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Adsorption of Fe^{3+} Ion from Aqueous Solution by Activated Carbon Prepared from *Leucas aspera*: Thermodynamic, Kinetic and Equilibrium Approach

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The adsorption process using low cost adsorbent carbon prepared from useless agricultural plant is very potential method to removing both organic and inorganic pollutants especially heavy metals. The present study reports the removal of iron(III) ions from contaminated aqueous solution by using roots and leaves of *Leucas aspera*. The adsorption process is also affected by various factors like temperature, contact time, adsorbent dosage and pH of the solution. The fitness of present study is also confirmed by verifying of Feundlich isotherm and Langmuir isotherm. The feasibility and nature of the process is also confirmed by thermodynamic parameters like ΔG, ΔH and ΔS. And also calculate kinetic parameters verifying by pseudo-second order kinetics, Elovich model and intra-particle diffusion model. Finally, the adsorption process is confirmed by FT-IR, XRD of before and after adsorption process of adsorbent.

Keywords: Iron, *Leucas aspera*, Adsorption, Thermodynamic, Kinetic parameters.

INTRODUCTION

There are various classes of pollutants *viz.* organic pollutants like phenols, amines, dyes, nitro compounds, halo compounds, *etc*. and inorganic pollutants like heavy metals are causing heavy poisoning to water especially ground water sources. Among the various organic and inorganic pollutants, the inorganic pollutants like heavy metals such as chromium, aluminium, copper, manganese, cobalt, *etc*. are toxic to human health. When the contaminated water by heavy metals taken by living organism causing severe toxic effects to the living organisms [1]. The Itai-Itai disease was reported in Jintsu river flowing in Japan due to present of cadmium metal in river aquatic environment [1,2].

There are several conventional methods are available to recycling the wastewater producing from various industries those methods are highly cost effective and also cause another adverse effects to the environment. On the other hand, the low cost effective green technologies are greatly attributed by the society. One of such a method is adsorption process using low cost carbon prepared from waste agricultural plants is effective method to removing heavy metals. The large number of literatures are available the removal of heavy metals by using the activated carbon from pine bark, wool waste, coconut shell, *etc.* [3,4].

The aim of present study is undertaken the removal of iron (Fe^{3+}) from the aqueous solution by adsorption process using low cost carbon prepared from *Leucas aspera* plant. In order to study of adsorption process, the mass-transfer principle, isotherm concept should be verified. The feasibility and kinetics of the adsorption process also checked by verifying the thermodynamic and kinetic parameters. Several authors are already investigated for removal various heavy metals and organic pollutants using adsorption process [5-14].

EXPERIMENTAL

Adsorbent: The activated carbon was prepared from dried *Leucas aspera* plant with 1:1 conc. H_2SO_4 and washed thoroughly with running water to become nearby neutral pH. Then, the black material was kept in muffle furnace at 600 °C for 8 h. It was ground well and the activated at 150 ± 5 °C. Finally it was used for the experiments.

All the chemicals used in experiments were highly purity AnalaR grade sample. 1000 ppm stock solution of iron (Fe^{3+}) was prepared by dissolving 8.635 g of ferric ammonium sulphate in 1000 mL distilled water. All other required concentration solutions were prepared by diluting the stock solution. The pH of the solution was adjusted to the required pH value using dilute HCl (or) NaOH.

The concentration of before and after adsorption of Fe^{3+} was determined with atomic absorption spectrophotometer at 480 nm absorbance.

Batch experiments method: The effect of various parameters like contact time, adsorbent dosage, effect of pH, on the removal of Fe^{3+} onto activated carbon of *Leucas aspera* was studied by batch series method at (30-60 °C). For each experimental step, 50 mL of known concentration of aqueous iron solution and pH were taken in a 250 mL iodine flask. A 100 mg adsorbent is added to the solution and shaken well at constant agitation speed (250 rpm). Then the samples were taken at regular time intervals (say 10 min) upto 60 min and the solution was filtered and adsorbent was removed. The residual concentrations of the solutions were analyzed to determine the Fe^{3+} ion concentration [15].

Similarly the effect of dosage of adsorbent, effect of concentration and effect of pH on the removal of iron ion was measured by batch experiments method.

Adsorption equilibrium isotherm was studied using 100 mg of activated carbon of *Leucas aspera* adsorbent for each 50 mL of iron solution were ranged from 10-60 ppm of initial concentration at 250 rpm for 1 h. Then the solution was separated and filtered and then the solution was separated from the mixture and analyzed for iron concentration. The adsorption capacity was calculated as follows:

$$Q_f = (C_i - C_f)\ V/M$$

where C_i and C_f being respectively called as the initial iron concentration (ppm) and final concentration, V is the volume of iron solution in liters [L] and M is the mass of adsorbent in grams [g]. The percentage removal of iron can be calculated as follows:

$$R\ (\%) = (C_i - C_f) \times 100/C_i$$

Batch kinetic method: The batch kinetic methods [16] were identical to the adsorption equilibrium method. All the kinetic methods took place at 30, 40, 50 and 60 °C at an initial concentration of 10, 20, 30, 40, 50 and 60 ppm. The amount of adsorption at time t, q_t (mg/g) was calculated by:

$$Q_t = (C_i - C_f)V/W$$

RESULTS AND DISCUSSION

Properties of adsorbent: The activated carbon prepared from the leaves and roots of *Leucas aspera* is an effective for the removal of many pollutant like organic, inorganic and biological from water and wastewater treatment. The adsorption capacity of solid adsorbent is due to the posses of micro porous fine structure, high surface area with different sizes and shapes [17]. All the above properties are possessed by activated carbon of *Leucas aspera* for adsorption process. The physical and chemical nature and pore structure actually determines the adsorption activity. The physico-chemical properties of activated carbon of *Leucas aspera* are listed in Table-1.

Effect of initial concentration: The value of C_f, Q_f and % removal of experiment of adsorption of Fe^{3+} ions on activated carbon of *Leucas aspera* at various initial concentrations for Fe^{3+} ions are listed in Table-2. It is suggested that the adsorption is highly dependent on initial concentration of metal ion.

TABLE-1
PROPERTIES OF THE ADSORBENT

Properties	Activated carbon of *Leucas aspera*
Size of particle (mm)	0.020
Density of particle (g/cc)	0.2015
Moisture (%)	0.1754
Weight Loss (%)	0.015
pH of aqueous solution	6.7

Effect of contact time: The contact time is important factor for adsorption process (Fig. 1) reveals that the curves are smooth and continuous leading to saturation, suggesting the possible mono layer coverage of the metal ions on acid activated cabon of *Leucas aspera* surface [18].

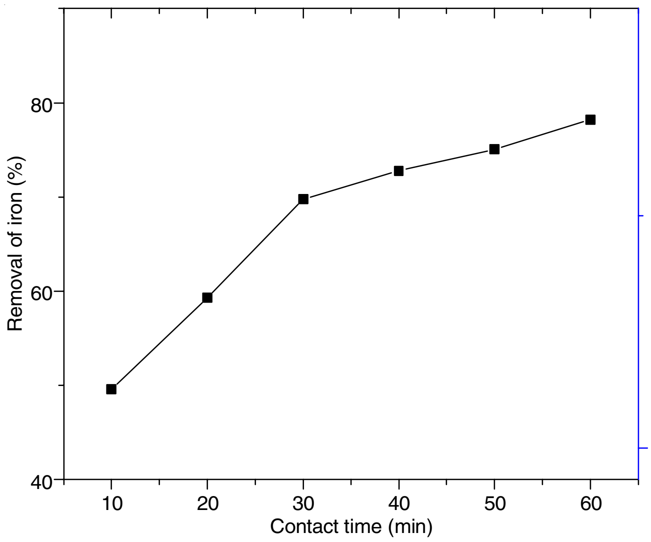

Fig. 1. Effect of contact time on the removal of Fe(III) ion, [Fe] = 20 mg/L; Temperature = 30 °C; Adsorbent dose = 100 mg/50 mL

Effect of adsorbent dosage: The adsorbent dosage is an important parameter, which influence the extent of metal uptake from the solution. The effect of varying doses of activated carbon of *Leucas aspera* at initial pH 6.5 shows an increase in percentage removal of iron with increase in dose of adsorbent [19] up to a certain limit (Fig. 2).

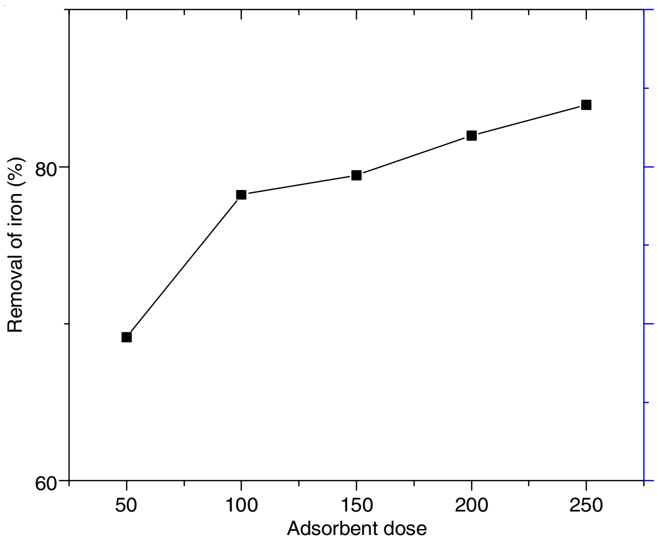

Fig. 2. Effect of adsorbent dose on the removal of Fe(III) ion, [Fe] = 20 mg/L; Temperature = 30 °C

TABLE-2
EQUILIBRIUM PARAMETERS FOR THE ADSORPTION OF Fe^{3+} ION ONTO ACTIVATED CARBON OF *Leucas aspera*

C_i (ppm)	C_f (ppm)				Q_f				Removal (%)			
	30 °C	40 °C	50 °C	60 °C	30 °C	40 °C	50 °C	60 °C	30 °C	40 °C	50 °C	60 °C
10	2.015	1.863	1.429	1.248	3.993	4.069	4.286	4.376	79.85	81.37	85.71	87.52
20	4.356	4.005	3.413	2.946	7.822	7.998	8.294	8.527	78.22	79.97	82.94	85.27
30	6.621	6.325	5.413	4.879	11.689	11.874	12.294	12.561	77.93	78.91	81.96	83.77
40	9.904	8.813	7.964	6.826	15.048	15.594	16.018	16.587	75.24	77.97	80.09	82.93
50	15.146	12.791	11.041	10.110	17.427	18.605	19.479	19.945	69.71	74.41	77.92	79.78
60	20.129	16.423	14.886	13.247	19.936	21.789	22.557	23.377	66.45	72.63	75.19	77.92

Effect of pH: The experiment was carried out at different pH shows that there is a change in the quantity of adsorbed Fe^{3+} ions on the solid phase of activated carbon of *Leucas aspera* over the entire pH range of 3 to 10 for iron (Fig. 3). The solution pH plays a major role in determining the amount of Fe^{3+} ions adsorption. After the certain range of pH value the metal ion forming as hydroxides as follows:

$$M^{3+} + nH_2O \longrightarrow M(OH)_n^{3-n} + nH^+$$

It is evident that the adsorption of iron is increased regularly with increasing upto certain level of maximum pH.

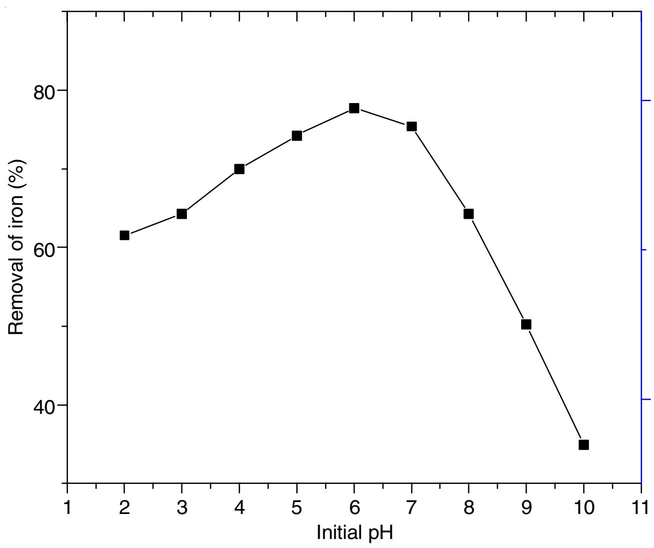

Fig. 3. Effect of pH on the removal of Fe(III) ion, [Fe] = 20 mg/L; Temperature = 30 °C; Adsorbent dose = 100 mg/50 mL

After that maximum, the adsorption capacity decreased due to formation of precipitate of metal hydroxides shown as above reaction. The maximum pH ~ 6.5, which reveals that the removal of Fe^{3+} due to only adsorption onto activated carbon of *Leucas aspera* not by precipitation.

Freundlich isotherm: The mathematical linear expression of Freundlich isotherm [20] is given by the expression as:

$$\log Q_f = \log K_f + (1/n) \log C_i$$

The value of K_f (adsorption capacity) n (adsorption intensity) can be calculated from the intercept and slope of the linear plot between log Q_f *vs*. log C_i, respectively. The value of n and K_f indicates that favourable adsorption condition [21-23]. The values of Freundlich parameters and it's statistical correlation value of r^2 which indicates that the fitness of model and listed in Table-3.

TABLE-3
LANGMUIR AND FREUNDLICH ISOTHERM PARAMETER FOR THE ADSORPTION OF Fe^{3+} ION ONTO ACTIVATED CARBON OF *Leucas aspera*

Temp. (°C)	Langmuir parameter		Fruendlich parameter		r^2
	Q_m	b	K_f	n	
30	38.651	0.2816	0.6049	1.687	0.9996
40	36.681	0.2149	0.6149	1.503	0.9999
50	32.243	0.1459	0.6241	1.416	0.9879
60	30.641	0.1346	0.6573	1.402	0.9999

Langmuir isotherm: Langmuir isotherm model [24] is actually based on the adsorption corresponds to a saturated monolayer of adsorption of adsorbate onto the surface of adsorbent. Mathematical linear expression of Langmuir written as:

$$C_f/Q_f = (1/Q_m b) + (Cf/Q_m)$$

The value of Langmuir constants such as Q_f (adsorption capacity) and b (rate of monolayer adsorption process) can be deduced by the slope and intercept of linear plot between C_f/Q_f (specific adsorption capacity) between C_f (final concentration), respectively. The values of Q_m and b with their statistical factor r^2 are listed in Table-3. It is possible to determine the feasibility of the Langmuir model, the isotherm can be expressed in terms of dimensionless separation factor G_L [25,26] given by the expression:

$$G_L = [1/(1+ bC_i)]$$

By following the expression, $G_L > 1$ unfavourable process, $0 < G_L < 1$ favourable process, $G_L = 0$ irreversible process, $G_L = 1$ linear adsorption.

The values of G_L between 0 to 1 reveals that favourable adsorption process for all initial concentration (C_i) and all fixed temperatures. The calculated G_L values are listed in Table-4.

TABLE-4
DIMENSIONLESS SEPARATION FACTOR (G_L) FOR THE ADSORPTION OF Fe^{3+} ION ONTO ACTIVATED CARBON OF *Leucas aspera*

C_i (ppm)	Temperature (°C)			
	30	40	50	60
10	0.29	0.31	0.32	0.33
20	0.17	0.18	0.19	0.20
30	0.12	0.13	0.14	0.14
40	0.09	0.10	0.11	0.11
50	0.07	0.08	0.09	0.09
60	0.06	0.07	0.07	0.08

Thermodynamic treatment of adsorption process: The thermodynamic parameters of adsorption such as standard free energy change (ΔG° expressed in terms of kJ/mol), standard enthalpy change (ΔH° expressed in terms of kJ/mol) and standard entropy change (ΔS° expressed in terms of J/K) were calculated as follows:

$$\Delta G^\circ = -RT \ln K_0$$

where, T is the temperature expressed in terms of Kelvin and R is the gas constant (8.314 J mol/K).The adsorption coefficient K_0 for the process was determined by the slope of the plot of ln (q_f/C_i) against C_f at different temperature according Khan and Singh [27]. The enthalpy change (ΔH°) and entropy change (ΔS°) can be expressed as a function of temperature, *i.e.*

$$\ln K_0 = (\Delta H^\circ/RT) + (\Delta S^\circ/R)$$

The value of ΔH° and ΔS° can be obtained by the slope and intercept of plot between ln K_0 against 1/T and listed in Table-5. The value of ΔG°, ΔH° and ΔS° from the Table-5 reveals that the adsorption of Fe^{3+} ion onto activated carbon of *Leucas aspera* is much favourable physisorption and endothermic nature. The increase in metal adsorption with increase in temperature indicates that the possibility of chemisorptions. The negative value of ΔG° indicates that the adsorption process is spontaneous, the positive value of ΔS° reveals that the randomness of the adsorption process increases during the adsorption of Fe^{3+} ion onto the activated carbon of *Leucas aspera*.

Adsorption kinetics: The kinetics study of adsorption explains about the rate and molecularity of the process between adsorbent-adsorbate interface. The various kinetics studies are available. Among those the pseudo second-order [28]. Elovich and intra-particle diffusion kinetic studies were analyzed. The well fitness of the models were expressed by parameters value with correlation coefficient (γ) and the values are near to unity (1). The high value of correlation coefficient (γ) reveals that the pseudo second-order model successfully explains the kinetics of Fe^{3+} ions adsorption onto the activated carbon of *Leucas aspera*.

Pseudo second-order equation: The integrated form of pseudo second-order adsorption kinetic rate equation with linear form is written as:

$$t/Q_t = (1/K_2Q_f^2) + (1/Q_f)t$$

where K_2 is the rate constant (g mg/min) and the initial adsorption rate (h)(mg $g^{-1}min^{-1}$) is:

$$h = K_2 Q_f^2$$

Then it follows as:

$$t/Q_t = (1/h) + (1/Q_f)t$$

The plot of (t/q_t) and t of eqn. 13 should give a linear relationship from which q_e and k_2 can be determined from the slope and intercept of the plot, respectively. The pseudo-second order rate constants (K_2), the calculated h values and the correlation coefficients (g) are summarized in Table-6 which reveals that the fitness of pseudo second order kinetic model.

Elovich equation: The differentiate form of Elovich model equation written as below:

$$dq_t/d_t = \alpha \exp(-\alpha q_t)$$

The above equation modified by Chien and Clayton [29] which describes the relationship between rate of initial adsorption (α) expressed in mg g^{-1} min^{-1} and desorption constant (β) expressed in g/mg at αβt >> 1.

$$Q_t = 1/\beta \ln(\alpha\beta) + 1/\beta \ln t$$

The value of α, β and correlation coefficient t (γ) is obtained by linear plot q_t *vs.* ln t should yield a slope (1/β) and intercept (1/β)ln (αβ) which are listed in Table-6. The values indicate that the fitness of Elovich model for adsorption on Fe^{3+} onto activated carbon of *Leucas aspera*.

Intra-particle diffusion model: Weber and Morris [30] proposed the rate equation for intra-particle diffusion process of adsorption as follows:

$$q_t = k_{id}t^{(1/2)} + C$$

The values of intra particle diffusion rate k_{id} (mg/g/min) and correlation coefficient (γ) is obtained from the slope of linear plot between q_t (mg/g) *vs.* ($t^{1/2}$) using origin software. The value of constant C reveals about thickness of the adsorption layer. The high value of correlation coefficient (γ) indicates that the fitness of model for adsorption of Fe^{3+} onto the surface of adsorbent (activated carbon of *Leucas aspera*) (Table-6).

FT-IR: The FTIR technique is used to confirm the adsorption process of metalo ions onto the surface of sorbent. The FT-IR spectra of before and after the adsorption the of activated carbon of *Leucas aspera* of Fe^{3+} shown in Fig. 4 (I) and Fig. 4(II) respectively. From Fig. 4(I and II), it is evident that the wide spetral range of Infra Red region confirms the adsorption of Fe^{3+} onto surface of activated carbon of *Leucas aspera* by physical attraction like van der Walls force between Fe^{3+} and activated carbon of *Leucas aspera* not by chemical forces like covalent or ionic forces.

XRD-evidence: The X-ray method is used to analyze the entire morphology of particles at different scattering angle. The interpretation of smaller particles at atomic level can be done by at wide range of scattered angels (WAXS). The XRD of WXAS of before and after adsorption of activated carbon of *Leucas aspera* on to Fe^{3+} shown in Fig. 5(I) and Fig. 5(II) respectively. The strong intense peak XRD reveals that the adsorption of Fe^{3+} ion well organized structure. This is

TABLE-5
THERMODYNAMIC PARAMETERS FOR THE ADSORPTION OF Fe^{3+} ION ONTO ACTIVATED CARBON OF *Leucas aspera*

C_i (ppm)	30 °C		40 °C		50 °C		60 °C		ΔH° (KJ/mol)	ΔS° (J/K)	r^2
	K_{ads}	ΔG° (KJ/mol)	K_{ads}	ΔG° (KJ/mol)	K_{ads}	ΔG° (KJ/mol)	K_{ads}	ΔG° (KJ/mol)			
10	3.993	-3489.6	4.368	-3838.4	5.998	-4812.9	7.013	-5394.9	341.8	3.1598	0.9976
20	3.591	-3222.1	3.994	-3605.3	4.859	-4247.2	5.788	-4863.2	302.4	2.8163	0.9989
30	3.531	-3179.7	3.743	-3436.4	4.542	-4065.9	5.149	-4539.2	246.1	2.0024	0.9949
40	3.039	-2801.5	3.539	-3290.4	4.023	-3738.8	4.859	-4378.6	198.3	1.8716	0.9968
50	2.301	-2100.4	2.909	-2780.1	3.528	-3387.2	3.946	-3802.1	141.2	1.2463	0.9999
60	1.981	-1722.9	2.653	-2540.2	3.031	-2979.2	3.529	-3492.8	109.7	1.0271	0.9968

TABLE-6
KINETIC PARAMETERS FOR THE ADSORPTION OF Fe^{3+} ION ONTO ACTIVATED CARBON OF *Leucas aspera*

C_i (ppm)	Temp. (°C)	Pseudo-second order				Elovich model			Intra-particle diffusion		
		Q_f	K_2	γ	h	α	β	γ	K_{id}	γ	C
10	30	9.429	0.0168	0.9999	5.65	1.42	0.742	0.9923	0.382	0.9946	0.160
	40	10.252	0.0249	0.9988	6.25	1.99	0.816	0.9999	0.342	0.9951	0.150
	50	12.972	0.0379	0.9968	9.49	3.83	0.986	0.9987	0.313	0.9953	0.130
	60	15.189	0.0561	0.9999	11.24	6.15	1.015	0.9961	0.271	0.9999	0.120
20	30	13.467	0.0199	0.9963	9.43	2.52	0.542	0.9943	0.652	0.9952	0.150
	40	16.217	0.0243	0.9976	12.66	6.49	0.566	0.9916	0.610	0.9983	0.160
	50	17.431	0.0301	0.9999	18.72	12.71	0.592	0.9989	0.576	0.9988	0.140
	60	18.146	0.0312	0.9986	20.24	18.26	0.606	0.9999	0.521	0.9999	0.120
30	30	16.634	0.0248	0.9988	19.46	11.26	0.506	0.9969	0.716	0.9953	0.160
	40	18.147	0.0205	0.9999	22.31	26.76	0.519	0.9958	0.659	0.9997	0.170
	50	21.402	0.0198	0.9986	29.40	40.21	0.546	0.9978	0.605	0.9979	0.130
	60	23.146	0.0385	0.9999	39.16	51.26	0.623	0.9963	0.572	0.9949	0.140
40	30	22.401	0.0294	0.9999	25.14	24.35	0.392	0.9949	0.846	0.9968	0.120
	40	25.249	0.0305	0.9979	29.65	49.16	0.409	0.9975	0.813	0.9999	0.125
	50	28.315	0.0298	0.9954	54.21	115.59	0.483	0.9913	0.719	0.9998	0.120
	60	30.215	0.0316	0.9969	71.29	167.20	0.540	0.9996	0.636	0.9999	0.110
50	30	33.149	0.0398	0.9999	42.10	102.39	0.381	0.9998	0.972	0.9919	0.110
	40	35.143	0.0421	0.9973	50.29	459.13	0.426	0.9949	0.943	0.9962	0.130
	50	37.098	0.0406	0.9997	68.45	871.29	0.584	0.9999	0.842	0.9986	0.136
	60	41.173	0.0362	0.9946	82.19	1029.46	0.629	0.9958	0.806	0.9999	0.140
60	30	37.981	0.0513	0.9987	49.27	875.16	0.216	0.9979	1.216	0.9989	0.160
	40	42.105	0.0501	0.9956	59.46	1198.21	0.252	0.9923	1.120	0.9986	0.163
	50	44.579	0.0486	0.9999	72.36	1452.04	0.320	0.9911	1.029	0.9999	0.170
	60	49.173	0.0421	0.9998	89.56	687.26	0.342	0.9999	1.009	0.9995	0.173

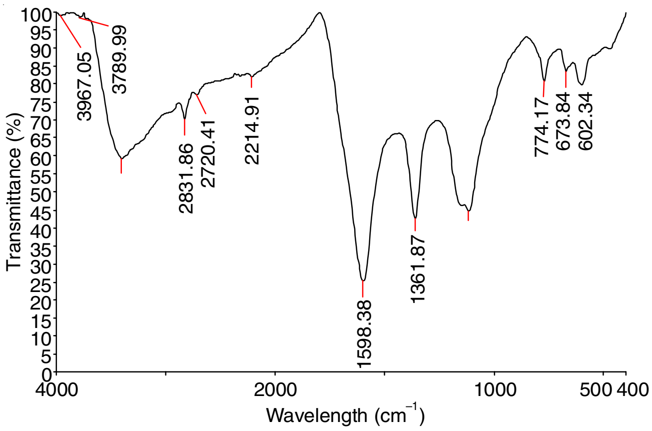

Fig. 4(I). FT-IR Spectrum of activated carbon of *Leucas aspera* before adsorption

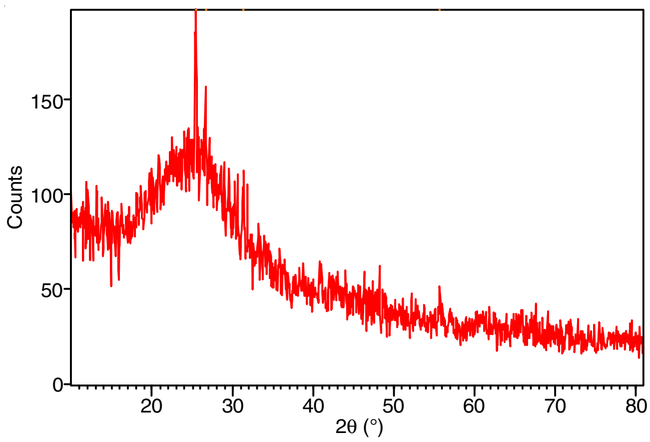

Fig. 5(I). XRD pattern activated carbon of *Leucas aspera* before adsorption

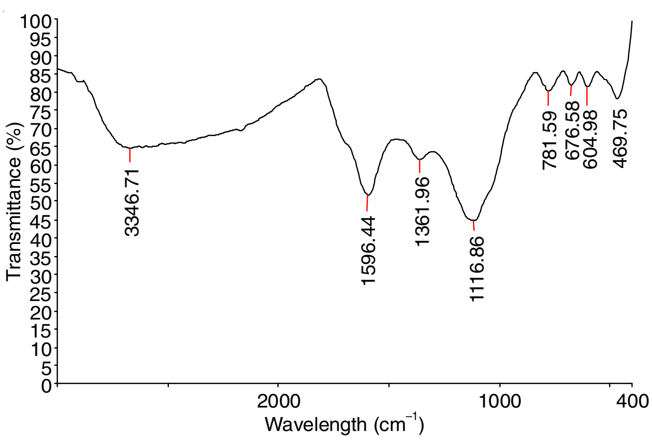

Fig. 4(II). FT-IR spectrum of activated carbon of *Leucas aspera* after adsorption of Fe^{3+}

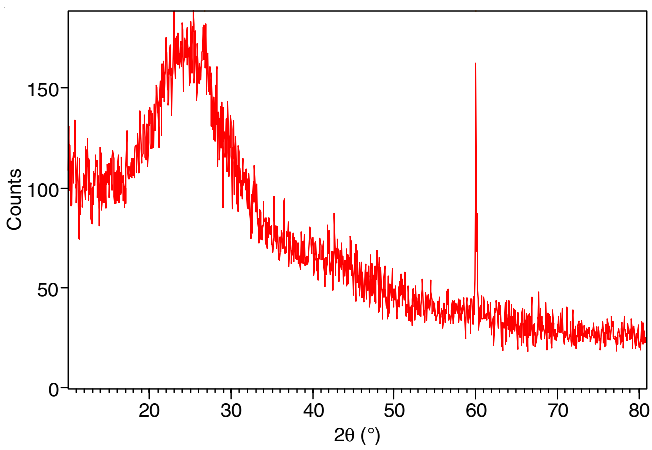

Fig. 5(II). XRD pattern of activated carbon of *Leucas aspera* after the adsorption of Fe^{3+}

confirmed that the adsorption of Fe^{3+} ion on the activated carbon of *Leucas aspera* surface by physisorption.

Conclusions

From the above experimental evidence the adsorption of Fe^{3+} ions onto the surface of activated carbon of *Leucas aspera*, the following points are concluded.

• The activated carbon prepared from *Leucas aspera* is an effective potential for removing Fe^{3+} ion from aqueous solution.

• The adsorption process of Fe^{3+} ion onto the surface of activated carbon of *Leucas aspera* depend upon the various factors such as concentration, dosage, contact time.

• The pH's of solutions affect the removal of Fe (III) ion on activated carbon of *Leucas aspera* surface up to certain limits of pH say pH ~ 6.5.

• The adsorption percent removal of Fe^{3+} ion decreased with rising initial concentration but increased with rising adsorbent concentration.

• The adsorption process for well fit with Freundlich and Langmuir isotherm.

• The values of various thermodynamic parameters like negative $\Delta G°$ indicate that the sorption process is physisorption.

• The sorption process of Fe^{3+} on activated carbon of *Leucas aspera* also follows that various kinetic model like pseudo second order, Elovich and intra-particle diffusion.

• Finally the mechanism of adsorption for removal percentage of Fe^{3+} is confirmed by FT-IR and XRD studies for before and after adsorption of activated carbon of *Leucas aspera*.

• All the parameters like $\Delta G°$, $\Delta H°$, $\Delta S°$ and kinetic parameters are calculated by using origin.

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