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Adsorption studies of Iron Removal from Aqueous Solution Using *Moringa Oleifera* Seed Pod Husk Activated Carbon as an Adsorbent.

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ABSTRACT
Moringa oleifera seed pod husk Activated Carbon was utilized as an adsorbent to remove Iron from aqueous solutions. The effect of pH, adsorbent dosage, initial concentrations, contact time, and temperature were studied using batch adsorption experiments. Characterization of adsorbent was identified by FTIR and XRD techniques. The pH dependence study of the adsorption process revealed that maximum pH for Iron removal was 8 with removal of 88.73% Temperature study reveals that the adsorption is endothermic as efficiency increases with the increase in temperature. The adsorption of Iron on Moringa oleifera seed pod husk activated carbon shows that the removal efficiency increases with increase in contact time and also increased as adsorbent dosage increases from 0.5gm/50ml to 2.5gm/50ml. The study showed that the method is a simple and efficient one to remove.

Keywords: Activated carbon, adsorption, batch adsorption experiments, Moringa oleifera seed pod husk, Iron removal.

INTRODUCTION

Iron is a common metallic element found in the earth's crust. Water percolating through soil and rock can dissolve minerals containing iron and hold them in solution. Occasionally, iron pipes also may be a source of iron in water.

The main anthropogenic sources of Iron are various industrial sources, including present and former mining activities, Power plants combustion, etc. It has adverse effects on the environment and human health and is toxic even at low concentration to human beings and other living and non-living things. Iron in water is normally found in the ferrous state. Oxidation of dissolved iron particles in water changes to red-brown solid particles that settle out of the water. Iron that does not form particles large enough to settle out and that remains suspended and leaves the water with a red tint. Standards for Iron concentration in drinking water[1]

Adsorption process is one of the easiest, safest and more effective methods for metal removal from industrial effluents [2,3] and this process is already established as a simple operation and an easy-handling process. Activated carbon is a commonly used adsorbent for the water and wastewater treatment. Previous research shows that there is growing interest of searching for a variety of materials as low cost adsorbents including cocoa shell [4] rice husk **[5]**, papaya wood **[6]**, maize leaf**[7]**, rice husk ash and neem bark [8] fly ash [9] and tea-industry waste [10]. The adsorbent like Sugarcane bagasse, Coconut coir having very good tendency for removal of total iron Balaji [11] the adsorption properties of coconut shell are due to the presence of some functional groups, such as carboxylic, hydroxyl, and lactone, which have a high affinity for metal ions [12]. Egg shell and Pongamiapinnata also known as Karanja tree bark is also used as adsorbents for the removal of iron from groundwater [13].

Earlier studies found that Moringa Oleifera is harmless and recommended it for use as a adsorbent in water treatment. When Moringa Oleifera seed powder used as bioadsorbent for removal of fluoride, it was found that the alkali treated seed powder was better than acid treated [14]. When Adsorption Studies for Arsenic Removal Using Activated Moringaoleifera leaves is carried out it was found that is an effective and alternative biomass for removing Arsenic from aqueous solution due to high bio-sorption capacity[15]. Various studies were carried out where Moringa oleifera seed powder was investigated as a best low cost biosorbent for the removal of toxic heavy metals from wastewater. Biosorption of Pb^{2+} from aqueous solution by biomass prepared from *Moringaoleifera* bark shows it is promising biosorbent material for the removal of heavy metal ions from wastewater/effluents [16]. Biosorption of Pb^{2+} from aqueous solution using Moringa oleifera pods also gives good results [17]. It was found that the highest level of metal removal was achieved at pH 5[18]. Moringa Oleifera is natural Phytoremedy for ground water treatment and effective in removal of Iron [19].

METHODOLOGY

Moringa Oleifera (Drum Sticks): It is the most widely cultivated species of the genus Moringa, which is the only genus in the family Moringaceae. English common name is drumstick tree from the appearance of the long, slender, triangular seed-pods. It is a fast-growing and widely cultivated as its young seed pods and leaves are used as vegetables. It can also be used for water purification and is sometimes used in herbal medicine.

Preparation of Moringa Oleifera seed pod husk charcoal: Moringa oliefera pods were collected from locally available trees, for this purpose mature seed pods are selected rather than the immature ones which are preferred for cooking purposes. The pods are sun dried for 3-4 days. The seeds are removed from the pods and their size was reduced by breaking it into small pieces. Then it was packed in an air tight in a cylindrical container with top completely sealed with a cover to prevent the entry of air during the process of charring. The sealed container was heated in furnace by slowly raising the temperature up to 350^0 C for 60 minutes and subsequently washed with distilled water, oven dried and sieved through 100 micron mesh sieve to obtain carbon powder.

Activation of carbon: The resultant charcoal obtained by above procedure was soaked in 2M KOH overnight. It was followed by washing with distilled water till the attainment of neutral pH, and then dried in the hot air oven at 80±5C temperature for 4 hrs to obtain activated carbon. The KOH saved as activating agent to introduce some functional groups and deepening of micropores' depth.

Stock solution as Adsorbates: Iron solution was prepared by dissolving 49.78g of $FeSO_4.7H_2O$ dissolved in 1000ml distilled water to make water equivalent to 10 g/lit and this served as a stock solution. A calibration curve was also plotted by analyzing the different concentrated solutions using UV-visible spectrophotometer

Batch adsorption experiments: Batch adsorption experiments were conducted to examine adsorption behavior of different adsorbent on Iron removal from aqueous solution under different adsorption condition. Adsorption studies were carried in different conditions namely adsorbent dose, initial concentration, contact time, pH and temperature. The adsorption experiments were conducted in 250 ml conical flasks. In each experiment, a known amount of adsorbent was contacted with 50ml of desired contaminated water with known pH and at a regular interval of time of 60 min. pH of the solution was measured using pH meter and adjusted using 0.1N HCl and 0.1 N NaOH. The solutions were filtered by using Whattman filter papers and filtrates were collected for analysis. In each experiment the conditions were kept constant except for the one in which its effect is studied.

Adsorbent Characterization: The adsorbent was characterized by FTIR analysis. In chemical activation, activating agent is expected to significantly affect the properties of substance. X-raydiffraction(XRD), Fourier transform infrared (FTIR)spectroscopy analysis performed to determine the structural and surface properties.

RESULTS AND DISCUSSION

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Effect of pH on Iron Removal

Concentration = 4.8mg/l; Contact time = 60 min,
Adsorbent dose = 1gm/50ml, Temperature = 30°C.

For activated Moringa Oleifera pod shell charcoal adsorption of iron is very high at all pH range and gradually increases as the initial pH of the solution is raised from 2 to 8. The maximum removal of iron was found to be 88.73% at pH 8. Hence, pH of the iron solution was maintained at 8 for further study.

Effect of Adsorbent dose on Iron Removal

Concentration=4.8mg/l; pH =8,
Contact time=60 min Temperature = 30°C.

The amount of activated Moringa Oleifera adsorbent was varied from 0.5 gms to 2.5 gms with a constant initial concentration of 4.8 mg/L and agitation time of 60 minutes at room temperature. The amount adsorbed is increase with increase in amount of adsorbent and became constant at 2 gms of adsorbent. The maximum removal was 94.23%. This result indicated that more surface area was made due to increased mass of adsorbent. Therefore, total number of sites increases [20].

Effect of contact time on Iron removal

Concentration = 4.8mg/l; pH =8,
Adsorbent dose = 1gm/50ml, Temperature = 30°C.

Removal percentage was recorded at contact time of 30 min to 160 min. evidently, more than 75 % iron removal occurred within 30 min showing that initially the rate of adsorption of iron is very fast and gradually increases attaining a steady value after reaching the equilibrium at about 120 min. It was observed that after certain time frame, the rate of adsorption remained constant which shows that the adsorption of took place because of the diffusion occurring in the pores on the surface of the adsorbent.

Effect of initial concentration on Iron Removal

Concentration = 4.8mg/l; pH =8,
Adsorbent dose = 1gm/50ml, Temperature = 30°C.

In case of adsorption by Moringa Oleifera, it was noticed that the percentage adsorption decreased very slowly with increase in concentration of Iron. Adsorption is very high in all range of concentration. The reason was probably with increase in concentration, the number of ions increased and so chances of contact between ions and adsorption sites also increased. This was responsible for higher uptake by unit amount of adsorbent.

Effect of temperature

Concentration=4.8mg/l pH =8,
Adsorbent dose = 1g/50ml, Contact time = 60 min,

Percentage adsorption of Iron on the surface of Morienga Oleifera does not vary much with temperature First it slowly increases with temperature and after 50^0C it slowly decreases with increase in temperature.

Adsorption isotherms

The Langmuir Adsorption isotherms: The Langmuir equation was applied for adsorption equilibrium

Ce/qe = 1/Qob + Ce/Qo

where, Ce is the equilibrium concentration (mg/L), qe is the amount adsorbed at equilibrium (mg/L) and Qo and b are Langmuir constant related to adsorption capacity and energy of adsorption respectively. The plots Ce/qe as a function of Ce for the adsorption was found linear suggest applicability of Langmuir model in present adsorption system.

Langumuir constants for Iron (II) ions are as follows-
B=1.4120, Q_0=3.5587, R_L= 0.9340- 0.5766

The higher adsorption capacity Q_0, b values obtained indicate higher adsorption capacities and good performance. According to R_L values which are between 0 to 1 indicate favorable adsorption for these adsorbent. Langmuir model is very applicable to this adsorbent in the present adsorption system.

Standards	Maximum Permissible Limit (mg/lit)
BIS (IS 10500)	0.3
USEPA	0.3
WHO guidelines	*0.3, No health based guideline value is proposed*

Figure1. Moringa Oleifera seed pod

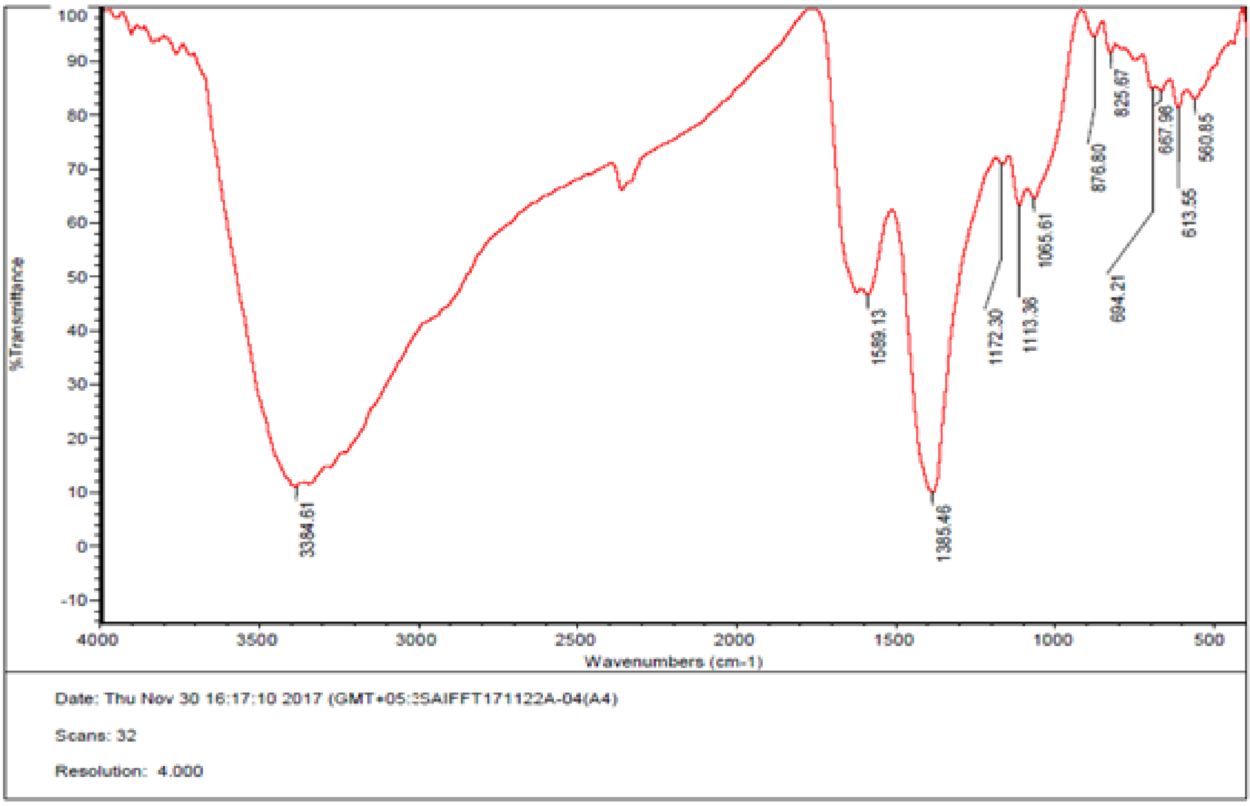

Figure 2. FTIR spectrum of KOH activated MoringaOleifera pod husk

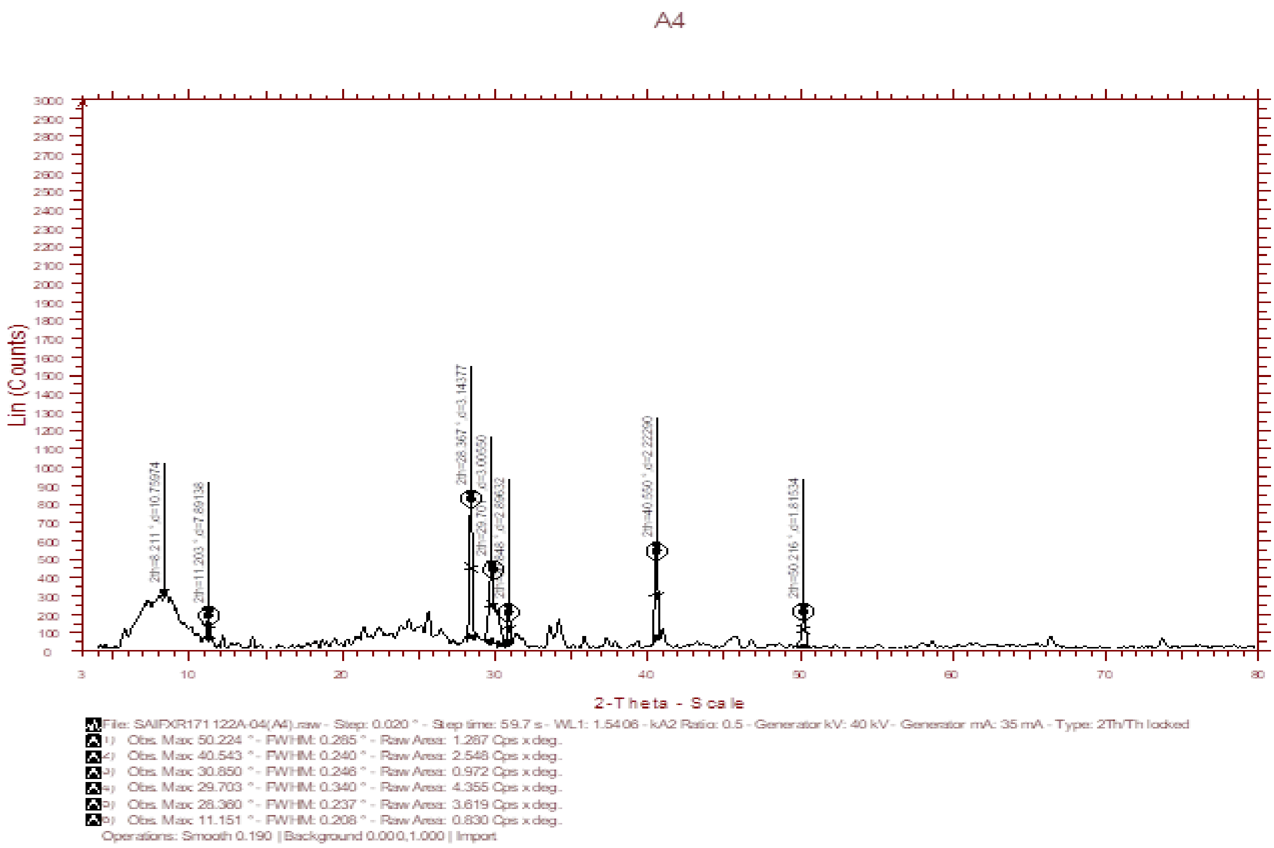

Figure 3.XRD of KOH activated MoringaOleifera seed pod charcoal

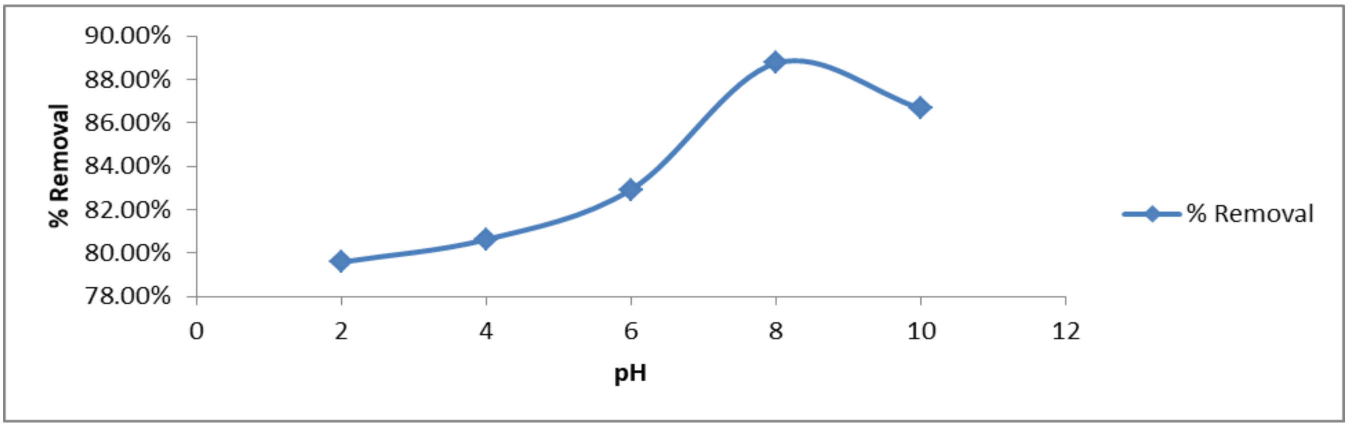

Figure 4.Effect of pH on Iron Removal

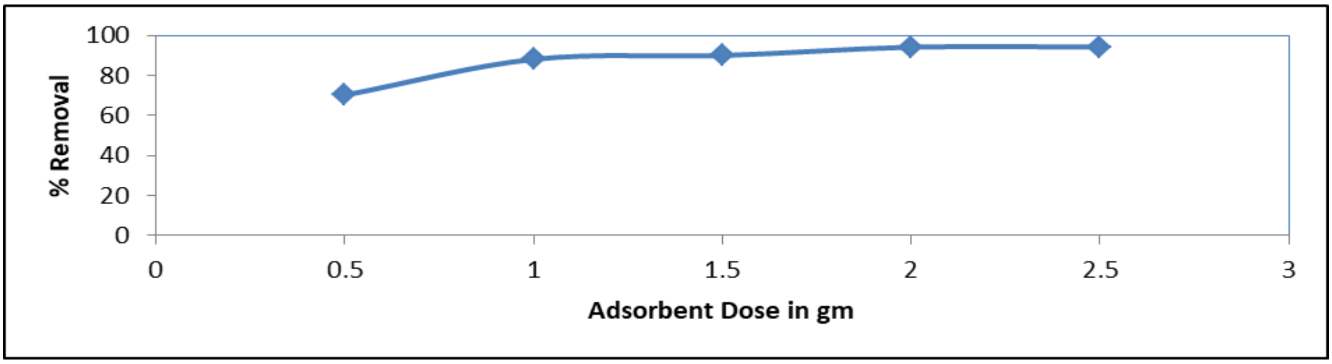

Figure 5.Effect of Adsorbent dose on Iron Removal

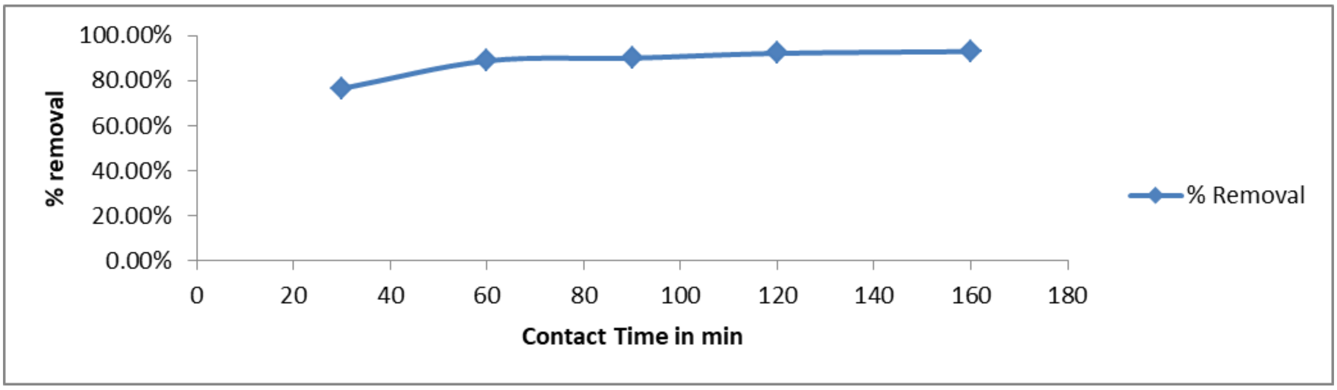

Figure 6. Effect of contact time on Iron removal

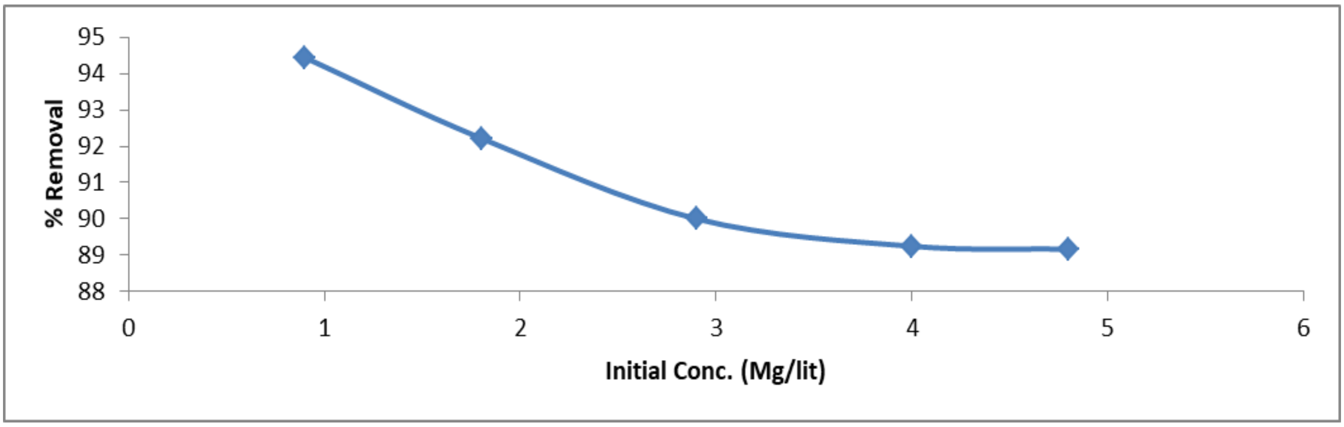

Figure 7.Effect of initial concentration on Iron Removal

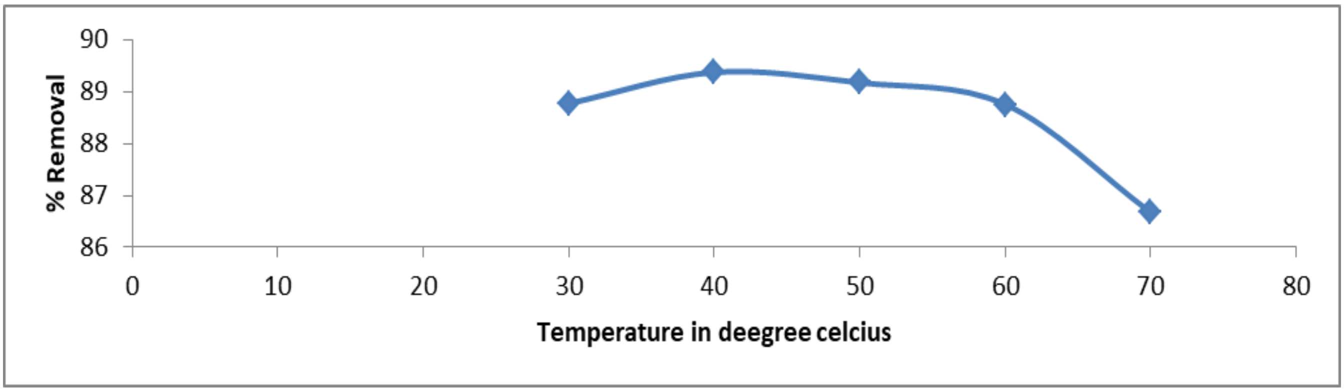

Figure 8.Effect of temperature

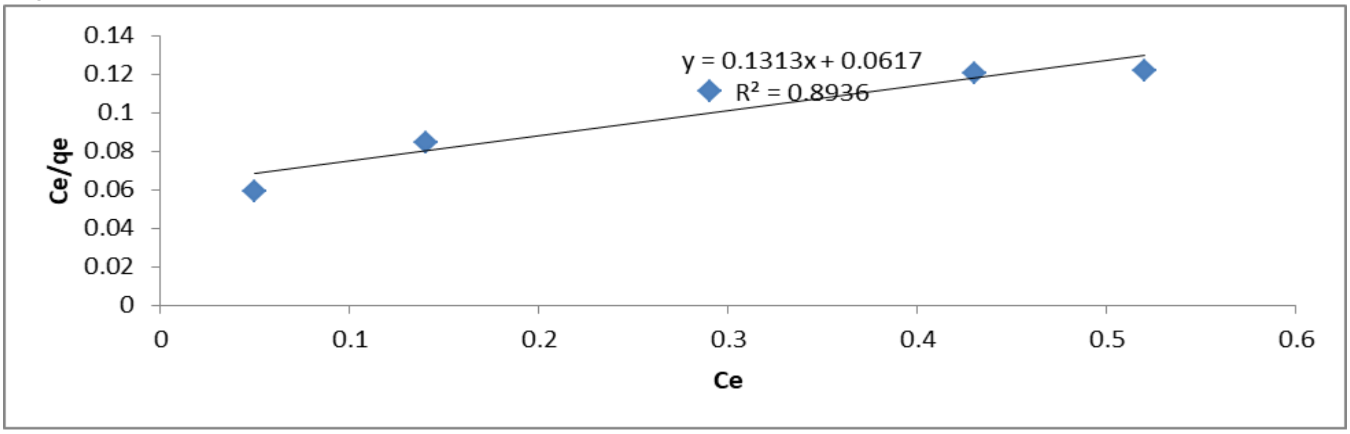

Figure 9.The Langmuir Adsorption isotherms for removal of Iron by MoringaOleifera

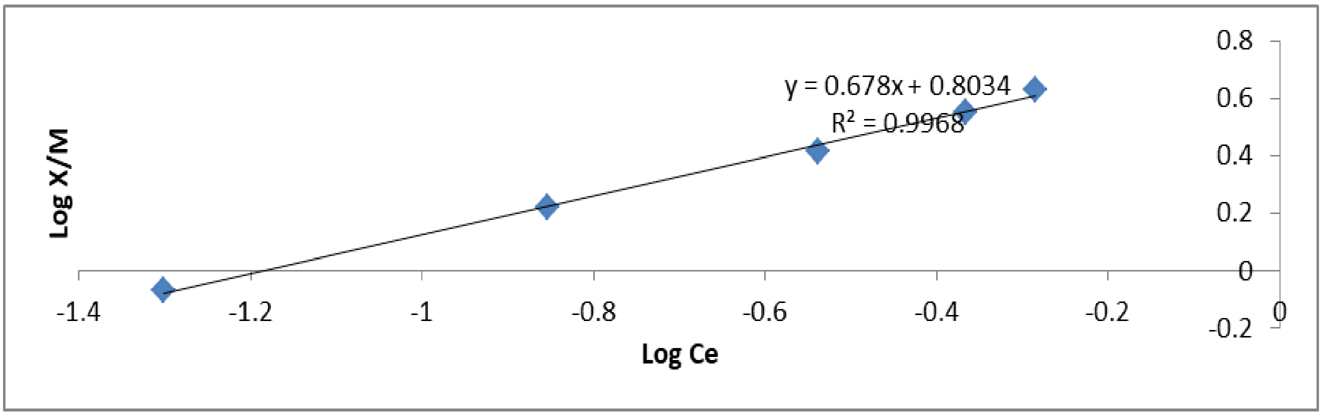

Figure 10.The Frendlich Adsorption isotherms for removal of Iron by MoringaOleifera

The Freundlich isotherm: The Freundlich isotherm is represented by the equation-

Log x/m = log K + 1/n (log Ce)

Where Ce is the equilibrium concentration (mg/L) and x/m is the amount adsorbed per unit weight of adsorbent (mg/g). Plots of log x/m *vs.* log Ce is linear. Figures 10show the Freundlich adsorption isotherm for Iron. The 'K' and 'n'values were calculated from the intercepts and slopesare 6.353 and 1.475 respectively. Value of n and K obtained indicate that the adsorbent are good for uptake of Iron from aqueous solution.The Correlation coefficient of Freundlich curves which is above 0.9 shows good linearity for adsorbent and also indicates strong binding of iron ions to the surface of adsorbent.

CONCLUSION

Activated Carbon was prepared through pyrolysis followed by chemical activation with KOH and used as an adsorbent for removal of Iron. Removal of Iron by Application of operational conditions such as contact time, adsorbent dose, pH, Temperature and concentration of adsorbate led to increase of Iron removal. Result clearly shows that adsorption of Iron on to activated materials was favored. The optimal dose was found to be 2gm and the maximum removal was seen within 120 minutes of contact time. Based on the results obtained in the present study, it is clear that Moringa Oleifera is effective in removal of Iron from aquaous solution. Since it is locally available, especially in regions of Chandrapur district where various mining activities takes places, problem of Iron contain in water is prevailing, then, this adsorbent is expected to be economically feasible forremoval of Iron from water.

Conflicts of interest: The authors stated that no conflicts of interest.

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