0-1, suggest that the favorable model for the adsorption process is Langmuir isotherm, and hence indicates the mono layer coverage of the adsorbent surface with the adsorbate is favorable.



Graph Nos. 1-a & b: Removal of Aluminum (III) Vs Time of Equilibration with Leaves based bio-adsorbents of *Withania Somnifera* plant

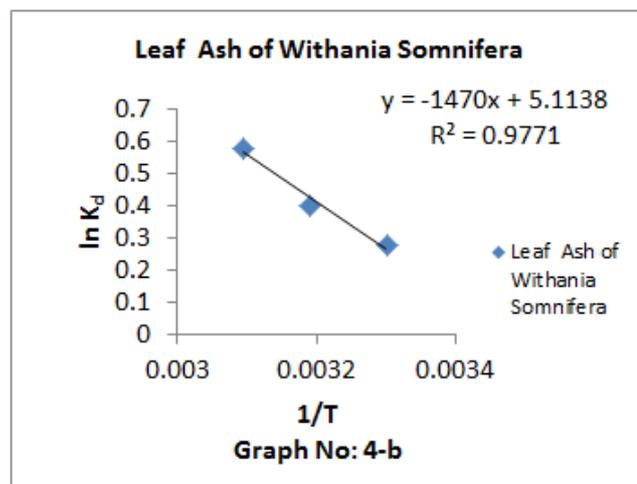
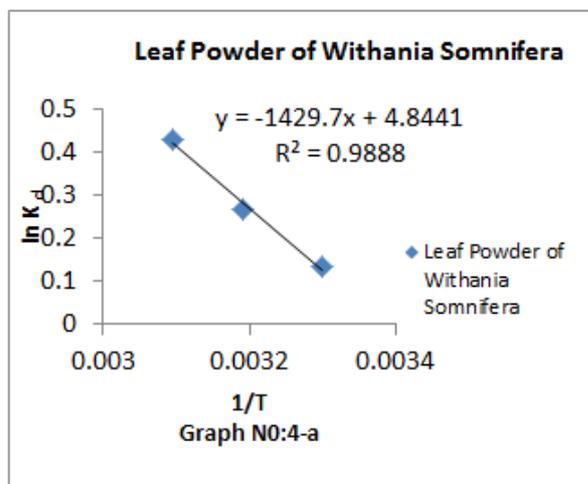
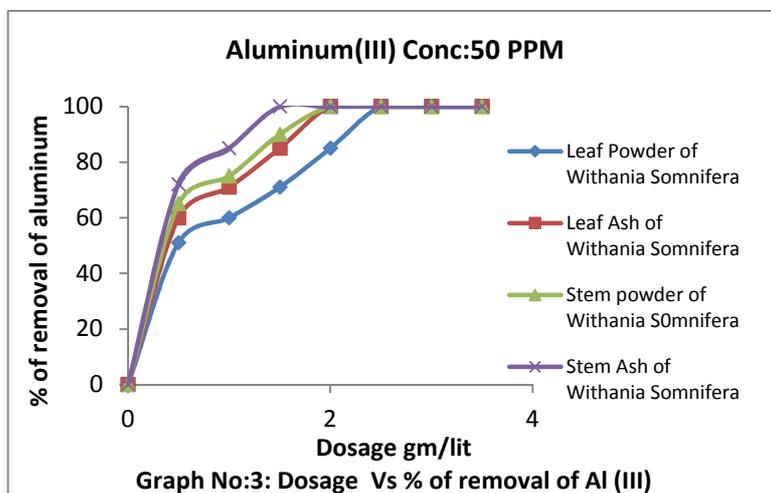
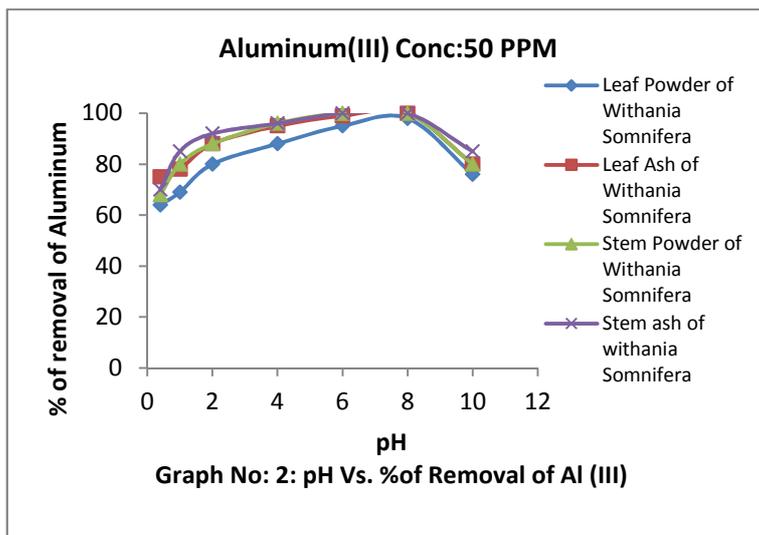


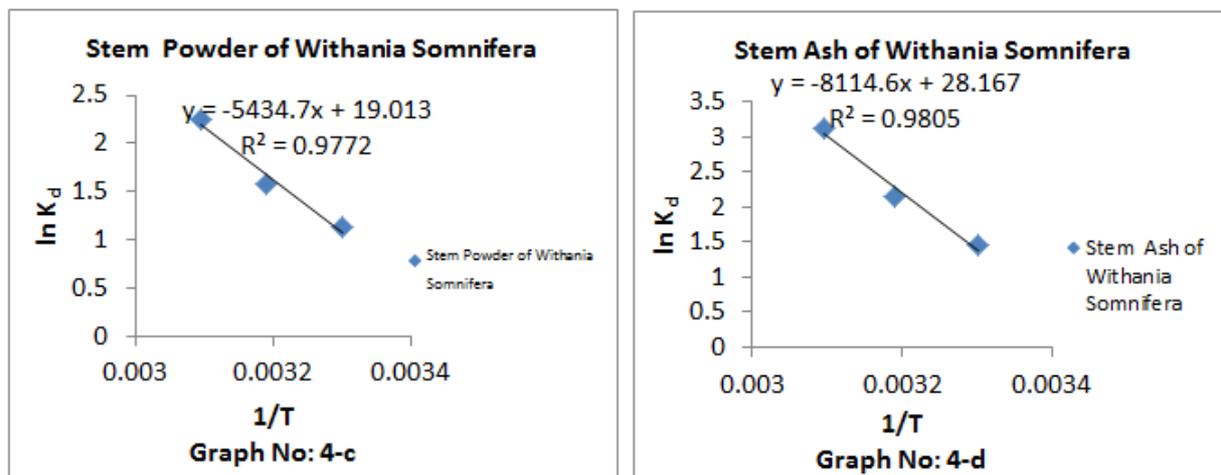
Graph Nos. 1-c & d: Removal of Aluminum (III) Vs Time of Equilibration with stem based bio-adsorbents of *Withania Somnifera* plant

Table 2-A: Adsorption isothermal parameters of Freundlich and Langmuir plots on different bio-adsorbents of *Withania somnifera* plant

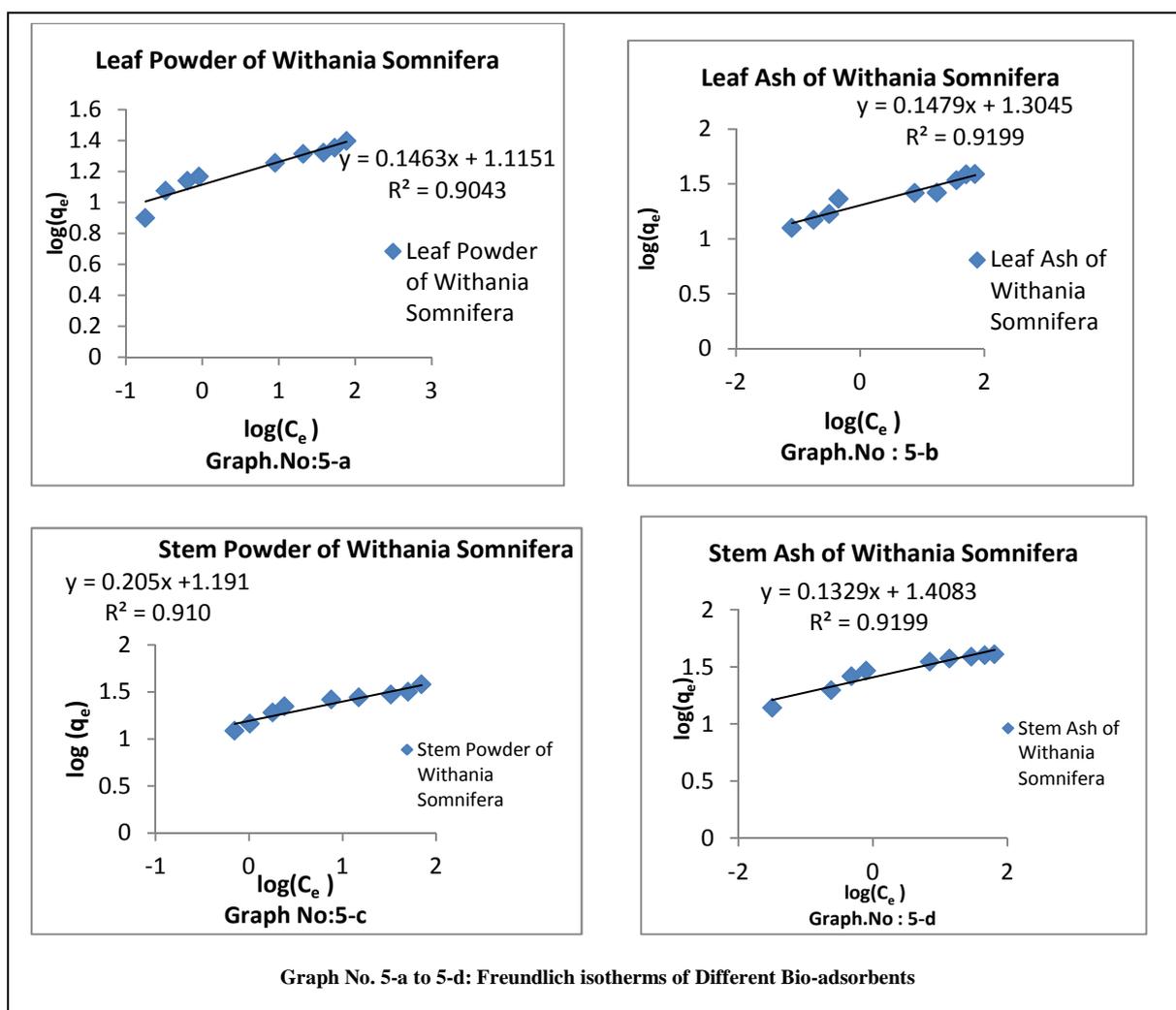
S.No:	Adsorption isotherms	Bio-sorbent		Slope	Intercept	R <sup>2</sup>
1	Freundlich isotherms	Leaf Powder		0.223	0.9485	0.8868
		Leaf ash		0.2033	1.0761	0.8605
		Stem Powder		0.2123	1.0558	0.8591
		Stem ash		0.1453	1.2408	0.9087
			<b>R<sub>L</sub></b>	<b>Slope</b>	<b>Intercept</b>	<b>R<sup>2</sup></b>
2	Langmuir isotherms	Leaf Powder	0.01594	0.0545	0.0341	0.9985
		Leaf ash	0.0142	0.0463	0.0259	0.9938
		Stem Powder	0.0212	0.0456	-0.0315	0.9689
		Stem ash	0.01174	0.0352	0.0219	0.9983

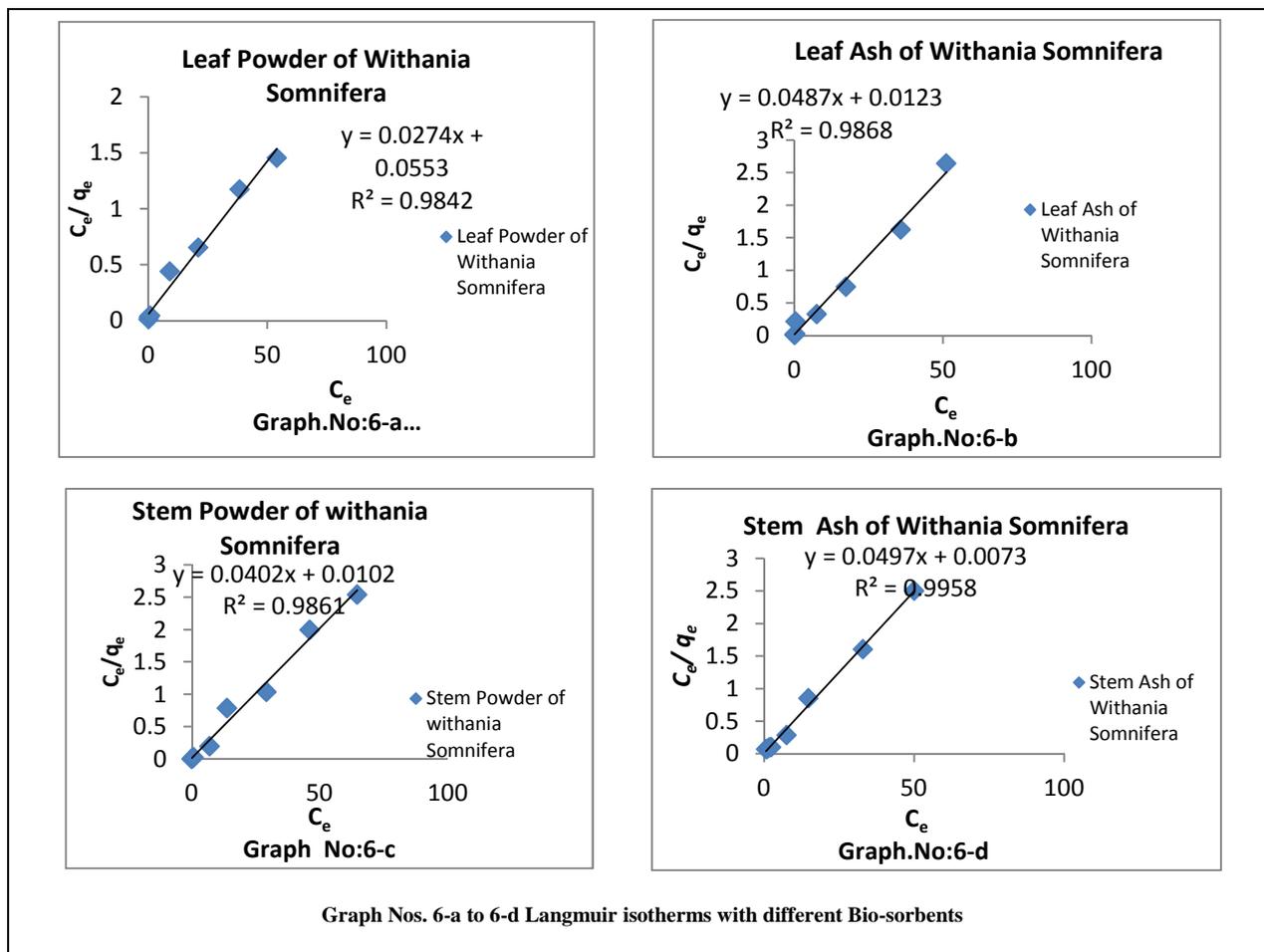
Further, the adsorption process is modeled with Linear form of Temkin equation:  $q_e = B \ln C_e + B \ln A$  where  $RT/b = B$ ; Linear form of Dubinin-Radushkevich equation :  $\ln q_e = -\beta \varepsilon^2 + \ln q_m$ , where  $\varepsilon = RT \ln(1+1/C_e)$ . The linear plots of these two adsorption isotherms are depicted in Graph.No :7-a to d & 8-a to d and isothermal constants along with the correlation coefficient values are presented in Table 2-B.



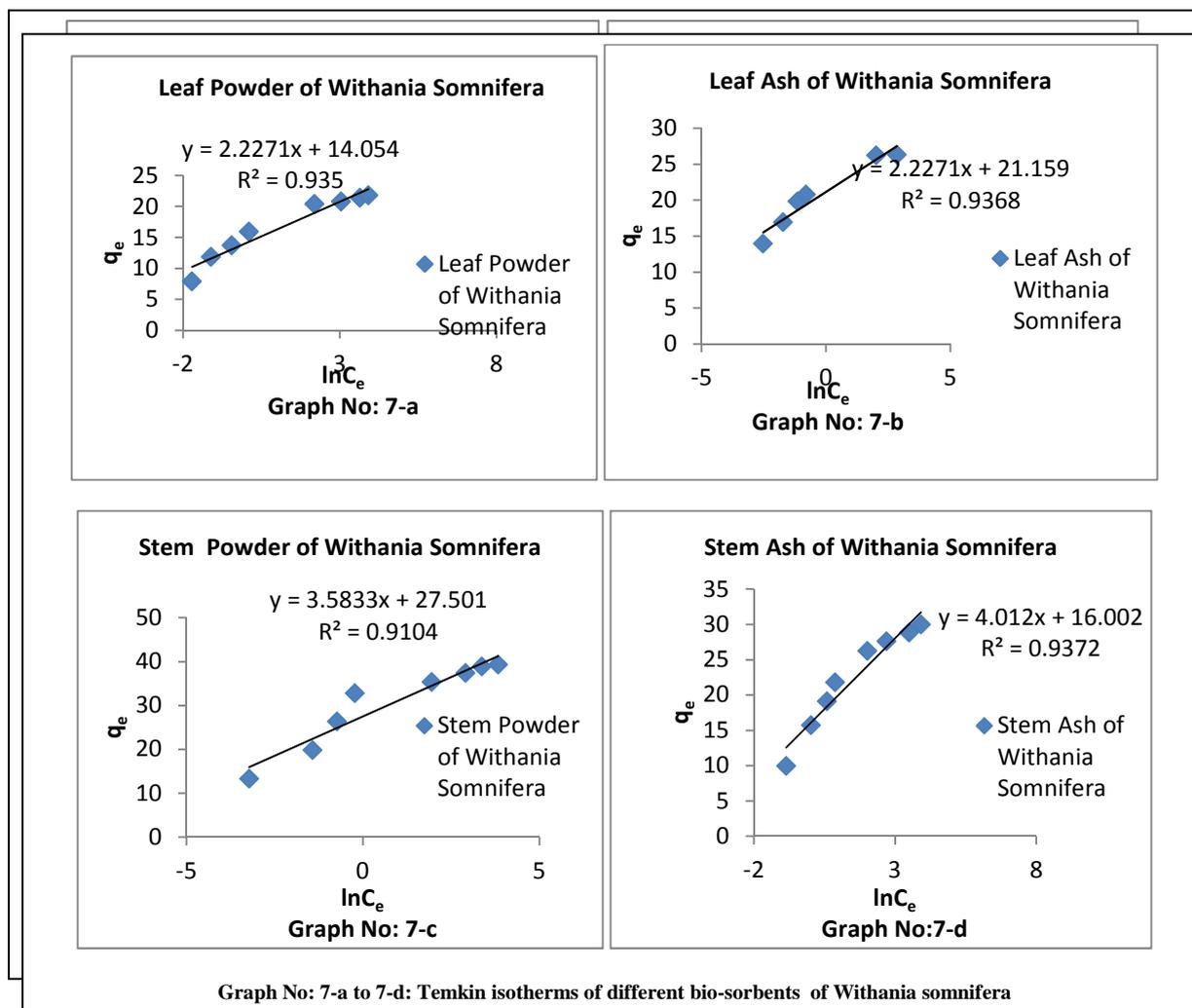


Graph Nos 4-a to 4-d: Effect of temperature on % removal of Aluminium(III) ion with different Sorbents





1	Temkin Isotherm	B	Slope	Intercept	R <sup>2</sup>
	Leaf Powder	2.271J/mol	2.271	14.05	0.935
	Leaf ash	2.2271J/mol	2.2271	21.159	0.9368
	Stem Powder	3.5833J/mol	3.5833	27.501	0.9104
	Stem ash	4.012J/mol	4.012	16.002	0.9372
2	Dubinin-Radushkevich isotherm	E	Slope	Intercept	R <sup>2</sup>
	Leaf Powder	5.0KJ/mol	-2E-08	-6.6101	0.9595
	Leaf ash	1.2909KJ/mol	-3E-07	-6.8447	0.9679
	Stem Powder	5.0KJ/mol	-2E-08	-6.8702	0.9676
	Stem Ash	3.162KJ/mol	-5E-08	-7.1041	0.9755

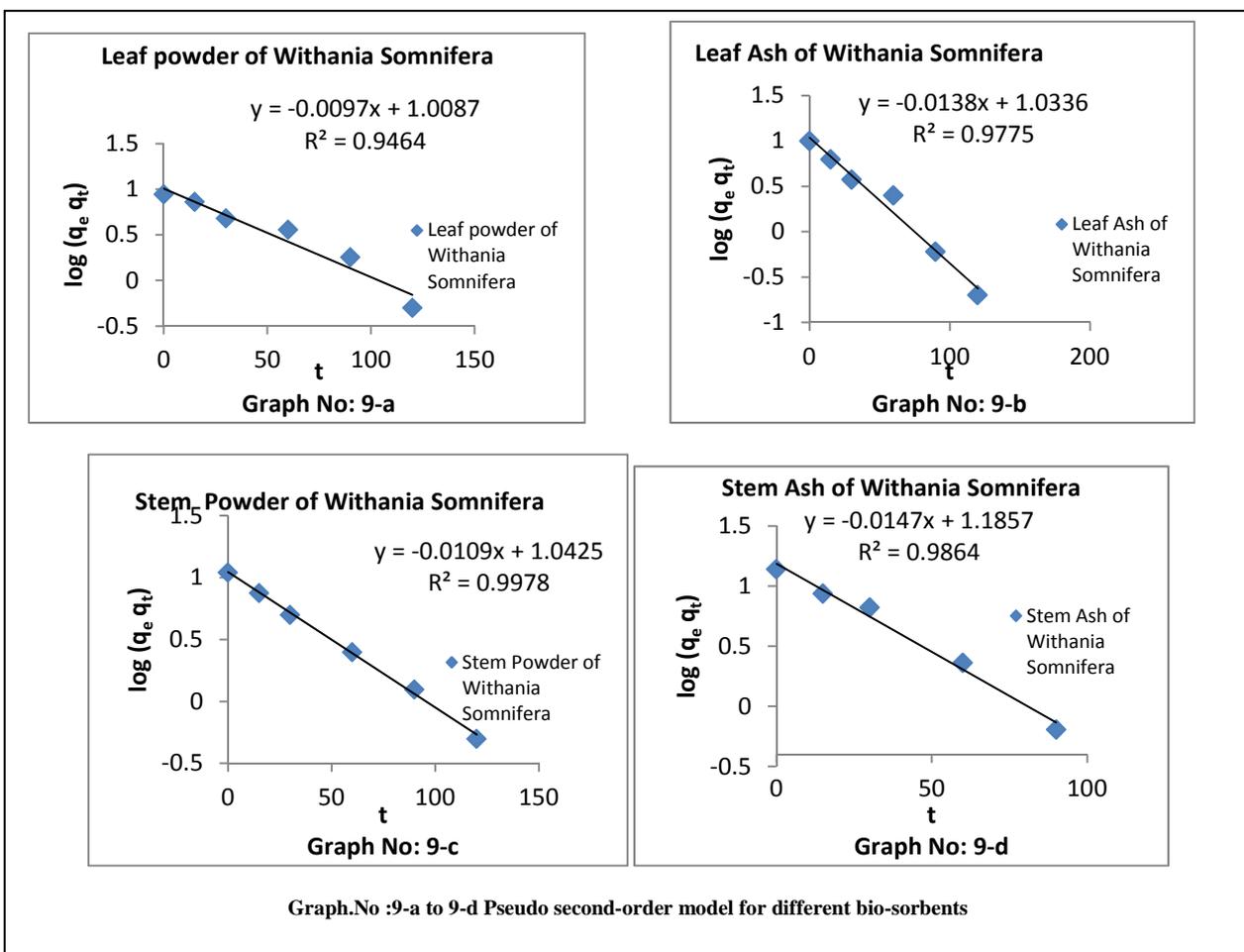
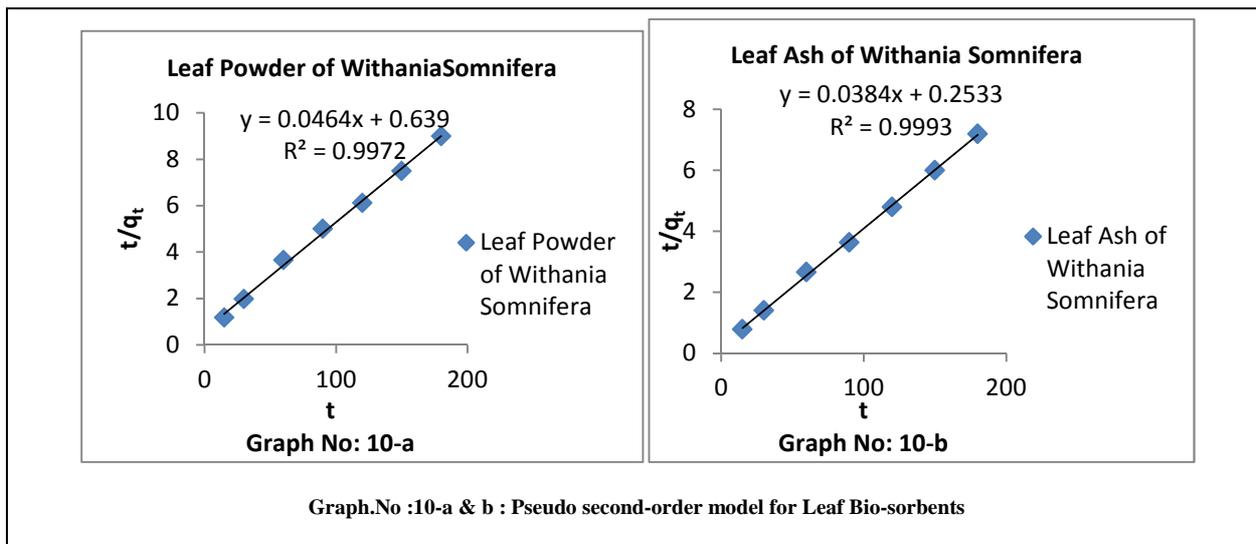


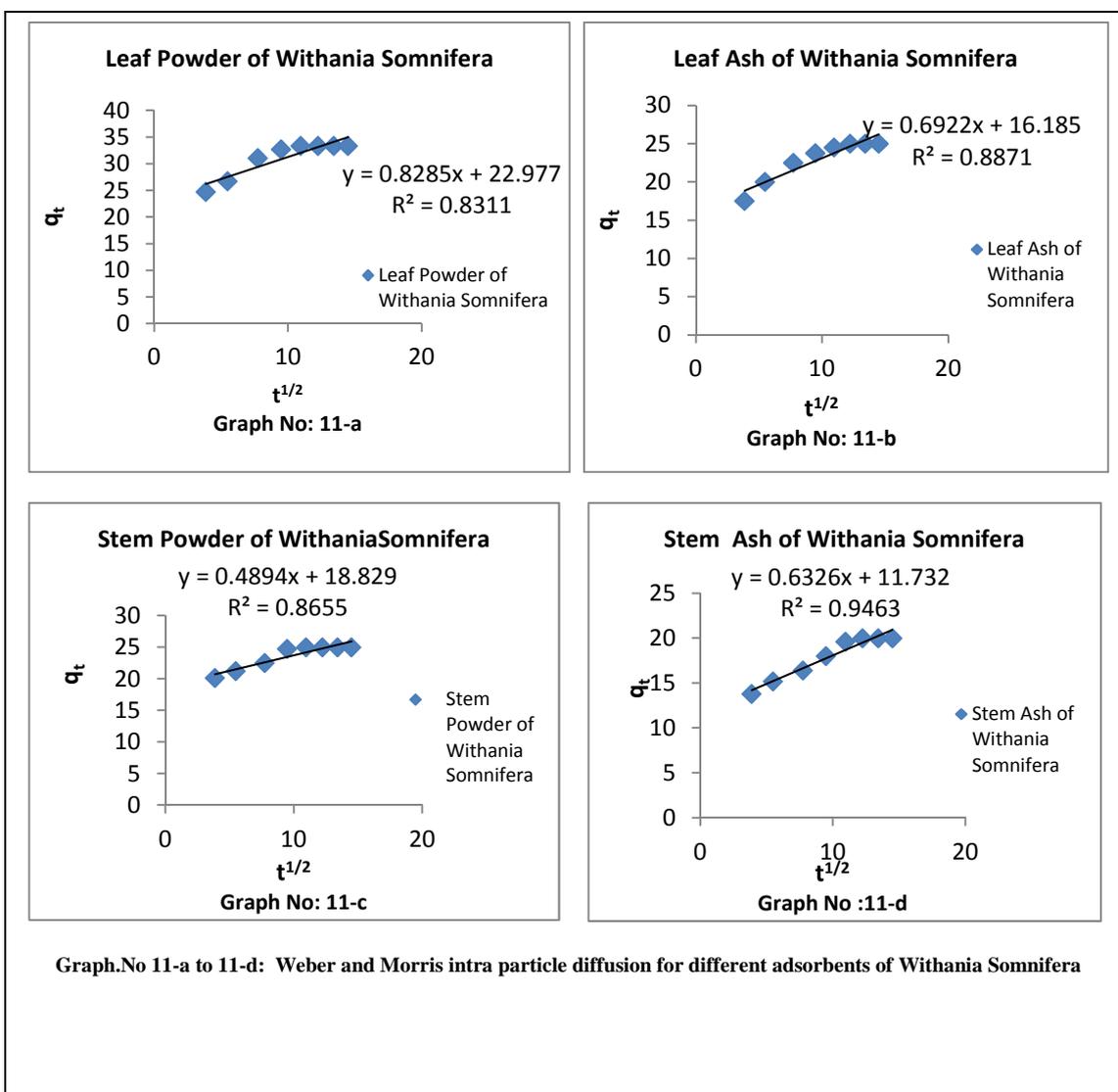
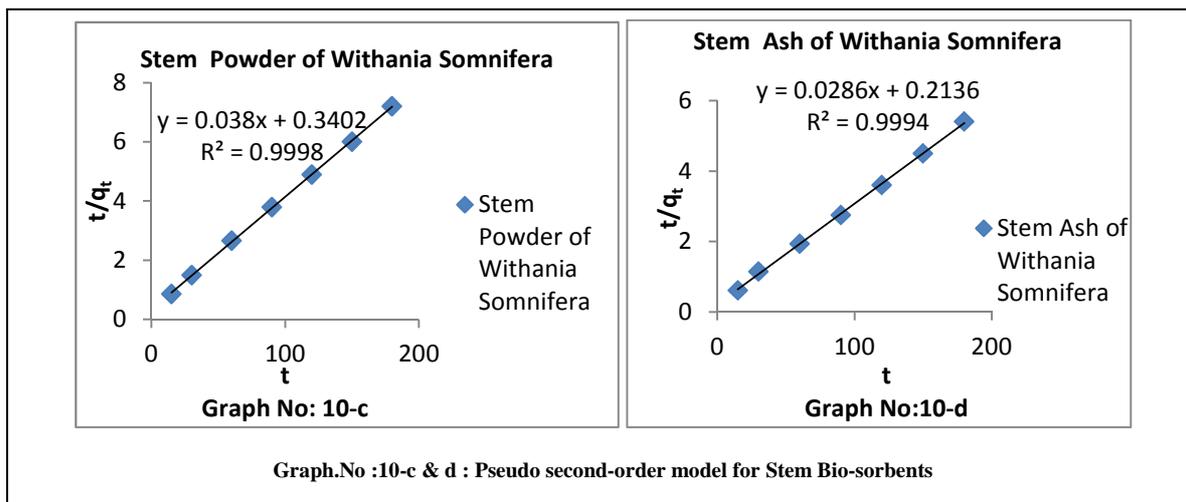
$R^2$ -values near to unity indicate the applicability of these two model adsorption isotherms confirming the heterogeneous surface of the adsorbent. The mean free energy ( $E$ ) and heats of sorption ( $B$ ) are characteristics of adsorption and as  $E$  values are less than 8 kJ/mol and  $B$  are less than 20kJ/mol, the adsorption is “physisorption” in nature i.e. non-specific adsorption due long range weak Vander Waals forces [64-68].

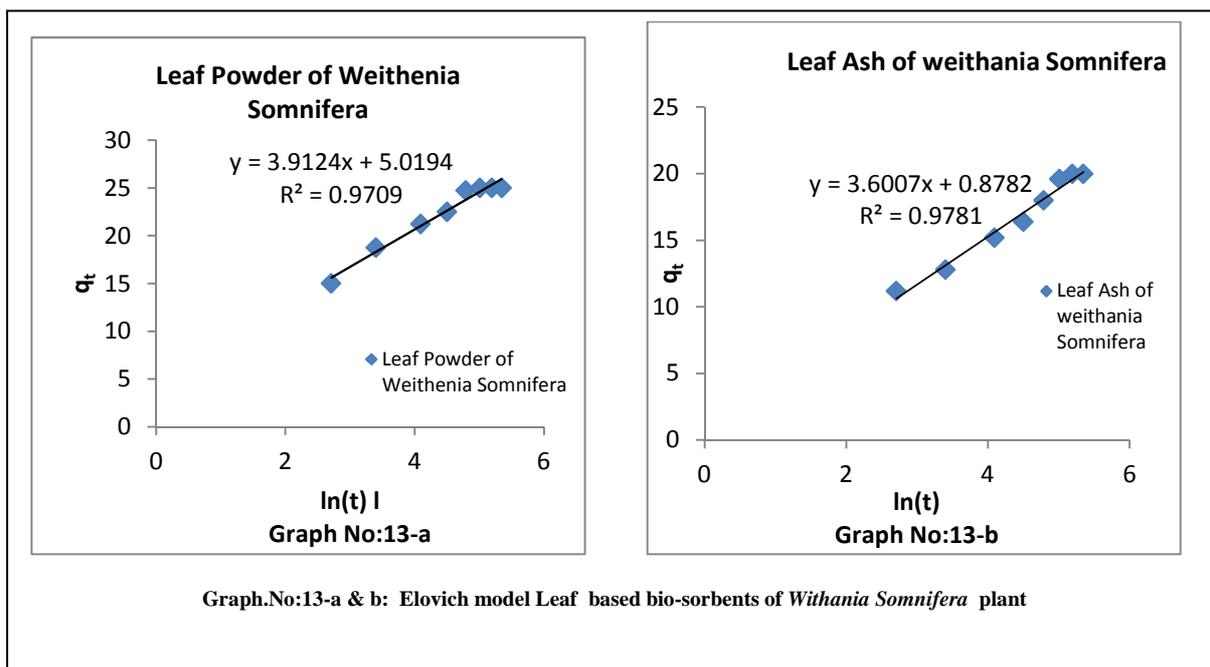
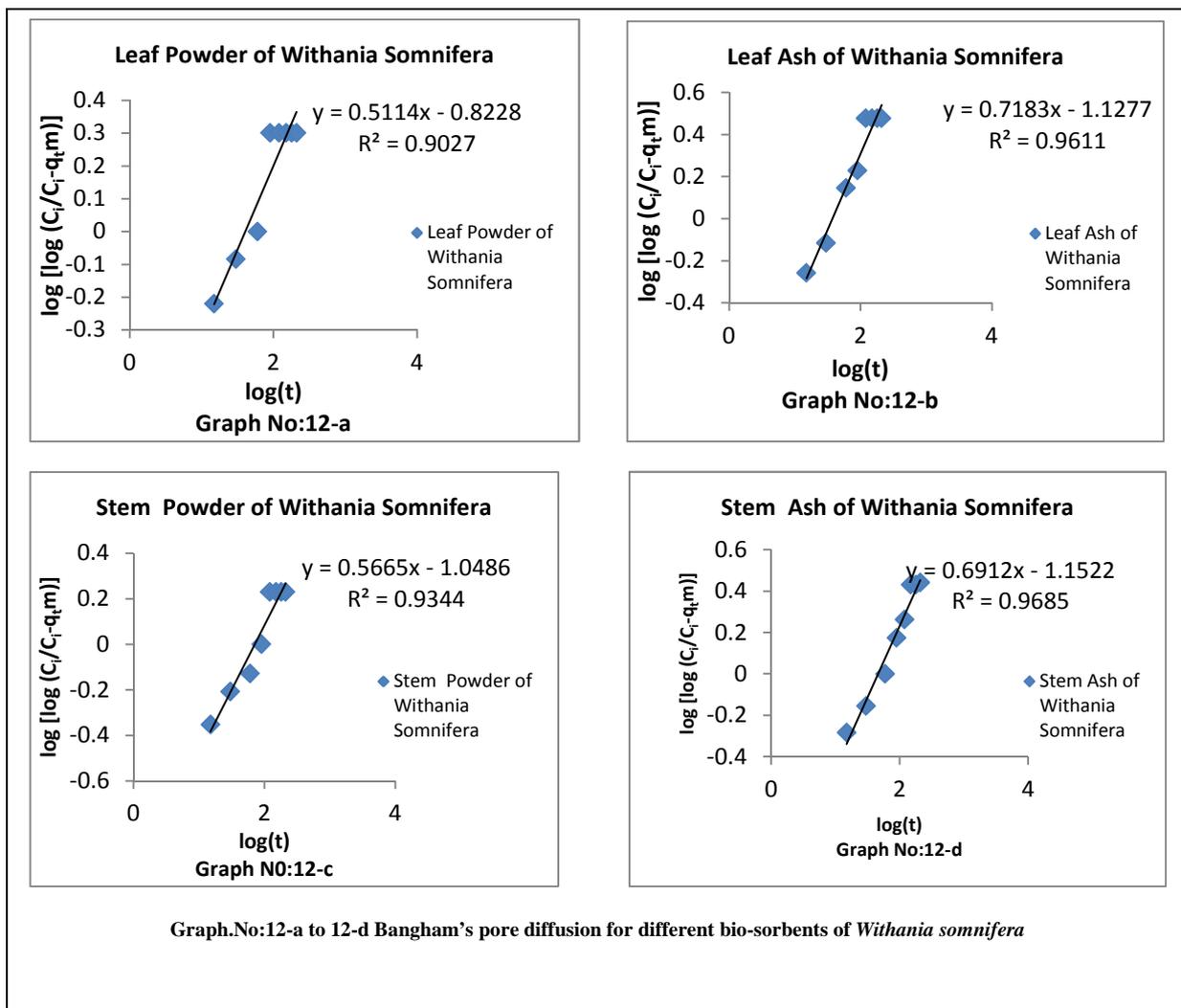
#### F: Adsorption Kinetics

Kinetics of adsorption is studied using pseudo first-order model [69, 70], pseudo second-order model [70, 71], Weber and Morris intraparticle diffusion model [72], Bangham’s pore diffusion model[73] and Elovich equations[74,75]. The pseudo first-order equation is  $\log (q_e - q_t) = \log q_e - k_1 t/2.303$ ; the pseudo second-order equation is  $t/q_t = 1/k_2 q_e^2 - (1/q_e) t$ ; Weber and Morris intraparticle diffusion equation is  $q_t = k_{ip} t^{1/2} + c$ ; Bangham’s pore diffusion equation is  $\log [\log (C_i/C_i - q_t m)] = \log (k_0/2.303V) + \alpha \log(t)$ ; Elovich equation is  $q_t = 1/\beta \ln(\alpha\beta) + 1/\beta \ln(t)$

The data of these five kinetic models are presented in Graph Nos: 9-a to d, 10-a to d, 11-a to d, 12-a to d & 13-a to d and rate constants along with the correlation coefficient values are presented in Table 3.









**G: Interfering Ions:**

S.No	Adsorbent	Maximum extractability at optimum condition	% of Extractability of Aluminum (III) in the presence of tenfold excess of interfering ions at optimum extraction conditions										
			SO <sub>4</sub> <sup>2-</sup>	NO <sub>3</sub> <sup>-</sup>	Cl <sup>-</sup>	PO <sub>4</sub> <sup>3-</sup>	F <sup>-</sup>	CO <sub>3</sub> <sup>2-</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Cu <sup>2+</sup>	Zn <sup>2+</sup>	Ni <sup>2+</sup>
1.	Leaves powder	100.0%; pH:6; 150 min.; 2.5gm/lit	94.4	94.1	64.1	100.0	60.1	92.6	91.0	94.2	93.1	93.9	91.3
2.	Leaves Ash	100.0%; pH:6;120 min. 2.0 gm/lit	98.4	98.1	71.1	100.0	63.1	97.1	95.2	98.4	97.0	97.9	95.8
3.	Stems powder	98.0%; pH:6;150 min., 2.0 gm/lit	97.5	97.1	70.2	100.0	62.1	96.3	94.1	97.6	96.1	97.0	94.6
4.	Stems Ash	100.0%; pH:6;120 min., 1.5gm/lit	99.5	99.1	69.2	100.0	65.2	98.2	96.2	99.4	97.4	98.5	96.6

The effect of the presence of tenfold excess of common co-ions viz., Sulphate, Nitrate, Chloride, Phosphate, Fluoride, Carbonate, Calcium, Magnesium, Copper, Zinc and Nickel ions on the % removal of Aluminium ions from synthetically simulated waters has been studied and the results are presented in **Table No.4**.

Cations have marginal effect and % of removal never comes down below 91.0% (Vide Table 4: Columns: 10-14).

Anions like SO<sub>4</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup> and CO<sub>3</sub><sup>2-</sup> marginally affected the % of extraction but Chloride and Fluoride affected the % of extraction significantly but Phosphates synergistically maintained at 100% of extraction.

**APPLICATIONS**

The methodologies developed in the present investigation have been applied to the samples collected from industrial effluents and polluted natural waters and the results are presented in the **in the Table No: 5**. It is inferred from the Table that the methods developed are successful as more than 90.0% of extraction is observed.

SAMPLES COLLECTED AT DIFFERENT PLACES	Conc. of Al(III) in the Sample	% of Maximum extraction of Aluminum(III)			
		<i>Withania somnifera</i>			
		Leaves Powders (mesh:75 μ) :pH:6;150 min& 2.5 g/lit	Leaves Ashes pH: 6;120 min & 2.0 g/lit	Stem Powders (mesh:75 μ) pH:6;150 min& 2.0 g/lit	Stem Ashes pH: 6;120 min & 1.5 g/lit
<i>Alum Industry effluents:</i>					
1					
2	20.0 ppm	95.5%	93.5%	90.5%	93.0%
3	25.0 ppm	93.4%	91.0%	91.4%	92.5%
	30.5 ppm	90.5%	95.0%	92.5%	90.5%
<i>Natural polluted Lake Samples(fed with known amounts of Aluminum (III)):</i>					
1	10.0 ppm	96.5%	96.5%	96.5%	95.5%
2	15.0 ppm	97.6%	97.6%	97.5%	96.5%
3	22.5ppm	98.7%	98.0%	98.5%	97.0%

**CONCLUSION**

- Bio-adsorbents from the leaves and stems of *Withania somnifera* plant have been prepared. Their affinity towards the removal of Aluminum (III) has been investigated with respect to pH, time of equilibration, sorbent dosage, agitation time, initial concentration of Aluminum ions, temperature, and presence of co-ions using simulated waters and by adopting batch methods of extraction. Optimum conditions for the maximum removal have been established. The interference of the common co-ions present in waters on the % of extraction, has been studied

- The adsorption process is analyzed with Freundlich, Langmuir, Temkin and Dubinin-Radushkevich (D-R) isotherm models and found the suitability of the Langmuir isotherm model indicating the mono-layer formation of the adsorbate on the adsorbent. The 'Physisorption' nature of the adsorption process has been inferred from Temkin and Dubinin-Radushkevich isotherm models and by calculating the mean free energy (E) and heats of sorption (B) from the plots of the isotherms.

- The kinetics of adsorption process has been analyzed using pseudo first-order, pseudo second-order, Weber and Morris intraparticle diffusion, Bangham's pore diffusion and Elovich equations and found that the adsorption process is well described by the pseudo-second-order model.
- The endothermic nature of the adsorption is found on the analysis of the thermodynamic parameters,  $\Delta H$ ,  $\Delta S$  and  $\Delta G$ .
- The methodologies developed are successfully applied to industrial sewages and polluted natural water samples.

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