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Removal of Heavy Metals from Wastewater Using *Tribulus terrestris* Herbal Plants Powder

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ABSTRACT

The potential of economically cheaper *Tribulus terrestris* was assessed for iron adsorption from aqueous solutions. The effects of pH, Contact time, sorbent dose, initial metal ion concentration and temperature on the uptake of iron were studied in batch process. Chemical treatment processes are ineffective and produce large quantity of sludge which requires further treatment. A simple and efficient treatment process for the removal of heavy metals is essentially required. Maximum iron removal was observed at pH 6 with adsorbent dosage of 0.6 g. The adsorbent data has been correlated with Langmuir and Freundlich adsorption models. FTIR and SEM before and after adsorption were recorded to explore the number and position of the functional groups available for iron binding onto the studied adsorbent and changes in surface morphology. The maximum percentage of iron removal was achieved at 87%. The results revealed that iron is considerably adsorbed on *Tribulus terrestris* and it could be economical method for the removal of iron from aqueous solutions. Pseudo second order model explains the iron kinetics more effectively.

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INTRODUCTION

The rapid industrialization has created water pollution. Wastewater from numerous industries such as paints and pigments, glass production, mining operation, metal plating and battery manufacturing processes are known to contain contaminates such as heavy metals. Heavy metals present in water produced many health disasters such as cancer, kidney failure, metabolic acidosis, oral ulcer, renal failure and damage in for stomach of the rodent. Removal of heavy metals from water and wastewater are important objectives [1-3]. Investigation into new and cheap methods of metal ions increased lately. The disposal of water with suspended solids have caused extensive amount of pollution to the natural water bodies. The rate of polluted water by domestic and industrial usages is increasing. Contaminants of environmental compartments [4, 5] with organic and inorganic compounds such as metals and pesticides has motivated the development of purification and extraction methods. The determination of trace of these ions in aquatic samples is particularly difficult because of the complex matrix and the usual low concentration of these elements in such samples requires various instruments such as ion exchange, solvent extraction, reverse osmosis, coagulation and filtration, adsorption on activated alumina (or) granular ferric hydroxide, membrane filtration, biological oxidation of Fe(II) and zero valent iron, precipitation, co-precipitation and adsorption [6-8]. However, chemical precipitation and electrochemical treatment are ineffective especially when metal ion concentrations in aqueous solutions are low and also alongside these removal techniques produce large quantities of sludge which require further treatment [9, 10]. Adsorption is considered as one of the effective and economical technologies for removal of heavy metals ions from wastewater. Natural material or certain waste from industrial or agricultural operation is one of the resources for low cost adsorbents. Natural products [11-13] usually considered peanuts shells and apple

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waste have been employed to extract metals from water. The use of biological material as adsorbent in heavy metals removal known as bioadsorption or biosorption [14-17]. The goal of this research was to develop a safe, cost effective and eco-friendly technique for wastewater purification [16-18]. *Tribulus terrestris* L is commonly known as puncture vine, caltrop, yellow vine, goat head and devil's horn. It is a member of the Zychophyllaceae family and is widely distributed in both tropical and mild temperature regions. The genus *Tribulus* contains 25 species and the innovative methodology developed in this study advanced adsorption and filtration by using *Tribulus terrestris.* There is a good confidences to use folk medicine as tonic, aphrodisiac, analgesic, astringent, stomachic, anti-hypertensive, diurteic, lithon-triptic and urinary anti-infection agents. The aim of this study is to propose new biomaterial involving application of *Tribulus terrestris* medicinal plant removal of iron from synthetic wastewater. The adsorbent dosage of *Tribulus terrestris* seed powder 0.6g for the removal of iron was confirmed by Langmuir, Freundlich and Kinetic studies. The highest percentage removal of iron was achieved at 87%. FTIR Spectra shows number of position of functional group for iron binding and SEM micrograph shows that surface morphology structure of *Tribulus terrestris* seed powder and this results showed that green sustainable and economically viable in wastewater treatment and purification.

MATERIAL AND METHODS

Preparation of Adsorbents

The seed powders of *Tribulus terrestris* was used for the removal of iron from synthetically prepared polluted water by optimizing various physico chemical properties viz pH, concentration of adsorbent and equilibrium time. It has been observed that the seeds of *Tribulus terrestris* have affinity towards the iron removal. The methodology herewith presented is only for these biosorbents. *Tribulus terrestris* native to warm temperate and tropical regions of southern India, Asia, Africa and Australia. *Tribulus terrestris* grows well in light textured soils; however, it grows over a wide range of soil types. Generally it can be found in cultivated crops, overgrazed pastures, road sides, lawns and neglected areas. *Tribulus terrestris* seed was dried and powdered using ordinary food processor. The structure of adsorbents are shown in Figure 1.

Preparation of aqueous solution of metal irons

The aqueous solution of 1000ppm of ferrous ion were freshly prepared from analytical grades salt with deionized water in 1% HNO_3 solution and water to obtain the working standard solutions. The pH adjustment of the solutions were made with aliquots of HCl and NaOH and checked with electronic pH meter which was calibrated using standard buffer solution.

Figure 1. Structure of adsorbents

Equilibrium studies

Equilibrium adsorption isotherms were performed in a batch process. The amount of metal ion adsorbed by the biosorbent at equilibrium Q_e mg $L^{-1}$was calculated using the following equation:

$$\%\ Removal\ of\ Fe = \frac{C_i - C_e}{C_i} X\ 100 \quad (1)$$

Where C_i and C_e (mg L^{-1}) are the liquid phase concentration of the metal ions at initial and equilibrium, respectively.

RESULTS AND DISCUSSION

The effect of pH on removal of metal iron

The pH of wastewater is one of the imperative factors governing the adsorption of the metal ion is shown in Figure 2. The prepared seed power from *Tribulus terrestris* used as adsorbents at a room temperature 30 °C for 0.6g of adsorbent at pH 2 to 12 were studied. It was found that the removal of metal ions has increased as pH of the aqueous solution increased and reached to maximum value at pH 5-6 for iron removal. The percentage of iron removal was very less at low and high pH. At low pH 3 this was due to the excess of H^+ ions surrounding the binding sites making adsorption unfavorable. The metal ions had the high tendency of precipitation as metal hydroxide.

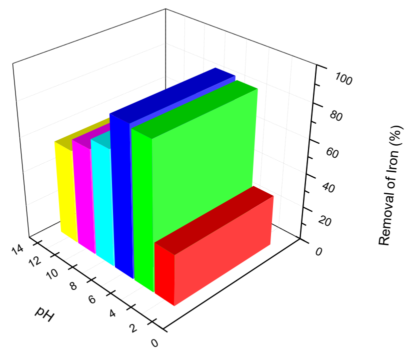


Figure 2. Effect of pH on the removal of 5 mg L^{-1} of Total iron on (2 to 12) pH 5 for 120 minutes contact time, 0.6 g adsorbent dosage

Effect of adsorbent dosage

Figure 3 Shows that the quantity of adsorbent is a significant factors; which is considered for effective adsorption. Performance of the *Tribulus terrestris* was evaluated for the percentage removal of iron was investigated. *Tribulus terrestris* adsorbent dosage was increased for the available exchangeable sites for iron. The adsorbent dosage was varied from 0.1 to 1.5g per 50 mL. The percentage of iron removal was highest in 0.6g of adsorbent dosage. Further studies were carried out at 0.6g as adsorbent dosage. Usually the percentage of iron removal decreases with an increase of iron concentration and the same results were reported by other researchers [18].

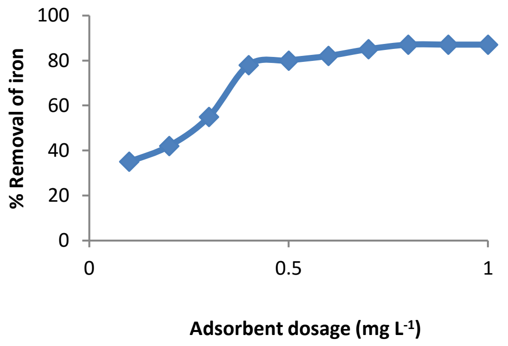


Figure 3. Effect of adsorbent dose on the removal of 5 mg L^{-1} of Total iron on 0.1 -1.5g adsorbent at pH 5 for 120 minutes contact time

Effect of Initial Concentration and Contact Time

The effect of contact time on adsorption of iron onto *Tribulus terrestris* was shown in Figure 4. The initial concentrations varied from 5 to 30PPM of iron Concentration at adsorbent dosage of 0.6g carried out at 393K as a function of time to evaluate the percentage of iron removal. The adsorption of iron removal increases with time and gradually equilibrium after 120 minutes.The maximum percentage removal of iron attained at 120minutes.The initial Concentration of iron removal increased from 5 to 30 ppm percentage removal of iron decreases.

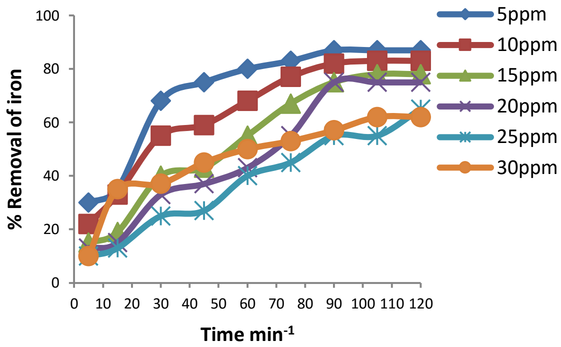


Figure 4. Effect of initial concentration and contact time on the removal of total iron on 0.6 g of adsorbent at pH 5 for 5 minutes to 150 minutes contact time

Effect of particle size

The variation in the percentages of iron removal by the sample with different particle sizes were investigated. The *Tribulus terrestris* with different particle sizes viz >60, 60-110, 110-180 ,180-230, 230-350mm. As the adsorption process is a surface phenomenon the highest percentage of iron removal was achieved at 100µm. The high efficiency was due to large surface area as shown in Figure 5. Smaller particle size having large surface area so adsorption efficiency increased.

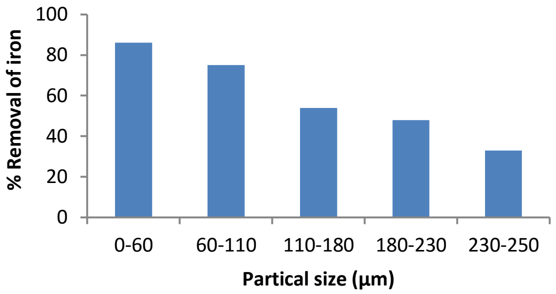


Figure 5. Effect of Particle size on the removal of total iron on 0.6g of adsorbent at pH 5 for 120 minutes contact time

Effect of Temperature

The effect of iron adsorption on the activated carbon at various temperature is shown in Figure 6. As observed from the figure *Tribulus terrestris* seed powder temperature increased resulted in an increase in the removal of the metal as expressed as percentages. Increasing temperature has increased the mobility and diffusion of ionic species [19, 20] when solution temperature increased. There would be enlargement of pore size due to activated diffusion of ionic species and also temperature increased created more surface area for adsorption process. The maximum percentage of iron removal achieved at 393K.

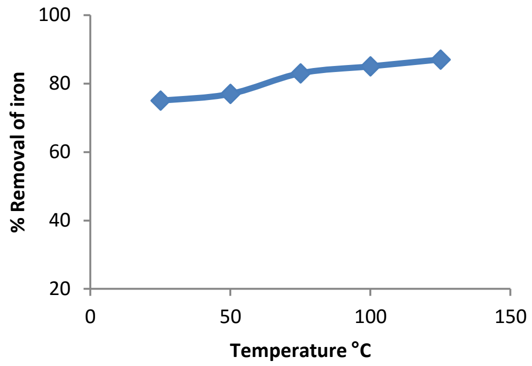


Figure 6. Effect of Temperature on the removal of Total iron on 0.6g of adsorbent at pH 5 for 120 minutes contact time

Langumuir and Freundlich adsorption isotherm

The equilibrium study is important for an adsorption process as it shows the capacity of the adsorption and the adsorption process was normally applied to describe the adsorption mechanism for the interaction of cations on the adsorbent surface. In the present study experimental data was analyzed to examine the adsorption isotherm using Langmuir and Freundlich models. The Langmuir isotherm was applicable to monolayer adsorption surface. Whereas the Freundlich isotherm was an empirical model that considers heterogeneous adsorption on the adsorption surface.

Langmuir model

Langmuir isotherm model was mainly based on the assumption that maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on the adsorbent surface. The Langmuir adsorption isotherm has been greatly used to many ground water effluent treatment processes and it has also been used to explain the adsorption of heavy metals by various adsorbents. Langmuir theory tells that adsorption takes place at specific homogeneous sites within the adsorbent. It is then assumed that once a metal ion molecule occupies a site after that no adsorption takes place adsorbent sites and adsorbed layer was unimolecular. The theory can be represented by the following linear form:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m K_L} + \frac{C_e}{Q_m} \qquad (2)$$

Where C_e is the equilibrium concentration (mg L^{-1}), Q_e is the amount adsorbed at equilibrium (mg g^{-1}), Q_m (mg g^{-1}) and K_L (L mg^{-1}) are Langmuir constants related to adsorption capacity and energy of adsorption respectively. Figure 7. shows the linear plot of C_e/Q_e vs. C_e for *Tribulus terrestris* powder. The values of Q_m and K_L were determined from slop and intercept of the linear plot of C_e/Q_e vs. C_e (see Table 1). The experimental data and the correlation coefficients (R^2) values of *Tribulus terrestris* was 0.983 indicates the applicability of the Langmuir isotherm model. The essential feature of Langmuir isotherm can be expressed by means of dimensionless constant referred to as the separation factor or equilibrium parameter, R_L which is defined by the following equation [21]:

$$R_L = \frac{1}{1 + K_L C_i} \qquad (3)$$

Where C_i is the initial metal ion concentration (mg L^{-1}). The value of separation factor R_L, indicates the nature of the adsorption process as given below:

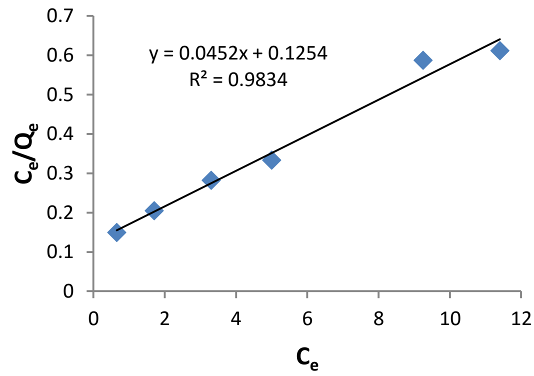


Figure 7. Langmuir isotherm for the adsorption of Total iron on *Tribulus terrestris* adsorbent

TABLE 1.

Nature of adsorption process	R_L value
Unfavorable	$R_L > 1$
Linear	$R_L = 1$
Favorable	$0 < R_L < 1$
Irreversible	$R_L = 0$

The values of R_L values calculated for this study are given in Table 2. The adsorption process will be favorable if the R_L values lie between 0 and 1. The R_L values given in Table 2 very well lie in this range and hence the adsorption process was favorable.

TABLE 2. The R_L Value

Initial Concentration (mg g-1)	The R_L Value
5	0.3571
10	0.2173
15	0.1562
20	0.1219
25	0.1
30	0.0847

Freundlich model

The Freundlich adsorption model is the most widely used isotherm for the explanation of adsorption of heavy metals on a wide variety of biosorbents. It is an empirical equation that can be used for non-ideal sorption that involves heterogeneous sorption. The empirical model was shown to be consistent with an exponential distribution of active centers, characteristic of heterogeneous surfaces. The amount of solute adsorbed, Q_e is related to the equilibrium concentration of solute in solution, C_e as following [22]

$$Q_e = K_F C_e^{\frac{1}{n}} \quad (4)$$

This expression can be linearized to give the following equation:

$$\log Q_e = \log K_F + \frac{1}{n}\log C_e \quad (5)$$

Where, K_F is a constant for the system, related to the bonding energy. K_F can be defined as the adsorption or distribution coefficient and respects the quantity of metal ion adsorbed onto adsorbent for a unit equilibrium concentration (a measure of adsorption capacity, mg g^{-1}). The slope $1/n$, ranging between 0 and 1, is a measure of adsorption intensity or surface heterogeneity (becoming more heterogeneous as its value gets closer to zero). A value for $1/n$ below one indicates a normal Freundlich isotherm while $1/n$ above one is an indicative of cooperative adsorption. A plot of log (Q_e) vs. log (C_e) was shown in Figure 8, where the values of K_F and $1/n$ are determined from the intercept and slope of the linear regressions (Table 3).

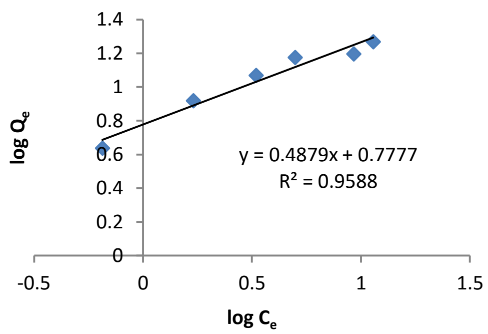


Figure 8. Freundlich isotherm for the adsorption of Total iron on *Tribulus terrestris* adsorbent

Table 3. Freundlich isotherm

Natural coagulants	1/n	K_F (mg g^{-1})	R^2
Tribulus terrestris seed powder	0.487	9.015	0.976

Tempkin isotherm model

Tempkin isotherm contains a factor that explicitly takes into account adsorbing species-adsorbate interactions. This isotherm assumes that the heat of adsorption of all molecules in the layer decreases linearly with coverage due to adsorbate-adsorbate interaction and adsorption was characterized by a uniform distribution of binding energies, up to some maximum binding energy. Tempkin isotherm has generally been used in the linearized and rearranged form as following:

$$Q_e = \beta\, lnK_T + \beta \ln C_e \quad (6)$$

where, KT is an equilibrium constant of binding corresponding to the maximum energy of binding (mg L-1) and the β is related to the heat of adsorption. Figure 9 shows a plot of Qe versus ln Ce, which enables the determination of the isotherm constants KT and β. The values of KT, β and correlation coefficient, R2 for Tempkin isotherm model are given in Table 4.

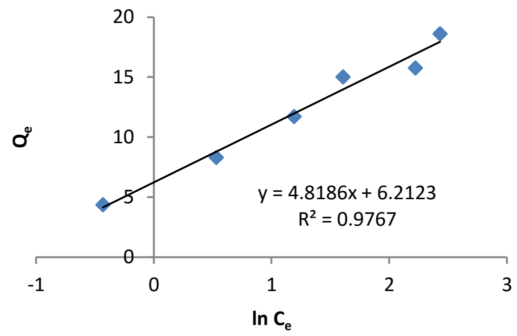


Figure 9. Tempkin isotherm for the adsorption of Total iron on tribulus terrestris adsorbent

TABLE 4. Tempkin isotherm

Natural coagulants	β	K_T (mg L^{-1})	R^2
Tribulus terrestris seed powder	4.818	1.4857	0.976

Adsorption kinetics

In order to investigate the mechanism of total iron adsorption onto adsorbent, three kinetic models were studied: Lagergren's first-order, Pseudo-second-order and Elovich [23-25].

Lagergren's first-order kinetic model

The pseudo-first-order kinetic model of Lagergren is more suitable for lower concentration of solute and its linear form is:

$$\log(Q_e - Q_t) = \log Q_e - \frac{k_1}{2.303}t \quad (7)$$

Where, Q_t (mg g^{-1}) is the amount of adsorbate adsorbed at time t (min); Q_e (mg g^{-1}) is the adsorption capacity in the equilibrium; k_1 (min^{-1}) is the rate constant of pseudo-first-order model.

The values of k_1 and Q_e for *Tribulus terrestris* adsorbents was determined from the plot of log (Q_e-Q_t) vs. time which is shown in Figure 9. The correlation coefficient, R^2 for *Tribulus terrestris* powder was presented in Table 5.

TABLE 5. Lagergren's first order kinetic model

Natural coagulants	$Q_{e\,(exp)}$	K_1x 10^{-2}	R^2
Tribulus terrestris seed powder	4.35	4.467	0.995

Pseudo second order kinetic model

Adsorption kinetics was explained by the pseudo-second-order model expressed as following linear equation:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \quad (8)$$

Where, k_2 is the second order rate constant (g $mg^{-1}min^{-1}$). The values of k2 for removal of iron by Tribulus terrestris adsorbents was calculated from the slopes of the respective linear plots of t/Qt vs. t (Figure 10). The correlation coefficients, R2 was 0.994 for Tribulus terrestris suggests a strong relationship between the parameters and also explain that the process follows pseudo second order kinetics (Table 6).

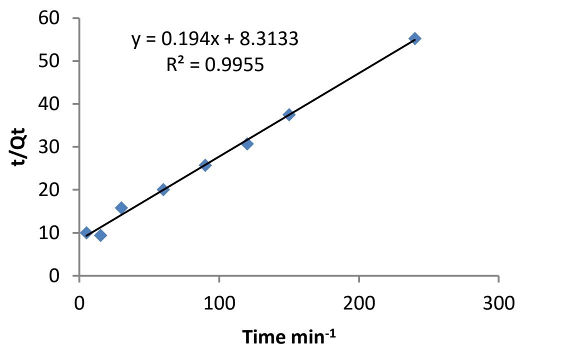


Figure 10. Lagergren first-order-kinetic model of Total iron adsorption onto 0.6g L^{-1} adsorbent, initial iron concentration 5mg L^{-1}, pH 5

TABLE 6. Pseudo second order kinetic model

Natural coagulants	$Q_{e\,(exp)}$	K_2x 10^{-2}	R^2
Tribulus terrestris seed powder	4.35	8.033	0.994

Elovich kinetic model

Elovich model suggests that the chemisorptions, i.e. a chemical reaction, is probably the Mechanism that controls the rate of adsorption. This model can be applied with success in liquid solution and the linear form of the Elovich equation is:

$$Q_t = \frac{1}{\beta}\ln\alpha\beta + \frac{1}{\beta}\ln t \quad (9)$$

Where, α (mg g^{-1}) is the initial sorption rate and β (g mg^{-1}) is the desorption constant. The values of α and β can be calculated from the slope and intercept of the plot of Q_t versus ln t (Figure11).

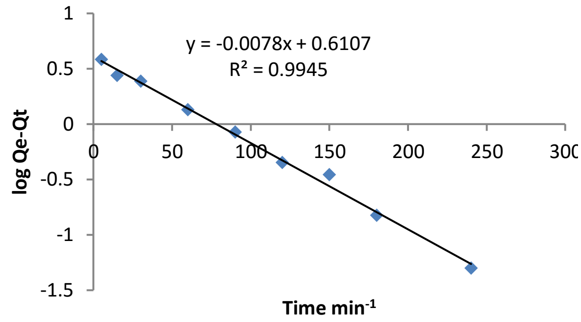


Figure 11. Pseudo-second-order kinetic model for the adsorption of Total iron onto 0.6 g L^{-1} of adsorbent with initial iron concentration of 5mg L^{-1} at pH 5

As can be seen from the Figure 12, the values of R^2 are closer to unity for pseudo second order model than pseudo first order model and Elovich model. Thus, adsorption of total iron onto adsorbent follows the pseudo second order model. Furthermore, values of $Q_{e\,(cal)}$ calculated from pseudo second order model were in good agreement with experimental values, $Q_{e\,(exp)}$ than those calculated from pseudo first order. The values of R^2 for pseudo first order and Elovich model are lower than the pseudo second order model and thus indicate that pseudo first order and Elovich model cannot be adequate to describe the kinetic of adsorption of iron metal onto *Tribulus terrestris* seed powder.

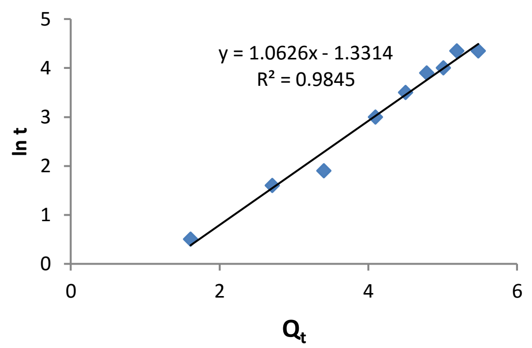


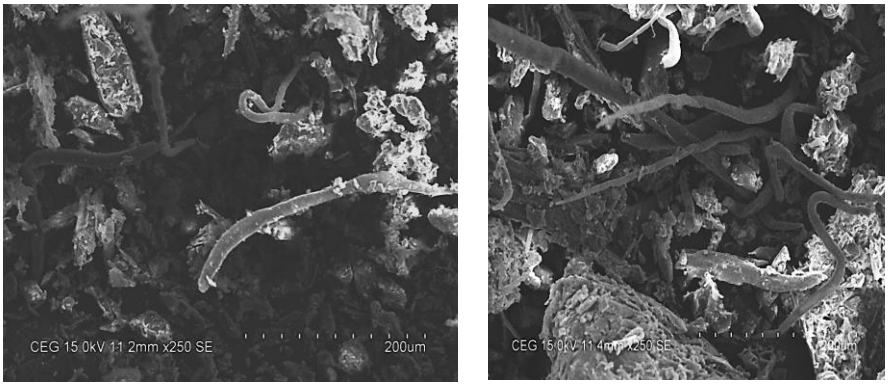
Figure 12. Elovich kinetic model for the adsorption of Total iron onto 0.6g L^{-1} of adsorbent with initial iron concentration 5mg L^{-1} at pH 5

Scanning electron Microscope and FT-IR Spectra

Scanning electron microscope (SEM) was used to observe the pore structure of the *Tribulus terrestris* seed powder. Pore present in *Tribulus terrestris* act as the active sites, where adsorption take place. The scanning electron microscope images of *Tribulus terrestris* before and after adsorption of surface morphological structure are shown in Figure 13a. SEM photograph showed that wide varieties of pores are present in the *Tribulus terrestris* which is more visible at 200 magnifications. The before adsorption SEM images clearly indicated that the surface structure of *Tribulus terrestris* was greatly changed from after adsorption. *Tribulus terrestris* seed powder having needle like fibrous and bulkier particles present in the surface of the fibre than in the case of iron adsorption. The after adsorption iron particles adsorbed on the adsorbent surface was confirmed by Figure13b. FT-IR Spectroscopy was also used to identify the changes on before and after adsorption of *Tribulus terrestris* seed powder correspondence with iron adsorption were shown in Figures 14a and 14b. The spectra was measured within the range of 400-4000 cm^{-1} in a Shimadzu spectrophotometer. Some fundamental FTIR frequencies of the *Tribulus terrestris* adsorbents, before and after adsorption are presented in Table 7. Figure14b shows that many other frequencies were shifted to different wave number with the adsorption of iron. From these predictions iron metal ion was adsorbed onto the *Tribulus terrestris* adsorbents through interaction with the active functional groups. *Tribulus terrestris* before adsorption the bands appearing at 3377 cm^{-1} and after adsorption the bands shifted to 3414 cm^{-1} predicting the presence of aromatic and aliphatic amine groups. The absorption bands of *Tribulus terrestris* before 1566 cm^{-1} and after adsorption the frequencies 1583 cm^{-1} assigned to conjugated C=C , C=O and carboxylate groups, 2922 cm^{-1} indicates C-H (str) group present in *Tribulus terrestris* seed powder. After adsorption of *Tribulus terrestris* seed powder additional band appear at 873 cm^{-1} and 779 cm^{-1} C-Cl stretching this observation confirmed that iron adsorbed on *Tribulus terrestris* seed powder.

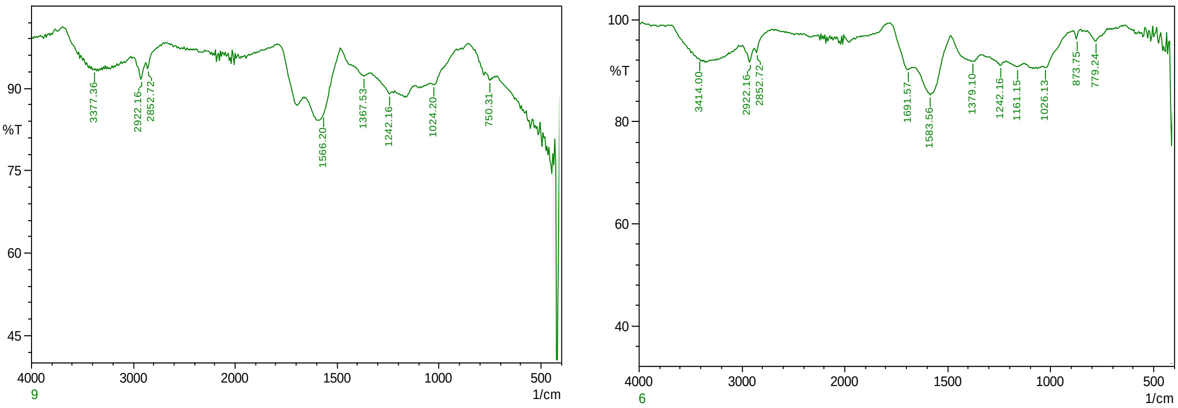
TABLE 7. FT-IR Absorption frequency (cm^{-1})

Functional group	*Tribulus terrestris* before	*Tribulus terrestris* after
C-H (str)	2922	2922
Conjugated C=C,C=O	1566	1583
O-H and N-H	3377	3414
C-Cl (str)	750	779



a b

Figure 13. SEM micrography of activated carbons a) *Tribulus terrestris* before adsorption b) *Tribulus terrestris* after adsorption of Total iron.



a b

Figure 14. FT-IR spectrum of activated carbon a) *Tribulus terrestris* before adsorption b) *Tibullus terrestris* after adsorption of total iron

Applications

Toxicity, safety and environmental impacts of any new technology have to be evaluated before the technology can be deployed with confidence. The natural adsorbent derived from *Tribulus terrestris* was used in water treatment but it leads to many diseases and side effects. The natural coagulants used in water purification developed in this research is safe and environmental friendly as it does not produce any secondary toxic byproducts. The purification process uses only natural coagulants. Which is abundant in developing countries that lack large and expensive infrastructure for waste water treatment. The natural coagulant used in water treatment is cost effective abundantly available and inexpensive.

CONCLUSION

A satisfactorily selective and simple method for the removal of iron from water samples was achieved. Powders of *Tribulus terrestris* have been found to have strong affinity towards iron at low pH values. Finally the technical applicability and cost effectiveness are the key factors that play major roles in the selection of most suitable treatment method for the removal of iron from wastewater. The most effective and inexpensive treatment in order to protect the environment. Studies on batch adsorption using synthetic wastewater samples in order to remove the iron indicated that the adsorbent has a good potential to remove the heavy metal ions from wastewater samples. *Tribulus terrestris* showed nearly 87% adsorption removal of heavy metal ions under optimized conditions of dosage 0.6g for aqueous solutions containing 5mg L^{-1} metal ions in 3hours.The Langmuir model is found to be in good agreement with experimental data on adsorptive behavior of all metal ions on *Tribulus* terrestris both Langmuir and Frundlich models. Kinetic data fitted well with the pseudo first order and second order models. The high values of Correlations coefficient indicated that there was a good agreement between the kinetic data and Pseudo second order model. This technique can be used to water treatment process.

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Persian Abstract

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چکیده

پتانسیل اقتصادی ارزان‌تر Tribulus terrestris برای جذب آهن از محلول‌ها ارزیابی شد. تاثیر pH، زمان تماس، مقدار جاذب، غلظت اولیه فلز آهن و دما در مصرف آهن در فرایند ناپیوسته بررسی شد. پیشتیمار شیمیایی فرایندها بی تاثیر بودند و مقدار زیادی لجن تولید می‌کردند که ملزم به پیشتیمار بعدی بودند. اساسا یک روش تصفیه فرایند ساده و کافی برای حذف فلزات سنگین نیاز بود. بیشترین میزان حذف آهن در pH 6 با مقدار جاذب ۰/۶ گرم مشاهده شد. داده‌های جاذب با معادله‌های جذب Langmuir و Freundlich مرتبط شد. FTIR و SEM قبل و بعد از جذب ثبت شد تا تعداد و مکان گروه‌های عاملی موجود برای اتصال آهن به جاذب‌های مورد مطالعه و تغییر در مورفولوژی سطح را تشریح کند. ماکزیمم درصد حذف آهن ۸۷٪ بدست آمد. نتایج نشان داد که آهن به طور قابل توجهی جذب Tribulus terrestris می‌شود و می‌تواند یک روش اقتصادی برای حذف آهن از محلول‌های آبی باشد. مدل شبه درجه دو سینتیک آهن را بطور موثرتری شرح می‌دهد.