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Activated Carbon *Merremia emarginata* (Acme) Adsorbs Hexavalent Chromium From Synthetic Aqueous Solutions

K. Kalpana[1], S. Arivoli*[2] and K. Veeravelan[3]

[1]PG and Research Department of Chemistry, M. R. Government Arts College,
(Affiliated to Bharathidasan University-620024), Mannargudi, Tamilnadu, India
[*2,3] PG and Research Department of Chemistry, Poompuhar College (AU),
(Affiliated to Bharathidasan University-620024), Melaiyur, Tamilnadu, India

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ABSTRACT

In this study, preparation of adsorbent from *Merremia emarginata* stem and its application for the adsorption of chromium (VI) from aqueous solution was studied. The batch adsorption experiments were carried out to study the effect of contact time, adsorbent dose, effect of pH and effect of other ionic concentration on the removal of chromium (VI) by activated carbon. The equilibrium adsorption data were analyzed by Freundlich and Langmuir isotherm models. The thermodynamic parameters such as change in free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) were determined. Kinetic modeling of the process of removal was carried out and the process of removal was found to follow a pseudo-second order model, Elovich model and Intraparticle diffusion model the value of rate constant for adsorption process was calculated. This study indicated that activated carbon *Merremia emarginata*, which is inexpensive, has efficiency to remove chromium (VI) from aqueous solution.

Key words: *Activated Carbon Merremia emarginata, (ACME), Chromium (VI) ion, Adsorption isotherm, Thermodynamic parameters, Kinetics, Equilibrium models.*

Introduction

Heavy metal ions have become an ecotoxicological hazard of prime concern and increasing significance, because of their accumulation in living organisms. Chromium (VI) is considered to be a bioavailable element due to its good solubility in water, strong oxidation properties and permeability through cell membranes. Unfortunately, chromium (VI) compounds cause toxic, mutagenic and carcinogenic effects on individual organisms (Arivoli *et al.*, 2010; Arivoli *et al.*, 2008). Chromium (VI) is one of the most toxic and its effects are carcinogenic, mutagenic, and teratogenic to humans and animals. The removal of these toxic metals from aqueous solution has a significant effect over the past decades on the reduction and their impact on the environment (Arthur *et al.*, 2009; Duwiejuah *et al.*, 2017). Several physical and chemical methods have been technologically advanced for the removal of toxic metals from aqueous solution. It is more and more often observed that their permissible concentration in surface waters is exceeded several times, which is why it is extremely important to search for effective and economical methods of removing them. Adsorption is one of the more effective, reusability of material, low- cost, ease of process, short time and eco-friendly approach. Activated carbon is the most widely used adsorbent and can be prepared from biomass (Houmei Liu *et al.*, 2017; Jun-jie Gao *et al.*, 2013). In this research, activated carbon prepared from *Merremia emarginata* stem, modified with conentrated H_2SO_4. Batch method static conditions, adsorption isotherms, thermodynamic parameters and adsorption kinetics were determined.

EXPERIMENTAL

The chemicals used in these experimental activities were all of analytical grade. The standard stock solution of chromium (1000 mg/l) was prepared by dissolving calculated amount of 99.9% analytical-grade $K_2Cr_2O_7$ in 1000 ml of distilled water. All the required solutions are made ready with analytical-grade reagents and double- distilled water. All experimental solutions were prepared by diluting the stock solution to the required concentration.

Preparation of adsorbent

The present work, a locally available plant, known by the local community as *Merremia emarginata*, was used as an adsorbent and this natural biomaterial is abundantly available at local area of Poompuhar. The stem of *Merremia emarginata* was washed with distilled water to remove the dirt and dust, successively dried in a hot air oven at 110 ^{o}C. Afterward, carbonization of the stem was carried out by adding w/v ratio Con. H_2SO_4 to get the primary carbon. The primary carbon was activated by thermal under mandatory conditions.

Batch experiments

The method of batch experiments (Kismir and Aroguz, 2011; Klimiuk *et al.*, 2003) were conducted to study the influence of important parameters like contact time, adsorbent dose, initial pH and ionic strength on the removal of Cr (VI) ions onto activated carbon *Merremia emarginata*. For Adsorption Isotherms, Cr (VI) ion solution of different concentrations (10-50 mg/l) and at different temperatures (30-60 ^{o}C) with known pH and known amount of adsorbent (0.025 g/l) were agitated at 120 rpm until the equilibrium was reached then the solution was kept to settle down and the residual concentration of Cr (VI) ion were analyzed by UV-Visible spectrophotometer at 540 nm. The amount of adsorption at time t, qt (mg/g), can be determined using the following formula,

$$q_t = V \times \frac{(C_0 - C_t)}{w} \quad .. (1)$$

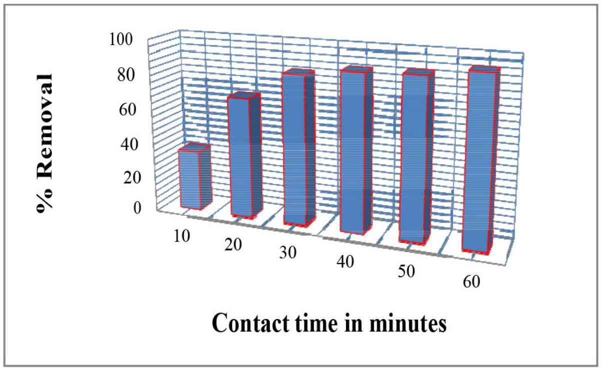
Where, q(t) is the mass of adsorbed Cr (VI)ion per unit mass of adsorbent (mg g -1), V is the volume of the treated solution (ml), w is the mass of adsorbent (g), (C0) and (Ct) are the initial and actual concentration (g dm-3) of Cr (VI)ion at time, respectively. The percentage of removal can be calculated as following mathematical formula,

$$\% \text{ Removal} = \left(\frac{C_0 - C_t}{C_0}\right) \times 100 \quad . (2)$$

Results and Discussion

Effect of contact time

The effect of contact time (Lanin *et al.*, 2017 ; Li *et al.*, 2009) on the removal of Cr (VI) on to ACME adsorbent and to determine the optimum contact time between the adsorbate and adsorbent, the experiment was carried out at room temperature and altered the contact time like 10, 20, 30, 40, 50 and 60 min. using 25 mg adsorbent dose and by optimal pH of the solution. These results were shown in Fig. 1 and the equilibrium data are given in Table 1. The Fig. 1 shows that the removal of Cr (VI) was initially rapid at that time maximum of the adsorption was complete with in 30 min; equilibrium was achieved with in 50 min. therefore all subsequent experiments were selected for 60 min.

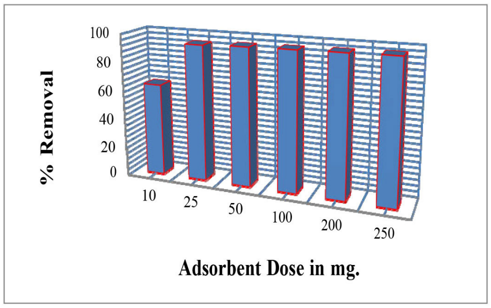


[Cr (VI) ion,]=10 mg/l; Temperature 30 ^{o}C; Adsorbent dose=0.025g /50 ml

Fig. 1. Effect of contact time on the removal of Cr (VI) ion

Effect of adsorbent dosage

The effect of ACME dose was studied by varying amount of adsorbent like 10, 25, 50, 100, 200 and 250 mg the adsorbent dose (Liu *et al.*, 2010) for 10 mg/l of Cr (VI) ion solution. The percentage of adsorption increased with increases in the ACME dose, which is attributed to increased carbon surface area and the availability of more adsorption sites. The results obtained from this study are shown in Fig. 2, it reveals that 25 mg of adsorbent dose enough to each experiments.

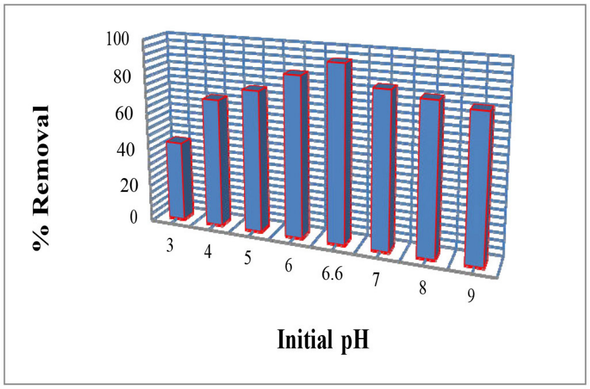


[Cr (VI) ion]=10mg/l; Temperature 30 ˚C; Contact Time 60 min

Fig. 2. Effect of Adsorbent dose on the Removal of Cr (VI) ion

Effect of pH

Effect of solution pH (Maria *et al.*, 2019) was studied in the pH range of 3-9 for removal of Cr (VI) ion from aqueous solution and keep on other parameters are constant. The percentage removal maximum at pH 6.8 that is pH zpc (Zero point charge) shown in Figure 3. The pH zpc of any adsorbent is a very important characteristic at which the surface has net electrical neutrality there is nocompetitive effect. The neutral surface of adsorbent occur support for the maximum removal of Cr (VI) ion from aqueous solution, as the result pH of solution was optimized as pH 6.8.

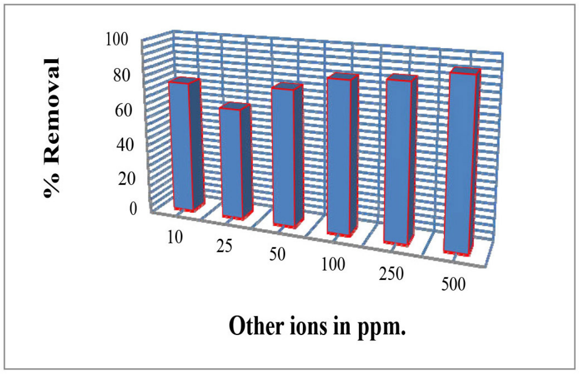


[Cr (VI) ion]=10mg/l; Temperature 30°C; Adsorbent dose= 0.025g/50 ml

Fig. 3. Effect of Initial pH on the Removal of Cr**(VI) ion**

Effect of ionic strength

The effect of ionic strength (Soltzberg *et al.*, 2007) was determined using different concentrations of chlorine solution such as 10, 25, 50, 100, 250 and 500 mg/l, and these results are shown in Fig. 4. Chlorine concentrations range from 10 to 25 mg when the removal capacity decreases reveals an increase in competitive absorption. Here after increases the ionic strength of solution as well as removal capacity for that reason an electric double layer is formed on the adsorbent.



[Cr (VI) ion]=10mg/L; Contact time=60 min.; Adsorbent dose= 0.025g/50mL.

Fig. 4. Effect of ionic strength on the removal of Cr (VI) ion

Adsorption isotherm Models

The adsorption isotherm study was reveals that adsorption mechanism, the Freundlich model (Syed Qummer Zia Gilani *et al.*, 2021) which is an indicative of surface heterogeneity of the adsorbent is described by the following equation,

$$log q_e = log K_f + \frac{1}{n_f} log C_e \qquad (3)$$

Where, Kf and 1/nf are Freundlich constants related with adsorption capacity and adsorption intensity respectively. The Freundlich plots drawn between log qe and log Ce for the adsorption of Cr (VI)ion were shown Figure 5, as a result listed in Table 2.

The Langmuir adsorption isotherm (Vijayakumaran *et al.*, 2009) equation which is valid for monolayer adsorption on to a surface is given below,

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \qquad (4)$$

Where, qe (mgg-1) is the amount adsorbed at the equilibrium concentration Ce (mol L-1), qm (mgg-1)

Table 1. Equilibrium parameters for the adsorption of Cr (VI) ion onto ACME

C_0	C_e (Mg /l)				q_e (Mg /l)				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	0.994	0.806	0.538	0.511	18.011	18.388	18.924	18.979	90.056	91.938	94.619	94.893
20	3.494	2.689	2.43	1.882	33.013	34.623	35.14	36.236	82.531	86.556	87.85	90.59
30	6.146	5.86	5.106	4.731	47.707	48.28	49.787	50.538	79.512	80.467	82.979	84.23
40	9.429	8.623	8.179	7.672	61.142	62.755	63.642	64.656	76.428	78.443	79.552	80.82
50	17.146	16.178	15.88	15.634	65.708	67.645	68.24	68.732	65.708	67.645	68.24	68.732

is the Langmuir constant representing the maximum monolayer adsorption capacity and KL (L mol-1) is the Langmuir constant related to energy of adsorption. The plots drawn between Ce/qe and Ce, shown in figure 6, the feasibility of isotherm by the essential characteristics of the Langmuir isotherm can be expressed in terms of dimensionless constant separation factor RL for the following equation.

$$R_L = \frac{1}{1 + K_L C_0} \quad ..(5)$$

Where, Co (mg/L) is the initial concentration of Cr (VI) ion and KL (L/mg) is Langmuir isotherm constant. The parameter RL indicates the nature of shape of the isotherm accordingly.

RL> 1 -Unfavorable adsorption 0 < RL< 1-Favorable adsorption RL = 0 - Irreversible adsorption RL = 1-Linear adsorption

The RL values in the middle of 0 to 1 indicate favorable adsorption for all initial concentration (Co) and temperatures studied. The calculated RL values are given in Table 3.

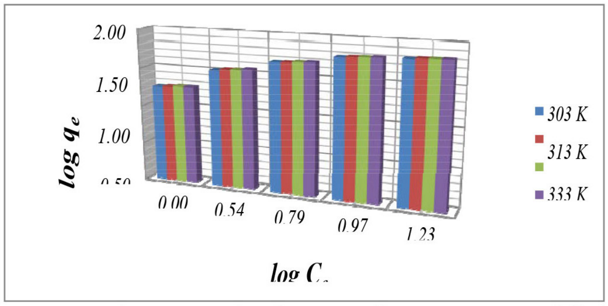


Fig. 5.1 Freundlich adsorption isotherm for the removal of Cr (VI) ion

Table 3. Dimensionless separation factor (RL) for the adsorption of Cr (VI) ion onto ACME

	Dimensionless separation factor(RL)			
(C0)	30 °C	40 °C	50 °C	60 °C
10	0.2967	0.2465	0.1924	0.1623
20	0.1742	0.1406	0.1065	0.0883
30	0.1233	0.0983	0.0736	0.0607
40	0.0954	0.0756	0.0562	0.0462
50	0.0778	0.0614	0.0455	0.0373

Thermodynamic treatment of the adsorption process

Thermodynamic (Xin-jiang Hu *et al.*, 2011) parameters studied the following equations, is physical adsorption process. The positive value of ΔH° further confirms the endothermic nature of adsorption process. The positive values of ΔS° showed increased randomness of the solid solution interface during the adsorption of Cr (VI) ion onto ACME.

$$\Delta G^0 = -RT\ln K_0 \quad (6)$$

$$\ln K_o = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (7)$$

Where, ΔG0 is the free energy of adsorption (kJ/mol), T is the temperature in (K), R is the universal gas constant (8.314 J mol/K), ΔH° is the standard heat change of sorption (kJ/mol) and ΔS° is stan-

Table 2. Freundlich and Langmuir isotherm parameter for the adsorption of Cr (VI) ion onto ACME

Model	Constant	Temperature (°C)			
		30	40	50	60
Freundlich	K_f (mg/g) (l/mg)$^{1/n}$	18.583	21.3	24.898	26.532
	n_f	2.068	2.204	2.497	2.558
	R^2	0.974	0.977	0.98	0.969
Langmuir	q_m (mg/g)	82.696	81.55	78.463	77.365
	K_L (l/mg)	0.237	0.306	0.42	0.516
	R^2	0.985	0.99	0.99	0.995

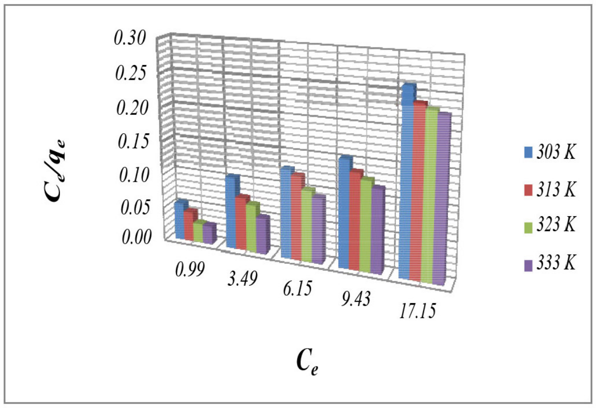


Fig. 5.2. Langmuir adsorption isotherm for the removal of Cr (VI) ion

dard entropy change (kJ/mol). The resultant data shown in Table 4, negative ΔG° values were confirming the spontaneous nature of adsorption Cr (VI) ion onto ACME. The lesser values of ΔG° suggest that adsorption.

Kinetic models

Pseudo-second-order equations can be used assuming that the measured concentrations are equal to surface concentrations. The linearized form of pseudo second order equation becomes.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right) t \quad (8)$$

Where, qt (mgg-1) is the amount of adsorbed Cr (VI)ion on the adsorbent at time t, qe the equilibrium sorption uptake and k2, is the rate constant of pseudo- second-order adsorption (min-1). The plots drawn between t/qtand t gives a straight line says second order kinetic model is applicable then q_e and

Table 4. Thermodynamic parameter for the adsorption of Cr (VI) ion onto ACME

(C0)	ΔG°				ΔH°	ΔS°
	30 °C	40 °C	50 °C	60 °C		
10	-5550.905	-6333.656	-7698.83	-8090.241	21.8	90.3
20	-3911.632	-4846.194	-5312.53	-6269.767	18.9	75.4
30	-3416.174	-3684.168	-4254.08	-4638.467	9.43	42.2
40	-2963.232	-3361.318	-3648.22	-3982.104	7.17	33.5
50	-1638.164	-1919.154	-2053.87	-2180.548	3.72	17.8

Table 5. The kinetic parameters for adsorption of Cr (VI) ion onto ACME

C_0	Temp °C	Pseudo second order				Elovich model			Intraparticle diffusion		
		q_e	k_2	R^2	h	α	β	R^2	K_{id}	α	R^2
10	**30**	19.8804	0.00669	0.9942	2.6446	27	0.361	0.9592	43.1899	0.175	0.9729
	40	19.7794	0.00912	0.9965	3.5675	120	0.445	0.9592	52.1091	0.135	0.97
	50	19.7595	0.0154	0.9986	6.0213	6700	0.667	0.9592	66.317	0.0844	0.9661
	60	19.764	0.0164	0.9988	6.4198	13000	0.703	0.9592	6.2444	0.0796	0.9658
20	**30**	36.2654	0.00386	0.9945	5.0708	63	0.205	0.9549	40.9415	0.1668	0.9684
	40	38.02	0.00351	0.9916	5.0699	61	0.195	0.9288	42.3489	0.1683	0.947
	50	38.3935	0.00401	0.996	5.9058	97	0.205	0.9601	45.8455	0.1556	0.9705
	60	38.9169	0.00452	0.9952	6.8519	220	0.226	0.9431	5.5152	0.1356	0.9567
30	**30**	51.4858	0.00292	0.9887	7.7374	200	0.165	0.857	42.9132	0.1421	0.8781
	40	52.2172	0.0037	0.9974	10.097	360	0.171	0.9402	46.7354	0.1323	0.9462
	50	54.4127	0.00312	0.9978	9.2507	170	0.147	0.9677	44.6012	0.1516	0.9733
	60	53.9586	0.00456	0.9999	13.283	640	0.173	0.9889	5.5192	0.1246	0.9848
40	**30**	66.0197	0.00335	0.9996	14.596	340	0.127	0.9734	43.5745	0.1414	0.9692
	40	67.6271	0.00314	1	14.379	350	0.124	0.9905	44.4777	0.1411	0.9855
	50	68.3123	0.00337	1	15.711	470	0.127	0.982	46.3373	0.1351	0.9761
	60	69.3416	0.00337	0.9999	16.209	540	0.127	0.9835	5.3504	0.1326	0.9783
50	**30**	68.4645	0.00531	0.9997	24.878	42000	0.2	0.999	47.2657	0.0805	0.9996
	40	70.8405	0.00424	0.9991	21.3	13000	0.176	0.9834	46.6208	0.0895	0.9878
	50	70.6735	0.00541	0.9991	27.004	400000	0.229	0.9426	51.4173	0.0671	0.9481
	60	71.6314	0.00516	0.9998	26.489	47000	0.192	0.998	5.4479	0.0802	0.9982

k_2 are determined from the slope and intercept of the plots respectively. The high regression value indicates the adsorption reaction exist a pseudo-second-order and these values are shown in Table 5.

The Elovich model equation is generally expressed as,

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t \qquad (8)$$

Where α is the initial adsorption rate (mg g-1 min-1) and β is desorption constant (g/mg) during any one experiment. If Cr (VI) ion adsorption fits with the Elovich model, a plots drawn between qt and ln(t) yields a linear relationship with a slope of (1/β) and an intercept of (1/β)ln(αβ). The Elovich model parameters α, β, and correlation coefficient (R^2) are summarized in Table 5. This model indicates that the initial adsorption (α) increases with temperature similar to that of initial adsorption rate (h) in pseudo-second– order kinetics models. Kinetic data was further analyzed using the intra-particle diffusion model based on the following equation.

$$\log R = \log K_{id} + \alpha \log t \qquad (9)$$

Where, kid is the intra-particle diffusion rate constant and it's related to the thickness of the boundary layer. According to above equation a plots drawn between log R and log t gives a straight line that's says the adsorption mechanism follows the intra-particle diffusion process in addition to the evidence of correlation co-efficient values are close to unity.

Conclusion

Due to its effectiveness in chromium ion adsorption from synthetic aqueous solution, this study advised using activated carbon *Merremia emarginata* as an alternative adsorbent for removal of heavy metal ions from the industrial effluents. The maximal monolayer adsorption capacity (qm) was determined to be 82.696 mg/l and the isotherm equilibrium values suited the Langmuir isotherm model well. Thermodynamic parameters demonstrated that the spontaneity of the Cr (VI) ion adsorption process was represented by the negative value of ΔG°, and the endothermic nature and growing randomness of the adsorption were shown by the positive values of ΔH° and ΔS°. Pseudo-second order, Elovich model, and intra-particle diffusion models all performed well in fitting the adsorption data, showing that a chemical reaction is involved in the adsorption process. It was discovered that three steps of diffusion processes governed the adsorption process. This work provides proof that ACME, or activated carbon *Merremia emerginata*, was an effective and efficient way to remove Cr (VI) ions from aqueous solutions.

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