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OPTIMIZATION OF BIOSORPTIVE REMOVAL OF COBALT(II) ONTO *EUCALYPTUS GLOBULUS L.*

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ABSTRACT

The objective of this study is to evaluate the efficacy of *Eucalyptus globulus* leaf powder in the removal of Cobalt(II) from aqueous solutions. Detailed batch mode experimentation using Response Surface Methodology (RSM) is undertaken and at optimum conditions, the maximum biosorption capacity of *E. globulus* leaf powder for Cobalt(II) loading is q_{max} = 7.194 mg/g. Freundlich equilibrium model is identified for adsorption process, after trying three standard isotherm models - Langmuir, Freundlich and Temkin isotherms. Pseudo-second-order kinetic model describes the adsorption process. Surface morphology studies through Fourier Transform Infrared Spectroscopy[FTIR] and Scanning Electron Microscopy [SEM] with Energy Dispersive X-ray spectroscopy(EDX) confirms the metal loading on the adsorbent surface.

KEYWORDS: ***Cobalt(II) removal, Biosorption, E. globulus L., Equilibrium, Kinetics, RSM***

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INTRODUCTION

Quality water is essential for the well being of all living systems. Unfortunate, its supply is diminishing due to continuous, unwanted interference with the ecosystem. The problem is compounded by the discharge of toxic exit streams of different processing industries, making water unfit for reuse. One of the major water pollutants is heavy metals- Co, Pb, Cr, Zn, Cd, Cu, Hg, As, Al and Ni, discharged by various industrial units. Levels of these ions in water are high enough to cause several health issues. Hence, there is a need to treat the water; to make it potable and adsorption is a good treatment option, because of it efficacy and efficiency[1, 2]. Numerous techniques available for removal of heavy metals from industrial waste water are chemical precipitation, reverse osmosis, ion exchange, coagulation, electro dialysis, ultrafiltration[3-8], but they are costlier and inadequate compared to adsorption. Biosorption of Cobalt(II) using different forms of agriculture waste material were tried earlier and the sorbent materials studied are functional ligand anchored nanomaterial(facial adsorbent)[9], Chrysanthemum indicum [10], leaves of Acacia nilotica[11], fruit wastes of Blighia sapida (Akee apple)pod[12], ficus religiosa[13] lemon[14-16] , lichen Evernia prunastri [17], rose waste biomass[18], crab and arca shell[19], Black carrot residues [20], crab[21], hazelnut shell[22], fruit peels of banana and orange [23]. Eucalyptus leaves contain essential oils like α-pinene, 1, 8-cineol and pinocarveol-trans at a level of 1.2% to 3% (w/w). The oil contains the highest antibacterial activity against microbes of *Listeria ivanovii and Bacillus cereus* [24-26]. In the present study *E. globulus* leaf powder is the sorbent chosen for the removal of Cobalt(II) and parametric dependence, equilibrium and kinetic modeling and surface modification characteristics are reported.

MATERIALS AND METHODS

Chemicals

All used chemical compounds are of analytic grade (Merck, Germany). A stock solution of Cobalt(II) (0.254mg/100mL) is prepared by dissolving 0.254 mg of 97% $Co(NO_3)_2 \cdot 6H_2O$ in 100 mL of distilled water. The stock solution is appropriately diluted to obtain desired concentrations of Cobalt(II) ion. pH of the metal solution is adjusted with 0.1 N NaOH and 0.1N H_2SO_4 solutions.

Preparation and Treatment of biosorbent

E. globulus leaves are collected from Sri Venkateswara University Campus, Tirupati, washed several times to remove impurities and then dried under sunlight. Dried leaves are ground to fine powder, using a mechanical grinder, to 240(63µm) mesh B.S.S. These particles are again washed with distilled water, dried at room temperature and stored in air tight bottles for further experimental work.

Biosorption procedure

Adsorption of cobalt with *E. globulus L.* is analyzed in a batch mode. 50 mL of solution having 20 mg/L of cobalt ion is taken into a 250 mL Erlenmeyer flask. 10 g/L of bio sorbent of 63 µm size are added to the flasks. Initial solution pH is adjusted by adding 0.1 N NaOH / 0.1 Nsulfuric acid. Samples are agitated on an orbital shaker at constant speed and at room temperature. At different time periods (1, 3, 5, 10…... 120 & 150 min), samples are collected, filtered and the filtrate is analyzed for Cobalt(II) concentration, using inductively coupled plasma optical emission spectroscopy (Optima 8000). It is observed that 60-minute contact time will take the procedure to equilibrium. The % Removal of Cobalt(II) is calculated using equation 1.

$$\frac{C_0 - C_e}{C_0} * 100 \qquad (1)$$

Effects of four factors - Cobalt(II) concentration, initial solution pH, adsorbent dosage and temperature of Cobalt(II) solution are then studied, keeping the particle size and processing time fixed. The equilibrium metal uptake capacity (q_e) is estimated by using the following equation 2.

$$q_e = \frac{C_0 - C_e}{m} * V \qquad (2)$$

Where c_o is initial Cobalt(II) concentration(mg/L), c_e is equilibrium Cobalt(II) concentration(mg/L), m is mass of adsorbent(g), V is volume of Cobalt(II) solution(ml)

Characterization of biosorbent

FT-IR (ALPHA interferometer (ECO-ATR)) Germany Spectroscopy, is used to record the IR spectrum before and after adsorption process in the range of 4000–500 cm^{-1} and to identify the functional groups involved in retention of Cobalt(II) ion. The structure and morphology *E. globulus L.* before and after adsorption of Cobalt(II) is observed using a scanning electron microscope (SEM–EVO MA 15).

RESULTS AND DISCUSSIONS

Optimization using Response Surface Methodology (RSM)

The contribution and inter relationship among the four independent variables and on the percentage removal of Cobalt(II) ions is studied and is fitted with a quadratic model using Design expert 10.0.3.0. Results from RSM (CCD) for Cobalt(II) biosorption by *E. globulus* L are as shown in Table 1 and Levels of different process variables in coded and un coded form for biosorption of Cobalt(II) using *E. globulus L.* leaf powder as shown in Table 2. ANOVA and estimated regression coefficients for the Cobalt(II) biosorption onto *E. globulus* L. are in Table 3. A comparison of optimal value (predicted) and actual percentage (experimental) values of Cobalt(II) and the normal plot for residuals are given in the form of Fig.1 and Surface contour plots for sorption of Cobalt (II) onto *E. globulus* leaf powder are grouped in to Fig.2. When a solution of 20 mg/L Co(II) is treated with 25 g/L *E. globulus L.* at a pH of 5.0 and temperature of 303 K, 80.849 % of Cobalt(II) present is removed, the following quadratic expression (equation 3) indicates the relative contributions of individual factors and their quadratic products to the percentage removal Cobalt(II) (Y).

$$Y = 70.26 -1.56*A+1.6*B+6.05*C+1.31*D+0.59*AB+0.027*AC+0.86*AD-0.12*BC-0.40*BD +0.25*CD+0.65*A^2+0.46*B^2-0.29*C^2-2.42*D^2 \quad \text{............ (3)}$$

Y is the estimated response is in equation 3 and suggests that maximum percentage of biosorption of Cobalt(II) is 79.519 % for the set of Cobalt(II) concentration(Co) = 15.126 mg/L, pH = 6.0, sorbent dosage (w) = 20.0 g/L, and absolute temperature(T) = 301.705K.

Table 1
Results from CCD for Cobalt(II) biosorption by E. globulus L.

Run	A(Co)mg/L	B(pH)	C(w), g/L	D(T), K	Biosorption of Cobalt(II) % Removal	Predicted
1	0.000	0.000	0.000	0.000	70.2648	70.2648
2	1.000	-1.000	1.000	-1.000	68.5800	68.3089
3	0.000	0.000	0.000	-2.000	57.2800	57.9842
4	1.000	1. 000	-1.000	1.000	65.3000	64.8751
5	-1.000	-1. 000	-1.000	-1.000	63.1000	62.4838
6	-1.000	-1. 000	-1.000	1.000	63.5800	63.6681
7	-1.000	1. 000	1.000	1.000	77.8900	77.4460
8	-1.000	-1. 000	1.000	-1.000	74.2290	74.2671
9	0.000	2. 000	0.000	0.000	74.3650	75.2950
10	-1.000	-1. 000	1.000	1.000	76.8000	76.4453
11	1.000	-1. 000	1.000	1.000	73.8100	73.9362
12	0.000	0. 000	0.000	0.000	70.2648	70.2648
13	1.000	1. 000	1.000	1.000	77.3050	77.2902
14	-1.000	1. 000	-1.000	-1.000	66.0500	65.5370
15	1.000	1. 000	-1.000	-1.000	62.1000	61.8237
16	1.000	1. 000	1.000	-1.000	73.7200	73.2451
17	0.000	0. 000	0.000	2.000	62.9000	63.2137
18	0.000	0. 000	0.000	0.000	70.2648	70.2648
19	0.000	0. 000	-2.000	0.000	56.3800	57.0242
20	1.000	-1. 000	-1.000	1.000	61.3120	61.0509
21	2.000	0. 000	0.000	0.000	69.5100	69.7710
22	1.000	-1. 000	-1.000	-1.000	56.3600	56.4172
23	0.000	0. 000	0.000	0.000	70.2648	70.2648
24	-1.000	1. 000	1.000	-1.000	77.2200	76.8501
25	0.000	0. 000	0.000	0.000	70.2648	70.2648
26	-2.000	0. 000	0.000	0.000	75.2364	70.2648
27	0.000	0. 000	2.000	0.000	80.8490	81.2227
28	-1.000	1. 000	-1.000	1.000	65.4990	65.1391
29	0.000	0. 000	0.000	0.000	70.2648	70.2648
30	0.000	-2. 000	0.000	0.000	68.8000	68.8878

A-Initial concentration(C_o) mg/L, B -pH of the solution, C- Biosorbent dosage(w) g/L, D(T) K- Temperature in kelvins CCD-Central Composite Design

Table 2
Levels of different process variables in coded and uncoded form for biosorption of Cobalt(II) using E.globulus L. leaf powder.

Varible	Name	Range and level -2	-1	0	1	2
A	Initial concentration(C_o) mg/L	10	15	20	25	30
B	pH of the solution	3	4	5	6	7
C	Biosorbent dosage(w), g/L	5	10	15	20	25
D	Temperature(T), K	283	293	303	313	323

Table 3
ANOVA and estimated regression coefficients for the Cobalt(II) biosorption onto E.globulus L.

Source	Sum of Squares	df	Mean Square	F-Value	p-value Prob > F	
Model	1261.92	14	90.14	297.31	< 0.0001	Significant
A	58.08	1	58.08	191.56	< 0.0001	
B	61.58	1	61.58	203.11	< 0.0001	
C	878.35	1	878.35	2897.18	< 0.0001	
D	41.02	1	41.02	135.31	< 0.0001	
A*B	5.54	1	5.54	18.27	0.00070	
A*C	0.012	1	0.012	0.039	0.84680*	
A*D	11.90	1	11.90	39.24	< 0.0001	
B*C	0.22	1	0.22	0.73	0.40650*	
B*D	2.50	1	2.50	8.26	0.01160	

C*D	0.99	1	0.99	3.26	0.09120*
A^2	11.74	1	11.74	38.74	< 0.0001
B^2	5.72	1	5.72	18.87	0.00060
C^2	2.23	1	2.23	7.37	0.01600
D^2	160.16	1	160.16	528.29	< 0.0001
Residual	4.55	15	0.30		
Lack of Fit	4.55	10	0.45		
Pure Error	0.000	5	0.000		
Cor Total	1266.47	29			

R^2 (Adj) = 0.9931 and R^2 (Pred) = 0.9793, *insignificant
ANOVA-Analysis of variance

Model F-value of 297.31 implies that the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Values of "Prob> F" and < 0.05 indicates the model terms are significant. In this case A, B, C, D, A*B, A*D, B*D, A^2, B^2, C^2, D^2 are significant model terms and A*C, B*C and C*D are not significant.

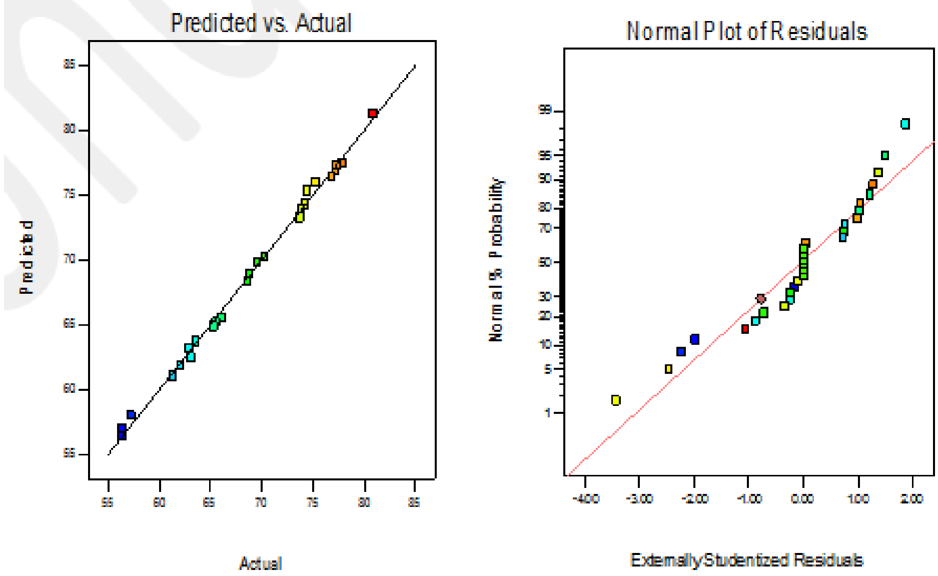

Figure 1
A comparison of predicted values and the percentage removal (actual values) and the normal plot for residual for Cobalt(II) onto E.globulus L

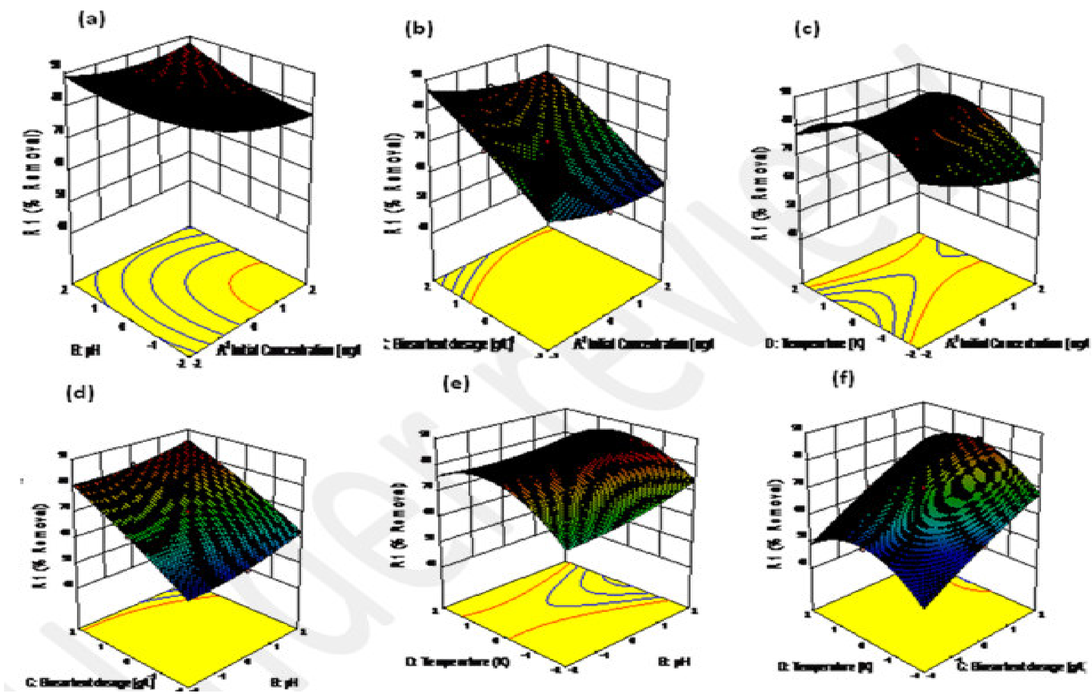

Figure 2
Surface contour plots, (a). pH -Initial conc –% Removal, (b). Initial conc– dosage – % Removal, (c). Temp.– Initial conc –% Removal, (d). pH – dosage -% Removal, (e). pH – Temp. – % Removal, (f). Temp. – dosage– % Removal.

Equilibrium Isotherms

Equilibrium isotherm is used to know the interactions between adsorption capacity and equilibrium concentration for metal removal. The equilibrium isotherms of Langmuir, Freundlich and Temkin models are adopted. Langmuir isotherm[27] model relates to coverage of molecules on a solid surface to concentration of a medium above the solid surface at a fixed temperature and is expressed by the following equation 4 and 5.

$$\frac{q_e}{q_{max}} = \frac{bC_e}{1+bC_e} \qquad (4)$$

And the linear form of the above equation is

$$\frac{C_e}{q_e} = \frac{1}{bq_{max}} + \frac{C_e}{q_{max}} \qquad (5)$$

A plot of (c_e / q_e) aganist (c_e) for *E. globulus* L. obtained for the experimental data indicates a slope and intercept. Where q_e is the amount of metal adsorbed at equilibrium (mg/g), q_{max} is the maximum adsorption capacity (mg/g). The separation factor, R_L of 0.93, which describes the essential characteristics of the Langmuir model and R_L can be defined in equation 5i and Langmuir isotherm for adsorption of Cobalt(II) onto *E. globulus L.* as shown in Fig.3.

$$R_L = \frac{1}{(1+bc_e)} \qquad (5i)$$

$0<R_L<1$ indicates favorable adsorption, $R_L>1$ indicates unfavorable adsorption, $R_L = 1$ indicates linear adsorption

$R_L = 0$ indicates irrepressible adsorption

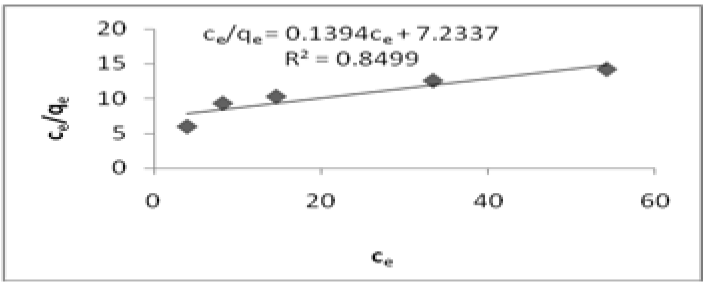

Figure 3
Langmuir isotherm for adsorption of Cobalt(II)

Freundlich isotherm[28]assumes that the adsorbent has heterogeneous surfaces on the sorbent and different functional groups are active in adsorption and is expressed by the following equation 6 and the linear form of above equation is in equation 7.

$$q_e = K_f c_e^{1/n} \qquad (6)$$

$$ln q_e = \ln K_f + \frac{1}{n} ln\, c_e \qquad (7)$$

The value of slope, n (1.444) in the equation satisfies the condition of 0<n<1 indicating suitability for sorption and Freundlich constant from slope and intercept of the equation from Freundlich isotherm for biosorption of Cobalt(II) in

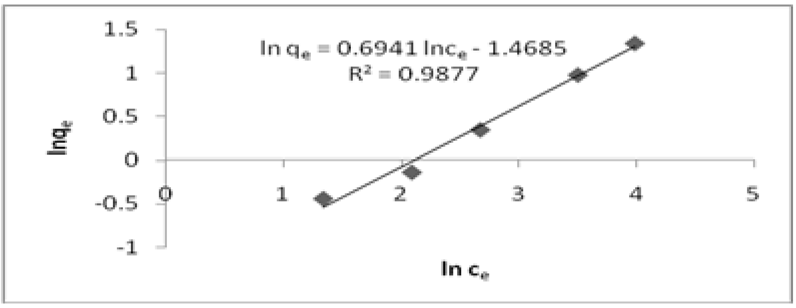

Figure 4
Freundlich isotherm for biosorption of Cobalt(II)

Temkin and Pyzhev isotherm[29] equation assumes low interactions between the adsorbent and adsorbate and the adsorption energy of all the molecules in the surface layer decrease at the cover surface and the linear form is in equation 8.

$$q_e = \frac{RT}{b_T} \ln A_T + \frac{RT}{b_T} \ln C_e \qquad (8)$$

Temkin constants can be calculated from slope and intercepts for the plot of q_e against ln c_e. Where R is universal gas constant (8.314 J/mol K) and T is the absolute temperature (K), A_T is the Temkin isotherm constant (L/g), Ce is the maximum binding energy and b_T is the Temkin constant related to the heat of sorption (J/mol). Temkin isotherm for biosorptionof Cobalt(II) onto *E. globulus L.* as shown in Fig.5. All isotherm constants evaluated at optimum conditions are provided in Table 4.

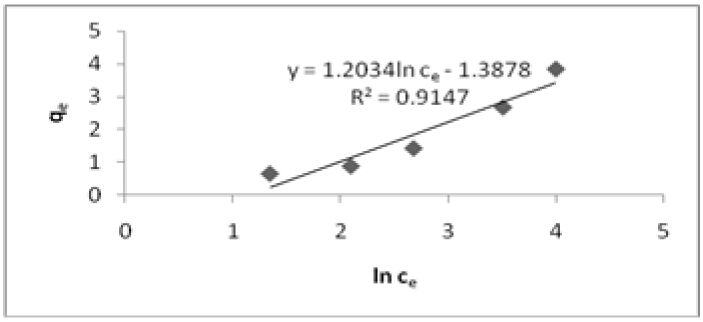

Figure 5
Temkin isotherm for biosorption of Cobalt(II) onto E. globulus L.

Table 4
Equilibrium Isotherm constants

Langmuir	Freundlich	Temkin
q_{max} = 7.194mg/g	K_f = 0.23 mg/g	b_T = 2094.04
k_L=b=0.0193 L/g	n=1.44	A_T = 0.3157
R^2=0.8499	R^2 = 0.987	R^2 = 0.914

q_{max}- Maximum adsorption capacity (mg/g), $k_{L=}$ b - Langmuir constant, R^2– Regression coefficient, K_f and n are Freundlich coefficients, A_T=Temkin isotherm constant(L/g), b_T- Temkin constant related to the heat of sorption (J/mol)

Kinetics of biosorption

Lagergren pseudo first kinetic model and pseudo second order kinetic models are in equation 9 and 10 , applied to the current study and the rate constants and equilibrium metal uptake capacity are evaluated from plot of log(q_e-q_t) against time (t) for pseudo first order and the intercept value should be equals the experimental q_e then the reaction can be categorized as pseudo first order rate equation[11] and from plot of t/q_t against t for pseudo second order model considers the rate-limiting step as the formation of chemisorptive bond involving electron sharing between the solute and sorbent. The rate constants, correlation coefficients for biosorption of Cobalt(II) onto *E.globulus* are tabulated in Table 5.

$$\log\left(q_e - q_t\right) = log q_e - \frac{k_1}{2.303}t \quad (9)$$

.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e{}^2} + \frac{1}{q_e}t \quad 10)$$

Table 5
The rate equations and coefficients for biosorption of Cobalt(II) onto E. globuslus L.

Kinetic model	Obtained equation	Rate constant,	R^2
Pseudo1st order	log (q_e–q_t) =-0.0111t–0.4141	k_1 = 0.0253 min^{-1}	0.9582
2nd order	(t/ q_t) = 0.771 t + 1.6387	k_2 = 0.363 g/mg.min	0.9974

Characterization of the E. globulus L. FT-IR analysis, SEM and EDX analysis

FTIR (ALPHA interferometer (ECO-ATR), Germany), Spectra are as shown in Fig.6(a1 -a2). Peaks at 3327 and 3249 cm^{-1} indicate strong O-H stretch, those at 2911 cm^{-1} C-H stretch, peak at 2354 cm^{-1} indicates alkyne triple bonds and nitrile triple bonds. Peaks at 1628 cm^{-1} C=C stretch, Peak at 1441 cm^{-1} -C-H bending, Wave number at 1317,1221, 1153 cm^{-1} C-N stretch and 1046 cm^{-1} are due to the skeletal vibration of the C-O stretch, Peak at and 850-550 C-Cl stretching vibrations. The Spectra of adsorbent before and after adsorption showed similar band characteristics, but the intensity of bands is either stronger or weaker. The peaks in Fig.6(a1) are shifted from 3327, 3249, 2911, 2354, 1628, 1441, 1317, 1221, 1153, 1046, 771, 662 and 582 cm^{-1} to 3338, 3244, 2906, 2348, 1631, 1531, 1441, 1324, 1224, 1018, 655 and 540cm^{-1} in Fig.6(a2) respectively. The change in band strongly indicates that adsorption has happened and Fig.6 (b1- b2) shows SEM images and Fig.6(c1- c2) shows EDAX spectra of the *E. globulus* L. before and after Cobalt(II) loading.

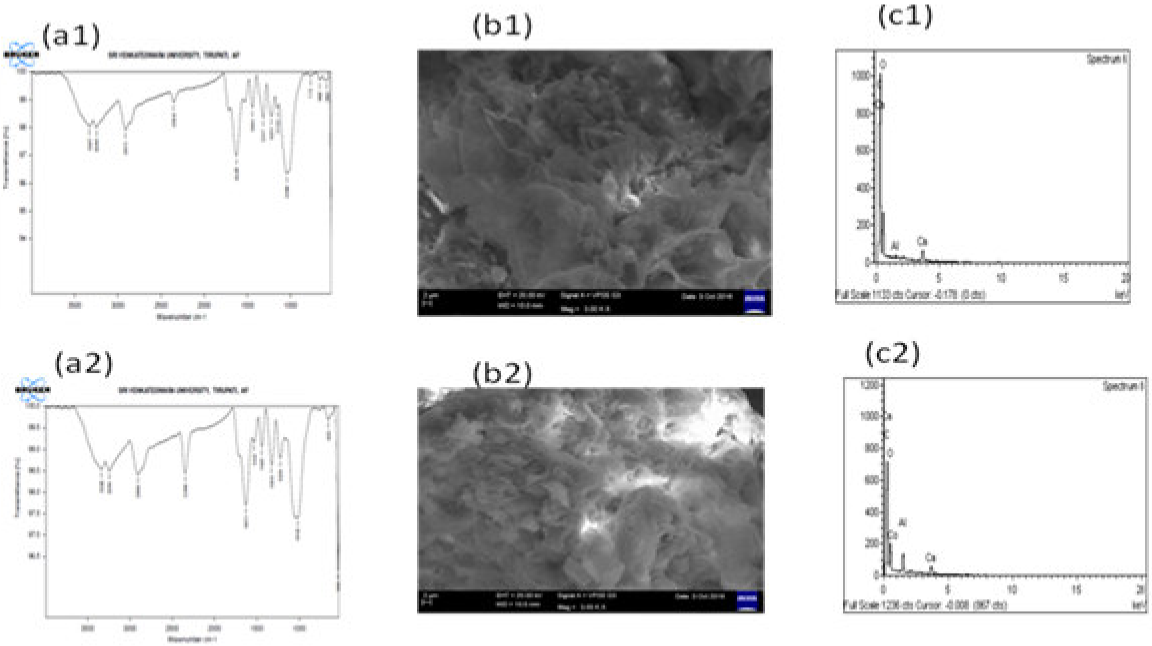

Figure 6
(a1- a2). FTIR spectra, (b1- b2). SEM images and (c1- c2). EDAX spectra of the E.globulus L. before and after Cobalt(II) loading.

Comparative study
A comparative picture of various studies, on removal of Cobalt(II) by adsorption process, is provided in Table 6.

Table 6
Comparison of maximum adsorption capacity, based on the Langmuir isotherm by different adsorbents for Cobalt(II) from aqueous solutions.

Adsorbent	**Concentration range, mg/L**	**Time, min**	**pH**	**Metal uptake, mg/g**
Acacia nilotica[12]	5-200	120	5.0	35.461
PET-TSE fibers[30]	10-400	180	5.0	78.08
Natural hemp fibers[31] (batch & fixed bed)	25–200	25	4. 5	13.58& 15.44
Apricot stone activated carbon[32] (ASAC)	40–80	90	9.0	111.11
Chrysanthemum indicum flower raw(CIFR) and biochar(CIFBC) [10]	25–200	60&45	5.0	14.84 45.44
Cystoseira indicia (C. indica)[33]	25 and 300	4h	5.0	59.524
Ficus carica[34] ($MgCl_2$ treated)	20-200	120	6.0	82.64
Facial adsorbent[9]	2–75.10	120	9.5	157.73
Diplotaxis harra & G. coronaria L[35]	25-200	60 & 45	6.5 & 7.5	33.02 & 24.52
E. globulus L.	**5 -150**	**60**	**5.0**	**7.192 Present study**

PET-TSE- Poly ethylene terephthalate thiosemicarbazide $MgCl_2$-Magnisium dichloride, CIFR- Chrysanthemum indicum flower raw, CIFBC- Chrysanthemum indicum flower biochar, G. coronaria L- Glebionis coronaria leaf.

CONCLUSION

The study conclusively establishes the suitability of *E. globulus L.* to remove Cobalt(II) after thorough investigation.

CONFLICT OF INTEREST

Conflict of interest declared none.

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