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Removal of Fe (II) ion from aqueous solution by sorption onto Activated *Orthosiphon aristatus* carbon (AOAC)

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ABSTRACT

This study deals with preparation and efficient utilization of activated *Ortho siphonaristatus* stem carbon (AOAC) for the removal of Fe (II) from aqueous solution. Batch experiments were conducted to analyze the effect of adsorption parameters viz, contact time, adsorbent dose, pH of solution and other ionic concentration on adsorption process. The results exposed that bivalent iron removal increased slowly but surely up to a maximum of 92.043% and removal gets maximum at 60 minutes. Complete removal of Fe(II) was observed at a dosage of 25 mg/50 ml of pH 6.7 and 60 minutes of contact time. Experimental data were fitted with Langmuir, Freundlich isotherm models and was observed to be better explained by Langmuir adsorption model and the monolayer adsorption was 56.324 mg/g of Fe (II) on AOAC. The 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. The thermodynamic parameters such as change in free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) were determined.

Key words : *Activated Orthosiphon aristatus carbon (AOAC), Ironbivalent ion (Fe(II)), Adsorption isotherm, Kinetics, Equilibrium models and Thermodynamic parameters.*

Introduction

Industries are given heavy metal pollutants into the environment at an extraordinary and ever-increasing rate. Due to their mobility and their toxicity in natural water ecosystems, the presence of heavy metals in surface water and groundwater causes major mineral pollution problems. Of those metal ions, even at low concentrations of iron produces undesirable effects on human and animal life. The removal of these iron bivalent ions from aqueous solution was a significant. Several physical and chemical methods have been technologically advanced for the removal of toxic metals from aqueous solution. Physical methods, primarily adsorption on several supports were recognized to be a promising and in effect process to remove metal ions from aqueous solution completely (Arivoli *et al.,* 2007; Arivoli). The foremost benefits of adsorption are the reusability of material, low-cost, ease of process and short time of action so it is necessary to develop in effect adsorbents for the removal of Fe (II) ion from aqueous solution. Activated carbon is the most widely used adsorbent and can be prepared by physical activation in which carbonation or pyrolysis of the carbonaceous material at higher temperature (>900°C) in an inert atmosphere followed by thermal activation. Several methods have been studied for the preparation of activated carbon from agricultural wastes (Vadivelan and Vasanthkumar, 2005; Wang *et al.*, 2015).

In this work, the adsorption of Fe (II) ion on activated carbon prepared from *Orthosiphon aristatus* stem by carbonization with concentrated. H_2SO_4. The kinetic and equilibrium adsorption data obtained were utilized to illustrate the sample systematized. The amounts and rates of adsorption of Fe (II) ion using above activated carbon from aqueous solution were then measured. The literature survey shows that no work has been done so for lift up the *Ortho siphonaristatus* stem as an adsorbent.

Experimental Methods

All chemicals used in the work were of analytical (AR) quality and were obtained from the scientific equipment company at Trichy. The prepared stock solutions dissolve the required amount of ferrous sulfate ($FeSO_4.7H_2O$) in 1000 ml of deionized water. All test solutions were prepared by diluting the stock solution to the required concentration.

Preparation of adsorbent

Ortho siphonaristatus stem were collected from east coastal area of Mayiladuthurai district and washed several times by de-ionized water in order to remove the primary impurities. The stem was placed in concentration H_2SO_4 (w/v) for 24 hours for carbonization and increasing the adsorbent's porosity. The prepared sample was washed several times by deionized water, grounded well to fine powder and sieved by a mesh. Finally, this was activated around 900 ^{0}C in muffle furnace for 12 hours to obtain the activated carbon (AOAC).

Batch adsorption method

To study the effect of effective parameters contact time, adsorbent dose, initial pH and other ions concentration on the adsorptive removal of Fe (II) ion, batch experiments were conducted. For each experimental run, 50 ml of different concentrations of the metal ion solution (10-50 mg/l) was agitated with 0.025 g of the adsorbent at 120 rpm until the equilibrium was achieved. Samples were withdrawn at different time intervals (15, 30, 45 and 60 minutes) and kinetics, isotherm and other parameters of adsorption was determined by analyzing the remaining dye concentration from aqueous solution. In order to evaluate the effect of the initial pH on Fe(II) ion adsorption, the equilibrium study was conducted at different pH levels 3, 4, 5, 6, 7, 8 and 9 and other equilibrium studies were continued at the optimum pH 6.7 that is pH_{ZPC}. The pH of the solutions was adjusted by adding 0.01 N aqueous solutions of NaOH and HCl. The amount of adsorption at time t, q_t(mg/g), and the percentage removal of Fe (II) ion were calculated using the following equation correspondingly.

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

Where, $q_{(t)}$ is the mass of adsorbed Fe (II) ion per unit mass of adsorbent (mg g^{-1}) (C_0) and (C_t) are the initial andactual concentration (g dm^{-3}) of Fe (II)ion at time, respectively V is the volume of the treated solution (ml), w is the mass of adsorbent (g). The removal percentage of Fe (II) ion can be calculated as following relationship.

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

Based on the adsorption kinetics experiments, the process time for the equilibrium adsorption experiments was chosen, long enough assuming that the considered sorbent/sorbate system is equilibrated. The following parameters of the process were changed during these experiments kind and amount of adsorbent (AOAC) the particle size: The initial pH of the solutions and the initial concentration of the considered ions, all experiments were performed at ambient temperature.

Results and Discussion

The effective primary parameters

Figure 1 illustrates the effects of contact time on the removal of Fe (II) ion, the adsorption of copper ions was rapid for the first 30 min because readily available active adsorption sites on the as surface. There after it continued at a slower rate and the maximum of adsorption equilibrium reached at 50 min as a result of saturation it as surface sites.Therefore, 60 minutes is sufficient for each subsequent experiment. The adsorption of the Fe (II) ion on AOAC was studied by varying the adsorbent dose (25–500 mg/50 ml) for 10 mg/l of metal ion concentration. The percentage of adsorption increased with increases in the AOAC 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 Figure 3. The amount of Fe (II) ion adsorbed per gram reduced with increase in the dosage of AOAC. This reveals that the direct and equilibrium capacities of Fe (II) ion are functions of the activated AOAC dosage and a dose of 25 mg of adsorbent is recommended for each experiment as a result of this particular trial. The effect of initial pH on adsorption of Fe (II) ion was studied from pH 3 to 9 at initial metal ion concentration of 10 mg/l, adsorbent dosage of 0.025 g and contact time of 60 min. Two possible mechanisms of adsorption of Fe (II) ion on the AOAC adsorbent may be considered include: (i) electrostatic interaction between the adsorbent and the Fe (II) ion, (ii) a chemical reaction between the Fe (II) ion and the adsorbent surface. When pH increases, the concentration of OH^- ions in the desired solution is increased, as well. This causes the surface of the AOAC to become deprotonated and, as a result, the negative charge of the used AOAC surface will be amplified. Therefore, the electrostatic attractive force between the Fe (II) ion, which has a positive charge and the adsorbent surface increases and consequently the rate of Fe (II) ion adsorption increases, as well. As Figure 3 depicts, the pH of the solution increased from 3 to 9, the rate of removal also increased up to pH_{zpc} 6.7 (Zero point charge) then decrease. Effect of chlorine ions [15] on the absorption process studied at different concentrations. The ions and contents added to the 50 mg/l Fe (II) ion solution were stirred at 30 °C for 60 min. The result shows in Figure 5 that the low concentration of Cl^- does not affect the percentage removal of Fe (II) ion, because the interaction of Cl^- at available sites of adsorbent through competitive adsorption is not so effective. While the concentration of other ions increases, the interference of these ions at the available surface sites of the sorbent in-

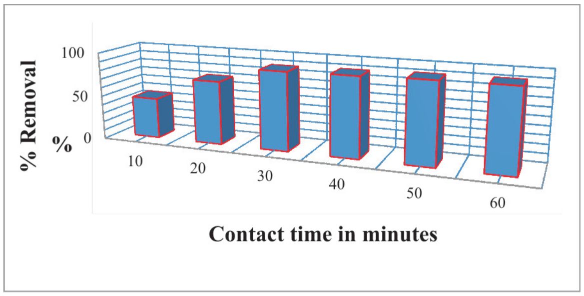

Fig. 1. Effect of Contact Time on the Removal of Fe (II)ion [Fe (II) ion]=10 mg/l; Temperature 30 °C; Adsorbent dose=0.025g/50 ml.

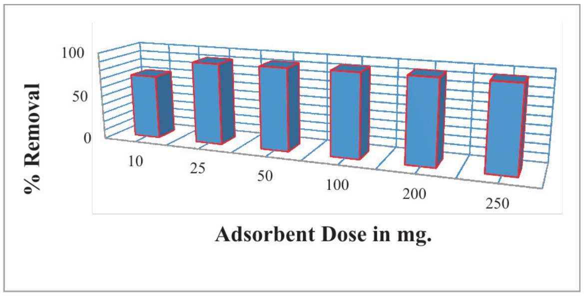

Fig. 2. Effect of Adsorbent dose on the Removal of Fe (II) ion [Fe (II) ion]=10 mg/l; Temperature 30 °C; Contact Time 60 min.

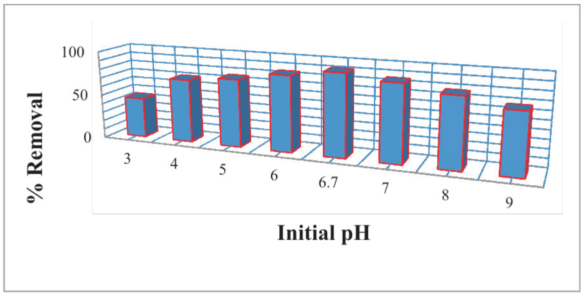

Fig. 3. Effect of Initial pH on the Removal of Fe (II) ion [Fe (II) ion]=10mg/l; Temperature 30 °C; Adsorbent dose=0.025g/50 ml.

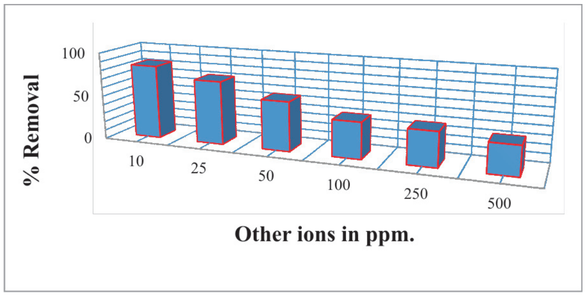

Fig. 4. Effect of other ionic strength on the removal of Fe (II) ion [Fe (II) ion]=10 mg/l; Contact time=60 min.; Adsorbent dose=0.025g/50 ml.

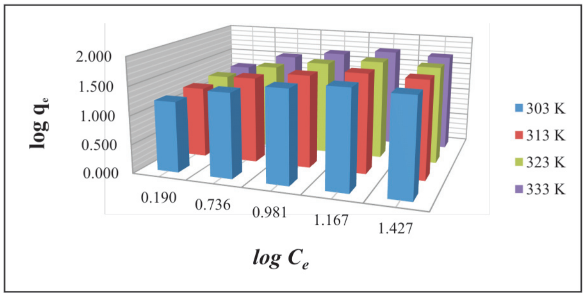

Fig. 5. Freundlich adsorption isotherm for the removal of Fe (II) ion

creases with competitive adsorption, which decreases the percentage adsorption.

Adsorption isotherm models

Adsorption properties and equilibrium parameters, commonly known as adsorption isotherms, describe how the adsorbate interacts with adsorbents, and comprehensive understanding of the nature of interaction. Several isotherm equations are available, and two important isotherms were selected for this study: the Freundlich and Langmuir models.

The linear form of Freundlich isotherm model is represented by the following equation:

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

Where, K_f and $1/n_f$ are Freundlich constants related with adsorption capacity and adsorption intensity respectively. The Freundlich plots drawn between log q_e and log C_e for the adsorption of Fe (II) ion were shown Figure 5. A linear form of the Langmuir isotherm model can be expressed as.

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

Where, q_e (mgg^{-1}) is the amount adsorbed at the equilibrium concentration C_e (mol L^{-1}), q_m (mgg^{-1}) is the Langmuir constant representing the maximum monolayer adsorption capacity and K_L (L mol^{-1}) is the Langmuir constant related to energy of adsorption. The plots drawn between C_e/q_e and C_e for the adsorption of Fe (II)ion was found linear and its shown in Figure 7. The values of monolayer capacity (q_m) and equilibrium constant (K_L) have been evaluated from the intercept and slope of these plots and given in Table 2.

Freundlich and Langmuir constant values and the correlation coefficients R^2 obtained from the linear regression are shown in Table 2. The equilibrium data analyzed using the linearized form of

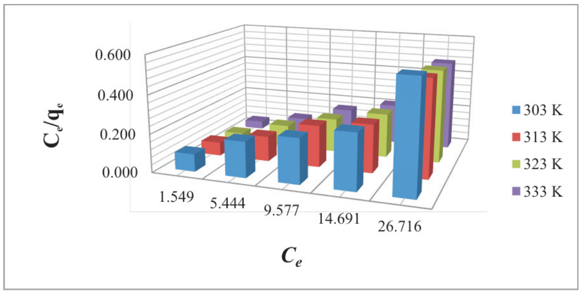

Fig. 6. Langmuir adsorption isotherm for the removal of Fe (II) ion

Table 3. Dimensionless Seperation Factor (R_l) for the Adsorption of Fe (II) ion onto AOAC

(C_0)	Temperature °C			
	30°C	40°C	50°C	60°C
25	0.2501	0.2124	0.1544	0.1225
50	0.1430	0.1188	0.0837	0.0652
75	0.1001	0.0825	0.0574	0.0445
100	0.0770	0.0632	0.0437	0.0337
125	0.0625	0.0512	0.0352	0.0272

Table 1. Equilibrium Parameters for the Adsorption of Fe (II) Ion Onto Aoac

C_0	Ce (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
25	1.549	1.256	0.839	0.796	16.901	17.488	18.323	18.409	84.506	87.438	91.615	92.043
50	5.444	4.189	3.786	2.932	29.113	31.621	32.427	34.135	72.781	79.053	81.068	85.339
75	9.577	9.131	7.956	7.372	40.846	41.739	44.087	45.256	68.077	69.565	73.479	75.427
100	14.691	13.435	12.744	11.954	50.617	53.129	54.511	56.091	63.271	66.411	68.139	70.114
125	26.716	25.207	24.743	24.360	46.568	49.586	50.513	51.280	46.568	49.586	50.513	51.280

Table 2. Langmuir and Freundlich Isotherm Parameter for the Adsorption of Fe (II) ion onto AOAC

Model	Constant	Temperature (°C)			
		30	40	50	60
Freundlich	K_f(mg/g) $(l/mg)^{1/n}$	15.081	17.393	20.601	22.081
	n_f	2.524	2.662	3.027	3.102
	R^2	0.918	0.928	0.932	0.904
Langmuir	q_m(mg/g)	54.214	56.324	55.502	55.493
	K_L (l/mg)	0.300	0.371	0.548	0.716
	R^2	0.979	0.987	0.989	0.992

Langmuir isotherm show a good linearity and the maximum adsorption capacity q_m were around 56.324 mg/g for AOAC.

In order to find out the feasibility of the isotherm, the essential characteristics of the Langmuir isotherm can be expressed in terms of dimensionless constant separation factor R_L by the equation.

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

Where, C_o (mg/L) is the highest initial concentration of Fe (II) ion and K_L (L/mg) is Langmuir isotherm constant. The parameter R_L indicates the nature of shape of the isotherm accordingly. $R_L>1$, $0<R_L<1$, $R_L=0$ and $R_L=1$ respectively unfavorable adsorption, favorable adsorption, irreversible adsorption and linear adsorption. The R_L values in the middle of 0 to 1 indicate favorable adsorption for all initial concentration (C_o) and temperatures studied and the calculated R_L values are given in Table 3.

Thermodynamic treatment of the adsorption process

The temperature is another important factor which influences the process of adsorption. The effect of temperature on adsorption of Fe (II) ion onto AOAC has been investigated at 303, 313, 323 and 333 K. Thermodynamic parameters for the adsorption are calculated using the equation:

$$\Delta G^0 = -\mathrm{RT}\ln \mathrm{K}_0 \qquad .. (5)$$

$$\ln \mathrm{K}_o = \frac{\Delta S^0}{\mathrm{R}} - \frac{\Delta H^0}{\mathrm{RT}} \qquad .. (7)$$

Where, ΔG^0 is the free energy of adsorption (kJ/mol), R is the universal gas constant (8.314 J mol/K), T is the temperature in Kelvin, K_0 is equilibrium constant (C_{solid}/ C_{liquid}), $\Delta S°$ is standard entropy change (kJ/mol) and $\Delta H°$ is the standard heat change of sorption (kJ/mol).

The values of $\Delta H°$ and $\Delta S°$ can be obtained from the slope and intercept of plot of $\ln K_0$ against 1/T and shown in Table 4. The negative ΔG° values were confirming the spontaneous nature of adsorption Fe (II)ion onto AOAC. The lesser values of ΔG° suggest that adsorption is physical adsorption process. The positive value of ΔH° confirms the endothermic nature of adsorption process. The positive values of ΔS° confirm the increased randomness of the solid solution interface during the adsorption of Fe (II) iononto AOAC.

Kinetic models

Kinetic study is important to the adsorption process because it depicts the uptake rate of Fe (II) ion and controls the residual time of the whole adsorption process. To explore the controlling mechanism of the adsorption process, the experimental data were tested for following kinetic models such as pseudo-second-order, Elovich model and intra-particle diffusion model.

The linearized form of pseudo second order equation becomes.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right) t \qquad .. (9)$$

Where, q_t (mgg^{1}) is the amount of adsorbed Fe (II)ionon the adsorbent at time t, q_e the equilibrium sorption uptake and k_2, (min^{-1}) is the rate constant of pseudo-second-order adsorption. The plot t/q_t versus t gives a straight line says second order kinetic model is applicable then q_e and $k_2$are determined from the slope and intercept of the plot, respectively. The Elovich model [21] equation is generally expressed as,

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

Where á is the initial adsorption rate (mg g^{-1} min^{-1}) and â is desorption constant (g/mg) during any one experiment. If Fe (II)ion adsorption fits with the Elovich model, a plot drawn between q_t and ln(t)

Table 4. Thermodynamic Parameter for the Adsorption of Fe (II) ion onto AOAC

(C_0)	ΔG°				ΔH°	ΔS°
	30°C	40°C	50°C	60°C		
25	-4273.444	-5048.969	-6421.231	-6777.971	22.785	89.357
50	-2477.708	-3456.130	-3905.826	-4876.588	20.645	76.492
75	-1907.812	-2151.217	-2736.598	-3105.051	10.755	41.604
100	-1370.109	-1773.933	-2041.394	-2360.873	8.451	32.507
125	-346.423	-43.110	-55.151	-141.757	5.106	15.904

Table 5. The Kinetic Parameters for Adsorption of Fe (II) ion onto AOAC

C_0	Temp	Pseudo second order				Elovich model			Intraparticle diffusion		
	°C	q_e	k_2	R^2	h	α	β	R^2	K_{id}	α	R^2
25	30	20.9534	2.86E-03	0.9811	1.2543	3.2E+00	2.32E-01	0.9592	21.9971	0.3221	0.9821
	40	20.1987	4.48E-03	0.9894	1.8295	7.6E+00	2.86E-01	0.9592	32.2346	0.2382	0.9771
	50	19.7908	8.63E-03	0.9962	3.3793	8.7E+01	4.28E-01	0.9592	50.5180	0.1416	0.9705
	60	19.7761	9.29E-03	0.9966	3.6324	1.3E+02	4.51E-01	0.9592	5.5916	0.1329	0.9698
50	30	36.4368	1.57E-03	0.9786	2.0822	5.1E+00	1.32E-01	0.9549	18.2892	0.3303	0.9793
	40	39.1323	1.47E-03	0.9687	2.2537	5.8E+00	1.25E-01	0.9288	20.4351	0.3202	0.9611
	50	39.1872	1.80E-03	0.9863	2.7614	8.1E+00	1.31E-01	0.9601	24.4668	0.2877	0.9782
	60	39.4272	2.20E-03	0.9848	3.4229	1.4E+01	1.45E-01	0.9431	4.4210	0.2426	0.9660
75	30	49.1870	1.24E-03	0.9491	3.0093	9.1E+00	1.06E-01	0.8570	19.5718	0.2889	0.8985
	40	49.6619	1.70E-03	0.9884	4.1966	1.4E+01	1.10E-01	0.9402	24.1237	0.2585	0.9519
	50	53.6930	1.38E-03	0.9911	3.9753	1.1E+01	9.41E-02	0.9677	22.3093	0.2920	0.9778
	60	51.8152	2.26E-03	0.9999	6.0552	2.4E+01	1.11E-01	0.9889	4.3613	0.2327	0.9806
100	30	60.7438	1.47E-03	0.9978	5.4327	1.4E+01	8.13E-02	0.9734	19.8470	0.2924	0.9639
	40	63.2280	1.41E-03	0.9999	5.6198	1.5E+01	7.97E-02	0.9905	21.1541	0.2854	0.9795
	50	63.9302	1.56E-03	0.9997	6.3663	1.9E+01	8.17E-02	0.9820	23.3932	0.2679	0.9697
	60	65.3924	1.59E-03	0.9997	6.7980	2.2E+01	8.18E-02	0.9835	4.0475	0.2583	0.9728
125	30	51.9010	2.62E-03	0.9989	7.0692	5.1E+01	1.28E-01	0.9990	21.3716	0.1908	0.9996
	40	55.9236	2.05E-03	0.9957	6.3997	3.7E+01	1.13E-01	0.9834	21.0608	0.2066	0.9926
	50	55.0217	2.83E-03	0.9952	8.5815	1.7E+02	1.47E-01	0.9426	26.8783	0.1500	0.9545
	60	56.7458	2.63E-03	0.9992	8.4614	7.5E+01	1.23E-01	0.9980	4.0304	0.1790	0.9979

with a slope of (1/β) and an intercept of (1/β) ln(αβ) are obtained.

The Elovich model parameters α, β, and correlation coefficient (R^2) a re 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. This may be due to increase the pore or active site on the AOAC adsorbent.

Intra-particle diffusion model [22] based on the following equation.

$$\log R = \log Kid + a \log t \qquad .. (9)$$

Where, k_{id} is the intra-particle diffusion rate constant and it's related to the thickness of the boundary layer and according to above equation a plot drawn between log R and log t.

The kinetics studies constant values and the correlation coefficients R^2 calculated and shown in Table 2. The experiment data are much related to pseudo-second-order because the R^2 values are close to 1.

Conclusion

In this study, natural activated *Orthosiphon aristatus* stem nano carbon was tested and evaluated as a possible adsorbent for removal of Fe (II) ion from its aqueous solution using batch sorption technique. The adsorption process is also dependent on effective factors such as contact time, adsorbent dose, the solution pH, other ionic concentration. The maximum removal was found between the pH ranges 3.0-6.7. The isotherm study indicates that the sorption data can be modeled by both Freundlich and Langmuir isotherms.. The adsorption kinetics followed pseudo-second-order kinetic model, Elovich model and intra-particle diffusion. Thermodynamic analysis suggests that the removal of Fe (II) ion from aqueous solution by AOAC was a spontaneous and endothermic process. The present findings suggest that AOAC may be used as an inexpensive and effective adsorbent without any treatment or any other modification for the removal of metal ions from aqueous solutions.

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